Alkali-Aggregate Reactions in Concrete
Properties, Classification and Testing of
Norwegian Cataclastic Rocks

Børge Johannes Wigum

December 1995

University of Trondheim
The Norwegian Institute of Technology
Department of Geology and Mineral Resources Engineering
Dissertation submitted of the requirements for the academic degree
DOKTOR INGENIØR
Success is failure turned inside out
the silver tint of the clouds of doubt
and you never can tell how close you are
it may be near when it seems so far
so stick to the fight when your hardest hit
it's when things seem worse that you must not quit.

- Unknown
ACKNOWLEDGEMENTS

I wish to thank my supervisor, Dr. S.W. Danielsen, for helpful advice and discussions during the work, and my co-supervisor, Dr. B. Brattli for helpful discussions and the introduction into the theory of cataclastic rocks.

I am grateful to Dr. W.J. French and all colleagues at Geomaterials Unit, Queen Mary and Westfield College, University of London, for their support during the course of my work.

Colleagues at the Department of Geology and Mineral Resources Engineering, University of Trondheim, and colleagues at the SINTEF- Structures and Concrete are thanked for their support and technical assistance.

I am grateful for the hospitality and helpfulness by the staffs at the Icelandic Building Research Institute (Iceland), Geomaterials Research Services Ltd. (UK), and at the Institute for Research in Construction, NRC (Canada).

Mr. A.L. Nissen at the Geological Survey of Norway assistance is appreciated during the course of collecting some of the samples.

This work has been funded by the Research Council of Norway. The financial support of the NorFa-, and NorCon-programs are also gratefully acknowledged.

Thanks to Dr. S. Lippard and Dr. C.D. Hills for their help in editing parts of this thesis.

Special thanks to Dr. H.H. Patel, for his effort in editing parts of this thesis and making critical comments during the course of the work.

Oslo/Trondheim, October 1995

Børge Johannes Wigum
ABSTRACT

This work reports on the alkali reactive potential of certain rock types through petrographic examination and accelerated mortar bar testing. The majority of the rocks studied were of cataclastic origin which had undergone ductile deformation.

Cataclastic rocks were commonly found in glaciofluvial aggregates in southern Norway. Some relationship were found between results from the currently used Norwegian petrographic examination and an accelerated mortar bar test.

The results from the accelerated mortar bar test were found to show good agreement with alkali-aggregate reaction observed under normal conditions. In this test the enhanced expansivity of cataclastic rocks was found to be related to the presence of microcrystalline quartz, and in particular to the high strain energy (dislocations) surrounding subgrain boundaries. For different types of reactive aggregates the rate of expansion was observed to depend on the test age and duration of the test.

From observations of the sequential expansion development it was evident that two different mechanisms were involved. These were dissolution of quartz along grain boundaries and crack generation within the aggregate, with the later being the main factor governing the degree of expansion produced by the reaction.

Various factors in the accelerated test were examined independently by two separate laboratories. It was evident that diagnosis of potentially highly reactive aggregate could be made with confidence, but that a large number of tests were required to assess marginal types of reactive materials.

A detailed examination of microstructural features of quartz, including determination of the total boundary area of quartz, was found to significantly improve the accuracy of predicting potential alkali reactivity in cataclastic aggregates. A logarithmic relationship was observed between the total grain boundary area of quartz, and the 14 days mortar bar expansion results. The study demonstrated that the grain size reduction of quartz, promoted by the process of cataclasis, enhances alkali reactivity by increasing the surface area of quartz grain boundaries available for reaction. These findings demonstrate the importance and reinforces the potential of using petrographic examination as an engineering tool to screen alkali reactive aggregates. It is recommended that by quantifying the reactive constituents in the aggregates, a more accurate classification of reactivity of aggregates will be possible.
Contents

Acknowledgements i

Abstract iii

1 Introduction 1

1.1 Background and purpose of the thesis 1
1.2 Organisation of the thesis 3

References

2 The Alkali-Aggregate Reactions 7

2.1 A variety of reactions 7
2.2 The amount and effect of the alkalies in the cement paste 8
  2.2.1 Enrichment, concentrations and recycling of alkalies in concrete 9
  2.2.2 Alkalies from other sources 10
      Leaching of alkalies from aggregate
      Contribution of alkalies from sea water/dredged aggregates
      External alkalies
      Alkali movement
  2.2.3 Replacement materials and additives 12
2.3 The reaction in relation to other damaging mechanisms - the holistic approach 13
  2.3.1 Freeze/thaw 14
  2.3.2 Secondary ettringite formation 14
  2.3.3 Corrosion of Reinforcing Steel 15
  2.3.4 Leaching 16

References

3 Properties of Alkali-Reactive Aggregates 21

3.1 Pattern of reaction for different types of aggregate 22
3.2 Petrographic properties of slow/late alkali-reactive aggregates 26
  3.2.1 The classification and properties of rocks subjected to deformation 28
  3.2.2 The undulatory extinction angle (UEA) of quartz 29
3.3 Other aggregate properties affecting the reaction 32
  3.3.1 Influence of reactive particle size 32
  3.3.2 The effect of grading 33
  3.3.3 Natural aggregates versus crushed aggregates 35
      Natural aggregate
      Crushed aggregate
3.4 Critical amounts of slow/late alkali-reactive aggregates 36

References
Natural aggregates 92
Crushed stones 93
Blends of aggregates 93
Discussion 94
The reactivity of different rock types 94
The usefulness of the petrographic examination, the NBRI mortar-bar test and the established limit of the critical amount of reactive rocks in aggregates 95
Conclusion 97
Acknowledgements 98
References 98

7 Sequential examination of slowly expanding alkali-reactive aggregates in accelerated mortar bar testing 103
B.J. Wigum and W.J. French

Introduction 105
Experimental Methods 106
Thin sections 107
Scanning electron microscopy 107
Soluble silica 108
Details of the aggregate 108
Meta-argillite 108
Mylonites 109
Metagreywacke 110
Microgranite 110
Mortar Bar Expansion 110
The sequential development of the reaction 112
Meta-argillite 112
Ultramylonites 113
Mylonite 115
Metagreywacke 115
Microgranite 115
Limestone 115
The composition of the gel reaction products 118
Quantitative aspects of mobilised silica and gel reaction product 120
Discussion 121
Magnitude of expansion 121
Aggregate grading 122
Pattern of expansion 123
Reactivity of the various aggregates 125
The composition of the gel reaction product - regeneration of alkalies 126
Quantity of silica involved in the reaction 127
The microstructure of the reaction product 127
Conclusion 128
Acknowledgements 129
References 129
8  Accelerated tests for the potential exhibited by aggregates for alkali-aggregate reactions 133
   B.J. Wigum, W.J. French, C.D. Hills and R. J. Howarth

Introduction 136
   ASTM C-227 Mortar Bar Test 138
   ASTM C-289 Chemical Test 139
   Accelerated Mortar Bar Test 140
      Alkali immersion methods 140
      Enhanced alkali method 143
Experimental Procedures 144
   Aggregate 144
   Cement 145
   Mortar bar sizes and composition 145
   Moulding and storage 145
   Method of measurement 146
Results 146
Discussion 152
   Expansion criteria 152
   Influence of volume of alkali 154
   Cement type 155
   Practical consideration 156
   Pessimum aggregate proportions 157
Statistical appraisal of test data 160
   Variation of standard deviation with mean expansion 160
   How many tests ? 163
   Assessment at an early age 165
   Contributions to variability 166
Towards a reliable test 171
   Procedural suggestions 172
Conclusions 173
Acknowledgements 174
References 175

9  Examination of Microstructural Features of Norwegian Cataclastic Rocks and Their Use For Predicting The Alkali-Reactivity in Concrete 181
   B.J. Wigum

Introduction 183
Theoretical background 186
   The important role of subgrain boundaries 186
   Activation energy, driving forces and preferential site of reaction 188
   Other preferential sites of reaction 189
      Inclusions
      Myrmekite
      The accessibility of the reagent
Experimental procedure 190
   Petrographic examination and grain size measurement 190
**Contents**

1. **Contents** ix

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Additional measurements related to the reactivity</td>
<td>192</td>
</tr>
<tr>
<td>Test procedure to determine potential alkali-reactivity</td>
<td>193</td>
</tr>
<tr>
<td><strong>Results</strong></td>
<td></td>
</tr>
<tr>
<td>Geological sampling and petrographic description of rock samples</td>
<td>193</td>
</tr>
<tr>
<td>Mineralogical composition and other experimental data</td>
<td>197</td>
</tr>
<tr>
<td>Mortar-bar expansion</td>
<td>198</td>
</tr>
<tr>
<td><strong>Discussion</strong></td>
<td>199</td>
</tr>
<tr>
<td>The quartz XRDA/DTA ratio</td>
<td>200</td>
</tr>
<tr>
<td>The Index of Crystallinity (CI)</td>
<td>201</td>
</tr>
<tr>
<td>The water absorption</td>
<td>202</td>
</tr>
<tr>
<td>The mean quartz grain size ($d_{50}$), and the total grain boundary</td>
<td>203</td>
</tr>
<tr>
<td>area of quartz</td>
<td></td>
</tr>
<tr>
<td><strong>Conclusion</strong></td>
<td>207</td>
</tr>
<tr>
<td>Acknowledgements</td>
<td></td>
</tr>
<tr>
<td>References</td>
<td>208</td>
</tr>
</tbody>
</table>

10  **Summary**  

11  **Conclusion**  

**INDEX**  

About the Author
Chapter 1

Introduction

1.1 Background and purpose of the thesis

Ever since T.E. Stanton in 1940 first observed and diagnosed the Alkali Aggregate Reactions (AAR) in concrete, the phenomena has been subject to a great deal of research. Since the first international conference regarding AAR which was held in Denmark in 1974, researchers have gathered together, on many occasions to present their findings on this subject. A series of nine international conferences have been held, which has attracted increasing numbers of research workers and engineers. The proceedings of these conferences provide the most important source of research information available. Studies have progressed rapidly in a number of different directions, ranging from identification of the aggregate mineral components and the mineralogical features which are involved in the reaction, through to the mechanisms which control the reaction themselves, to diagnosis, testing and assessment of the reaction on concrete properties. However, the phenomena of AAR is complex, and there are many interacting and interdependent parameters that influence its occurrence which still leaves many questions unanswered.

In 1973, Gogte, following Brown (1955) and Mielenz (1958) suggested that AAR should be viewed from a new angle. They claimed that crystalline quartz-bearing rocks could act as potentially alkali-reactive aggregates as well, however, by a slow or delayed mechanism in comparison to the well known rapid alkali-aggregate reactions. As a consequence an increasing interest has been shown into the new potentially alkali-reactive rock types. In order to study these rock types new petrographic methods have been investigated to determine and quantify their presence. Investigations brought interest particularly into the occurrence of undulatory extinction angle (UEA) of quartz as a quantitative indicator of the alkali-reactivity. The introduction of a new method to measure the undulatory angle of strained quartz by Dolar-Mantuani (1981, 1983), was used to a large extent as a tool to quantify the alkali-reactivity of quartz-bearing rocks exhibiting strained quartz. However, many scientists questioned the examination of the undulatory angle of strained quartz and the value of the method (Andersen and Thaulow 1989). Today the method is no
longer considered to be reliable for predicting the alkali-reactivity of slow/late alkali-reactive aggregate (Grattan-Bellew 1986, 1992 and French 1992).

The application of an accelerated mortar bar procedure, presented in 1986 by Oberholster and Davis as the NBRI Mortar-Bar Test, has frequently been used in many countries to determine the potential alkali-expansivity of various types of aggregates and, modified versions have recently become standard test procedures in some countries (CSA 1994, ASTM 1994). Evaluation of the test has shown it to be reliable and reproducible, both for rapid and slow/late alkali-reactive aggregates. However, there is still a need to develop a reliable and reproducible petrographic method, as a first step in the quantitative assessment of potentially slow/late alkali-reactive aggregates.

The awareness of slow/late alkali-reactive aggregates as a cause of degradation of Norwegian concrete structures is rather new. This made the Norwegian researchers able to take advantages of the broad international experiences which were available, when starting to study the problem. Since 1989, a major research project regarding AAR has been carried out at SINTEF Structures and Concrete (Jensen and Danielsen 1993, Øye et al. 1993 and Meland et al. 1994), including a Doctor Technicae thesis (Jensen 1993). Laboratory testing, aiming to substantiate suitable test methods, resulted in 1992 in the establishment of an optional arrangement for declaration and approval of Norwegian aggregates for concrete purposes (DGB - Deklarasjon- og Godkjenningssordning for betongtilslag). Aggregates for concrete purposes are now tested on a voluntary basis according to the procedures given by the Norwegian Concrete Society, Publication NB, No. 19 (Norsk Betongforening 1991). The procedures include both the use of a new petrographic method and, an accelerated mortar bar test, adopted from the primary NBRI Mortar-Bar Test.

When using such test procedures in the screening of potentially alkali-reactive aggregates, ambiguities will always occur regarding the use of appropriate specification limits. This is a problem in particular in Norway, due to the high amounts of potentially alkali-reactive rock types in natural aggregates. The frequent occurrence of e.g. cataclastic rocks, which are considered as the most common and widely distributed source of alkali-reactive aggregates in Norway (Jensen 1993), along with other frequently occurring slow/late alkali-reactive rock types, requires a full description, including quantitative measurements of microstructural features of all the rock types, rather than the simple use of standard terminology. The relationships between the petrographic examination and the final stage of accelerated mortar bar testing must be established, along with precise guidelines for both methods. In addition these test results should be compared with known field performance of similar materials. Without all these assessments it is difficult to make proper judgements on the significance of test results or to set appropriate specification limits for the properties being tested.
The purpose of the present work is to study the properties, classification and testing of certain Norwegian aggregates. The cataclastic rocks were selected as these are the most frequent alkali-reactive rock types found in Norway. The aim was to achieve a better understanding of the properties governing the alkali-reactivity of such rocks, thereby enabling improvement of existing test procedures and guidelines for engineering practice. It is necessary to improve the existing petrographic examination methods if it is to be used as a reliable and reproducible tool, and as a first step, in the screening of potentially slow/late alkali-reactive rock types. This must be based upon the quantitative determination of microstructural features. The relationship between the petrographic examination and the accelerated mortar bar test, as the precision assessment of the accelerated mortar bar test itself, must be established.

1.2 Organisation of the thesis

The thesis is divided into three main sections. The first section (chapter 2 to 4), covers background topics and literature reviews concerning alkali-aggregate reaction, aggregate properties and test methods. This section gives a general introduction of the subjects, and the experimentally techniques are covered in more details in the second section. In the literature review, AAR is covered in outline as this area is already covered in detail in existing reviews. Nevertheless, some topics regarding the AAR are given a more extensive coverage because these are regarded to be of importance when assessing the complex phenomena of AAR, and which are to a limited extent covered earlier.

The main results of this study are presented in the second section (chapter 5 to 9), which comprises of five papers published, accepted for publication or submitted for publication in international proceedings and journals as follows:

Wigum, B.J. and Jensen, J.: Petrographic examination of Norwegian glacifluvial aggregates: interpretations of mechanisms leading to high contents of Cataclastic rocks. Accepted for publication in NGU Bull., Norway.


Wigum, B.J. and French, W.J.: Sequential examination of slowly expanding alkali-reactive aggregates in accelerated mortar bar testing. Accepted for publication in Magazine of Concrete Research, London, UK
The choice of experimental techniques used in the present work was primarily to observe the following trends: In chapter 5, the paper discusses the observed high amount of cataclastic rocks in Norwegian glaciofluvial deposits, which is a basis to understand the frequent occurrence of cataclastic rocks and the problems regarding AAR in Norwegian structural concrete.

The three following papers (chapter 6 to 8) consider the various aspect of testing and evaluating these types of aggregates. Chapter 6 assesses the amounts of alkali-reactive rocks in aggregates, from an existing petrographic method of examination, which is compared with the expansion measured by the Norwegian version of the accelerated mortar bar method. Chapter 7 considers cataclastic rocks along with certain other types of slow/late alkali-reactive rock types and, looks at how these aggregates respond in the accelerated mortar bar test. Chapter 8 deals with the accelerated mortar bar test itself, the accuracy of the test, and how certain parameters, including cement type and mortar-bar size, influence the measured expansion.

After considering the background for the occurrence of cataclastic rocks and the various test methods, experimental techniques are considered which could more accurately predict the potential alkali-reactivity of cataclastic rocks in chapter 9. In this chapter the quantitative measurements of various types of cataclastic rocks are determined by a new petrographic method of examination, and correlated with the measured expansion of the accelerated mortar bar test.

Finally, chapter 10 summarises the findings, which are presented as main conclusions from the experimental work in chapter 11.
References


Chapter 2

The Alkali-Aggregate Reactions

2.1 A variety of reactions

Alkali Aggregate Reaction (AAR) is a chemical reaction where sodium and potassium ions in solution react with certain types of aggregate in the concrete. It is generally agreed that the chemical reaction between the alkali hydroxides and reactive silica is essentially a dissolution reaction. This occurs as a result of the increased solubility of amorphous, disordered or poorly-crystallised forms of silica minerals in high pH solutions. The reaction forms a hygroscopic alkali-silica gel which imbibes water and swells. The swelling forces generated may be sufficient to disrupt aggregate and the surrounding concrete, causing expansion, cracking, and associated deterioration. The mechanisms of reaction have been described by various authors (e.g. Dent Glasser and Kataoka 1981a,b, Chatterji et al. 1986, 1987, 1989), and an extensive literature review is given by Jensen (1993). The mechanisms of reaction will not be considered further in this section.

The AAR can be divided into several reactions, which are associated with the type of alkali-reactive aggregates and the mechanism and rate of the reaction. Alkali-silica reaction (ASR), which was identified first, is the most rapid reaction occurring with various heterogeneously structured, porous, and sometimes hydrous silica minerals. Slow/late-expanding alkali-silicate/silica reaction, appears to occur with a delayed onset and at a slower rate and is associated with crystalline quartz-bearing rock types as the reactive constituent. The so-called alkali-carbonate reaction which has been reported with some dolomitic limestones will not be considered in this work. The properties and potential alkali-reactivity of different types of aggregates are discussed further in chapter 3.
2.2 The amount and effect of the alkalies in the cement paste

The alkalies in the cement are derived from the raw materials (clay, limestone, chalk and shale) used in the manufacture of the cement. If coal is used as a fuel, alkalies can also come from the coal ash. The final proportion of the alkalies present in a clinker will be dependent on the proportion of mica, illite or feldspar in the feed and upon the details of the cement manufacture.

Usually, in determining the safe alkali content of the pore solution in concrete made with an alkali-reactive aggregate, only the alkalies derived from the cement are considered. The purpose of this section is to assess possible uncertainties arising from the method adopted to evaluate the total alkali content of the pore solution in the concrete system. Different factors may affect the total content of alkalies observed, and these will be reviewed and discussed.

In order to assess the total content of available alkalies present in a cement or concrete it has become standard practice to express the alkali content in terms of "sodium oxide equivalent":

$$\text{Na}_2\text{O}_{\text{eq}} = \text{Na}_2\text{O} + 0,658 \times \text{K}_2\text{O} \text{ (in weight percent)}$$

The great majority of concrete structures reported internationally which show deterioration due to AAR were made using a high-alkali cement. That is to say, in excess of 1% alkalies as Na_2O_{eq}. The alkali content of cement clinker may be differentiated into two generic types; alkalies which are normally condensed on the surface of clinker grains, and alkalies which are locked into the crystal structures of the clinker minerals. Alkalies present as water soluble sulphates go into solution almost immediately, whereas alkalies locked into clinker grains become available to the solution rather more slowly. The overall characteristic release rate, will vary from one cement to another, depending on the distribution of alkalies between rapid-release and slow-release sources, as well as on total alkali content (Glasser 1992).

Since the work of Power and Steinour (1955), it has been suggested that an alkali content of less than 0.6 weight-% Na_2O_{eq} in the cement is normally harmless with respect to the generation of AAR. Although such a limit will greatly minimise the risk of AAR, it ignores the basic fact that the alkali content of concrete is determined by both the alkali content of the cement, and by the total cement content of the concrete.

Oberholster (1983) pointed out various difficulties that arise in trying to set a clear cut-off limit for the alkali content of a cement above which deleterious expansion with a certain aggregate would occur. He proposed certain guidelines in terms of the cement content of the concrete and the active alkali content of the cement to assess the
potential that a concrete would undergo expansion due to AAR. The guideline limits are illustrated in fig. 2.1.

Since 1986, Denmark has specified a requirement of a maximum content of 3kg Na₂Oₐq/m³ of concrete for moderate and aggressive environments (Jensen 1993). In UK several sources recommend using the same alkali level limit of 3kg Na₂Oₐq/m³ of concrete, based on the certified average alkali level supplied by the Portland Cement manufactures (Hobbs 1988). Similar recommendations are proposed in Norway (Dahl et al. 1993).

![Figure 2.1](image)

**Figure 2.1** Relationship between the active alkali content of cement, the cement content of concrete and the alkali content of concrete and the resultant potential of the concrete for AAR (From Oberholster 1983)

### 2.2.1 Enrichment, concentrations and recycling of alkalies in concrete

In addition to the total content of alkalies in a concrete system, the level of alkali distribution will also be of interest. It has been suggested that moisture mobility through concrete can cause alkali metal salts to migrate and create temporary or permanent concentrations of these salts in some regions of the concrete. One example, of where this can occur is in foundation blocks where the tops are exposed, allowing water to evaporate from the surface (Concrete Society 1987).

Various authors (French 1989, Lagerblad and Trägårdh 1992), following Power and Steinour (1955), have observed the phenomenon of recycling of alkalies during the alkali-aggregate reaction. The alkali gel formed within the grains changes composition when it comes in contact with the paste, and becomes richer in calcium, and loses alkalies to the binder. The change in composition is caused by either
dilution of the alkalies by a calcium compound from the cement paste, or by a cation exchange reaction where calcium replaces alkalies. Lagerblad and Trägårdh (1992) suggest that the cation exchange reaction would cause the displaced alkali to return to the cracks in the aggregate particles and no alkalies are consumed. According to this phenomenon the reaction may theoretically continue until all alkali-reactive material is transformed into gel.

2.2.2 Alkalies from other sources

In addition to the alkalies obtained from the cement, the concrete system may also be supplied with alkalies from other sources.

Leaching of alkalies from aggregate

Certain types of aggregate within an alkaline medium such as concrete, may release sodium and potassium ions into the pore solution. In an investigation where different types and sizes of aggregates were stored in a saturated Ca(OH)₂ solution at 40°C, the maximum amount of leached alkali after 1 year exceeded 1.0 weight % Na₂Oeq. (Jensen 1993). Grain size strongly influences the release of alkalies with lowest values observed for the coarsest fraction. In spite of the very high values obtained, it is however speculation as to whether such a high amount of alkali is actually released in the concrete. Similar results and conclusions were obtained in an investigation carried out by Batie et al. (1990). Kawamura et al. (1989) found alkalies to be continuously released from reacting andesitic aggregates into the pore solution until at least 90 days. The rate of removal of alkali is also dependent on the state and type of the aggregate, with fine, freshly crushed material rapidly giving up alkalies in outer micrometer or two, then reaching equilibrium (W.J.French, pers.comm.).

St John and Goguel (1992) investigated the aggregate/pore solution interaction and found that leaching of alkalies from concrete during curing and weathering can be significant. They claim that these results provide an explanation as to why some concrete structures containing low-alkali cement have undergone alkali-aggregate reaction with sands containing rhyolite and andesite.

Examination of pore solutions in concrete cores from Ontario, Canada, showed that the alkali contents were higher than the estimated original value (Grattan-Bellew 1994). It was claimed that the excess of alkalies in the pore solution in the 30 years old concrete could possibly have been derived from the clay minerals in the limestone aggregate. Laboratory tests estimated the water soluble alkalies in pulverised limestone aggregate to be 0.45 kg (Na₂Oeq.) where the concrete contains 2000 kg of aggregate/m³. Grattan-Bellew (1994) reports that the alkali contents in concrete cores from a number of dams, containing a variety of aggregates, appear to be higher, in large concrete structures, than the estimated initial values. He claims that these
observations suggest that alkali contribution to the pore solution from the aggregate, may not be uncommon.

**Contribution of alkalies from sea water/dredged aggregates**

When sodium chloride is present in the aggregate or mix water, the tricalcium aluminate in Portland cement may react with the chloride taking some of the chloride out of the solution with the separation of sodium ions in solution. Similar enhancement of alkalies has also been found to occur for sulphates and for nitrates (Metha 1978).

In Iceland, sea-dredged aggregates were formerly used as the main source for concrete. Work by Olafsson and Helgason (1983) showed the importance of the contribution of alkalies from these sea-dredged aggregates. In this case the water content of the sea-dredged aggregates was so high that no additional water was added to the concrete mix. The calculation of the total alkali level in table 2.1, per m$^3$ of a concrete mix from Reykjavik, shows clearly the enhanced alkali-level in the concrete.

**Table 2.1** Calculation of the contribution of alkalies in an Icelandic concrete mix. (With 285 kg of OPC-cement, sea-dredged-sand with 10% water content, and sea-dredged gravel with 3-3.5% water content) (Olafsson and Helgason 1983).

| Amount of alkalies in the cement (Na$_2$O/m$^3$) | 4.1 kg |
| Amount of alkalies contributed from sea-dredged sand (Na$_2$O/m$^3$) | 2.0 kg |
| Amount of alkalies contributed from sea-dredged gravel (Na$_2$O/m$^3$) | 0.6 kg |
| **Total amount of alkalies (Na$_2$O/m$^3$)** | **6.7 kg** |

This high Na level will bring with it a high chloride level that is certain to cause reinforcement corrosion as well as AAR.
External alkalies

It is not uncommon for alkali ions to enter concrete from the outside. This happens commonly on the coast and on roads and bridges where de-icing salts are employed.

Alkali movement

Nixon et al. (1979) have shown that alkalies can diffuse towards the surface of concrete on wetting and drying. Xu and Hooton (1993) reported in addition that constant humidity gradients, freezing/thawing cycles and electrical potentials, can produce strong ion movements as well. More recently electrical potentials are being applied to concrete in chloride removal which leads to realkalization. Such movements of chloride ions could generate significant local alkali enhancements, and initiate and sustain AAR.

2.2.3 Replacement materials and additives

One way to prevent problems when using potentially reactive aggregates, is to replace a sufficient quantity of cement by supplementary cementing materials such as condensed silica fume, pulverised fly ash (PFA) and ground granulated blast furnace slag (GGBFS).

Condensed silica fume is an amorphous SiO\(_2\) rich material in the size range of 0.02 to 0.50 µm. These particles, which due to the chemical and crystalline properties are highly alkali-reactive, have ironically been used with success as an admixture, to prevent AAR in different countries. In Iceland since 1979 the State Cement Works has only produced cement intermixed with silica fume, replacement at first being 5%, but since 1983 being 7.5% (Olafsson 1992). This new cement is now regarded as a final solution to the AAR problems in Iceland. Several mechanisms have been proposed to explain the positive influence of silica fume. One of the explanations is that the extremely fine particles react very quickly and consume alkalies even before the concrete has set. However, in some cases it has been demonstrated that fused silica particles can react, leading to expansion at later stage. The probable reason for this is that commercial silica fume have not been properly dispersed and broken up during the mixing, thus forming silica rich agglomerates (Kawamura et al. 1986, Pettersson 1992, Fidjestøl 1991).
When supplementary cementing materials are used in concrete to minimise the risk of damage from AAR, these materials might enhance the total level of alkalies. The British Working Party report (Concrete Society 1987) recommends the use of the equations below when calculating the total contribution of alkalies according to the 3.0 kg/m³ limit:

For factory-made cements the reactive alkali content of concrete is calculated from:

$$A = \frac{(C*a)}{100} \quad [1]$$

For site combination of Portland cement with either GGBFS or PFA the reactive alkali content of the concrete is calculated from:

$$A = \frac{(C*a) + (E*d)}{100} \quad [2]$$

where

- $A$ = reactive alkali content of concrete (kg/m³)
- $C$ = target mean Portland cement content of concrete (kg/m³)
- $a$ = certified average acid soluble alkali content of Portland cement (%)
- $E$ = target mean content of either GGBFS or PFA in the concrete (kg/m³)
- $d$ = water soluble alkali content of GGBFS or PFA provided by the manufacturer (%)

### 2.3 The reaction in relation to other damaging mechanisms - the holistic approach

Better knowledge of deleterious processes and its behaviour in concrete, has shown that emphasis cannot be placed on only one single process or mechanism when assessing the durability or reason for damage of concrete. In most cases different processes and mechanisms will occur simultaneously and their deleterious effect will depend upon each other, different mechanisms can interact with each other, and one process can initiate another.

In terms of getting a better understanding of the behaviour and durability of concrete the holistic approach is outlined by Mehta (1994). The term holistic from the Greek word "holos", refers to an understanding of a phenomenon or a structure in term of an integrated whole whose properties cannot be deducted from the sum of the properties of the constituent parts (Mehta 1994).

Regarding the Alkali-Aggregate Reaction in relation to other deleterious mechanisms, the classical question about which process occurs first will always be asked. Often it has been suggested that other processes and mechanisms and secondary
deleterious processes are more damaging to a structure than the Alkali-Aggregate Reaction.

The purpose of this section is to evaluate the consequences of other deleterious mechanisms on potential Alkali-Aggregate Reactions.

### 2.3.1 Freeze/thaw

Bolton (1992) investigated the secondary effects of freeze/thaw on concrete exhibiting AAR based on the suggestion that freeze-thaw action may contribute to the continued deterioration of the concrete. From laboratory test it was found that high strength concrete, which is commonly regarded as "immune" from freeze/thaw damage, become more susceptible when cracking is induced by AAR. The ultimate expansion caused by combination of AAR together with freeze/thaw was shown to be greater than that caused by AAR cracking alone. It was however mentioned by the author that the results were for plain unreinforced concrete, and consequently might not be directly applicable to real structures.

Xu and Hooton (1993) investigated the possible interaction between freezing/thawing cycles and the migration of alkali ions. Their results revealed that migration and concentration of alkali ions does occur under freezing/thawing cycles.

It has been proposed by several authors (Jensen 1993, Lagerblad and Trägårdh 1992) that the access of alkalies into slow/late alkali-reactive aggregate occurs via interfacial cracks between detrital grains and crystals. It may be reasonable to believe that processes such as freezing and thawing of aggregates in concrete will increase the width of these cracks. This will subsequently lead to increased migration of alkalies to the reaction site within reactive particles. On the other hand, initial expansion and cracking resulting from AAR caused by the reaction may subsequently accelerate the deterioration by frost.

The frost resistance of concrete is influenced by the content of air voids. It is well known that lack of sufficient amount of entrained air makes concrete potentially vulnerable to freezing and thawing damage. In concrete with AAR the gel product will fill up some of the voids and decrease the total volume of air-voids, and hence decrease the frost resistant of the concrete.

### 2.3.2 Secondary ettringite formation

Simultaneous presence of alkali-silica gel and ettringite in cracks in concrete has been reported by several authors. The so-called delayed ettringite formation is the formation of ettringite from the reaction products of previously decomposed ettringite which was formed during the initial hydration and setting of the cement paste. Such
ettringite is formed subsequent to heat curing at temperatures above about 60-70°C, and can be expansive and can cause expansion/cracking in the cement paste of concrete (Johansen et al. 1993). The simultaneous presence of both gel and ettringite leads to the question about what in such cases is the primary cause of damage.

Jones and Poole (1986) investigated the interaction between AAR and the ettringite development. They stated that the common replacement of alkali-silica gel by ettringite indicates that AAR must have reached an advanced stage before ettringite growth occurred.

Shayan and Quick (1991) investigated the AAR in cracked and uncracked prestressed steam-cured concrete railways sleepers. They suggested that microcracking of the sleepers was initiated by AAR and loading in the railway track, and that ettringite subsequently filled these existing cracks as a result of moisture penetration. They also claim that this may have contributed to further widening of the existing cracks, but was considered to be a consequence of the initial cracking and not the cause of it.

Oberholster et al. (1992) investigated the reason for cracking of prestressed concrete railway sleepers. They found that cracking was observed only in sleepers made with potentially alkali-reactive aggregate, but not in those made with innocuous aggregate. It was concluded that although it is possible that expansion associated with delayed ettringite formation plays a contributory role in the overall expansion of cracked sleepers, it appears that AAR is a prerequisite for this to occur.

2.3.3 Corrosion of Reinforcing Steel

In principle, lower corrosion is expected due to the highly alkaline environment associated with AAR. However, many investigators have observed that microcracking of concrete resulting from factors other than corrosion, plays a major role in increasing the penetration of water, oxygen, and CO₂ which are instrumental in the initiation and progress of corrosion-related damage (Metha 1994).

Kawamura et al. (1989) investigated the influence of AAR on the corrosion of steel reinforcement in concrete, especially when NaCl contaminated the mortar. They found a higher corrosion rate for steel bars embedded in mortar with alkali-reactive aggregate. The increase in the corrosion rate was explained by the increase in the Cl⁻/OH⁻ ratio in the pore solution, as well as to some changes in the morphology of hydrate products within the mortar phases due to the AAR.

Page et al. (1992) investigated whether AAR-induced expansion could be stimulated by applying cathodic protection to steel in concrete of "near-threshold" mix proportion. Concrete specimens containing steel electrodes were made from a mix that contained an alkali-reactive aggregate, but insufficient alkali concentration to cause
expansion due to AAR. Expansion in the vicinity of the steel was found to be dependent on the level of applied polarisation and was associated with local development of AAR in a zone of enhanced alkalinity formed around the cathode.

Sanjuan and Andrade (1994) investigated experimentally the influence of AAR on the corrosion of embedded steel reinforcement of concrete structures. The work showed that exposure of concrete exhibiting AAR, to a chloride containing environment, reduced the time needed by the chlorides to reach the rebar, and increased the risk of corrosion of steel reinforcement.

**2.3.4 Leaching**

Hydrated cement paste consists of about 65% lime, where part of it is present as free calcium hydroxide. The solubility of calcium hydroxide in water is about 1.7 g/litre, and it can be leached out by water (Soroka 1979). Structures such as bridges, piers, dams and part of structures below ground-water, are permanently or frequently exposed to water, and might be damaged due to leaching. The penetration of the water will be dependent on the permeability of the concrete. Surface cracking of structures due to AAR will contribute to an enhanced permeability of the concrete. Such structures frequently exhibit lime on the surface associated with map-cracking.
References


Chapter 3

Properties of Alkali-Reactive Aggregates

It is generally accepted that the alkali-aggregate reactions involve two main types of aggregates; rapid- and slow/late alkali-reactive aggregates. Primarily, alkali-reactive aggregates were related to various heterogeneously structured, porous, and sometimes hydrous silica minerals such as opal, chert, chalcedony, flint, and some sorts of volcanic glass, which were found to be extremely reactive under the right conditions. More recently well crystallised and more dense quartz-bearing rock types (e.g. meta-greywacke, meta-sandstone, argillite, phyllite, cataclastic rocks and various other types of rock exhibiting signs of deformation) have been observed to show reactions in concrete as well, by a slower mechanism which leads to a delayed expansion and damage. Microcrystalline quartz and/or quartz with crystal lattice defects (strained quartz) caused by some sort of deformation, are assumed to be one of the reasons for the alkali-reactivity for such slow/late alkali-reactive aggregate. The so-called alkali-carbonate reaction, which occurs with some types of dolomitic limestones, is not considered in this work. Gillott et al. (1973) suggested that the expansion observed in association with some types of greywacke, phyllite and argillites was due to the occurrence of swelling phyllosilicate minerals (vermiculite). However, the mechanism for this reaction still remains unclear.

During the last decades many researchers have reported an increasing number of new rock types which react in concrete due to AAR. Dolar-Mantuani (1983) collected these information and presented a list of potentially alkali-reactive rock types. Such list should however always be read with caution, and are of relatively little value for the assessment of polycrystalline slow/late alkali-reactive rock types, unless a further description of the microstructural features, are carried out. It is clearly incorrect to consider rock type only as a criteria for an aggregate's potential for reactivity.

It is suggested by French (1991) that a full description of potentially alkali-reactive rocks is required, rather than the use of standard nomenclature. Also Jensen (1993) suggested that the alkali-reactivity of slowly expansive rocks should be evaluated by microstructural analyses and known field experience, rather than list of rocks based on traditional classification.
3.1 Pattern of reaction for different types of aggregates

The formation of cracks in hardened concrete may be associated with many factors, such as drying shrinkage, thermal stresses, weathering, corrosion of reinforcement, poor construction practices, construction overloads, externally applied loads, deleterious chemical reactions, etc. (Fournier and Bérubé 1993). However, the occurrence of reaction products (gel) in micro-cracks is the main diagnostic symptom confirming that AAR has occurred. Microcracking due to AAR is generated through forces applied by the expanding aggregate particles and/or swelling of alkali-silica gel within and around the boundaries of reacting particles.

Examination of thin section of concrete under the petrographic microscope, is probably one of the most widely used technique for assessment of AAR in concrete. Different types of dissolution, cracking, micro-cracking and disintegration of aggregate particles can be observed in different types of reacted particles; Delamination might occur in cataclastic rocks with a strong foliation texture due to ductile deformation, while inter- and intragranular fissures as well as polygonization occur in some quartzite with separation of individual monocrystalline crystals. In rapid alkali-reactive, fine-grained particles (e.g. chert), random micro-cracking and dissolution can be observed both within and around the boundaries of reacting particles.

Different types of reaction products can be observed in micro-cracks in reacted particles, in micro-cracks in the cement paste, as lining of gel in air-voids, or in gel in air-voids filling up parts of the void. Reaction products occurring in the hardened cement paste has a completely different morphology from the reaction products located in the reacting aggregate particles. Within aggregate particles reaction products typically exhibit a crypto-crystalline appearance, while near the paste-aggregate interface the reaction products transform into a transparent, isotropic morphology. The reaction products eventually form into a layered, non-transparent morphology away from the cracked aggregate particle.

In this chapter different pattern of reaction for different types of reactive materials are reported and exemplified by micrographs. Crack pattern and typical microstructural features of different types of alkali-reactive aggregates are presented. The thin-sections presented in this chapter were examined by the author at SINTEF Structures and Concrete (Norway), The Icelandic Building Research Institute (Iceland), Geomaterials Research Services Ltd. (UK), and Institute for Research in Construction, NRC (Canada).

Rapid alkali-reactive aggregates (figs. 3.1, 3.2, 3.3, 3.4 and 3.5a), such as opal, chert and volcanic glass, will react by dissolution to form alkali-silica gel at both the surface of the reactive particle and inside it. The reaction occurs in zones or patches within the aggregate, associated both with micro-cracking and dissolving processes which transform parts of the particles into gel. Smaller particles will have a much higher specific surface area than coarser ones, and consequently they will react quicker.
For slow/late alkali-reactive aggregates (figs. 3.5b, 3.6, 3.7, 3.8, 3.9, 3.10 and 3.11) the reaction probably initiates at original cracks, inhomogenieties or grain boundaries, which act as pathways for the alkaline pore solution. Only certain parts of reactive particles should be considered as alkali-reactive. In such cases only minimal gel formation is produced, however capable of separating the grain boundaries and leading to expansion and cracking of the aggregate. The resulting cracks will appear more marked, longer and more defined. Those cracks with a longer extension will be more susceptible to the stress field in the concrete structure.

**Figure 3.1** Reactive chert (UK) in ordinary light shows gel-filled crack running from the particle into the cement paste. Part of the particle surface is transformed into gel. (Geomaterials Research Services Ltd).

**Figure 3.2** In polarised light it is shown that the reacted chert exhibits fine quartz crystals (< 5 µm). The reaction product shows recrystallisation. (Geomaterials Research Services Ltd).

**Figure 3.3** Reactive porphyritic volcanic glass (Iceland) in ordinary light. Zone of brownish palagonized glass is partly transformed into brownish gel. The palagonized glass is distinguished from the reaction product by the porphyrites and lack of drying shrinkage cracks. (The Icelandic Building Research Institute).

**Figure 3.4** Reacted rhyolite (Iceland) in ordinary light. Layered reaction product runs from the particle into the cement paste. Air void is completely filled with reaction product. (The Icelandic Building Research Institute).
Jensen (1993) proposed a hypothesis on the sequential development of AAR in Norwegian slow/late alkali-reactive aggregate based on petrographic observation and SEM/EDX analyses of reacted concrete. It was proposed that the access of alkalis into the aggregate occurs via interfacial cracks between detrital grains or crystals. As the reaction starts to take place some cracks will develop at the outer zone of the aggregate due to tension. These cracks will be oriented parallel to the preferred orientation and/or lamination of the rock and develop at the longest diameter of the aggregate. Lagerblad and Trägårdh (1992b) proposed a similar reaction mechanism, noticing the need for initial cracks to be present in the alkali-reactive materials. As the reaction takes place, the reactive grains are dissolved from the inside and an alkali gel is formed within the grains. In such a situation it will take some time to initiate the reaction process. The gel in contact with the paste and its pore solution changes composition and becomes more viscous and richer in calcium. The cation exchange would cause the displaced alkali to return to the crack in the aggregate grain. Consequently, they claim there is no pessimum for slow/late alkali-reactive rocks due to the non consummation of alkalis.

In this work the sequential development of reaction was followed by using the accelerated mortar bar test (chapter 7). Two different reaction styles were observed at the reaction sites involving dissolution of quartz along grain boundaries and crack generation within the aggregate, with the later being the main factor governing the amount of expansion produced by the reaction.

![Figure 3.5a & b](image) Sketches of difference in expansion and cracking between rapid- and slow/late reactive aggregates. Left (a); rapid reactive aggregate (e.g. opal). Right (b); slow/late reactive aggregate (e.g. mylonite).
Figure 3.6 Reacted cataclastic rocks in ordinary light. Layered reaction product runs from the particle into the cement paste. (Institute for Research in Construction NRC).

Figure 3.7 In polarised light relatively coarse quartz grains are observed. (Institute for Research in Construction, NRC).

Figure 3.8 Reacted ultra mylonite (NBRI test, D=56) exhibits in UV-light microcracking and dissolution. (SINTEF Structures and Concrete).

Figure 3.9 In polarised light it is observed that reaction is associated with subgrained parts. Coarser quartz grains are observed not to be affected. (SINTEF Structures and Concrete).

Figure 3.10 Reacted schist exhibits in ordinary light dissolution and disintegration along grain boundaries with infilling of gel. (Geomaterials Research Services Ltd).

Figure 3.11 In polarised light it is observed that quartz grains are relatively coarse. (Geomaterials Research Services Ltd).
3.2 Petrographic properties of slow/late alkali-reactive aggregates

Mather (1975) considered all types of aggregates to be potentially alkali-reactive, the differences are only in the rate and magnitude of reaction. However, for engineering practice, the magnitude of potentially alkali-reactivity has to be quantitatively evaluated. The nature and characteristics of aggregates susceptible to attack by alkalies in concrete have to be assessed with emphasis on their petrographic characters and microstructural features.

The factors which might be considered to affect the alkali-reactivity of a rapidly reacting silica-containing aggregate are considered by Zhang et al. (1990) to be as follows; 1) The phases in which the silica occurs, 2) porosity, 3) crystal particle size, 4) defects or strain within the crystals.

For slow/late alkali-reactive aggregates, very much the same factors can be considered as affecting the alkali-reactivity. However, the volume of alkali-reactive constituents for such aggregate types is different. The critical amounts of slow/late alkali-reactive aggregates will be considered later in this chapter.

The present work is primarily concerned with the properties of cataclastic rocks which are relatively dense crystalline rocks, where crystal particle size, subgrain development (total grain boundary area of quartz) and defects or strain with the crystals, will be the most significant factors affecting the alkali-reactivity. The effect of these factors on the alkali reactivity is discussed in chapter 9, based on results from the experimental determination of the various microstructural features of Norwegian cataclastic rocks.

AAR can occur in very fine-grained non deformed quartz (e.g. argillite), owing to its relatively large grain boundary-energy. However, strain energy and enhanced grain boundary area in the form of subgrain development or strain lamellae are probably necessary to promote reactions in relatively coarse-grained rocks, in which the original grain-boundary energy are relatively small. Experimental results of the effects of subgrain and strained quartz exhibiting undulatory extinction are considered in chapter 7 and 9. The minimum size of grains and subgrains which significantly enhance the solubility and hence the alkali-reactivity, is not yet established. However, the effect of the total grain boundary area of quartz are considered in chapter 9.

Some authors have made attempts to examine and assess microstructural features and other factors influencing the alkali-reactivity. However, insufficient attention has been given to quantifying the microstructural properties of the rock, and to relate this to the alkali-reactivity.

Grattan Bellew (1986, 1992) suggested that rather than the occurrence of undulatory extinction in quartz, the alkali-reactivity of certain aggregates may be due to the occurrence of microcrystalline quartz. French (1992a) claimed also that wherever meta-quartzites or rocks containing strained quartz are alkali-reactive, the quartz grains exhibit...
either strain lamellae or are cataclased or otherwise contain microcrystalline grains on larger grain boundaries or along fracture planes of various types. In further work French (1992b) proposed relationships between degree of reaction, mean particle size of alkali-reactive material, and alkali content for various types of aggregate (Figure 3.12).

Figure 3.12  Approximate relationships between degree of reaction, mean particle size of reactive material and alkali content for various types of aggregate (French 1992b).

Zhang et al. (1990) investigated the microstructure of various alkali-reactive aggregates by using analytical transmission electron microscopy (TEM). The study suggested that the source of alkali-reactivity is associated with large areas of grain boundary arising from a smaller quartz grain size, and the high density of dislocation present within many of these grains.

In an investigation performed by Kerrick and Hooton (1992) mylonites were studied for their alkali-reactivity. Besides the effect of microcrystalline quartz, it was suggested that the alkali-reactivity depends upon the degree of foliation (schistosity) of the rock. The investigation concluded that the reaction primarily occurs at the grain boundaries of microcrystalline quartz formed by dynamic recrystallisation of strained quartz.

In Sweden and Norway cataclastic rocks (especially mylonites) exhibiting strained quartz and subgrain development are found in structures in several areas exhibiting deleterious expansion due to AAR (Lagerblad and Trägårdh 1992a, Jensen 1990, 1993). According to observations on Norwegian structures, cataclastic rocks are now considered the most common source of alkali reactive rock types (Jensen 1993). An interpretation of the reactivity based on microstructural features of these rock types has not yet been established.
Thomson and Grattan-Bellew (1993) and Thomson, Grattan-Bellew and White (1994) investigated the alkali-reactivity of certain mineralogical and textural constituents in metamorphically deformed rocks. The study showed that the most reactive constituent appeared to be the microcrystalline quartz that had undergone significant subgrain development, but not complete recrystallisation. It was also observed that zones of notable undulatory extinction were not significantly more alkali-reactive than non deformed quartz. It was emphasised that a simplified approach involving measurement of undulatory extinction angles may lead to an incorrect estimation of strain, and therefore an incorrect estimation of the potential alkali-reactivity. The study left the relative importance of grain size versus the degree of subgrain development unresolved.

There is a necessity for engineering practice to develop methods and procedures to enable more accurate determination of potentially slow/late alkali-reactive aggregates, based on examination and quantification of microstructural features. More emphasis should be put on the complete petrographic description of rocks with the aim of quantifying certain microstructural features associated with alkali-reactivity, in particular the subgrain development. In order to evaluate the alkali-reactivity of aggregates, an accurate measurement of the size of subgrains in an aggregate may be necessary. To achieve this accuracy new analysis techniques have to be assessed.

3.2.1 The classification and properties of rocks subjected to deformation

Since Lapworth (1885) first described the mylonite (from the Greek "mylon" - a mill) from the Moine Thrust in Scotland, there have been many petrographic descriptions and classifications of the texturally distinctive rocks found associated with fault zones. However, ambiguities have surrounded the use of proper nomenclatures, descriptive terminology and, the classification of the various rocks. The term cataclastic rocks was introduced by Water and Cambell (1935) as a collective name for all rocks of the gouge - breccia - cataclasite - mylonite kindred. Also Higgins (1971) used this collective term, however, he realised the dilemma with multiple terms for the same type of rock and single terms for rocks of widely varying characteristics and origins. He cited the lack of literature which the uninitiated could refer to in order to learn about cataclastic rock nomenclature, theory, and characteristics or where the more experienced worker could turn for reference and comparison. As a consequence he produced an extensive paper concerning the classification and nomenclature of cataclastic rocks in an attempt to clarify the ambiguities. Sibson (1977) decided not to use the term cataclastic rocks, because the misleading implication that such rocks had developed solely by cataclasis, which he claimed in the strict sense involves the brittle fragmentation of mineral grains. He preferred the term fault rocks as a collective term. However, text books dealing with the microstructural features of rocks associated with fault zones, often use the general term; deformed rocks (Hobbs et al. 1976, Barker 1990).

Some cases have been reported where rocks associated with fault zones have shown to be alkali-reactive. However, it has been the tendency with researchers to describe the
rocks by a nomenclature based on mineralogical composition rather than microstructural features. As a consequence the names of many rock types have been included in lists of alkali-reactive rock types, when they in fact should be regarded as innocuous. In many instances rock types containing strained quartz have been reported slow/late alkali-reactive, without any further assessment or classification based on the microstructural features. Gogte (1973) was one of the first to report on the occurrence of slow/late alkali-reactive rock types, concluding that the potential alkali-reactivity of the Indian rock types granites, granodiorites, gneisses, charnockites, quartzites and schists was related to the relative amount and straining effects in quartz. Rao and Sinha (1989) investigated the textural and microstructural features of what they called alkali-reactive granitic rocks, including rock types such as biotite gneisses and mica granite. Shayan (1993) evaluated alkali-reactive aggregates in Western Australia, what he calls deformed granitic rocks. Kerrick and Hooton investigated the alkali-reactivity of aggregate quarried from a fault zone. The rocks were named deformed rocks, however, specific samples were classified as various sorts of mylonites.

The nomenclature and classification of cataclastic rocks in the experimental part of this work (chapter 9) are carried out according to Higgins (1971). However, it should be emphasised that the classification is based upon the quantification of microstructural features rather than lithologic composition.

3.2.2 The undulatory extinction angle (UEA) of quartz

The term undulatory extinction of quartz refers to an optical characteristic of strained quartz, when observed in thin section and viewed in cross polarised light using a petrological microscope. Permanent bending of crystal lattices result in a change in lattice and optical directions (displacement of the c-axes) across a crystal so that it does not extinguish as a whole. A region of extinction sweeps across the crystal as the stage is rotated and the crystal is said to exhibit undulose, undulatory or strain extinction.
Since Brown (1955), Mielenz (1958) and Gogte (1973) first claimed that quartz-bearing rocks could act potentially as alkali-reactive, the awareness of AAR in concrete caused by such rock types has been shown increasing interest. As mentioned earlier, Dolar-Mantuani (1981, 1983), measured the undulatory extinction angle (UEA) of strained quartz as a measurement of the amount of dislocation in the crystal lattice and related this to the alkali-reactivity of the material. The method had previously been used to a large extent as a tool for quantifying the alkali-reactivity of quartz bearing rocks exhibiting strained quartz. Buck and Mather (1984) proposed a minimum criteria for recognising aggregates containing potentially alkali-reactive strained quartz as being 20% strained quartz with an average undulatory extinction angle of 15°. However, many scientists have questioned the examination of the undulatory angle of strained quartz and the value of the method, and have claimed that other factors have a greater influence then UE-angle on enhanced alkali-reactivity of deformed rocks. Because the principle of the method has limitations and shows large variations (Andersen and Thaulow, 1989), the measuring of the undulatory angle of strained quartz is no longer considered to be a reliable parameter for predicting alkali-aggregate reactivity (Rogers 1990, Grattan-Bellew 1986, 1992 and French 1992a).

Because the method has shown to inaccurately echo the true value of the extinction angle, attempts have been made to develop more accurate techniques. Smith et al. (1992) proposed a new measuring method, using an universal stage mounted on the normal microscope stage, which enabled the determination of the true angle between c-axes of quartz.

However, even though improved methods have been identified to obtain more accurate values of the extinction angles, it is necessary to ask to what extent the undulatory extinction of quartz mirrors the alkali-reactivity. To get answer, the origin of undulatory extinction, along with other microstructural features produced during deformation and recovery processes of quartz, must be assessed.
The ductile deformation of quartz produces several microstructural features, observed and recognised by a variety of different names, both during the deformation, annealing and recrystallisation period. The literature apparently shows some disagreement about the origin of the different microstructures and to what period they are associated with. Suppe (1985) regarded the detailed pattern of undulose extinction to reflect the degree of polygonization of a crystal. If the extinction sweeps smoothly across a deformed crystal, it is an indication of little or no polygonization, at least on an optical scale (figure 3.15a). However, if the extinction sweeps stepwise across a crystal, the crystal is subjected to recovery, and exhibits polygonization at an optical scale (figure 3.15b). During the recovery processes the dislocations will move and group themselves into stable low energy configurations that usually take the form of low angle boundaries. These low angle boundaries which separate regions of slightly differing extinction position of the order of 1-5°, represent subgrain boundaries (Hobbs et al. 1976). Smaller subgrains bounded by low-angle walls (1° or less) may occur inside the larger subgrains, causing segmented undulose extinction within the larger subgrains when viewed in the optical microscope (Vernon 1976).

**Figure 3.15** Polygonization of a deformed crystal. (a) Segment of a bent crystal lattice. (b) After annealing, the dislocations have arranged themselves by climb into walls to form subgrain boundaries, producing a polygonized crystal in which each subgrain has a slightly different orientation (After Suppe 1985).
As the radius of lattice curvature becomes smaller (i.e. the crystal is more sharply bent) the undulose crystal may be composed of narrow regions of slightly different orientations refereed to as deformation bands (Spry 1976). Hobbs et al. (1976) claims that deformation bands are planar regions within grains that have suffered a different kind of deformation to that in adjacent part of the crystal. White (1973) investigated optical features in deformed quartz by studying identical microstructural features in both optical- and electron microscope. The aim of the study was to determine what kind of lattice defects were responsible for these optical features, and to clarify the nomenclature ambiguities related to them. The investigation showed that all the microstructural features, including undulatory extinction, deformation bands, deformation lamellae and subgrains, could be related to dislocation sub-structures, in particular to the development of subgrains. Therefore, he interprets, as a result of such conditions, the lattice is systematically bent at each subgrain wall, and the net result is seen in the optical microscope as undulatory extinction. Smaller subgrains are formed within larger ones with low angle boundaries of 1° or less. These cannot be detected individually in the optical microscope, but their presence may be indicated by the development of segmented undulatory extinction within the large sub-grains.

Various microstructural features, including subgrain development, are considered both theoretically and experimentally in chapter 9.

3.3 Other aggregate properties affecting the reaction

3.3.1 Influence of reactive particle size

Various researchers (Hobbs and Gutteridge 1979, Diamond and Thaulow 1974, Lagerblad and Trägårdh 1992, Jensen 1993) have shown that AAR is dependent on reactive particle size. The influence appears to differ between rapid- and slow/late alkali-reactive aggregates.

According to French (1994) the alkali-reaction is most damaging for certain rock types when the reactive rocks occur in a particular size range. It has been observed that for reactive aggregates, such as chert and volcanic glasses, the most damaging size range appears to be between 3 to 7 mm. However, for aggregate types such as recrystallised sandstones and meta-quartzites the most damaging size range appears to be between 10 to 20 mm. Greywackes and argillites have been found to be most damaging when they occur as coarse aggregate and damage has been observed in cases where the aggregate reaches several centimetres across (French 1994).

An experimental investigation was carried out by Nishibayashi and Yamura (1992) to clarify the effects of reactive fine aggregates on the expansion characteristic of concrete due to AAR. In Japan, only a few examples of deterioration due to AAR in structures containing reactive fine aggregate have been reported. By using accelerated test methods, it was observed that the expansion of concrete containing only reactive fine aggregate
increased rapidly to a constant level during the early period of the reaction. On the other hand, the expansion of concrete containing only reactive coarse aggregate initially increased slowly, but continued for a longer period.

Hobbs and Gutteridge (1979) tested mortar specimens containing reactive opaline rocks. They found that for a given reactive aggregate content, expansion increased as the particle size of the opaline rock decreased. It was also suggested that the reaction rate primarily was a function of particle volume and not particle surface. Also Diamond and Thaulow (1974) made similar observations when investigating the influence of the size of the alkali-reactive aggregate. They found that expansion occurred suddenly and was completed in a relatively short time for fine reactive particles, while with larger sizes the expansion was slower and much more gradual. Sprung (1975) found that the pessimum expansion also changed as the size of the aggregate changed. It has also been demonstrated that there is some kind of pessimum size of particles for rapid reactive aggregate. Experimental work showed the pessimum size of opal particles to be about 0.2 mm when testing expansion according to mortar-bar testing (Kawamura et al. 1983).

Lagerblad and Trägårdh (1992b) and Jensen (1993) have observed that for slowly alkali-reactive aggregates, certain aggregate size fractions in particular contribute more to the deleterious reaction than others. It was observed that finer slow/late alkali-reactive particles do not seem to take part in the reaction in real structures. As far as the author is aware of, no explanation has been found for these observations. From figure 3.5b it can be seen that slow/late alkali reaction occurs along discontinuous structures in the aggregate, such as grain boundaries and foliation. The ingress of alkalies and following reaction will especially be preferable where these discontinuous structures contain a high amount of finer grains and subgrains. To contain these discontinuous structures, particles have to be of a certain size. Particles finer than the width between parallel foliation or finer than original crystal sizes (monocrystalline) in the rock, will not contain any discontinuous structures. In such fine particles there will be no initial discontinuous structures which provide access to the alkalies.

### 3.3.2 The effect of grading

During the last four decades much attention has been drawn to the alkali-reactivity of different aggregate types. However, in spite of the voluminous research, the effect of grading upon the reactivity and expansivity of aggregates, have not been considered to any great extent. The grading may influence in different ways, the degree of alkali-reactivity for a concrete system containing alkali-reactive aggregate. The total amount of alkalies within the system will among other things be dependent on the excess of cement in the system, which is governed by the density of the aggregate particles, which in turn is governed by the grading curve. The relative amount of alkalies available to take part in the reaction will be governed by the available surface area of the aggregates.
The principle of maximum density means specifying a grading curve which gives a minimum of open space (voids) between the particles. This volume of voids will stand in relation to the necessary amount of cement to be added to fill this space. It has been found that aggregate graded to give a maximum density makes a harsh and somewhat unworkable mix. To improve the workability of the fresh concrete, an excess of cement above that required to fill the voids is added. This additional amount of cement will give an overfill, and an increasing spacing between the particles will start to occur. The effect of this overfill, will increases the amount of alkalies. The degree of the increase will vary according to the different type of grading, and consequently this will have an effect upon the alkali-reactivity of the concrete system. A grading curve for a typical structural concrete will give an open space of about 30%. Commonly, 33-35% of cement paste is added to the system, which gives an overfill of 3-5% of cement. Sometimes it is difficult to evaluate the effective level of alkali present because of its relationship to the grading curve (French 1994). When designing grading curves for dam concrete, extremely coarse particles will be used to avoid high heat generation and shrinkage due to the large volume of the concrete. If the whole concrete is considered, the alkali level might be found to be low, however, if the potentially reactive sites are considered, the alkali concentrations might have to be regarded as locally high.

Concerning the total amount of alkalies in a certain concrete system, the aggregate sizes and grading in smaller domains should be considered. To symbolise this, two theoretical types of concrete domains with exactly the same aggregate/cement ratio of 2.25:1 are showed in figure 3.16. Both of the concrete domains will have the same total amount of alkalies due to same volume of cement. The concrete with a finer grading curve has consequently a higher aggregate surface area/cement ratio than the coarser concrete to the right. An aggregate particle of a given size in the coarser graded concrete (right) will be surrounded by more cement paste and hence have available a higher amount of alkalies per unit surface area than a similar particle in the finer graded concrete.
3.3.3 Natural aggregates versus crushed aggregates

A distinction can be made between natural aggregates eroded to present size by natural agents and crushed aggregates obtained by a deliberated fragmentation of rocks. Both natural aggregates (glaciofluvial- and fluvial aggregates) and crushed aggregates (crushed bedrock or crushed oversized natural aggregates), or a combination of these types, are used in concrete. Both the physical factors (shape factors) and chemical factors might to a certain degree, differ between these types of aggregates. This section will introduce some possible differences affecting the alkali-reactivity of these distinctive types of aggregates.

Natural aggregate

Natural aggregates generally contain rounded particles due to modifications during transportation. Well-rounded particles may be expected to require less water or cement paste for equal workability than angular particles of equal sphericity and similar surface texture (Popovics 1979). This is particularly true for fine aggregates. On the other hand, experimental evidence indicates that for certain types of coarse aggregate, roughness increase the tensile and flexural strength of the concrete by improving the bond between the cement paste and the aggregate surface. Deposits of natural aggregates will be exposed to weathering by the infiltration of surface water. Natural weathering above the ground water level will leach out sodium, potassium, calcium and magnesium (Danielsen et al. 1984). In addition the weathering processes reduce the mechanical strength of the aggregate. It is also necessary to considered the possibility of enhancing amount of certain alkali-reactive rock types during transportation (chapter 5).
Crushed aggregate

Crushed aggregates will contain more angular particles, showing a higher roughness, which gives a relatively higher surface area than for natural aggregate of same size. This might favour the attack of alkalies. Lagerblad and Trägårdh (1992b) and Jensen (1993), pointed out the need for initial cracks to be present in reactive particles. During the crushing process the density of cracks will be enhanced. In addition to the original cracks in the parent rock, new cracks will be induced during the crushing processes. Thereby the density of cracks in natural aggregate will decrease during transportation due to the fragmentation of particles along existing cracks.

3.4 Critical amounts of slow/late alkali-reactive aggregates

The volume of rapidly alkali-reactive particles required to produce deleterious expansion needs only be very small. However, in the case of slow/late alkali-reactive aggregates the required amount of alkali-reactive rocks to produce deleterious expansion still is ambiguous. It has been suggested that no pessimum value exists for slow/late alkali-reactive rock types (Lagerblad and Trägård 1992b). The pessimal proportion of reactive particles might be 100%, however, even still unclear, this might vary for different types of slow/late alkali-reactive rocks.

The critical amount of slow/late alkali-reactive particles in natural aggregates has not been subject of much investigation. However in some cases proposals of critical amounts have been given. Magni et al. (1986) suggested a maximum of 15 percent of slow/late alkali-reactive rock types such as argillites, greywackes, quartz arenites and arkose to be allowed in concrete coarse aggregate. Field investigations in the region of Ontario, Canada, showed that if the quantity of potentially alkali-reactive rock types (argillite, siltstone, greywacke, sandstone/arkose) was more than about 30 percent of the coarse aggregate, structures usually showed cracking within about 15 years and sometimes within four years (Rogers, 1994). Rogers (1994) suggested that if the amount of alkali-reactive rock types was less than 15 percent, the aggregate may be used in concrete without further testing. If more than 15 percent of alkali-reactive rock types are present, it was proposed that the accelerated mortar bar test and the new version of the concrete prism test should be conducted before acceptance.

Bragg and Foster (1992) investigated relationship between petrographic examination and the results of the accelerated mortar bar test. A petrographic rating system was introduced based on the amount of known alkali-reactive rocks. Aggregates with greater than 15 percent, but less than 40 percent of known alkali-reactive rocks were rated as good, tending to reactive, while aggregates with greater than 40 percent of known alkali-reactive rocks were rated as highly reactive. The accuracy rate between petrographic rating and the accelerated mortar bar test was found to be 83 percent.
It is suggested by French (1994) that combination of potentially reactive materials sometimes leads to one type of lithology taking part in reaction while others do not. It is claimed that argillite tends to be reactive when combined with greywacke which might in other circumstances have proven innocuous.

In Norway further testing by the accelerated mortar bar test is recommended when the petrographic examination indicates a critical limit of ≥20 percent potentially- and alkali-reactive rock types in an aggregate. An aggregate containing <20 percent is accepted as innocuous, and no further testing is recommended (Norsk Betongforening 1991). This critical limit should be regarded as a preliminary criteria (Jensen 1993), however the Norwegian optional arrangement for declaration and approval of aggregates (DGB) has fixed this criteria until the end of 1996. The criteria is primarily based upon an investigation where different blending proportions of an innocuous aggregate and reactive rhyolite were tested. This rhyolite is assumed to be one of the most alkali-reactive rock types in Norway. By extrapolating the line of expansion for various blending proportion, the critical limit of 20 percent rhyolite corresponded with the critical mortar-bar expansion of 0.10% after 14 days (Berg 1991).

The results of further investigation of the critical limit is given in chapter 6, where it was found that this limit appeared sufficient in most cases. However, the study failed to show a linear relationship between the amount of potentially- and alkali-reactive rocks and the mortar bar expansion. In this study, the work in chapter 6 proposes the phenomenon of marginal quantity. However, later considerations give reason to believe that the relationship is explained by a logarithmic distribution.

Relatively high variations are observed when comparing the amounts of potentially- and alkali-reactive rocks obtained by petrographic examination, with the expansion values obtained by the accelerated mortar bar test. These variations could be due to the fact that only the potentially- and alkali-reactive particles in the aggregate are considered. Possible contribution of expansion from the innocuous particles is not considered. Mortar bar expansion of aggregate particles classified by the petrographic examination as innocuous rock types, such as fine grained granite, quartzites and gneisses, have shown expansion values close to 0.10% after 14 days (chapter 7).

When discussing the critical amount of slow/late alkali-reactive aggregates, two different approaches are possible. As mentioned above, the amount of potentially- and alkali-reactive particles are said to represent the reactive quantity in the aggregate. However, this estimation ignores the basic fact that even though a particle is classified as reactive, the volume of the whole particle should not be considered as a reactive constituent. Particles made of polycrystalline rocks will contain different minerals of varying crystal sizes, and different types of alkali-reactive rocks (e.g. ultramylonite vs. blastomylonite) might constitute different amounts of reactive components. As pointed out by various researchers (French 1992a, Deloye and Divet 1992, Furusawa et al. 1994), and considered in chapter 7, only a very small fraction of the rock, within 1% of the aggregate, takes part in the reaction. The volume of reactive constituents for
slow/late alkali-reactive aggregate will be the volume of quartz within a relatively small distance from the dislocation line, subgrain- or grain boundary. This is thus less than the total volume of quartz, and hence much less than the total volume of reactive particles. As far as the author knows, no attempts have been made to determine a quantitative value for this reactive constituent. However, by determining the quantitative value of the total grain boundary area of quartz, very promising results have been obtained which correlate closely with the expansion results (chapter 7).
References


Chapter 4

Predicting AAR - Test methods

There is a need for reliable test methods which can distinguish between innocuous and potentially alkali-reactive aggregates. Reliable and repeatable test methods will give opportunities to make better predictions of possible behaviour of aggregate in the concrete environment. Such test methods must be easy to carry out and give reliable results in a short time, and it is essential that the results obtained reflect actual field performance.

4.1 Petrographic examination

The petrographic examination of concrete and concrete aggregates has become a well-established procedure and is routinely used in the design and construction of concrete structures, in failure analysis and forensic studies in the prediction of service life under various environments, and in research investigations to advance knowledge in concrete technology. The petrographer examines the nature of the materials and observes the relationships of texture, structure, composition, and alterations to its properties. By seeing the relationships, the petrographer fits the pieces of the puzzle together to form a picture and explain complex phenomena (DePuy 1990).

Petrographic examination of aggregates for structural application can, as mentioned above, serve a number of purposes. This section however will emphasise the use of petrographic examination as a preliminary examination of aggregate for concrete purposes, as a screening tool in the assessment of alkali-reactivity. Petrographic examination alone cannot provide information on the expansiveness of a particular cement-aggregate combination, however, experienced petrographers can predict the likely behaviour of aggregates with which they are familiar (Grattan-Bellew 1989).

Petrographic examination is an essential first step in evaluating the potential alkali-reactivity of an aggregate, partly because this may obviate the need for further testing and partly because knowledge of aggregate composition will facilitate interpretation of subsequent test results. Where certain minerals, rock types or specific microstructural features are known to cause deterioration of concrete, recognition of these constituents
in an aggregate, by petrographic examination, may be sufficient evidence to reject the aggregate.

However, difficulties exist in the classifications employed in normal geological usage because the aggregate samples often are removed from their field association. Hence they must be named specifically with respect to their lithology and texture (French 1991). Ambiguities also arise in the use of rock names which may in practice convey little meaning for the engineer. Full description of the rocks is required, rather than the simple use of standard terminology. The difficulties of nomenclature also mean that it is generally unsatisfactory to make lists of rocks that are either innocuous, potentially alkali-reactive or known alkali-reactive.

The procedures for the petrographic examination of aggregates for concrete purposes are given in national codes, such as ASTM C295 (ASTM 1985) and BS 812, draft Appendix C (BSI 1987).

The ASTM C295 says that the petrographic examination should identify and call attention to potentially alkali-silica reactive and alkali-carbonate reactive constituents, determine such constituents quantitatively, and recommend additional tests to confirm or refute the presence in significant amounts of aggregate constituents capable of alkali reaction in concrete. The procedure presents a list of slow/late alkali-reactive rock types and alkali-reactive constituents found in aggregates. These petrographic procedures include also the additional use of X-ray diffraction, electron microprobe, SEM and thermoanalytical techniques.

However, the different procedures are primarily based on the recognition of relatively minor constituents or contamination of rapid alkali-reactive rock types which may be non-uniformly distributed within the aggregate. Despite the lists of potentially slow/late alkali-reactive rock types, non of the existing procedures include ways to recognise or quantitatively determine the possible microstructural features of slow/late alkali reactive rock types. Hence, at the present stage the reliability of the petrographic examination according to standard procedures is likely to be poor and ancillary testing is generally needed to confirm the reactivity. Consequently, more research is needed to develop accurate petrographic procedures for proper determination and screening of potentially slow/late alkali-reactive aggregates.

4.1.1 The petrographic examination in Norway

The awareness of slow/late alkali-reactive aggregates as a cause of degradation of Norwegian concrete structures is rather new. The establishment of a new petrographic examination was essential because of the frequent occurrence of high amounts of potentially slow/late alkali-reactive rock types in Norwegian natural aggregates. Contamination or minor amounts of rapid alkali-reactive constituents are not regarded as a problem in Norway. The widely distributed occurrence of various types of
slow/late alkali-reactive rock types in natural aggregates, requires a full description, including quantitative measurements of all the rock types, rather than the simple use of standard terminology. At present the petrographic examination in Norway is mainly based upon qualitative assessments and the recognition of known alkali-reactive aggregates. However, microstructural features are assessed on a semi-quantitative basis. It is therefore recommended that the petrographic examination has to be performed by geologists familiar to Norwegian alkali-reactive rock types. On the basis of the petrographic examination, the aggregates are divided into three main categories, with totally 12 different rock assemblages (Lindgård et al. 1993). These rock assemblages are presented in the papers in chapters 5 and 6.

The purpose of the petrographic examination is to identify, quantify and group different rock types in the aggregate. The classification and identification of the different alkali-reactive rock types are based on; not only knowledge of field performance and petrographic nomenclature, but also on semi-quantitative assessment of the mineralogy and microstructures. Instead of characterising the tested aggregate by name only, which often is very difficult in small grains, the microstructural features are used to evaluate the aggregate, and an assessment is made on the amount of potentially reactive components. The microstructural assessment includes evaluation of grain-size of quartz, subgrain development in quartz, degree of deformation and recrystallisation.

As mentioned earlier, aggregates for concrete purposes are now tested on a voluntary basis according to an optional arrangement for declaration and approval in the procedures given by the Norwegian Concrete Society, Publication NB, No.19 (Norsk Betongforening 1991). The procedures include the use of the new petrographic method as a first step. If results from the petrographic examination indicates <20 percent alkali-reactive rock types in an aggregate further testing is not required.

The petrographic examination in Norway was primarily based on thin-section examination of aggregate particles in the fraction 4-8 mm. The aggregate was first investigated by visual observation using a binocular microscope, to separate the particles into different groups. A few representative particles from each group were selected and prepared for more detailed identification by thin-sections. The amount of alkali-reactive rock types was given as percentages of the number of grains counted in the binocular microscope. Due to the high metamorphic grade and degree of deformation of certain Norwegian rocks, the identification and classification of various rock types were very complex. Occasionally the thin-section examination made differences between particles from the same group, difficult to identify. Sometimes one particle could be classified as reactive and other particles from the same group as innocuous. In these cases the evaluation of the reactivity was uncertain.

Therefore a modified method was developed, based on point-counting in thin-sections under the petrographic microscope. After sieving the sample, two fractions (1-2 mm and 2-4 mm) are selected for further petrographic examination. The
aggregates are embedded in an epoxy resin, which allows the preparation of thin sections for petrographic examination. Two thin-sections (25 x 35 mm) are made with particles in the fraction 2-4 mm and one in the fraction 1-2 mm. Approximately 1000 points are counted in each fraction. The content of alkali-reactive rock types in an aggregate is obtained by calculating an average of the results from both fractions. The amount is given as volume percentage. A description of the method is given by Haugen and Jensen (1993) and Lindgård et al. (1993).

In order to compare the effectiveness of the primary petrographic method with the current new method, the classification of some aggregates investigated by both methods was included in this study. As evident from figure 4.1, the primary method in most cases gave a higher estimate of the amount of potentially- and alkali-reactive rocks compared with the current new petrographic method. To investigate the accuracy of the current petrographic method, 13 aggregate samples containing various amounts of potentially- and alkali-reactive particles, were investigated by two independent operators. Both operators had a similar experience of recognising Norwegian alkali-reactive rock types. As evident from figure 4.2, all the results from operator II are on average 6 percent higher for potentially- and alkali-reactive aggregates than the results from operator I, except for one.

![Figure 4.1](image-url)  
**Figure 4.1** Comparison of results from the current used new petrographic method and the primary petrographic method.
A third method of petrographic examination was developed in this study to quantify microstructural properties of certain cataclastic rocks (chapter 9). Besides a general petrographic examination, quartz grain sizes, including sub-grains, were measured for each rock type by point-counting in thin-section. By using an automatic point counter (Swift-model F), quartz grains along lines in the thin-section were measured. Approximately 200 points for each thin-section were counted. For foliated rocks with elongated quartz grains, the direction of point counting was 45° to the parallel foliation of the rock in order to obtain an average grain size diameter. As a measurement of the grain size, the length of a quartz grain lying at the line was measured. For determination of the mean grain size of quartz, the $d_{50}$ was used.

In order to make a simple estimation of the grain boundary area of quartz, each quartz grain, including sub-grains, was assumed to be cubical in shape. Certain selected grain sizes (fig. 4.3) were used to calculate the grain boundary area for specific parts of the quartz grain size grading. The area obtained was multiplied by the proportion of quartz grains within each specific part of the grading, and all areas added together. By multiplying the grain boundary area by the amount of quartz in the rock type, as determined by XRD-analyses, an estimate for the total grain boundary area of quartz (m²/cm³) in each sample was obtained.
4.2 Accelerated mortar bar testing

In recent years attempts have been made world-wide to improve the quantitative test methods in order to enable better prediction of the potential expansivity of aggregates for concrete purposes. The need for quick and reliable test methods has been evident for some time. Currently several methods are being employed for the screening of aggregates for potential alkali reactivity. Most of these methods have disadvantages of one sort or another. Test methods such as the ASTM C-227 Mortar Bar Method (ASTM 1986a) and the Canadian CSA A23.2-14A, Concrete Prism Test (CSA 1994) which have traditionally been used to evaluate the potential alkali-reactivity of aggregates take too long to complete. The ASTM C-289 Chemical Test (ASTM 1986b) which is a rapid method, is not suitable for use with all types of aggregates and furthermore it does not give an estimate of the potential expansion of the aggregate.

A critical review of the use and precision of the ASTM C-227 Mortar bar test, and the ASTM C-289 Chemical Test is given in chapter 8.

Figure 4.3 Example of grading of quartz grain size obtained by petrographic examination in thin-section. Average quartz grain size and proportion of quartz grains between certain main fractions are indicated.
4.2.1 The development and use of accelerated mortar bar tests

Numerous accelerated mortar bar tests have been developed, most of which centre on the procedure introduced by Oberholster and Davies (1986) known as the NBRI Mortar Bar Test. The procedures are based on storage of mortar bars in 1M NaOH at 80°C. In spite of the fact that these methods have shown some limitations and disadvantages, there is a general agreement that such accelerated tests provide qualified classification of aggregates. The method is used to evaluate the quantitative expansivity for both rapid and slow alkali-reactive aggregates. The accelerated mortar-bar tests have the advantage of being rapid compared to other performance tests for alkali-aggregate reactivity. The disadvantages are that such tests are not suitable for evaluation of different cement-aggregate combinations due to insensitivity to cement alkali content. Both the heat and the NaOH-solution, which is rich in alkalies, accelerate the reaction, and the tests have been found to give useful results on 14 days in comparison with one year taken by the conventional ASTM C-227 Mortar bar test. Despite the fact that such accelerated tests are relatively new, the test procedure has been evaluated and investigated in many countries since the NBRI mortar bar test was first introduced. According to a survey published by Nixon and Sims (1992), accelerated tests based upon the NBRI mortar bar test are now in use on a regular basis in Australia, Belgium, Canada, Norway, South Africa and USA. The accelerated mortar bar tests have now been adopted as the regular standards; ASTM C-1260-94 (ASTM 1994) and CSA A23.2-94 (CSA 1994), and is introduced as a voluntary arrangement for the declaration and approval of aggregate in Norway (Norsk Betongforening 1991).

The precision and variation of the results obtained by the accelerated test procedures are discussed in chapter 8. The effect of different variables including the size of the mortar bars, the type of measurement points employed, the precision of the aggregate grading, the ratio of the volume of alkali to the surface area of the mortar, and the cement type employed are also investigated. This chapter also gives a review of different proposed expansion limits applied in order to distinguish between innocuous and alkali-reactive aggregates, while introducing the probability of an aggregate being alkali-reactive in service, if it exhibits a particular level of expansion in the accelerated test.

4.2.2 Norwegian use of accelerated mortar bar testing

In Norway several quantitative test methods were assessed at SINTEF - Structures and Concrete with different types of Norwegian aggregates with known field performance. A modified version of the NBRI mortar bar test was found suitable for distinguishing between alkali-reactive and innocuous aggregates (Jensen 1993). This accelerated mortar bar method is now included in the procedures of the optional arrangement for declaration and approval of aggregates for concrete purposes. (Norsk Betongforening 1991). The test is used to investigate natural aggregates, crushed stone
and blends of aggregates. Testing of blends of aggregates is often realistic in cases where utilisation of maximum amounts of potential alkali-reactive aggregates is advantageous for economical or other reasons (Dahl et al. 1992). The expansion is monitored by measuring length change of the mortar-bars (40x40x160mm) after 4, 7, 12, 14, 28 and 56 days respectively. More recently the test has also been carried out on a commercial basis by other institutions in Norway.

4.2.3 Effects of various exposure conditions, and other factors affecting expansion of mortar bars

The following section contains a review of some additional variables which could potentially affect the test results in the accelerated mortar bar test.

The water/cement ratio

Changing the w/c ratio in mortar bars affects not only the alkali concentration of the pore solution but also the physical properties of the mortar. These changes effect the expansion of mortar bars made with alkali reactive aggregates. It is suggested by Grattan-Bellew (1989) that the optimum w/c ratio for expansion of mortar bars containing alkali-reactive aggregates is probably in the range of 0.4 to 0.6. The exact value depends on the physical and chemical properties of the aggregates. Several workers observed that expansion of mortar bars generally increased with increasing water/cement ratio (Davies and Oberholster 1987). Fournier and Bèrubè (1991) made mortar-bars with different w/c-ratio and reported that the expansion generally increased with increasing w/c, but decreased when the w/c ratio exceeded 0.50. For this reason it is recommended by Bèrubè et al. (1992) to use a fixed w/c ratio of 0.50 for coarse aggregates and manufactured sands, and 0.44 for uncrushed sands. These criteria are now incorporated into the Canadian standard (CSA 1994) and the ASTM standard (ASTM 1994).

Investigation in this study on the effect of different w/c ratio (w/c 0.45 and 0.50) in mortar bars made with innocuous aggregates (figure 4.4) shows that an increased w/c ratio gives an increased effect with time.
The particle size

The effect of particle size on the expansion of mortar bars containing opal shows a pessimum particle size of about 0.2 mm (Kawamura et al. 1983). According to Grattan-Bellew (1989), no such effect has yet been documented with slowly expanding aggregates.

In Norway, generally the coarser aggregates (>1mm) have been observed to react in concrete structures. However, in the laboratory, the Norwegian modified NBRI mortar-bar test, shows that all particle sizes seem to take part in the reaction (Jensen 1993). Lagerblad and Trägårdh (1992) reported similar experience with slowly Swedish alkali-reactive aggregates. Based on a Danish accelerated expansion test (NT Build 295), the smaller particle sizes were observed to cause more expansion than the coarser. This contradicts what they found in reacted concrete from real structures.

In a study carried out in Canada with slowly/late alkali-reactive aggregates from Nova Scotia, results from the ASTM C227 mortar bar test showed the expansion rate to increase with decrease in average particle size of alkali-expansive rocks (Duncan et al. 1973).

Similar results were obtained in an investigation carried out in Norway by Mørtsell and Wigum (1992). An innocuous crushed aggregate was substituted with 100% reactive material in the lower part (<0,8mm) of the required grading curve. For

---

**Figure 4.4** Effect of different water-cement ratio for the accelerated mortar bar expansion for the innocuous rock types gneiss and granite.
another sample the upper part of the curve (>0.8mm) was substituted with 100% reactive material. Expansion tests were carried out for both these two blends of aggregates according to the Norwegian modified NBRI mortar-bar test. As evident from fig. 4.5, mortar-bars containing reactive materials in the lower part of the grading curve exhibit higher expansion than the mortar-bars containing reactive material in the upper part. In addition, mortar-bars containing aggregate substituted by 50% reactive materials in all the grading fractions were tested. These mortar-bars show a lower expansion than the previous two. However all these effects only appeared after 14 days of exposure in NaOH solution.

![Figure 4.5](image)

Figure 4.5 Expansion results from the Norwegian Modified NBRI mortar-bar test with reactive materials substituted in different fractions in an innocuous aggregate.

This study also included the comparison of the mortar bar expansion of a set of mortar-bars with the expansion of concrete-bar containing the same greywacke aggregate (Chapter 7). It was shown that the mortar-bars exhibited higher expansion rates only after the first 12 days of exposure. After this period the concrete bar showed rapid expansion rates, and overall exhibited a higher expansion than the mortar-bars after approximately 18 days.
The NaOH concentration

It has been reported by Davies and Oberholster (1987) that fluctuations in the concentration of the NaOH solution could affect the precision of the accelerated mortar bar test. In most cases the 1N NaOH solution exhibited the greatest expansion. The mortar bar expansion with 0.5N and 1.5N NaOH fell below the minimum expansion measured for the 1N NaOH solution. Fournier and Bèrubè (1991) investigated the effect of the alkali concentration of the storage solution by testing similar sets of mortar-bars in NaOH solutions of different concentration. It was suggested that the composition or the nature of the cement may influence the test results when NaOH solutions of moderate concentration are used.

It is shown in chapter 8 that the amount of expansion increases progressively as the ratio of the volume of alkali to the surface areas of the aggregate increases.

The temperature.

Davies and Oberholster (1987) carried out the NBRI mortar bar test by exposure of the mortar bars at different temperatures. In this work the mean expansion at 80°C was higher than at 70°C and 90°C.
References


Mørtsell, E. and Wigum, B.J. 1992: *Praktiske bruksegenskaper av tilslag i betong - iblanding av 0-4 mm knust sand i siktet natursand*. (In Norwegian). Dr.ing. student rapport, University of Trondheim. 11 pp.


Chapter 5

Petrographic examination of Norwegian glacifluvial aggregates: interpretations of mechanisms leading to high contents of Cataclastic rocks

BØRGE JOHANNES WIGUM & VIGGO JENSEN


*B.J. Wigum, Department of Geology and Mineral Resources Engineering, University of Trondheim, The Norwegian Institute of Technology, 7034 Trondheim, Norway*

*V. Jensen, SINTEF, Structure and Concrete, 7034 Trondheim, Norway*
Petrographic examination of Norwegian glaciofluvial aggregates: interpretation of mechanisms leading to high contents of Cataclastic rocks

BØRGE JOHANNES WIGUM & VIGGO JENSEN


Norwegian glaciofluvial aggregates have been examined microscopically and classified according to a newly established petrographic method. The results of this study and previous data show that there are high amounts of particles of cataclastic rocks in many Norwegian glaciofluvial aggregates. These rocks are unsuitable because of the expansive effects of such rock-types in concrete, due to alkali-aggregate reactions (AAR). Some interpretations and reasons are put forward to account for the more extensive occurrence of cataclastic rocks in certain areas. Regional examination of glaciofluvial aggregates shows that cataclastic rocks can resist erosion over relatively long transport distances, and that particles within specific size ranges show higher enrichment in these rock-types. It is recognised that the concentration of cataclastic rocks within particular particle fractions, is governed by the provenance, distance from the source rock, and the mechanical properties of specific types of cataclastic rocks.

Børge Johannes Wigum, Department of Geology and Mineral Resources Engineering, University of Trondheim, The Norwegian Institute of Technology, 7034 Trondheim, Norway. Present address: Kontrollrådet for betongprodukter, P.O.box 53 Blindern, N-0313 Oslo, Norway

Viggo Jensen, SINTEF, Structures and Concrete, 7034 Trondheim, Norway.

Introduction

Research into the lithology and transport processes of glaciofluvially derived materials has a long tradition, mainly as a tool for mineral exploration. Most classifications of sand and gravel fractions have usually been done by sieve analysis and binocular microscope. The most commonly used parameters in modern classification and description of glacial deposits are grain size and shape. Pettijohn et al. (1973) pointed out the need for more thin-section studies of sand, by which varieties of lithic fragments can be identified.
Norwegian glaciofluvial sand and gravel deposits have for some years been assessed and classified for their volume and quality, and recorded in computerised databases at the Geological Survey of Norway (Neeb 1993). However, over the last few years there has been a growing awareness of the importance to study the petrographic and microstructural composition of natural aggregates, mainly glaciofluvial materials used for concrete purposes. This has become necessary in order to meet the more stringent control for detecting aggregate which could exhibit slow/late-expansive alkali-aggregate reactions, which during the last few years has been recognised as a concrete durability problem in Norway (Jensen 1990, 1993, Jensen & Danielsen 1992, 1993, Dahl et al. 1992, Lindgård et al. 1993, Meland et al. 1994).

In concrete, alkali-aggregate reaction is a chemical reaction between sodium and potassium ions in the pore solution and certain types of aggregates. Such types of alkali reactive aggregates contain siliceous components, particularly in the form of microcrystalline and ductile deformed quartz. The reaction forms a hygroscopic alkali-silica gel that can imbibe water and swell. The swelling forces generated may be sufficient to disrupt the surrounding concrete, causing expansion and associated deterioration. In 1992 an optional arrangement for declaration and approval of aggregates for concrete was introduced in Norway (DGB - Deklarasjon-og Godkjenningssporing for Betongtilkjøring). It suggests that aggregates should be tested in accordance with the procedures outlined by the Norwegian Concrete Society, publication NB 19 (Norsk Betongforenings Publikasjon Nr.19, 1991). The procedures recommend that the first step should involve testing by petrographic examination of the aggregate. If a low content of reactive or potentially reactive rock-types (<20%) is observed, the aggregate is classified as innocuous with respect to its alkali-aggregate reactivity. If, however, a high quantity of reactive rock-types (≥20%) is present, the aggregate is classified as reactive. In addition, it is recommended that the aggregate is tested by an accelerated mortar bar test to confirm the reactivity of the aggregate before it is used in concrete structures. Reactive aggregates are not recommended to be used in concrete structures situated in humid environments unless precautions are taken regarding cement type, protection, etc.

At SINTEF Structures and Concrete an improved petrographic method for thin-sections has been developed which has been used successfully to recognise more accurately reactive aggregates (Jensen 1993). This technique has been used to examine a number of glaciofluvial aggregates in Norway. As a result a more accurate picture has emerged with regard to the petrographic and microstructural composition of Norwegian glaciofluvial aggregates. An important feature of the method is that it is able to recognise and classify microstructural features of quartz-bearing rocks. These microstructural features cannot be recognised by ordinary binocular microscope examination. Investigations of a large number of samples from glaciofluvial deposits in Norway have revealed the occurrence of cataclastic rocks in a majority of the samples. Petrographic examination of aggregate from concrete samples obtained from
structures suffering from AAR, have also shown a high content of cataclastic rocks. Such rock-types are now considered as the commonest and most widely distributed source of alkali reactive aggregates in Norway (Jensen 1993).

In Norway, due to the intense thrusting and faulting, cataclastic rocks are widely present and therefore should be expected to occur in many glaciofluval deposits. During the comminution and transportation of glaciofluval materials, more fragile materials abrade more rapidly, leading to an enrichment (or maintenance of a high level) of quartz-bearing rocks exhibiting high abrasion resistance, in certain fractions in the deposits. The effect will be more marked for longer transport distances.

The aim of this work was to examine the relative occurrence and distribution of cataclastic rocks in Norwegian glaciofluval materials and to assess various mechanisms and processes which could account for the high occurrence of cataclastic rocks. The provenance, comminution and transportation conditions of glaciofluval deposits were also taken into account when interpreting the results. Together with the results from SINTEF Structures and Concrete, two further areas were selected for investigation in order to obtain a more detailed picture.

**Classification and properties of cataclastic rocks**

In order to understand the mechanism which led to enrichment of cataclastic rocks in glaciofluval deposits it is essential to be familiar with the classifications and properties of such rock-types. All rocks formed by cataclasis are termed cataclastic rocks and are generally felsic and/or silicic in composition. Cataclastic rocks include metamorphic rocks that are deformed at low temperature with primary cohesion due to a combination of crystalloblastic and cataclastic processes. Higgins (1971) has classified cataclastic rocks with primary cohesion into two main categories, depending on whether cataclasis is dominant over neomineralisation-recrystallisation in their formation, or vice versa. Further classification is based on the occurrence of fluxion structures. Cataclasites are formed under conditions of brittle deformation of the rock, showing random fabric, while various types of mylonites are formed during ductile deformation (flow) of the rock, showing fluxion structures. Fluxion is a synonym for flow, and the term reflects the occurrence of the comminuted matrix of mylonites flowing around the porphyroclasts in layers separated by thin lines marked by concentrations of fine micaceous minerals. Finally, rocks without fluxion structure and cataclasism dominance are defined as microbreccia and cataclasite, while rocks with fluxion structure and cataclasism dominance are defined as protomylonite, mylonite and ultramylonite. Rocks with fluxion structure where neomineralisation is dominant over cataclasis are defined as mylonite gneiss and blastomylonite.
The physical properties of the cataclastic rock are governed by its microstructural features, in particular the state of the quartz. Brattli (1994) investigated the influence of cataclasis on abrasion resistance of granitic rocks. He observed that some types of ductile deformed cataclastic rocks, such as protomylonite, mylonite and ultramylonite, appeared to have extremely high abrasion resistance, while at the same time exhibiting a high brittleness. This was attributed to intense ductile deformation which occurs under relatively low temperatures. Under these conditions, high concentrations of very tightly bounded dislocations are produced in the quartz grains, causing hardening of the minerals, equivalent to cold-working in metals.

**Distribution of cataclastic rocks in southern Norway**

Cataclastic rocks generally occur in thrust and fault zones resulting from dynamic metamorphism. Fig. 1 shows the outline of mapped cataclastic rocks (mylonite) and major fault and thrust zones in southern Norway where cataclastic rocks may or may not occur. The map has been drawn from the 1:1 mill. bedrock map of Norway (Sigmond et al. 1984). Two larger areas with mapped cataclastic rocks (mylonite) occur in the south-eastern part of Norway. These are the Precambrian Mjøsa-Vänern mylonite zone which can be followed into Sweden (to lake Vänern); and further south, the mylonite zone from Øyern to the Swedish border (Oftedahl 1980). According to Oftedahl (1980) the mylonite zones were caused by a series of microcontinental collisions in the Precambrian.

Fault zones occur in many areas of southern Norway, e.g. the southern and southeastern Precambrian regions, the Oslo Region and in the areas of the Caledonian nappes. Thrust zones are also prevalent in many areas, and reflect the extent of the Caledonian overthrusted rocks as a result of nappe movements. The map in Fig. 1 shows that cataclastic rocks are widely distributed in South Norway and should therefore be expected to occur in many glaciofluvial deposits in these areas.
Fig. 1. The outline of mapped cataclastic rocks (mylonite) and major fault and thrust zones in southern Norway (Jensen 1993).
The provenance, comminution, transportation and deposition of glaciofluvial materials

To understand the end product of a glaciofluvial process it is necessary to look at the sedimentary cycle starting with the parent rock at the basal traction zone of glaciers, through transportation in the aqueous environment, to the eventual sedimentary deposit. The origin of glaciofluvial materials is either the bedrock, till or englacial debris. The glaciofluvial materials could be defined as the net result of; *plucking and abrasion* of lithic fragments in the glacial environment, and modification during *recycling in aqueous environments* (Slatt & Eyles 1981). These two factors will ultimately influence the final petrographic composition of the glaciofluvial materials. To understand the environmental influence upon the potential enrichment of cataclastic rocks in such materials, these two processes will be discussed further.

*Plucking and abrasion of lithic fragments in the glacial environment*

In the basal traction zone of glaciers, coarser clasts and sand-size lithic fragments are detached from underlying bedrock surfaces by plucking, abrasion and crushing due to shear stresses exerted by the overriding ice. The physical properties of the rocks and minerals have an influence on their resistance to fracturing. Shear fractures propagate along intracrystal, as well as intercrystal, planes of weakness (Slatt & Eyles 1981). As a result of abrasion, materials beneath the glacier will rapidly be crushed into fine-grained sediments, while plucking might incorporate the loosened bedrock material into the sole of the glacier and then be transported within the glacier. Cataclastic rocks that exhibit very fine quartz grain-sizes, or microstructural features including zones of undulatory extinction, planes of bubble wall inclusions, sub-grain boundaries and water-weakened dislocations, might favour shear fractures along these planes of weakness. On a macroscopic scale, plucking might also exploit pre-existing joints whereby large joint-bounded blocks may be pulled away from the bedrock and be incorporated into the glacier (Boulton 1979).

*Modification during transportation in aqueous environments*

As the material enters the glaciofluvial system it becomes involved in a process of reworking. This reworking is influenced by many factors during subglacial meltwater transportation such as the viscosity of the water which can be high when temperatures are very low. In addition, when there is a combination of heavy load and high velocity flow, then the meltwater can exert an extremely high abrasive action. At lower velocities abrasion is the most important mechanism causing erosion, while cavitations are important at higher velocities. Another important mechanism acting during transport is abrasion due to impinging suspended particles during flow
(Lilliesköld 1990). Also during glaciofluvial transportation, lithic fragments are subjected to impact-loading, which induces tensile stresses, and which in turn cause extensional fractures to propagate preferentially along intercrystal boundaries (Slatt & Eyles 1981). Harrel & Blatt (1978) found very little size reduction of polycrystalline quartz granules (2-4 mm) during tumbling experiments. They concluded that mechanical durability was inversely proportional to the size of the crystal or grain in an aggregate. Therefore, in a finely polycrystalline particle, the crack path will cross more grain boundaries and grains of different crystallographic orientation. As a result the rate of energy dissipation increases, which in turn leads to a greater hindrance of the crack propagation. This type of behaviour is characteristic of cataclastic rocks as they commonly exhibit very fine grain sizes.

Haldorsen (1982) observed that quartz grains, because of their great mechanical resistance, generally erode to form particles of coarser size fractions than compared for instance to feldspars which have a much lower mechanical resistance. Glaciofluvial materials which originated by erosion of tills were investigated. It was found that the glaciofluvial materials had a sand fraction significantly richer in quartz than the original tills. Results from a grinding test were applied to explain the enrichment of quartz in the sand fraction. It was claimed that glacial transport involves both abrasion and crushing, whereas the glaciofluvial transportation is dominated by abrasion. During abrasion mainly silt is formed. The silt is enriched in feldspar and sheet silicates, and the remaining sand in quartz.

**Transportation distance**

It is generally agreed that over long transport distances the volume fraction of various grain-size classes of glacial materials is affected by their differential resistance to glacial abrasion. The transport distance is generally greater in glaciofluvial material than in the till from which it is delivered, and thus the source area is more difficult to assess. Further complications are introduced as a result of sorting by water and clast weight. The transport distance might range from kilometres to tens of kilometres, according to the energy level of the glacial meltwater system, the grain size and the resistance of the rocks (Lilliesköld 1990). Most pebbles in glaciofluvial deposits are not particularly far travelled, which explains their relatively poor degree of rounding. In southwest Wales, it has been found that most of the rock-types represented in glaciofluvial deposits are of strictly local origin. There is seldom more than 5% of exotic pebbles which have travelled more than 5 km from their source (Sugden & John 1985). Lee (1965) found that most pebbles in a Canadian esker had travelled less than 10 km from their source, whereas sand and gravel particles had travelled much further.
Deposition

The mode of deposition will control the lithology, the stratigraphy and the facies assemblages. The lithological variation in different beds usually reflects the grain-size distribution (Lilliesköld 1990). It has been reported that subglacial glaciofluvial deposits in eskers have commonly followed zones of structural discontinuity in the bedrock, such as faults (Shilts 1984).

Petrographic method

Most Norwegian alkali reactive aggregates are very fine grained (microcrystalline); therefore identification and classification of aggregate grains cannot be made accurately without the use of thin-section microscopy. In order to obtain more realistic classifications of aggregate for use in concrete, an improved petrographic examination which involves point counting has therefore been developed by SINTEF Structures and Concrete. This method has been used to assess rock constituents in glaciofluvial sands in the present investigation. The preparation of samples of sand for this test is as follows: After sieving, two representative samples of the fractions 1-2 mm and 2-4 mm, are selected for further petrographic examination. The samples are then impregnated with an epoxy resin, in order to prepare thin-sections for petrographic examination. Two thin-sections (25 x 50 mm) are made with particles from the fraction 2-4 mm and one thin-section with particles from the fraction 1-2 mm. Approximately 1000 points are counted in each fraction. The volume percentage of reactive rock-types is based on the average of the results from both fractions. A more detailed description of the method is given by Haugen & Jensen (1993) and Lindgård et al. (1993). The classification and identification of the different alkali reactive rock-types are based on; knowledge of past field performance, petrographic nomenclature and, mineralogical and microstructural criteria (Jensen 1993). During microstructural examination the following factors were taken into account; grain-size of quartz, subgrain development in quartz, degree of deformation and recrystallisation. In order to obtain reliable data it is recommended that the petrographic examination is carried out by a geologist who is experienced in identifying reactive rock-types prevailing in that particular country (Lindgård et al. 1993).
In the present work the rocks were classified into the following three main categories in order to simplify interpretation of results and for use in data processing:

**Category 1. - Reactive aggregates** (with known reactive field performance):
- sandstones (1), cataclastic rocks (2), acid volcanic rocks (3), argillaceous rocks (4), greywacke (5) and other rock-types with microcrystalline quartz (6).

**Category 2. - Potentially reactive aggregates:** Quartzite (fine grained*) (7), Other rock-types containing finely divided* quartz (8).
  *(crystal sizes 0.06-0.13 mm).

**Category 3. - Innocuous aggregates:** Rock-types with coarse grains and/or minor amounts of quartz, e.g. volcanic rocks/gabbro (9), granites/gneisses (10), mafic rocks/pure limestone (11) and other rocks (12)

In addition, results from petrographic analyses carried out by SINTEF Structures and Concrete were used to determine the distribution and content of cataclastic rocks obtained from different sources.

**Investigated areas**

In addition to the results from work by SINTEF Structure and Concrete, we selected two further areas for this study. This ensured data on aggregates sourced from close to and remote from the original parent cataclastic rocks. The work also attempted to compare samples on a regional and local basis, and to compare the difference between different types of cataclastic rocks. In the first investigated area which is part of the southeastern Precambrian province, two major mylonite zones are included, and from the second area, on the Fosen Peninsula, smaller fault zones containing cataclasites are included.

**The southeastern Precambrian province**

The southeastern Precambrian area lies between the Permo-Carboniferous Oslo Paleorift and the Oslo fjord to the west, and the Swedish border to the east. There are two major mylonite zones located in the area (Fig. 5). The northernmost zone is the Mjøsa-Vänern mylonite zone, which lies south of the Solor gneisses and the strongly deformed Odal granites. Further south in this region lies the second mylonite zone,
which separates the Romerike grey gneisses (mostly metatonalites) from the Østfold grey gneisses in the south (Oftedahl 1980).

The deglaciation and the glacial deposits in this Precambrian area have been reported by Sørensen (1979, 1983). The Ra moraine in the outer Oslofjord area was formed during the Early Younger Dryas, whereas the second most prominent ice-marginal deposit in the region, the Ski Moraine, was formed at the end of the Younger Dryas. Both the glacial striae older than the Younger Dryas and the glacial striae formed during the Younger Dryas indicate a glacial movement towards the south-southwest in the region.

A total of nine samples containing glaciofluvial aggregates were collected from six different locations within the area. The locations nos. 1 and 2 lie just south and downstream of the Mjøsa-Vänern mylonite zone, with location 3 lying between the mylonite zones, while location 4 was situated along the southern mylonite zone. Locality 5 is situated south of this mylonite zone, at the southern end of lake Øyern. The southernmost location was at the prominent ice-marginal deposit at Mona, which is part of the Ski-Ås moraine complex. At this location four samples were collected (nos. 6 to 9) in order to investigate lithological variations between different layers in the deposit. At the distal part of the ridge, in an approximately 1 m-thick part of the deposit, samples were collected from three different layers; no.6 was collected from a coarse upper layer, no.7 from a 15 cm-thick layer of fine material, while sample no.8 was collected from a medium-coarse layer. Additionally, one sample (no.9) was collected from a coarse layer deep in the middle of the ridge.

Verrabotnen - the Verran Fault system

The valley of Skaudalen runs ENE-WSW on the Fosen Peninsula (Fig. 6). The valley was developed along the Verran Fault, forming a topographic lineament running from Rissa to Verrasundet. The fault system near Verrabotnen displays a variety of fault rocks produced by both brittle and ductile deformation (Grønlie et al. 1991). The glacial system has produced an ice-marginal glaciofluvial deposit, Younger Dryas in age, in the valley just west of Verrabotnen (Reite 1994). In most valleys and fjords in this area, glacial striae indicate an ice movement strongly dependent on topographical conditions (Reite 1994). It is therefore believed that the ice moved along Verrabotnen and down Skaudalen to the southwest. A total of three aggregate samples were collected at the ice-marginal glaciofluvial deposit, along the valley profile west of Verrabotnen.
Fig. 2. Cataclastic rocks in glaciofluvial materials in 88 aggregate samples from locations in southern Norway; based on petrographic examination at SINTEF Structures and Concrete 1991-1995.

Fig. 3. Relationship between cataclastic rocks and alkali reactive rock types in 88 aggregate samples from locations in southern Norway; based on petrographic examination at SINTEF Structures and Concrete 1991-1995.
Results

The results from this work and those from petrographic analyses carried out on a commercial basis by SINTEF Structures and Concrete are presented here. Figures 2 and 3 show data analysed from 88 samples which were collected from different locations of glaciofluvial sands in southern Norway. The distribution of cataclastic rocks in aggregate samples from different counties in southern Norway is presented in Fig. 4.

![Diagram](image)

Fig. 4. Distribution of cataclastic rocks in 88 aggregate samples located in different counties in southern Norway; based on petrographic examination at SINTEF Structures and Concrete 1991-1995.

The petrographic compositions of the twelve samples investigated in this study are given in Table 1. Further information, such as the locations of the samples, the graphical presentation of some rock assemblages in the 1-2 mm fractions, as well as the ratio of cataclastic rocks between the two fractions, are given in Figs. 5 & 6.
Table 1. The rock composition for all twelve tested glaciofluvial samples, given as volume percentages of the 1-2 and the 2-4 mm fractions.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Location No.</th>
<th>Rock assemblages (%)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>051094.05, 1-2 mm</td>
<td>1</td>
<td>23 - - - - - 10 - 1 51 15 -</td>
</tr>
<tr>
<td>051094.05, 2-4 mm</td>
<td>-</td>
<td>- 32 - - - 13 - 2 49 4 -</td>
</tr>
<tr>
<td>051094.02, 1-2 mm</td>
<td>2</td>
<td>16 - - 1 - 3 - 4 72 3 1</td>
</tr>
<tr>
<td>051094.02, 2-4 mm</td>
<td>-</td>
<td>- 20 - - 1 - 9 - 64 6 -</td>
</tr>
<tr>
<td>061094.09, 1-2 mm</td>
<td>3</td>
<td>19 - - - 8 - 2 67 4 -</td>
</tr>
<tr>
<td>061094.09, 2-4 mm</td>
<td>-</td>
<td>- 13 - - 17 - 3 61 3 -</td>
</tr>
<tr>
<td>061094.09, 1-2 mm</td>
<td>4</td>
<td>16 - 2 3 - 9 - - 3 61 9 -</td>
</tr>
<tr>
<td>061094.09, 2-4 mm</td>
<td>-</td>
<td>- 21 - - 2 - 3 50 11 -</td>
</tr>
<tr>
<td>061094.05, 1-2 mm</td>
<td>5</td>
<td>10 - 3 3 - 9 - 1 64 10 -</td>
</tr>
<tr>
<td>061094.05, 2-4 mm</td>
<td>-</td>
<td>- 12 - 3 4 - 11 - 7 58 5 -</td>
</tr>
<tr>
<td>061094.01, 1-2 mm</td>
<td>6</td>
<td>13 - - 2 - 4 - 1 72 7 1</td>
</tr>
<tr>
<td>061094.01, 2-4 mm</td>
<td>-</td>
<td>11 - - 13 - 8 - 5 57 6 -</td>
</tr>
<tr>
<td>061094.02, 1-2 mm</td>
<td>7</td>
<td>12 - 2 3 - 7 - 2 64 8 1</td>
</tr>
<tr>
<td>061094.02, 2-4 mm</td>
<td>3</td>
<td>9 - - 4 - 9 4 7 59 5 -</td>
</tr>
<tr>
<td>061094.03, 1-2 mm</td>
<td>8</td>
<td>1 12 - 2 1 - 9 - 3 66 5 1</td>
</tr>
<tr>
<td>061094.03, 2-4 mm</td>
<td>-</td>
<td>- 23 - 3 7 - 7 - 4 50 6 1</td>
</tr>
<tr>
<td>061094.04, 1-2 mm</td>
<td>9</td>
<td>15 - 1 3 - 5 - 6 65 5 -</td>
</tr>
<tr>
<td>061094.04, 2-4 mm</td>
<td>-</td>
<td>13 - 3 1 - 14 3 6 53 7 1</td>
</tr>
<tr>
<td>131094.01, 1-2 mm</td>
<td>10</td>
<td>8 - - - - 2 - 89 - 1 -</td>
</tr>
<tr>
<td>131094.01, 2-4 mm</td>
<td>-</td>
<td>7 - - - - - - 91 - 2 -</td>
</tr>
<tr>
<td>131094.02, 1-2 mm</td>
<td>11</td>
<td>11 - - - - - - 88 - 1 -</td>
</tr>
<tr>
<td>131094.02, 2-4 mm</td>
<td>-</td>
<td>18 - - - - - - 78 4 -</td>
</tr>
<tr>
<td>131094.03, 1-2 mm</td>
<td>12</td>
<td>17 - - - - - - 82 1 -</td>
</tr>
<tr>
<td>131094.03, 2-4 mm</td>
<td>-</td>
<td>- - - - - - 92 - -</td>
</tr>
</tbody>
</table>

*Description of the different rock assemblages: 1) sandstones, 2) cataclastic rocks, 3) acid volcanic rocks, 4) argillaceous rocks, 5) greywacke, 6) other rock types with microcrystalline quartz (e.g. marl), 7) fine grained quartzite, 8) other rock types containing fine divided quartz (crystal sizes 0.06-0.13 mm), 9) volcanic rocks/gabbro, 10) granites/gneisses, 11) mafic rocks/limestone, 12) other rocks.
Fig. 5. Sample locations investigated in the southeastern Precambrian province. Graphical presentation of some rock assemblages in the 1-2 mm fraction, and the ratio of cataclastic rocks (1-2 mm/2-4 mm). The main glacial movement has been towards the SSW in the region. (Geological map modified from Sigmond et al. 1984).
Fig. 6. Sample locations in the investigated area in Verrabotnen. Graphical presentation of some rock assemblages in the 1-2 mm fraction, and the ratio of cataclastic rocks (1-2 mm/2-4 mm). The main glacial movement has been along Verrabotnen, down Skaudalen to the south-west (Geological map modified from Grønlie et al. 1991).

Discussion

It is necessary to point out that a more accurate interpretation of Norwegian glaciofluvial sand would have been possible if the aggregate samples had been sampled randomly. However, the results presented here will provide a clearer picture about the content and distribution of cataclastic rocks in Norwegian glaciofluvial aggregates. It is evident from Fig. 2 that about 90% of the samples contain various amounts of cataclastic rocks. A high proportion, about 45%, contain 0-5% of cataclastic rocks and only a small percentage about 5%, exhibit greater than 20% of
cataclastic rocks, which also is the limit of alkali reactive aggregates according to the Norwegian optional arrangement for declaration and approval of aggregates for concrete (DGB). Fig. 3 shows the relationship between cataclastic rocks and alkali reactive rock-types. In this figure, cataclastic rocks constituted the majority of all alkali reactive rock-types in several of the samples. In a few samples cataclastic rocks constitute the main component of the alkali reactive rock (plots located on the broken line). The distribution of samples with cataclastic rocks is shown by county region within southern Norway in Fig. 4. The diagram shows that cataclastic rocks are present in varying degrees in samples analysed from all the counties. Generally, the cataclastic rocks constitute less than 20% of the volume fraction of the aggregate. Aggregate samples from Telemark, Rogaland, Aust-Agder and Møre og Romsdal contain cataclastic rocks which fall into two or less % volume fraction categories. Only samples from the counties of Sør-Trøndelag and Oppland exhibited cataclastic rocks with a volume fraction greater than 20 %, which likely is governed by local lithology.

The main objective of collecting samples for this study was to examine glaciofluvial materials located over a range of transportation distances from the origin of the cataclastic rocks. It would have been preferable to obtain more information about the particular glacial and sedimentary environments within these two investigated areas but this was not within the scope of the presented work. It is recommended that such a study would enable assessment of the regional effects of glaciofluvial transportation to be made and would help to understand the influence of other regional factors upon the end product of the glaciofluvial material. In outlining our data here, account needs to be taken of the limited number of samples analysed; with the exception of one location, where four samples were collected within the same deposit, only one sample was taken at each location. The different sedimentary units within the various deposits also needs to be taken into consideration when selecting the samples.

The interpretation of the results is based on petrographic examination of the 1-2 mm and 2-4 mm fractions according to the technique described in the experimental section. Hence, the occurrence and content of cataclastic rocks in the coarser fractions, with regards to the effect of transportation, will not be discussed.

Results from the southeastern Precambrian province show the samples to be dominated by the rock assemblage granites and gneisses. Even though minor fractions of other associated rock-types are observed, all the nine samples examined exhibited a relatively high content of cataclastic rocks, and they appear to be the second most dominant rock assemblage in most of the samples. The cataclastic rocks were all classified as mylonitic rocks, showing fluxion texture with a matrix of microcrystalline and subgranular of quartz, and larger porphyroblasts of feldspar. The highest amounts of cataclastic rocks were found in samples close to the mylonite zones, in general agreement with previous observations (Figs. 7 & 8), and the %
volume fraction declined with increasing transportation distance from these zones. It is evident from Fig. 7 that the content of cataclastic rocks declines (for both investigated particle sizes) at distances greater than 5 km from the mylonite zone. The maximum content of cataclastic rocks in both fractions is observed to occur at approximately 6 km from the zone.

The sample from location 4 was taken directly above the mylonite zone. In this case, questions might be raised whether the mylonite particles found in this aggregate are derived locally, or if the particles are the result of glaciofluvial transportation from the northern mylonite zone. The low ratio between the content of cataclastic rocks in the 1-2 mm and 2-4 mm fractions indicates, however, that the aggregate is derived locally.

Even though the amount of cataclastic rocks declines with increasing transportation distance, the relatively high content of such rocks in samples located more remotely, and downstream from the mylonite zones, indicate a high survival potential for these rock- types. It is evident from Figs. 5, 7 & 8 that samples close to the mylonite zones contain a relatively higher amount of cataclastic rocks in the 2-4 mm fraction than in the 1-2 mm fraction. This trend is reversed for samples more remote from the major mylonite zones. It appears that cataclastic rocks are dominant in the 2-4 mm fraction in comparison with the 1-2 mm fraction for transportation distances up to about 20 to 25 kilometres downstream from both mylonite zones. For transportation distances greater than 20 to 25 kilometres, cataclastic rocks in the 1-2 mm fraction are more prevalent. The high survival potential of cataclastic rocks could be explained by the observation that such rocks are more durable to mechanical abrasion than most other rock types (Brattli 1994). Therefore, cataclastic rocks will be able to survive greater transport distances than other rock-types of similar origin without significant erosion of material. Those particles in the 2-4 mm fraction will contribute to the amount of cataclastic rocks in the 1-2 mm fraction, as a result of their undergoing erosion and comminution after travelling long distances.
Fig. 7. Distribution of mylonitic rocks, in two different fractions, from locations in the southeastern Precambrian province, related to the distances (km) from the Mjøsa - Vänern mylonite zone. Numbers (1, 2 & 3) represent the locations of the samples.

Fig. 8. Distribution of mylonitic rocks, in two different fractions, in the southeastern Precambrian province related to the distances (km) from the second mylonite zone. The numbers (4, 5, 6, 7 & 9) represent the different locations of the samples. For locations 6-9, an average value has been applied. The outlier (No. 8) is not included in the graph.
The four samples (nos. 6-9) which were obtained from the same location at Mona demonstrate the homogeneity between different layers within the same glaciofluvial deposit (Fig. 8). No significant differences were observed in the 1-2 mm fraction for samples from the coarse-, fine- and medium-graded layers. However, in the 2-4 mm fraction an unusually high content of cataclastic rocks was found in the medium layer (no.8). No reasons are given for this anomalous result; however, such uncharacteristic behaviour could significantly influence the statistical variation when testing and approving materials for concrete purposes.

Some of the aggregate samples (nos.3 and 6-9) which contained particles of cataclastic rocks were located up to 40 km downstream from their origin in the mylonite zones, in relation to the main ice movement. However, glaciofluvial transportation of materials will not necessarily follow the main ice flow direction, rather it will be governed by local topography. Hence, the true transportation distance for glaciofluvial materials will in most cases be longer than that indicated by the main ice movement.

The three samples from Verrabotnen all show a simple mineralogical composition, consisting of only a few rock assemblages. The petrographic examination indicates a dominance of granites and gneisses (Fig. 6), which are the predominant rock-types in the area. The second most frequent rock assemblage is that of cataclastic rocks, in this case classified as cataclasite. The three samples were collected from within a much smaller area than the nine samples from the southeastern Precambrian area.

From Fig. 9, it is evident that for transport distances greater than 7 km downstream from the origin of the fault rock, cataclasite rocks in the 1-2 mm fraction are more abundant than the 2-4 mm fraction. However, the amount of cataclasite in the 2-4 mm fraction reaches its maximum at the second location (no. 11), only 1 km downstream from the origin of the fault rocks, and beyond these distances the volume fraction decreases near the third location (no. 12). In comparison to results analysed for Figs. 7 and 8, the data for Fig. 9 were only from a profile of 10 km. As a consequence of the difference in the area profiled and the difference in the mechanical properties of cataclastic rock-types between the two investigated areas, it is unwise to make any realistic comparison of the trends observed. However, regarding the amount of cataclasite in the 2-4 mm fraction, it appears that the cataclasite shows a lower potential to survive transportation over longer distances than mylonitic rocks. This is in accordance with the observations of Brattli (1994) who attributed this behaviour to the lower abrasion values for brittle deformed cataclastic rocks (cataclasites) and various granites in comparison to the ductile deformed behaviour of mylonites.
Conclusion

The following main conclusions can be drawn from the present work:

The results from the data analysis of glaciofluvial materials, even those that were not considered to be representative for Norwegian glaciofluvial sediments, showed that cataclastic rocks are a common constituent in the majority of glaciofluvial sediments. This is in good agreement with the geological bedrock map of southern Norway.

In some locations cataclastic rocks constitute the major component for all alkali reactive rock-types in the aggregate samples analysed in the present work. Only about 5 % of the investigated samples contained more than 20 % volume fraction of cataclastic rocks. These types of aggregate samples were only observed in the counties of Sør-Trøndelag and Oppland.
Based on literature studies, both the provenance and the various processes associated with the comminution and transportation of glaciofluvial materials have been identified as the factors which can lead to enhanced amounts of cataclastic rocks occurring in glaciofluvial materials. Examination of glaciofluvial materials, located at various transportation distances from two major mylonite zones, showed relatively high contents of cataclastic rocks, in both the 1-2 mm and the 2-4 mm fractions; whereas glaciofluvial materials near mylonite zones show a higher content of cataclastic rocks in the 2-4 mm fraction than in the 1-2 mm fraction. The opposite trend is observed for samples located further away from the mylonite zones, particularly in the direction of downstream ice movement. In samples taken up to 30-40 km downstream from the parent rock, a high content, or an enrichment of cataclastic rocks (mylonites), was found in the fine fraction (1-2 mm).

The occurrence of particles of cataclasite in glaciofluvial materials follows similar trends to those described for the mylonite. However, the cataclasite appears to be enriched in the 1-2 mm fraction rather than the 2-4 mm fraction, and occurs much closer to the fault zone than for mylonites. This would seem to indicate that cataclasites are less durable to mechanical abrasion, when transported over such long distances, than mylonitic rocks.

**Acknowledgements**

The staff at the Department of Geology and Mineral Resources Engineering, University of Trondheim and at the SINTEF Structures and Concrete, are thanked for their technical assistance. The work is a part of the first author's PhD. study, which has been funded by the Royal Norwegian Council for Scientific and Industrial Research. He would like to acknowledge the contribution of Dr. S.W. Danielsen and Dr. B. Brattli, in supervision and discussion during the course of the PhD study. SINTEF Structures and Concrete is acknowledged for access to their database, and special thanks to Dr. S. Lippard and Dr. H.H.Patel for their help in the editing of this manuscript.
References


Chapter 5 - Paper 1


Sørensen, R. 1979: Late Weichselian deglaciation in the Oslofjord area, south Norway. Boreas, 8, 241-246.

Chapter 6

Test Methods for Alkali-Aggregate Reactions in Norwegian Aggregates: Petrographic Examination and the South African NBRI Mortar-Bar Test

BØRGE JOHANNES WIGUM and JAN LINDGÅRD


B.J. Wigum, Department of Geology and Mineral Resources Engineering, University of Trondheim, The Norwegian Institute of Technology, 7034 Trondheim, Norway

J. Lindgård, SINTEF, Structure and Concrete, 7034 Trondheim, Norway
SP 145-41

Test Methods for Alkali-Aggregate Reactions in Norwegian Aggregates: Petrographic Examination and the South African NBRI Mortar-Bar Test

by B.J. Wigum and J. Lindgård

Synopsis: Petrographic examination and the South African mortar-bar test have been performed at SINTEF - Structures and Concrete during the last 2-3 years to evaluate the reactivity of Norwegian aggregates to be used in concrete structures. This paper presents and discusses the relationship between these two test methods.

The purpose of the petrographic examination is to identify, quantify, and group different rock types in an aggregate. These groups are: reactive aggregates (with known reactive field performance), potentially reactive aggregates and innocuous aggregates.

In Norway further testing by the mortar-bar test is recommended when petrographic examination indicates ≥20% reactive or potentially reactive rock types in the aggregates. The mortar-bar expansion after 14 days of exposure is used for the evaluation of potential expansivity of the aggregates.

One main conclusion from the investigation is that the mortar-bar expansion increases up to an upper level with increasing content of reactive rocks in the aggregates. Beyond a "marginal" amount of reactive rocks in aggregates, the mortar-bar expansion increases no further. A significant difference in mortar-bar expansion between different reactive rock types has not been found. The established limit of 20% reactive rocks in aggregates appears in most cases sufficient for classifying aggregates as innocuous, however, no verification of the limit has been made.

Keywords: Accelerated tests; aggregates; alkali aggregate reactions; concretes; expansion; mortar bar test; petrography; rocks
Børge Johannes Wigum holds his Master`s degree from the Norwegian Institute of Technology (NTH) in Trondheim, Norway. He is now working on his Dr.ing.-study at the Norwegian Institute of Technology, Department of Geology and Mineral Resources Engineering, Trondheim Norway.

Jan Lindgård holds his Master`s degree from the Norwegian Institute of Technology (NTH) in Trondheim, Norway. He is now a research engineer at SINTEF Structures and Concrete, Trondheim, Norway.

INTRODUCTION

During the last 5-6 years, slow/late-expansive alkali-aggregate reaction (AAR) has been recognised as a concrete durability problem in Norway (1, 2). In Norway the reaction is a very slow phenomenon which takes a minimum of 15-20 years to manifest itself in a structure that contains the necessary ingredients for the reaction. Therefore, accelerated tests have to be used to evaluate the expansivity of aggregates for concrete structures. Increasing the rate of chemical reaction in the laboratory makes it possible to measure harmful expansion in concrete after a short time. The main purpose of an accelerated test is to optimise the classification of aggregates regarding field performance.

The evaluation of the usefulness of different test methods in Norway is based on petrographic examination of the aggregates and field experience from structures containing different aggregate types. Two out of several test methods investigated seem to be suitable for Norwegian aggregates (3). These are the South African NBRI mortar-bar test (14 days) (4), and the Canadian CSA A23.2-14A concrete-prism test (one year). A modified version of the mortar-bar test has been used in Norway the last 2-3 years for distinguishing reactive, potentially reactive and innocuous Norwegian aggregates (3).

The aggregate committee of the Norwegian Concrete Society has proposed an arrangement for declaration and approval of aggregates used for concrete (5). The arrangement (named "DGB") was introduced in Norway in the autumn 1992 on a voluntary basis. In this arrangement the testing procedure consists of three stages (6), starting with a petrographic examination (step 1). If a low content of reactive or potentially reactive rock types (<20%) is
observed, the aggregate is classified as innocuous. On the other hand, if a high quantity of reactive rock types (≥20%) is present, the aggregate is recommended to be tested according to the modified version of the South African NBRI mortar-bar test (step 2).

Finally, the Canadian CSA A23.2-14A concrete-prism test (step 3) is recommended if the aggregate is classified as reactive according to the mortar-bar test. Step 3 (modified method) is used only to find innocuous concrete compositions, i.e. investigate the effect on the concrete-prism expansion of changing cement type, changing cement content, adding pozzolans etc. However, step 3 is not used to confirm sometimes questionable NBRI mortar-bar results.

The main purpose of the research work presented in this paper is to investigate possible relationships between petrographic composition of the aggregates and expansion results obtained by the mortar-bar test. Natural aggregates, crushed stones and blends of aggregates are included in the investigation. Subsequently, the reactivity of different rock types will be discussed. Finally, the usefulness of the petrographic examination, the mortar-bar test and the established limit of critical amount of reactive rocks in aggregates will be discussed in light of present knowledge of field performance of different Norwegian aggregate types.

The foundation of the research work is a number of tests performed at the laboratory of SINTEF - Structures and Concrete, during the last 2-3 years. The testing of natural aggregates is mainly performed on a commercial basis, while testing of crushed rocks and blends of aggregates are performed both on a commercial basis and in connection with research projects (7).
LABORATORY TESTING

Petrographic examination

The purpose of the petrographic examination is to identify, quantify and group different rock types in an aggregate to be used in concrete structures. In Norway the classification and identification of the different alkali-reactive rock types are based on knowledge of field performance, petrographic nomenclature, and mineralogical/microstructural criteria. On the basis of the petrographic examination, the Norwegian aggregates are divided into three main groups (8):

*Reactive aggregates* (with known reactive field performance). Aggregates containing sandstone, siltstone, cataclastic rocks, acid volcanic rocks, argillaceous rocks, greywacke, or other rock types with microcrystalline quartz or another unstable phase.

*Potentially reactive aggregates*. Aggregates containing fine-grained quartzite, other rock types with fine-grained quartz or another unstable phase.

*Innocuous aggregates*. Aggregates containing rock types with coarse grains and/or minor amounts of quartz.

Due to the high metamorphic grade and degree of deformation of the Norwegian rocks, the identification and classification of various rock types may be very difficult. It is therefore recommended that the petrographic examination has to be performed using thin-section examination, by a geologist familiar with Norwegian reactive rock types (6).

The petrographic examination used at SINTEF - Structures and Concrete was primarily based on thin-section analysis of aggregate grains in the fraction 4-8 mm. The aggregate was first investigated by visual observation using a stereomicroscope, and divided into separate groups. A few representative grains from each group were then selected and prepared for more detailed identification by thin-section analysis. The amount of reactive rock types was given as percentages of the number of grains counted. Unfortunately the thin-section analysis occasionally made a difference between grains from the same group. Sometimes one grain could be classified as reactive and other grains from the same
group as innocuous. In these cases the evaluation of the reactivity was uncertain.

Therefore a modified method was developed, based on point-counting in thin-sections under the petrographic microscope (8). Two thin-sections (25x35 mm) are made with grains in the fraction 2-4 mm and one is made with grains in the fraction 1-2 mm. It is possible to point-count approximately 1000 points in each fraction. The content of reactive rock types in an aggregate is obtained by calculating an average of the results from point counting both fractions. The amount is given as volume percentage.

The South African NBRI mortar-bar test

An accelerated method which is based on expansion of mortar-bars in a 1N NaOH solution at 80°C has been found effective for classifying reactive aggregates. Both the heat and the NaOH solution, which is rich in alkalies, accelerate the chemical reactions. This accelerated test method is referred to as the "South African NBRI mortar-bar test" (4, 9). At SINTEF - Structures and Concrete, a modified version of the test has been practised. The expansion is monitored by measuring length change of the mortar-bars (40x40x160mm) after 4, 7, 12, 14, 28 and 56 days respectively (8).

The expansion after 14 days in the NaOH solution is used for evaluating the potential expansivity of the aggregates (3). Aggregates causing 14-day expansion of:

\[
\begin{align*}
\leq 0.10\% & \text{ should be considered } \textit{innocuous}; \\
> 0.10\% \text{ but } < 0.25\% & \text{ should be considered } \textit{potentially deleteriously alkali reactive - slowly expansive}; \\
\geq 0.25\% & \text{ should be considered } \textit{potentially deleteriously alkali reactive - rapidly expansive}.
\end{align*}
\]

In Norway further testing by the mortar-bar test is recommended when the petrographic examination indicates ≥ 20% reactive rock types in an aggregate. An aggregate containing < 20% is accepted as innocuous, and no further testing is recommended (5). This critical limit is based on one investigation using different blending proportions between an innocuous aggregate and a reactive rhyolite. This rhyolite is assumed to be one of the most reactive rock
types in Norway. With 20% of reactive rhyolite in the blended aggregate, the critical mortar-bar expansion of 0.10% after 14 days was obtained (10).

The mortar-bar test is used in Norway to investigate natural aggregates, crushed stone and blends of aggregates (3).

Testing blends of aggregates is done most frequently to investigate the reactivity of a reactive sand combined with an innocuous crushed stone. To make a consistent and reproducible test, the blending proportions of the separate aggregates are the same in each fraction. When classifying blends of aggregates, the mortar-bar test is always performed in addition to the petrographic examination.

RESULTS

In presenting the results from the petrographic examinations and the mortar-bar tests, we have distinguished between natural aggregates (glaciofluvial sediments), crushed stone and blends of aggregates.

Some aggregates were investigated only by the primary petrographic method. However, when the results from this test method were regarded as uncertain, the aggregates were re-examined by the modified petrographic method. An evaluation of, and comparison between, these two methods is not yet completed. However, preliminary results indicate the primary method to slightly over-estimate the content of reactive aggregate, although the modified method gives a more specific description of the petrographic composition.

Natural aggregates

Results from the investigations of natural aggregates are given in Figure 1. Most of the natural aggregates tested contain between 25 and 50% of reactive rocks. The mortar-bar expansion of these aggregates varies mainly between 0.15 and 0.30% after 14 days of exposure to the NaOH solution. As mentioned earlier, the mortar-bar test is recommended only when the content of reactive rocks exceeds 20%. Therefore only one natural aggregate classified as innocuous by petrographic examination was tested by the mortar-bar method. The figure shows some scatter, but the trend is that the
mortar-bar expansion increases with an increasing content of reactive rocks, as determined by petrographic examination.

**Crushed stones**

Results from the investigations of crushed stones are given in Figure 2. Most tested aggregates contain only one rock type. Therefore, the content of reactive rocks is usually 0 or 100 %. In the figure we have divided the rock types into four groups: cataclastic rocks, rhyolite, sedimentary rocks and innocuous rocks.

All crushed stones classified as innocuous by petrographic examination show an expansion less than 0.05 % after 14 days of exposure to the NaOH solution.

The mortar-bars made of crushed stones with 100 % reactive rocks show expansions mainly between 0.15 and 0.30 % after 14 days of exposure in the NaOH solution. There is no significant difference in expansion between the different rock types. The expansion of mortar-bars containing cataclastic rocks show a large variation, from 0.12% to 0.30% after 14 days of exposure to the NaOH solution.

**Blends of aggregates**

Results from the investigations of blends of aggregates are given in Figure 3. When blending an innocuous aggregate and a reactive aggregate, the mortar-bar expansion decreases compared to the expansion of mortar-bars containing only the reactive aggregate. In some cases there seems to be a nearly linear relationship between the mortar-bar expansion and the proportion of reactive rocks in the blended aggregate. One example is the tests mentioned earlier, with different blending proportions of reactive rhyolite (crushed) and an innocuous natural aggregate (10).

In the other tests with blends of aggregates, reactive natural aggregates are combined with different amounts of innocuous crushed stone. In these tests there is no linear relationship between the proportion of innocuous crushed stone in the blended aggregate and reduction in mortar-bar expansion. For some reactive natural aggregates substitution with an innocuous crushed stone (up to 50 %) only produces minor reduction in the mortar-bar expansion. In such cases the mortar-bar expansion of the blended aggregate is higher than for natural aggregates containing similar amounts of
reactive rock. In one case the mortar-bar expansion of the blended aggregate exceeds 0.10 %, even though the content of reactive rocks in the blended aggregate is less than 20 %.

DISCUSSION

The reactivity of different rock types

In Norway sedimentary rocks (sandstone, siltstone, greywacke and argillaceous rocks), acid volcanic rocks (rhyolite) and cataclastic rocks are classified as reactive or potentially reactive. In addition, rock types containing microcrystalline quartz (e.g. some quartzites) are reported to be reactive (2, 5). Depending on the geographical position, various natural aggregates (glaciofluvial sediments) in Norway contain from 0% to 100% of reactive rock types. Different types of cataclastic rocks are frequently the dominant proportion of these reactive rock types. It is believed that the cataclastic rocks are particularly reactive due to a high content of quartz, which is often microcrystalline or strained, caused by ductile deformation.

In Figure 4, all tested natural aggregates which contain more than 50% of cataclastic rocks, as the reactive part, are given an index. The purpose was to investigate the hypothesis that aggregates with a major content of reactive cataclastic rocks, cause larger expansion than aggregates containing similar amounts of other reactive rocks, such as sandstone, siltstone, greywacke and argillaceous rocks. The Figure shows no significant difference in expansion between the different rock types. A corresponding conclusion was drawn from the tests with crushed stone (Figure 2). However, in these tests the different cataclastic rocks also demonstrated a large variation in expansivity, which could be explained by the broad range of cataclastic rocks. Both the degree of deformation and the grain size are very variable in the group of cataclastic rocks. The hypothesis of a large expansivity may only be valid for some of the cataclastic rocks. Further research is required to clarify this subject.
The usefulness of the petrographic examination, the NBRI mortar-bar test and the established limit of the critical amount of reactive rocks in aggregates

In Figure 5, results from the testing of both natural and blended aggregates are shown together. The results indicate that when the content of reactive rocks in the aggregate exceeds 25%, the expansion increases rapidly. The results also indicate that with a "marginal" amount of approximately 40% reactive rocks in the aggregate the expansion reaches a possible upper limit of 0.25-0.30%. In Figure 2 it can be observed that crushed stone containing 100% reactive rocks expand no more than aggregates containing less reactive rock. Even the most expansive crushed stones (mylonite) do not exceed an expansion of 0.30% after 14 days, supporting the theory of a "marginal" effect. The possible occurrence of a "marginal" amount of reactive rocks to reach a maximum expansion, leads to the question of the pessimum effect (a worst possible proportion).

This effect is an internationally known phenomena that occurs with rapidly expansive rock types (e.g. opal). However, in the mortar-bar test, the pessimum effect cannot be observed, since there is an excess of alkalies (11). Another explanation of the possible "marginal" effect, may be found in the performance of the mortar-bar test, but that will not be discussed in this paper.

A general observation is that the mortar-bar expansion increases with increasing content of reactive rock in the aggregates. Two separate regression analyses were made on the test results with natural aggregates (Figure 1) and blends of aggregates (Figure 3). Three separate regression analyses were made on the collected results from both natural and blends of aggregates (Figure 5). The results indicate a linear relationship exists between the content of reactive rock in the aggregates and the mortar-bar expansion. However, all the regression analyses, except perhaps one, show a lack of significant correlation. The best correlation (R-sq = 0.83) was obtained on test results from aggregates containing \( \leq 20\% \) reactive rocks. The purpose of the regression analysis was to investigate the critical limit of reactive rocks. From this analysis it would be reasonable to consider aggregates containing \( \leq 20\% \) reactive rocks as a separate population, which is also in agreement with the rapidly increasing expansion of mortar-bars containing >20% reactive rocks in the aggregates. However, more test results
with aggregates containing \( \leq 20\% \) reactive rocks is required to confirm this theory. Aggregates containing \( > 20\% \) reactive rocks show no linear correlation (R-sq = 0.30).

The investigation does not clearly confirm the critical limit of 20% reactive rocks used to classify aggregates as innocuous. Nevertheless, in this investigation 9 aggregates (natural, crushed stones and blends of aggregates) classified as innocuous by petrographic examination were tested. Only one of them (a blended aggregate) was classified as slowly reactive (0.12% expansion after 14 days with a content of 16% reactive rock).

On the basis of the results presented, it may be necessary to modify or alter the established critical limit of reactive rocks, alternatively the critical limit of the mortar-bar expansion, or the time of exposure in the NaOH solution. Before modifying or altering these criteria, it is important to compare test results with field performance of different aggregates in real structures. Petrographic examination and measuring the quantity of reactive rock types in these aggregates should also be performed. The Norwegian experience shows that aggregates which react in structures exhibit mortar-bar expansion from 0.10-0.30% after 14 days of exposure in the mortar-bar test (7).

Some earlier investigations with the mortar-bar test have shown it has some disadvantages. One investigation claims that the mortar-bar test is severe for many aggregates with good field performance (12). Another investigation reports that the mortar-bar method is unsuitable for distinguishing certain deleteriously reactive rocks containing micro-crystalline quartz associated with strained quartz as the only reactive component (13).

In Norway, reacted aggregates in structures have only been observed in coarse-aggregate and coarser sand fractions (>1-2 mm) (1). The effect of particle size on expansion of mortar-bars with slowly expanding aggregates has not yet been investigated (14). It cannot be ignored that there is a possible difference in the mechanism of reaction for slowly reactive Norwegian aggregates in field performance and in the mortar-bar test. However, an investigation reports that the reaction products formed under the conditions of the accelerated test are similar to those formed in concrete subjected to the conditions of standard tests, although they are formed at a faster rate under the accelerated conditions (15).
A limitation of the test is that it can only be used to investigate the reactivity of aggregates. Evaluation of the reactivity of different concrete compositions (e.g. change of cement type or cement content, adding pozzolans etc.), cannot be performed, due to the excess of alkalies (6).

**CONCLUSION**

In this paper it has been shown that there is a relationship between results from petrographic examination and results from the mortar-bar test. The mortar-bar expansion increases with increasing content of reactive rocks in the aggregate. However, no significant correlation by statistical analyses has been established.

With increasing content of reactive rocks in aggregates over 25%, the mortar-bar expansion increases rapidly. The results indicate a "marginal" amount of approximately 40 % reactive rocks in the aggregates will produce an upper limit of (0.25-0.30%) mortar-bar expansion. A larger content of reactive rocks in aggregates produces no additional expansion.

No significant difference in expansion between different reactive rock types was found. The established limit of 20% reactive rocks in aggregates appears sufficient in most cases from the results obtained by both the petrographic examination and the mortar-bar test. However, this investigation does not verify the limit.

The usefulness of both test methods for evaluating the expansivity of aggregates to be used in concrete structures appears to be sufficient in most cases. To optimise the classification of aggregates, further research should emphasise a comparison, related to local experience, between results from both petrographic examination, the mortar-bar test and field performance of aggregates in different structures.
ACKNOWLEDGEMENTS.

This paper is published with the permission of the Norwegian Research Institute SINTEF - Structures and Concrete (including the previously Cement and Concrete Research Institute, SINTEF FCB) Thanks are extended to Viggo Jensen and Svein Willy Danielsen for helpful advice and discussions, and to Stephen Lippard for improving the English text. The paper is a part of B.J.Wigum's Dr.ing.-study, which is supported financially by the Royal Norwegian Council for Scientific and Industrial Research.

REFERENCES


Fig.1--- Percentage of reactive rocks in natural aggregates (determined by petrographic examination) versus percentage expansion of mortar-bars after 14 days of exposure in the NaOH solution.
Fig. 2 – Percentage of reactive rocks in crushed stone versus percentage expansion of mortar-bars after 14 days of exposure in the NaOH solution.

Fig. 3 – Percentage of reactive rocks in blends of aggregates (determined by petrographic examination) versus percentage expansion of mortar-bar after 14 days of exposure in the NaOH solution.
Fig. 4 – Grouping of reactive natural aggregates. The figure gives percentage of reactive rocks (determined by petrographic examination) versus percentage expansion of mortar-bars after 14 days of exposure in NaOH solution.

Fig. 5 – Percentage of reactive rocks in both natural- and blends of aggregates (determined by petrographic examination) versus percentage expansion of mortar-bars after 14 days of exposure in NaOH solution.
Chapter 7

Sequential examination of slowly expanding alkali-reactive aggregates in accelerated mortar bar testing

BØRGE JOHANNES WIGUM and WILLIAM J. FRENCH

Wigum, B.J. and French, W.J. 1995: Sequential examination of slowly expanding alkali-reactive aggregates in accelerated mortar bar testing. Accepted for publication in Magazine of Concrete Research, London, UK

B.J. Wigum, Department of Geology and Mineral Resources Engineering, University of Trondheim, The Norwegian Institute of Technology, 7034 Trondheim, Norway

W.J. French, Geomaterials Unit, School of Engineering, Queen Mary and Westfield College, University of London, Mile End Road, London E1 4NS, UK
...DO NOT PRINT THIS SIDE
Sequential examination of slowly expanding alkali-reactive aggregates in accelerated mortar bar testing

B. J. Wigum† and W. J. French‡

† Department of Geology and Mineral Resources Engineering, University of Trondheim, The Norwegian Institute of Technology, 7034 Trondheim, Norway.

‡ Geomaterials Unit, School of Engineering, Queen Mary and Westfield College, University of London, Mile End Road, London E1 4NS, UK.

The NBRI Mortar-bar test, now adopted with slight modification as ASTM C1260-94, has been used to study the sequential development of alkali aggregate reaction for certain slow/late expanding types of reactive rocks. The presented work is concerned with the expansion produced by mylonites, meta-argillite, meta-greywacke, granite, limestone and silica glass. At different stages in the test, the situation at the reaction site and the formation of the gel reaction products, have been investigated using both optical- and scanning electron microscopes. The composition of the gel reaction products and the amount of mobilised silica have also been measured.

The accelerated test method appears to echo the alkali-aggregate reaction occurring in field concrete. The different rock types show different rates of expansion, and two different reaction styles are observed at the reaction sites. One of these two phenomena involves dissolution of quartz along grain boundaries and the other involves crack generation within the aggregate. The process of cracking and the amount of cracking appears to be the main factor governing the amount of expansion produced by the reaction. No correlation was found between the amount of alkali-silicate gel produced as the reaction product and the expansion of the mortar bar.

Introduction

The first reports of alkali-reactive aggregates related to various heterogeneously structured, porous, and sometimes hydrous silica minerals such as opal, volcanic glass and chert, which were found to be extremely reactive under particular conditions. The reaction is well documented and potentially reactive rocks of this type are unambiguously recognised by the NBRI accelerated mortar bar test1. Reaction in the field is normally seen after a few years and is strongly developed in some two decades. More recently well crystallised and more dense quartz-bearing rock types have also been observed to show significant levels of reaction involving a slower mechanism which leads to relatively delayed expansion and damage. A wide range of
rock-types take part in these reactions and are characterized by the presence of microcrystalline quartz and coarser quartz with crystal lattice defects caused by ductile deformation. Strained quartz grains - especially those with strain lamellae - are commonly involved. According to Stark\textsuperscript{2} a few such rocks including quartzites with strained quartz and gneiss, known to be reactive in the field, may not be detected by this test.

The accelerated mortar bar procedure, described in 1986 by Oberholster and Davis\textsuperscript{1} and known as the NBRI Mortar-Bar Test, is widely used to determine the potential for alkali expansivity of various types of aggregates in concrete and has become a standard test now established as the ASTM C1260-94\textsuperscript{3} accelerated test for reactive aggregates. Evaluation of the test has shown it to be reliable and reproducible, both for rapid and usually for the slow/late expanding reactive aggregates. It appears that the reaction while being greatly accelerated echoes the process occurring in field concretes very closely. It was therefore considered that the method might prove suitable for the investigation of the sequential development of Alkali-Aggregate Reactions\textsuperscript{4,5} and the nature of the reactive components in the deleterious aggregate. The acceleration makes it possible to follow the reaction and harmful expansion with reactive aggregate after only 14 days and in the present work the reaction has been followed for up to 56 days. The rate of expansion falls to negligible after this period.

The main objectives of this study were to investigate the sequential development of AAR by this accelerated test, in an attempt to identify possible differences in the behaviour of different types of slow/late expanding reactive aggregate. In association with the expansion development, it was thought to be of interest to examine the characteristics and the sequential development of the products at the reaction site, and to evaluate the formation, properties and amount of the gel reaction produced.

**Experimental Methods**

The aggregates investigated include meta-argillite (Malmesbury, South-Africa), meta-greywacke (Wales), micro-granite (Norway), and two different types of mylonite (Norway). Additionally, as low and high controls of the expansion, an inert limestone aggregate and a quartz sand combined with 15\% of reactive silica glass, were included in the investigation.

The aggregate samples were crushed and washed to meet the size required by the test procedure - those specified for ASTM C-227\textsuperscript{6}. The meta-greywacke aggregate was unusual because it was obtained from concrete cores from the former dam at Maentwrog in Wales. This dam has been reported by Blackwell and Pettifer\textsuperscript{7} to show severe deterioration due to AAR and the quartz was shown to contain numerous crystal dislocations\textsuperscript{8}. Concrete cores from the structure were crushed, and aggregate
particles were separated by hand from the cement paste. The aggregate was then crushed to meet the required grading. In addition, one of the concrete cores was tested by cutting out a concrete bar with the same dimension as the mortar-bars used in the test. This concrete-bar contained the original grading, much coarser than the specified NBRI grading, but was tested in exactly the same manner as the corresponding mortar bars.

The mortar bars measured 25x25x250 mm and were made according to the ASTM C-227 specification for grading and mix design. All the aggregates were crushed materials. For each type of aggregate, four mortar bars were prepared, with an aggregate:cement:water ratio of 2.5:1:0.5. The bars were cured for 48 hours in closed fog containers before being demoulded. They were then immersed in water in closed containers and kept at constant temperature in an oven at 80°C. After 48 hours in the oven, the initial readings of the lengths of the mortar bars were taken. The bars were then immersed in molar NaOH solution in the original tightly covered polypropylene containers and returned to the oven at 80°C. The length of each bar was then measured after 4, 7, 14, 28, 42 and 56 days respectively with each bar being measured four times, the measurements being made in different sequences.

Thin sections

Following the mortar-bar expansion measurements after 4, 14, 28 and 56 days, one bar of each type was removed and transverse petrographic thin-sections (25mm x 25mm) were prepared from the bars after impregnating the mortar with a fluorescent dye. These thin sections were used to follow the sequential development of the reaction. During storage and before the preparation of thin sections, the samples were impregnated and sealed with epoxy resin to prevent carbonation. The use of thin sections made from specimens impregnated with a resin containing fluorescent dye for petrographic study is one of the most widely used techniques for assessment of micro-cracking in concrete and was found to be particularly well suited to the assessment of the development of the mortar bars.

Scanning electron microscopy

Following the mortar-bar expansion measurements after 14 and 56 days, polished and broken surfaces of cross-sections of the different mortar-bars were prepared. These specimens were used to examine the chemical composition and microstructural properties of the gel, using an Hitachi S450 Scanning Electron Microscope fitted with a secondary electron detector. Also attached to the sample chamber were a backscattered electron detector and an energy dispersive X-ray detector. The X-rays detected was processed by an Oxford Instruments AN10000 microanalysis system.
using a ZAF correction programme. Where possible, both gel in cracks in aggregate, and gel lining air-voids were examined but in general only the gel in voids gave reliable analyses because the gel filled cracks were too thin for the analysis to avoid the aggregate. For the meta-greywacke aggregate, polished sections from the original reacted concrete were also examined. These specimens were also used to examine the development of the reaction and the details of the reaction site.

**Soluble silica**

An attempt was made to estimate the total amount of mobilised silica produced by the different reactive materials. The procedure was based on a method described by Müller and Schneider that was introduced to determine biogenic silica (opal) in sediments. Portions of the 14 days old mortar bar samples were crushed to pass a 500µm sieve. Aliquots of the powder (4 g) were placed into 25ml of a Molar NaOH solution at 80°C for 5 and 20 minutes respectively, to extract soluble silica from the gel in the crushed mortar. In an attempt to make a correction for the amount of silica dissolved from the cement-paste, the soluble silica was also measured for the mortar bar containing limestone, with presumably no gel, and this result was subtracted from the results for bars containing other aggregates. The amount of silica in the NaOH solution prepared in this way was determined by atomic absorption spectrophotometry. The ratios of silica to the anhydrous analytical total obtained by the EDX-measurement for the different mortar-bar samples were used to estimate the percentages of gel produced in each type of mortar bar.

**Details of the aggregate**

*Meta-argillite*

The meta-argillite aggregate from Malmesbury, South Africa, has previously been reported to be reactive, and the aggregate was originally used as a standard reactive material in the NBRI test introduced by Oberholster and Davies in 1986. In thin section the rock appears microcrystalline, with quartz and feldspar making a slightly foliated texture with mica. However, in this rock it is difficult to observe individual quartz and feldspar grains and in thin section the maximum crystal grain size of the quartz is approximately 15µm. The scanning electron microscope shows that the rock exhibits a granular structure with a mean grain size of approximately 5µm (Fig. 1.)
Fig. 1. SEM micrograph showing the typical open microstructure of the argillite aggregate, with grain sizes approximately 5-10 \( \mu \text{m} \).

Mylonites

Two of the Norwegian aggregates belong to the group of cataclastic rocks exhibiting ductile deformation. This group, which includes rock types such as protomylonites, mylonites and ultramylonites, is classified by Higgins\(^1\) as rocks with fluxion structure, where reduction in grain size by movement (cataclasis) dominates over mineral growth (neomineralisation). One of the samples tested is classified as an ultramylonite in this scheme, and shows the presence of relatively small porphyroclasts of feldspar surrounded by a fine grained granular quartz matrix in which the quartz has strain induced subgrains with a mean grain size at approximately 10 \( \mu \text{m} \). The second cataclastic aggregate is classified as a mylonite with a mean grain size of quartz of approximately 40 \( \mu \text{m} \). This rock type exhibits some strong micro-folding with layers of secondary mica. Some carbonate minerals are also present.
Metagreywacke

The aggregate obtained from the concrete cores from Maentwrog dam in Wales, is as a meta-greywacke, composed of clastic material including quartz, feldspar, and granitic fragments in the size range of 0.30-0.15 mm. The rock is characterised by signs of low grade metamorphism with secondary mica formation, and the matrix occasionally exhibits microcrystalline recrystallised quartz. Some grains show subgrain structure with obvious strain lamellae. These are interpreted as layers one or two micrometers thick which contain clusters of crystal dislocations. The rock is reported to show slow/delayed alkali-aggregate reactive after some decades of service life.

Microgranite

The third Norwegian sample is a microgranite, with uniform quartz grains (170µm across) exhibiting no obvious signs of deformation or subgrain development. The feldspars here exhibit some sericitization so that small secondary quartz grains are probably present within the alteration product.

Mortar Bar Expansion

The varied average expansion for the different sets of mortar-bars (Fig. 2), all show an induction phase of 3 or 4 days before the main expansion starts. The meta-argillite and the ultramylonite exhibit the highest main expansion, while the mylonite and the metagreywacke show a slower development, though the main phase for mylonite lasts longer than that for the metagreywacke. The microgranite, which was considered to be innocuous before testing because of the relatively coarse quartz grains, exhibits unexpected slow and gradual expansion. The limestone, as expected, exhibits no expansion at all.

The percentage expansion for the metagreywacke aggregate is compared with that of the concrete bar in Figure 3. The mortar bars exhibit a higher percentage expansion during the first 18 days of exposure but after that the concrete exhibits a faster expansion rate, and reach a higher total expansion than the mortar-bars.
Fig. 2. Average percentage expansion for all the different types of aggregate.

Fig. 3. Average percentage expansion of the mortar-bars and the percentage expansion of the concrete-bar, both made with the greywacke aggregate.
The sequential development of the reaction

Thin sections made of mortar bars of the different types of sample at all the different test ages, and samples of polished and fractured surfaces of pieces of two selected ages, were used to examine the sequential development of the reaction at the reaction site. A summary of the various observations at different ages is given in table 1.

Meta-argillite

Observations in thin section, show microcracks in the outer parts of the meta-argillite after 4 days. These occur in particles near the surface of the bar. At the same time small amounts of gel appear in air-voids. In the 14 days old mortar these cracks are more open (Fig. 4), and the cracks begin to make connection between aggregate particles. More alkali-silicate gel is observed lining the air voids. In the thin sections made of mortar after 28 and 56 days no further structural changes are observed, but the amount of gel increases. In the polished SEM section, made from 14 days old mortar, well-developed cracks in aggregate particles are observed. Some particles show cracks connecting with air-voids and these voids are lined with gel (Figs. 5 and 6).

Fig. 4. Thin-section micrograph showing micro-cracks in an argillite particle in 14 days old mortar-bar specimen (Plane polars).
In the thin-sections from the 4 and 14 days old mortar-bar made of the ultramylonite aggregate, single cracks are observed in aggregate particles near the edge of the mortar-bar cross-section. Some gel occurs in the 4 days specimen, and the amount of gel lining air-voids increases significantly after 14 days. After 28 days particles near the edge of the cross section start to exhibit typical dissolution structures and aggregate particles closer to the middle of the cross section, begin to develop single cracks. No obvious increase in reaction product is observed in the sample of this age. After 56 days aggregate particles all over the cross section show quartz dissolution structures along with single microcracks. Particles showing the dissolution structures, have outer rims that are not affected by the dissolution, and aggregate particles dominated by dissolution tend to show no cracking extending into the paste. The reaction appears to be associated with microcracks and dissolution along the
boundaries of microcrystalline and subgrained quartz. The cracks do not necessarily follow the fluxion structure of the mylonite, and are occasionally observed normal to the fluxion structure. Aggregate particles below the size of approximately 0.30 mm appear not to participate in the reaction. There is little increase in the amount of gel reaction product after 56 days, but at this stage brownish reaction products are also present. Specimen of polished surface of the 56 day old sample shows dissolution on the surfaces of 5-10 µm quartz grains (Figs. 7 and 8). The energy dispersive analysis of spots between the quartz grains shows incipient gel development. The composition of this early gel-formation was found to be 91.0%SiO₂, 6.3%CaO, 1.5%Na₂O and 0.7%K₂O but this composition includes much quartz and the gel zone is only a few micrometres thick.

Figs. 7, 8. SEM micrographs showing dissolution structures around microcrystalline quartz grain boundaries in the ultramylonite aggregate, in 56 days old polished section of the mortar-bar specimen.
**Mylonite**

In the thin section from the 4 day old mortar-bar made of the mylonite aggregate only traces of gel reaction products are found. In the 14 day old specimen, a few microcracks appear in particles near the edge of the cross section of the bar and gel reaction products begin to coat air voids. As for the other samples, cracks are occasionally observed normal to the fluxion structure of the mylonite. After 28 days the situation has changed significantly. Both microcracks and dissolution structures are then found all over the cross-section and after 56 days these effects have increased. For this sample, it also appears that aggregate particles below the size of approximately 0.30 mm do not participate in the reaction. As shown in Figures 9 and 10, the reaction after 56 days is associated with dissolution around the subgrain boundaries of quartz. Other parts of the same aggregate particle exhibiting undulatory extinction show very little signs of reaction.

**Metagreywacke**

In thin sections of mortar-bars with the metagreywacke aggregate, traces of gel reaction products are observed in the 4 day old specimen. The amount is found to be higher in the 14 days specimen. The first affects of the reaction in aggregate particles are seen after 28 days when a few particles exhibit microcracks between quartz grains. In the 28 days specimen a significant increase in amount of gel reaction product is observed and is associated with brownish reaction products. After 56 days, much more dissolution is observed, the amount of gel increases and microcracks start to link aggregate particles.

**Microgranite**

No affects of reaction are observed in aggregate particles in the thin section of the 4 day old mortar bar made with the microgranite aggregate and no traces of gel are found in air voids. After 14 days a few dissolution structures occur along quartz grain boundaries and some traces of gel are found in air voids. The dissolution structures are more marked in the thin section of the 28 day old bars, where more gel is observed in air voids. After 56 days the dissolution structures have increased further, with increase amounts of gel and the brownish reaction products. The feldspar grains are not affected (Figs. 11 and 12) and no microcracks are observed in aggregate particles. The reaction for this material appears to be associated with dissolution around grain boundaries of relatively coarse quartz grains.

**Limestone**

No affects of reaction or differences in properties are observed for all the thin-sections of different test ages of mortar-bars made of the Limestone aggregate.
Fig. 9. Thin-section micrograph showing dissolution structures in subgrained part of mylonite particle in 56 days old mortar-bar specimen (Plane polars).

Fig. 10. Same as above. Note the dissolution structures are associated with the right part of the particle which shows subgrain development, while no dissolution structures are observed in the left part of the particle which shows strong undulatory extinction (Cross polars).
Fig. 11. Thin-section micrograph showing dissolution structures around relative coarse grains of quartz in micro-granite particle in 56 days old mortar-bar specimen (Single polars)

Fig. 12. Same as above. The grains of feldspars show no sign of dissolution structures (Cross polars).
### Table 1. Summary of the reaction development observed in thin-sections at different test ages.

<table>
<thead>
<tr>
<th>Aggregate types</th>
<th>Observations of reactions in thin-sections, after certain test ages.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>4 days</td>
</tr>
<tr>
<td>Meta-argillite</td>
<td>Micro-cracking starts in outer part of particles, in the edge of the cross-section.</td>
</tr>
<tr>
<td>Ultramylonite</td>
<td>A few micro-cracks appear near the edge of the cross-section.</td>
</tr>
<tr>
<td>Mylonite</td>
<td>Slight micro-cracking starts near the edge of the cross-section.</td>
</tr>
<tr>
<td>Greywacke</td>
<td>Dissolution starts to occur.</td>
</tr>
<tr>
<td>Microgranite</td>
<td>Dissolution starts</td>
</tr>
</tbody>
</table>

### The composition of the gel reaction products

The mean compositions and ranges encountered for the reaction products for different 14 days old mortar bar samples are given Table 2. It was possible to measure only gel reaction products lining air voids. Sufficient amounts of reaction products were not found in cracks in aggregate particles to provide unambiguous analyses. All gel analyses give a high content of CaO, while the K₂O contents are low. These compositions can be compared with the sequence of gel analyses obtained from the reactive silica glass in other experiments which show a progressive change in composition from the aggregate into the binder. These analyses, also given in Table 2, suggest that some reaction with the binder has occurred for the gels in voids in the present samples.
Chapter 7 - Paper 3

The composition of the analysed gel reaction product from the old concrete core, containing the metagreywacke aggregate, is given in Table 3. In this specimen it was possible to recognise sufficient gel in both cracks in the aggregate particles and in the air-voids for analysis to be possible. The composition of the gel in the old metagreywacke concrete, shows a relatively low CaO and Na2O content, in comparison to the metagreywacke mortar bar composition, and much higher K2O.

Table 2. Gel compositions
Gel compositions in 14 days old mortar bars obtained by SEM/EDX analysis.

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Meta-argillite</th>
<th>Ultra-mylonite</th>
<th>Mylonite</th>
<th>Greywacke</th>
<th>Micro-granite</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO2</td>
<td>63.3 ± 0.8</td>
<td>64.9 ± 1.3</td>
<td>69.9 ± 4.6</td>
<td>59.1 ± 0.6</td>
<td>74.6 ± 1.9</td>
</tr>
<tr>
<td>CaO</td>
<td>24.9 ± 1.4</td>
<td>25.9 ± 2.5</td>
<td>18.8 ± 4.6</td>
<td>32.9 ± 1.0</td>
<td>16.2 ± 1.9</td>
</tr>
<tr>
<td>Na2O</td>
<td>8.4 ± 0.5</td>
<td>7.7 ± 1.7</td>
<td>9.2 ± 1.1</td>
<td>6.0 ± 0.3</td>
<td>5.6 ± 0.5</td>
</tr>
<tr>
<td>K2O</td>
<td>1.9 ± 0.1</td>
<td>1.3 ± 0.1</td>
<td>0.7 ± 0.2</td>
<td>1.0 ± 0.0</td>
<td>1.8 ± 0.2</td>
</tr>
<tr>
<td>Na2Oeq</td>
<td>9.6</td>
<td>8.6</td>
<td>9.7</td>
<td>6.7</td>
<td>6.8</td>
</tr>
</tbody>
</table>

Gel compositions associated with silica glass

<table>
<thead>
<tr>
<th>Oxide</th>
<th>In aggregate</th>
<th>Aggregate surface</th>
<th>In paste</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO2</td>
<td>63.8</td>
<td>58.3</td>
<td>49.1</td>
</tr>
<tr>
<td>CaO</td>
<td>13.8</td>
<td>27.5</td>
<td>36.6</td>
</tr>
<tr>
<td>Na2O</td>
<td>0.7</td>
<td>0.6</td>
<td>0.3</td>
</tr>
<tr>
<td>K2O</td>
<td>19.5</td>
<td>12.1</td>
<td>10.9</td>
</tr>
<tr>
<td>Na2Oeq</td>
<td>13.6</td>
<td>8.6</td>
<td>7.5</td>
</tr>
</tbody>
</table>

Table 3. Gel composition for concrete sample (metagreywacke aggregate) obtained by SEM/EDX analysis.

<table>
<thead>
<tr>
<th>Oxide</th>
<th>In cracks</th>
<th>In air-void</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO2</td>
<td>70.1 ± 1.2</td>
<td>63.2 ± 0.2</td>
</tr>
<tr>
<td>CaO</td>
<td>15.0 ± 0.3</td>
<td>17.6 ± 1.3</td>
</tr>
<tr>
<td>Na2O</td>
<td>0.6 ± 0.2</td>
<td>1.9 ± 0.5</td>
</tr>
<tr>
<td>K2O</td>
<td>12.4 ± 0.8</td>
<td>16.8 ± 0.8</td>
</tr>
</tbody>
</table>
Quantitative aspects of mobilised silica and gel reaction product

The amount of SiO₂ extracted from the different types of powdered mortar, after 5 and 20 minutes in the NaOH solution, is summarized in Figure 13. The average amount of extracted SiO₂ after 5 and 20 minutes, after subtraction of the amount of silica extracted from the limestone mortar-bar, were used as measurements to indicate the possible gel content. The equivalent values for the percentage of gel in the different mortar bars are given in Table 4. This calculation is based on the silica content measured in the gels and excludes water.

Fig. 13. The extracted amount of SiO₂ (mg/litre) for various samples after 5 and 20 minutes.
Table 4. Quantitative aspect of the mobilised silica extracted after 14 days for different types of aggregate. The amount of silica extracted from the limestone mortar bar has been subtracted. The results are expressed as weight percentage of the original powdered mortar.

<table>
<thead>
<tr>
<th></th>
<th>Meta-</th>
<th>Ultra-</th>
<th>Mylonite</th>
<th>Meta-</th>
<th>Micro-</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>argillite</td>
<td>mylonite</td>
<td>greywacke</td>
<td>granite</td>
<td></td>
</tr>
<tr>
<td>Extracted (mean) SiO$_2$ (mg/l)</td>
<td>48</td>
<td>152</td>
<td>56</td>
<td>48</td>
<td>62</td>
</tr>
<tr>
<td>Mobilised SiO$_2$/mass of mortar</td>
<td>0.030 %</td>
<td>0.095 %</td>
<td>0.035 %</td>
<td>0.030 %</td>
<td>0.039 %</td>
</tr>
<tr>
<td>Equivalent gel/mass of mortar</td>
<td>0.050 %</td>
<td>0.150 %</td>
<td>0.050 %</td>
<td>0.050 %</td>
<td>0.050 %</td>
</tr>
</tbody>
</table>

Discussion

Magnitude of expansion

It was expected that the silica glass aggregate would show the greatest expansion, that the limestone and microgranite would be inert, and that the other lithologies would be found to be reactive after 14 days. The boundary between innocuous and reactive was taken to be 0.1% after 14 days. Both the limestone and microgranite have been found to be innocuous from field experience.

The separation of reactive and innocuous aggregates has been made at different expansion limits after 14 days in the literature using this accelerated test. There is an agreement that the limits must always be decided in relation to known field performances for the tested aggregate and this experience suggests that the commonly used expansion limit of 0.10% after 14 days, now widely accepted and included in standard procedures$^{11}$, is safe or even conservative. Hooton 1991$^{12}$ following extensive testing of Canadian aggregates has proposed a limit of 0.15%.

The results of the present study show that there can be an induction phase in which there is little expansion. Hence while the usual expansion limit seems adequate for identification of the reactive aggregates with a relatively short induction phase, for aggregates with a longer induction phase and a delayed but relatively high later expansion, this limit might not give an accurate long term prediction of the reactivity of the aggregate. This is the case for example for the mylonite aggregate, which at 14 days shows an expansion near the 0.1% limit, but later shows a high expansion rate. The same uncertainty occurs for the metagreywacke aggregate where the field performance is known. This aggregate, with an expansion of 0.10% after 14 days, would have been classified as on the limit between marginal reactive or innocuous. Previous tests of this aggregate gave expansion of around 0.17% and the field record
of exactly the same aggregate, showed substantial damage after several decades of service. The separated aggregate might not of course correspond fully with that generating the reaction in the field because it represents a small amount of rock taken from existing concrete. These results introduce potential difficulties in the use of the mortar-bar test in prediction of reactivity, and in particular making estimations of the predicted service-life for real structures. The reason for the induction period is not clear but it could relate to the experimental conditions with the volume ratio of alkaline solution to bar volume being critical. The influence of the quality of the mortar on the rate of alkali ingression could also be important and it is evident that reaction and crack generation began in the outermost part of these bars. It is therefore concluded that the test may need some further investigation but that if the limit of 0.1% expansion is taken as the boundary between innocuous and reactive then it is prudent to take the period of the test as beginning at the time that expansion commences. Alternatively, the test could be continued to 28 or even 56 days as recommended by Hooton 199112.

Aggregate grading

The mortar bar test requires a specific grading of the aggregate. Various researchers have observed that for slowly reactive aggregates, certain aggregate sizes in particular contribute more to the deleterious reaction than others. Lagerblad and Trägårdh13 observed that in an accelerated expansion test the smaller particle sizes caused more expansion than the larger. This contrasts with what they found in reacted concrete from real structures. Also, Jensen14 reported that in Norwegian structures suffering from AAR, reacted aggregates have only been observed in the coarse aggregate and in the coarser sand fraction (>1-2 mm), while in the NBRI mortar-bar test with similar aggregates, all the particle sizes seemed to react. However, for most of the samples in the present study, it was observed that no reactions occurred below a particle size, of approximately 0.30 mm.

The observations of the greywacke aggregate in this study, in the freshly made mortar bars and in the old concrete bar, clearly showed a different expansion development. The coarser aggregate particles in the concrete bar ultimately gave a higher total expansion than the mortar bars. The expansion of the concrete bar also demonstrated the possibility that concrete originally reacting in a structure, might react again if additional alkalies are made available. As mentioned in an earlier investigation by Brown15, this implies that the reaction could be continued in the original structure, provided that the supply of alkalies was not exhausted or alkalies were added to the structure.

It is an interesting question as to why smaller slow/late reactive aggregate particles do not seem to take part in the reaction in the mortar bars or the real structures. As far as
the authors are aware, no attempts have been made to explain this observation. It is commonly found that more serious cracking relates to clustering of reactive aggregate particles, and many cracks are developed as a consequence of the accumulation of chains of reaction particles. Coarser particles create longer cracks running out into the cement paste from the aggregate than finer particles, and consequently contribute to longer interconnected cracks along chains of reactive clusters. The expansion forces required to crack the concrete might be dependent upon this accumulation of reactive particles of a particular size. In real concrete, coarser particles will also have access to a greater concentration of alkalies due to the low surface area to volume ratios than would a similar volume of small particles because of the lower surface area to volume ratio. This might not be the situation in accelerated tests where there is large excess of alkalies but it is also evident that larger pieces of aggregate will have a greater potential for carrying the minute amounts of actually reactive lithic material and a greater potential for internal crack development than the smaller grains.

Pattern of expansion

Oberholster and Davies\(^1\) and Grattan Bellew\(^{16}\) have divided the expansion in the accelerated mortar-bar test into three different phases; the induction phase, the main expansion phase and the late expansion phase. These phases will differ in duration for different types of reactive aggregates. Aggregate exposed for a long time in real structures follows the same pattern as aggregate in the accelerated mortar bar test even where the aggregate in the mortar bar is taken from comparable concrete. The present experiments show that different types of reaction take place with some aggregates showing mainly microcrack development while others show gel formation along the grain boundaries in composite quartz grains. Expansion seems to be related to microcrack development rather than total gel development. Hence the changing pattern of expansion could relate to the changing ability of the microcracking to generate expansive forces on the weakened mortar or, in the field, on the host concrete.

Consequently, it is of interest to assess the rate of expansion for different time intervals and in relation to the observed reaction development at the reaction site for different types of aggregates. The mean percentage expansions for different mortar bars for each day at different time intervals are therefore presented in Figure 14. When assessing the effect of time and rate of expansion, it is important to emphasise that signs of the reactions were first recognised in aggregate particles near the edge of the mortar bars cross-section and it appears that the reaction develops successively through the cross-section of the mortar bars.
All the investigated siliceous aggregates in this study showed signs of reactions but different signs were found at different test ages and at different locations in the mortar bars. In the early period from 0 to 4 days, the silica glass exhibited its highest rate of percentage expansion (0.063% each day and off scale in Figure 14). It appears that the main expansion phase for this material starts almost immediately. The meta-argillite and the ultramylonite exhibit their highest rate of expansion in the period from 4 to 7 days and microcracks are formed in the aggregate particles in this period. The peak rate of the expansion for the mylonite is in the period from 7 to 14 days. No affects are observed in the thin section after 4 days but after 14 days a few microcracks appear in particles near the edge of the cross section. The microgranite exhibit the highest rate of expansion in the period of 14 to 28 days. No cracking is observed in this material, but the sign of dissolution increases at 28 days. The expansion rate for metagreywacke remains steady from 0 to 14 days, without any defined peak of expansion. Signs of reaction in the thin section are first recognised after 28 days.
From these observations it appears that the highest rate of expansion is associated with the incidence of cracking rather than the dissolution. No cracks running into the cement paste were observed in association with the process of grain boundary dissolution. This echoes the relationships found in field concrete\textsuperscript{17} where much gel can be produced by grain boundary reactions without notable cracking appearing. In field concrete the gel produced by grain boundary reaction tends to spread along aggregate surfaces and may enter pores and voids while in other types of reaction the gel fills cracks inside the aggregate and issues into the binder along cracks which often begin at the corners or edges of aggregate fragments. Hence the expansion appears to relate mainly to crack opening with the expansive force exerted by the gel being confined to a narrow zone at the tip of the generating crack. The quality of the binder itself will also have an effect upon the rate of expansion. For an aggregate type with a high water demand (i.e. the mylonite with a high mica content) the mortar will show an increased volume of pore space. When the gel is generated this pore space may first be filled and hence expansion may be postponed.

*Reactivity of the various aggregates*

The reactivity of a specific rock type does not necessarily depend on the mineralogical or the petrographical nomenclature of the rock, but rather on the occurrence of special and perhaps localized textural and microstructural features of the materials. For slowly reactive aggregates, quartz always appears to be the reactive constituent. However, the nature of these quartz minerals varies significantly, both between different types of aggregates and within an individual aggregate. A relatively large grain boundary energy, has promoted alkali-aggregate reactions in the microcrystalline meta-argillite. This lead to the cracking of aggregate particles as observed in this study. For the coarser rock types, including the various kinds of metagreywackes and mylonites, stored strain energy, and subgrain development in particular, are probably necessary to promote the reactions. Subgrain development enhances the reactivity of a material through high dislocation density surrounding subgrain boundaries, which provide preferential sites for the inception of silica dissolution. The occurrence of subgrains may also promote the ability of the alkali-rich fluid to gain access to the reaction site. This process of dissolution was recognised in all the samples except for the argillite sample, and was in particular observed in samples from 28 days of ages. In field concrete these two processes are also observed\textsuperscript{9}, but it appears that the NBRI mortar-bar test, due to the severe conditions, enlarges the dissolution process. Kerrick and Hooton\textsuperscript{18} also examined thin-sections of specimens from the accelerated mortar bar test and in specimens 56 days of ages, they similarly found that signs of reaction are most abundant within foliated layers and at the grain boundaries of microcrystalline quartz.
The measurement of the undulatory angle of strained quartz has previously been used to a large extent in attempts to predict the reactivity of various aggregates. However, more recently it has been suggested that rather than the undulatory extinction of quartz, the reactivity may be due to the occurrence of microcrystalline and subgrained quartz\textsuperscript{19,20,21}. In this study the mylonite aggregate particles contain quartz which exhibits both strong undulatory extinction and subgrain development. No significant reaction could be associated with the undulatory extinction, even though the subgrain boundaries in the same particle showed severe dissolution.

In this study the quartz bearing microgranite was not expected to be reactive, due to the relatively coarse strain-free quartz grains. However, as has been suggested by Mather\textsuperscript{22}, all aggregates are potentially reactive, they vary only in the kind of reaction, including the degree and rate of the reaction. Even the quartz grains of the microgranite, under the severe condition of the accelerated test, show clear indications of some reaction and expansion, albeit at a very slow rate. For such an aggregate, containing only relative coarse quartz grains, the activation energy is higher, the available grain boundary surface area is lower, and consequently the generated reaction products and expansion forces lower than would occur in material of with larger reactive surfaces.

**The composition of the gel reaction product - regeneration of alkalies**

The occurrence of the gel reaction product is the main diagnostic symptom confirming that AAR has occurred. The gel may vary in elemental composition and properties. Shayan & Quick\textsuperscript{4} investigated the microstructure and composition of gel reaction products in samples from NBRI mortar-bar testing. Although expansion of a given specimen occurred much more rapidly under the NBRI mortar-bar test than under field conditions, they found that the reaction products appeared to be the same, indicating that reaction mechanisms were the same but differ only in rates. The gel reaction product observed in this study, in the specimens after 14 days exposure in the NaOH solution, revealed signs of reaction similar to those found in real structures. As expected, Na-rich gels develop in the NaOH solution. However, the high calcium content of the gel in the paste after only 14-days in the NaOH solution was not expected though Davies and Oberholster\textsuperscript{23} found such a high amount of CaO in 14 days old mortar bar specimens. Various authors\textsuperscript{13,14,24} following Power and Steinour\textsuperscript{25}, have observed the phenomenon of regeneration of alkalies during the alkali aggregate reaction. The alkali-silicate gel formed within the grains changes composition when it comes in contact with the paste, and becomes richer in calcium, and loses alkalies to the binder. The change in composition is caused by either dilution of the alkalies by a calcium compound from the cement paste, or by a cation exchange reaction where calcium replaces alkalies. The cation exchanges would make the displaced alkali available for further reaction so that alkali consumption is
minimized. Hence the reaction may theoretically continue until all reactive materials are transformed into gel and Lagerblad and Trígàrdh\textsuperscript{13} claimed that as a consequence there is no pessimum for slow/late expanding reactive rocks.

**Quantity of silica involved in the reaction**

No clear correlation has been found between the amount of gel and the expansion forces responsible for the deterioration of concrete\textsuperscript{26,27}. French\textsuperscript{28} claimed that in the case of aggregates including greywacke, siltstone or argillite, the observed amount of gel is low where greywacke is involved but can be high in the case of argillites. Deloye and Divet\textsuperscript{29} assessed the quantitative aspects of the gel reaction product regarding AAR. Their results, which were obtained on structures affected by AAR, show that the silica mobilised ranges from 0.2 to 1.0\% with respect to the total mass of concrete, depending on the severity of the reaction. Also French\textsuperscript{21} suggested that the substances taking part in the reaction are always a very small fraction of the rock. Even with severe cracking, it has been considered that not more than about 1.0\% of the silica in the rock would have been converted into gel. Furusawa et al.\textsuperscript{30} obtained similar values for the amount of reaction product in mortar-bar test using Pyrex glass, both as theoretically estimations, and as measured values.

In the present study the calculated amount of dissolved silica in 14 days old mortar-bars does not relate to the expansion after 14 days. The ultramylonite shows a significantly higher amount of dissolved silica, while the other aggregate types, all show similar values. Even the micro-granite with a low expansion after 14 days gave the same result as the three aggregates which produced the lower amount of soluble silica. By using an 80\textdegree\textcelsius NaOH solution to extract the gel reaction products, it is possible that some of the original silica in the form of microcrystalline quartz might be dissolved especially where the grinding process has reduced the particle size of the quartz. The values of extracted silica after 5 and 20 minutes of extraction showed some divergence and some of the samples showed a lower amount of silica when extracted for 20 minutes than after 5 minutes. Hence the results are regarded as giving only an indication of possible maximum levels of soluble silica produced in the test and they might be on the high side. They are consistent with the low amount of actually reactive material found for field concretes and in other experiments.

**The microstructure of the reaction product**

With time gel might develop different microstructural properties, which always appear to be related to an increase in calcium ions\textsuperscript{31}. Fournier and Bérubé\textsuperscript{32} investigated the microstructures of gel reaction products in SEM specimens obtained from the NBRI mortar-bar test. The well-formed classical rosette-like phase of gel
was not observed in specimens of 18 days old mortar-bars but in specimens of 80 days old mortar, air dried at room temperature for one year, the classical texture was observed. They suggested that the crystallisation processes for this phase are time dependent and possibly not involved in the short term expansion by the AAR. In the present study, examination of gel reaction products in SEM specimens of 14 day old mortar bars, revealed only the occurrence of massive gels. However, in specimens of 56 day old mortar, classical rosette-like phases could be observed in gel lining air void for all the different types of reactive materials.

Conclusions

* The NBRI mortar bar test seems satisfactory to echo the naturally occurring alkali-aggregate reaction, distinguishing between different types of reactive aggregate, and the properties of the gel reaction products. However, for extremely slow reactive aggregate, the accelerated test might show some limitations regarding the prediction of in-service performance.

* In assessing the potential for reaction of an aggregate under test it may be desirable to measure the expansion for a period following the inception period or to continue the test for 28 or 56 days where the initial test results might be regarded as ambiguous.

* Results from this study imply that slow/late expanding AAR could be continued in old structures, provided that the supply of alkalies is not exhausted or alkalies are added to the structure and the concrete retains sufficient structural integrity.

* Coarser aggregate particles seem to contribute more to the total expansion than finer for these late expanding aggregates.

* The rate of expansion differs in time for the different types of reactive aggregate. Two main processes seem to appear during the reaction; a process of dissolution, and a process of cracking which are mostly mutually exclusive. Even though the process of dissolution appears to produce the largest amount of the gel, the process causing cracking appears to contribute most to the expansion and relates most closely to the rate of expansion.

* The reaction appears to be associated with microcrystalline quartz, in particular with the occurrence of subgrain development. No significant signs of reaction could be associated with coarser quartz grains dominated by extensive undulatory extinction.
* Na-rich gel reaction products were observed in 14 day specimens, however, a high CaO content gives indications of the phenomenon of reaction with the binder and possible regeneration of alkalies.

* No relation was found between the amount of gel reaction products and the total expansion but the amount of silica extracted is in keeping with a very low proportion of actually reactive lithic material.

* Massive gel reaction products were observed using the scanning electron microscope in air voids in 14 days old specimens for all the different types of reactive materials. Classical rosette-like phases of gel reaction products were found in air voids in the corresponding 56 days old specimens.

Acknowledgements

The paper is a part of B.J.Wigums' PhD. Study, which is supervised by Dr. S.W.Danielsen. The study was supported financially by the Royal Norwegian Council for Scientific and Industrial Research. The authors wish to thank the staff and colleagues at the Geomaterials Unit, Queen Mary and Westfield College, University of London, and in particularly Mr. Stuart Adams and Mr. Chris Mole for assistance with the various measurements.

References


6. ANNUAL BOOK OF ASTM STANDARDS, Volume 4.02, Concrete and Aggregates, C227-90, Standard method for potential alkali reactivity of cement aggregate combinations, mortar bar metho, 126-130.


Chapter 8

Accelerated tests for the potential exhibited by aggregates for alkali-aggregate reactions

BØRGE J. WIGUM, WILLIAM J. FRENCH, COLIN D. HILLS and RICHARD J. HOWARTH


B.J. Wigum, Department of Geology and Mineral Resources Engineering, University of Trondheim, The Norwegian Institute of Technology, 7034 Trondheim, Norway

W.J. French, Geomaterial Unit, School of Engineering, Queen Mary and Westfield College, University of London, Mile End Road, London E1 4NS, UK

C.D. Hills, Concrete Durability Group, Department of Civil Engineering, Imperial College, University of London, London SW7 2BU, UK

R.J. Howarth, Department of Geology, University of London, Gower Street, London WC1, UK
Accelerated Tests for Assessing the Potential Exhibited by Concrete Aggregates for Alkali-aggregate Reaction

B. J. Wigum, *a W. J. French, b R. J. Howarth c & C. Hills, d

a Department of Geology and Mineral Resources Engineering, Norwegian Institute of Technology, University of Trondheim, Trondheim, Norway
b Geomaterials Research Services Ltd, 1 Falcon Park, Basildon SS14 3AL, UK
c Department of Geological Sciences, University College London, Gower Street, London WC1E 6BT, UK
d Department of Civil Engineering, Imperial College, London SW7 2AZ, UK

Abstract

A number of tests are available for assessing the potential of aggregates for alkali-aggregate reaction. These are listed, for example, in ASTM C33-93. The majority of these test procedures have been widely used in researching aggregates, but there are serious reservations concerning the application of some of these tests to the selection of materials for structures. In recent years, many papers have been published detailing investigations of the tests. An examination of the literature shows that most of the tests are of little value for making engineering decisions. This paper reviews the literature relating to three commonly used tests, the ASTM C-289 Rapid Chemical Test, the ASTM C-227 Mortar Bar Test, and what has become ASTM C-1260-94 Accelerated Mortar Bar Expansion Test. Attention has been given to a detailed practical study of the accelerated mortar bar tests since these are becoming very widely used. The experimental work, in essence, represents a determination of the errors to be expected from application of the test to a single aggregate and the factors that influence these errors. The repeatability of the tests suggests an upper bound standard deviation of 0.0365% expansion. This figure is used to establish discriminant criteria for the recognition of potentially damaging materials. It is evident that diagnosis of potentially highly reactive materials can be made with confidence, but that a large number of tests are required to assess marginal types. Petrographic evaluation of the mortar following the test is essential, particularly where the results are borderline, though the presence of traces of gel has to be interpreted with caution. Much of the uncertainty in the test results derives from inherent aggregate variability. A test schedule can be designed that has small contributions to the error from the experimental steps, but the error due to aggregate variability remains. This can lead to the requirement for the manufacture of multiple batches of mortar for establishing acceptance criteria for even a single aggregate batch, and further consideration is needed as to the procedures necessary for the assessment of aggregate sources. © 1997 Elsevier Science Ltd. All rights reserved.

Keywords: ASR, accelerated tests, mortar bar test, repeatability, petrographic evaluation, acceptance criteria.

INTRODUCTION

From the engineering point of view, it is essential that tests are available for the detection of potentially reactive aggregates that are reliable and capable of giving a measure of the probability that the aggregate will be innocuous in service. Many of the tests currently in use are possibly suitable for research purposes but do not provide an adequate basis for discrimination in the selection of aggregate for construction. This paper reviews some of the more commonly used tests from the engineering
standpoint and describes an investigation into the factors influencing expansion in one of the preferred accelerated mortar bar tests. The repeatability of the method is also assessed as is the definition of limits providing an acceptable probability that aggregates will be appraised correctly.

The test explored in detail is based on the original work of Oberholster and Davies, which was designed to discriminate between innocuous and deleteriously expansive reactive aggregates when they are used in concrete. The procedure that we have used is similar to that now adopted as the recent designation ASTM C-1260-94 and the Canadian Standard A23.2-25A, which was published in June 1994.

Since Stanton characterised the expansive and damaging reaction between alcalies in the pore fluid of concrete and aggregate, numerous tests have been devised in an attempt to discriminate between supposedly potentially reactive and innocuous aggregates. This discrimination presumably ought to mean potentially reactive or potentially innocuous under the most severe conditions imaginable, though many tests specify particular compositional and environmental parameters that are not necessarily the most severe that could be applied. Some argue that all aggregates should be regarded as potentially reactive if the conditions are sufficient aggressive. There are good geological reasons for supposing that any aggregate source can contain, at some stage in its exploitation, unstable lithologies. The amount of material actually reactive is likely to be less than 1% by volume of the aggregate in even the most severe examples, and will be unevenly distributed. It is also to be expected that some potentially harmful components when present in particular concentrations will, in practice, produce no damage in service.

Early, if not the first tests, are those introduced by ASTM. In 1950, the mortar bar test ASTM C-227 was introduced, and 2 years later, the chemical method ASTM C-289 was added. Both take advantage of ASTM C-295 for the petrographic examination of aggregates, which was also first issued in 1952. Except for minor modifications, these tests remain in the current book of standards. The limitations of these procedures are shown, however, by the publication of some 25 papers on testing that appeared in the proceedings of the 8th International Conference on Alkali-Aggregate Reaction in Kyoto in 1989, and about 35 papers were published in the proceedings of the 9th International Conference held in London in 1992. The introduction of the accelerated mortar bar test ASTM C-1260 in 1994 provides further evidence of the deficiencies in the previous standards and the need for improvement. There are also various tests based on concrete prisms that can test the aggregate in the size ranges to be employed in practice. Of these, perhaps that most widely employed is the Canadian standard. However, concrete prism tests require at least a year for their completion, and there is evidence that a much longer period may be required, especially for some slowly reactive aggregates.

What is needed, therefore, is a practical test that allows defective materials to be identified under the most extreme conditions likely to be encountered in service and that can be carried out in a time scale that is appropriate to construction planning. Each such test also requires definition of criteria for the necessary discrimination to be made under the given environmental conditions. This implies a need for statistical knowledge of the errors attached to each method and the definition of the probability of avoiding damage at a level that the user will find acceptable. In general, the amount of information on the repeatability and reproducibility of the standard procedures is meagre, and the impact of poor reproducibility on the interpretation of test results is rarely considered. In some cases, including some standards, the repeatability of the tests is not defined or considered. A range may be stated without explanation as to its meaning. For the present work, the definitions used conform with current BSI and ISO practice and are as follows.

Standard deviation: \( s = \sqrt{\frac{1}{n-1} \sum (x-\bar{x})^2} \), where \( n \) is the number of measurements of a value and \( x \) the values obtained.

Standard error:

\( s/\sqrt{n} \)

Repeatability:

\( r = 1.96s/\sqrt{20} \), where \( r \) is a measure of the random error occurring once in 20 measurements and associated with one operator obtaining successive
results by the same method applied to the same material.

Reproducibility:

\[ R = 1.96 \sqrt{\bar{s}_1^2 + \bar{s}_2^2}^{0.5}, \]

where \( \bar{s}_2 \) is the contribution to the variability due to test operators working in different laboratories, \( \bar{s}_1 \) is within lab variation, and \( R \) is a measure of the random error occurring once in 20 measurements with the operators using the same method and materials. \( R \) is typically about one and a half to double the value of \( r \).

**ASTM C-227 mortar bar test**

The ASTM C-227\(^7\) Mortar Bar Test has been widely used, though it may require a year or more for completion. The mortar bars are made with cement that has more than 0.60% by weight of alkali as Na\(_2\)O equivalent, and the bars are stored over water at 38 ± 1.7°C, presumably meaning that the full range must be kept within these limits or the test result rejected. ASTM C-33-93\(^{13}\) specifies that the alcalies should be substantially above 0.60% and preferably about 0.8% as Na\(_2\)O equivalent, but difficulties arise because this level of alkalies is not rigorously defined so that expansion could relate to the selected alkali level. The expected alkali levels in the eventual concrete are not related to the test method or result. This standard also states the following:

While the line of demarcation between non-reactive and reactive combinations is not clearly defined, expansion is generally considered excessive if it exceeds 0.05% at three months and 0.10% at six months. The expansion is measured to the nearest 2 micrometres. All measurements should be within ±0.003% of the mean or the range of results should be ±15% of the average if the mean results are 0.02% or higher.

Presumably, the ranges given mean that the result is rejected if it falls outside the range. At 0.10%, the expansion is about 250 \( \mu \)m, and the acceptable range is therefore ±38 \( \mu \)m. The discriminant criterion is therefore 0.10% with a standard deviation of about 0.002% expansion.

<table>
<thead>
<tr>
<th>Mean expansion</th>
<th>Standard deviation</th>
<th>Reproducibility (% expansion)</th>
<th>Number of laboratories</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.013</td>
<td>0.005</td>
<td>0.015</td>
<td>5</td>
</tr>
<tr>
<td>0.015</td>
<td>0.003</td>
<td>0.008</td>
<td>5</td>
</tr>
<tr>
<td>0.024</td>
<td>0.015</td>
<td>0.041</td>
<td>5</td>
</tr>
<tr>
<td>0.026</td>
<td>0.025</td>
<td>0.073</td>
<td>5</td>
</tr>
<tr>
<td>0.049</td>
<td>0.055</td>
<td>0.154</td>
<td>5</td>
</tr>
</tbody>
</table>

This exacting repeatability is unlikely to be achievable in practice. The standard does not provide data on the repeatability of the test or its reproducibility. Sorrentino et al.\(^{14}\) showed that the repeatability of the test can be as low as ±0.02% expansion at an expansion of 0.28%, i.e. more than ten times that required by the standard. Moreover, Oberholster\(^{15}\) quotes various published researches and an inter-laboratory study that show that known reactive aggregates are not detected by the test. He also shows that the reproducibility of the method from the inter-laboratory study is highly variable (Table 1) with means and standard deviation depending on the alkali content of the cement.

The number of laboratories involved in this test was small, but these results clearly show a variation that indicates that the reproducibility worsens at a higher rate than the rate of increase in expansion, and the magnitude of the reproducibility suggests that the analytical measurements may not be normally distributed. To define a limit of 0.10% expansion means that the result might need to be recorded as, at best, 0.10 ± > 0.10 given the data of Table 1. The test will have to be repeated many times for a statistically valid discrimination to be made. This result alone shows that the test leaves much to be desired from a practical engineering point of view. The reason for the large standard deviation is discussed later, but one feature may be that the laboratories used slightly different methods.

Ranc et al.\(^{16}\) discussed in detail earlier reservations with respect to the efficacy of this test and confirmed the reservations by showing that six aggregates known to be reactive in service were not detected at all as reactive by the test. These aggregates gave a mean expansion of 0.029 ± 0.009% after 6 months and 0.029 ± 0.017% with no aggregate reaching
0.10% after 12 months. These results echo the work of Shayan et al. Rogers and Hooton also found the procedure to be unsatisfactory and explained the lack of expansion as due to leaching of alkalies by condensation on the surface of the mortar bars. Sorrentino et al. considered that the lack of expansion might, for some reactive aggregates, be due to the removal of aggregate below 0.15 mm as required by the standard. The procedure was improved by Ranc et al. by increasing the alkalies to 1.25% as Na₂O equivalent by weight of cement and by preventing condensation. Nevertheless, the test again failed to detect the reactive aggregates, and Ranc et al. discuss various reasons for this behaviour.

This test therefore is clearly unacceptable for assessment of a range of aggregate types and in particular may not be suitable for discrimination of the aggregates that generate slow or delayed expansion. There is also a need for more careful definition of the experimental conditions. Doubt must be attached, for example, to the statements defining the required alkali concentrations in the test. The results obtained may only be acceptable if it can be shown that the range of lithologies and microtextural features present in the rock are the same as those for which the test has proved to be satisfactory in the past. In the absence of suitable data for the reproducibility of the method, the reported failure to detect a wide range of potentially damaging aggregates, and the length of time required for the test, it can be difficult to interpret the observed magnitude of expansion in the test in terms that are useful in any way from the engineering point of view. The test is unfortunately still specified but should be abandoned.

ASTM C-289 chemical test

This test has been widely used, often in combination with the C-227 mortar bar test. It was designed specifically for testing rocks that are mainly silica, and is inappropriate for rocks containing carbonates, serpentine minerals, and probably for a wide range of rocks that are impure silicate lithologies. Grattan-Bellew regarded this test as superseded, and Ranc et al. have shown that the test is unreliable in that two of six aggregates known to be reactive in service were not identified as such. Figure 5 of Katayaki et al. suggests that some 10% of aggregates may be identified incorrectly as reactive, while a smaller percentage is found to be innocuous by this test but reactive with the mortar bar test. Fournier and Berube and Barisone and Restivo have used acid treatment to remove carbonates in order to improve the discrimination effect by the procedure.

From an engineering point of view, the poor precision attached to the likely behaviour of the aggregate in this test must be regarded as unacceptable. In the authors’ experience, there are two main problems associated with the test. The first of these is its widespread use on rocks for which it was not designed, and the second is the influence of minor or trace amounts of rock that are not in the specified size range for the test. In particular, it is sometimes necessary to wash the aggregate many times before traces of turbidity produced by suspended rock particles are removed. The presence of very finely divided siliceous material is considered likely to have a significant affect on the result of the test and can lead to rocks giving results that are indicative of potential for deleterious behaviour. Conversely, Sorrentino et al. point out that removal of the finest fraction could mean removing the reactive fraction, and they have substantially modified the test to allow the fine fraction to be included. Their modifications mean that the test must be carried out, possibly repeatedly, over a few weeks and that both very high and very low results can result from aggregate considered to be innocuous.

No precision trials have been reported for this test, and since no controls are available, the bias of the method cannot be assessed. According to the standard, replicate results are considered to be satisfactory if they fall in the range ±12 mmol/l when the test result is 100 mmol/l or less for the two variables measured, and 12% where the measured values exceed 100 mmol/l. Presumably, tests giving results outside this range are regarded as invalid. This means that there must be a broad band of uncertainty along the defined discriminant boundary. The standard also makes it plain that the result of the test does not predict the reactivity of slow–late expanding aggregates such as those ‘containing strained quartz, microgranulated quartz, or aggregates composed of meta-greywacke, metasiltstone, metaquartz and similar rocks’. There are, therefore, many conditions that must be met before the test result can be of value for practical purposes. Only
results indicating that the aggregate is strongly reactive are meaningful because of the uncertainty of the discriminant boundary.

**ACCELERATED MORTAR BAR TESTS**

**Alkali immersion methods**

Numerous accelerated mortar bar tests have been developed, most of which centre on the procedure published by Oberholster and Davies, which is known as the NBRI method. In this procedure, the mortar bar is immersed in 1 M NaOH solution at 80°C. In a comparison of several test procedures, Hooton and Rogers found the NBRI method to be the most effective. The version of the NBRI test now adopted as ASTM C1260-94 has been tested as the ASTM provisional specification P-214. Bérubé et al. has pointed out that this test may be too severe in that some innocuous aggregates have been found to react expansively. Conversely, it is reported by Hooton that Stark found some reactive aggregates that are not detected. Possibly, these differences reflect slight differences in the way in which the test is carried out, as discussed below.

There is now a great deal of published information on tests of this type, which shows that the test detects reactive aggregates satisfactorily, particularly if the test bars are examined microscopically at the end of the test, and if the test is carried out for up to 56 days. Some data on the reproducibility of this type of test were given in Hooton. The test used by Hooton was based on the NBRI specification but is quoted in ASTM C-1260 and was based on the work of six laboratories using three aggregates and three cements. The inter-laboratory precision was found to correspond with a coefficient of variation of 9.55% (one standard deviation) when the expansion was greater than 0.15%. More recently, more detailed studies of the repeatability have been published, including a very detailed interlaboratory study published by Rogers et al. for one aggregate that shows that the reproducibility of the test is superficially rather poor. These studies demonstrate a need for further research and more careful definition of the experimental parameters and of the relationship between the test result and the cause of the expansion. Hooton has published an account of the application of this test to a large number of aggregates with known service records. The test has also been adopted in Canada as CSA A23.2-25A and the Aggregate Committee of the Norwegian Concrete Society has also proposed a voluntary arrangement for the declaration and approval of aggregates used in concrete, which includes a slightly modified version of the NBRI test. A proposal for a new Rilem standard currently under consideration is also based on this method.

It is this alkali immersion test that has therefore been chosen for the investigation reported here. No firm criteria have yet been established for the method, and slightly different procedures and criteria are recommended by the differing authorities. It is highly desirable that the minor differences between the various test specifications should be eliminated and that firm criteria should be established for the discrimination of innocuous and reactive materials. The aim of the present work is to examine the factors influencing the results obtained in the test and to discuss the precision and variation of the results obtained. Variables studied in the present work that can influence the results obtained are the size of the mortar bars, the type of studs embedded in the ends of the bars, the precision of the aggregate grading, the ratio of the volume of alkali to the surface area of the mortar, and the cement type employed. It is difficult to assess such factors unambiguously because of the apparently poor precision of the method. The tests were carried out independently by two separate laboratories, with one laboratory running all the tests at essentially the same time, while the other carried out the tests one at a time. Details of the test procedure used in the present study are summarized in Table 2. Some of these steps are more stringent than is necessary but were required for the evaluation of details of the procedure.

**Enhanced alkali method**

In these procedures, alkalis are added to the mortar during the mixing stage. A number of variants of the method have been developed in which the mortar is treated at an increased pressure in an autoclave. The use of increased pressure and temperature means that results can be obtained in a few days or even a few hours in some instances. Following the work of Tang, Fournier et al. have reviewed the procedures for rapid autoclave methods and
Table 2. Basic procedure used in the Mortar Bar tests

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Grade the aggregate to produce the grading curve specified in ASTM C227-94.</td>
</tr>
<tr>
<td>2.</td>
<td>Blend 800 g of cement with water to produce a water/cement ratio of 0.45 (crushed aggregate).</td>
</tr>
<tr>
<td>3.</td>
<td>Blend into this mixture 2000 g of the aggregate to be tested and mix for 2 min.</td>
</tr>
<tr>
<td>4.</td>
<td>Cast four mortar bars, filling in two or three stages and using vibration after each stage. (The bars used measured 160 × 40 × 40 mm or 250 × 25 × 25 mm.)</td>
</tr>
<tr>
<td>5.</td>
<td>Cover with polythene and cure in a sealed container containing water for 24 h with the mould mounted above the water and with the inner sides of the container being coated with cotton fabric that extends into the water.</td>
</tr>
<tr>
<td>6.</td>
<td>Carefully remove the bars from the moulds and place in water in a sealed polypropylene box. The water must cover the bars by at least 3 mm. Thin plastic rods or a corrugated plastic sheet are placed beneath the bars.</td>
</tr>
<tr>
<td>7.</td>
<td>Place the box in an oven and bring the temperature to 80 ± 1.5°C over about 4 h and maintain this temperature for 48 h.</td>
</tr>
<tr>
<td>8.</td>
<td>Remove the bars one at a time and measure their length to 0.002 mm in not more than 20 s. Replace the bars in the water immediately after measuring.</td>
</tr>
<tr>
<td>9.</td>
<td>Repeat this measurement a further three times changing the order in which the bars are measured and after returning the bars with their charge to the oven for 2 h.</td>
</tr>
<tr>
<td>10.</td>
<td>Decant the water and add the same volume of 1 M NaOH solution, which has been previously warmed to 80°C.</td>
</tr>
<tr>
<td>11.</td>
<td>Return the box to the oven and repeat the length measurements as before at four convenient times over a period of 14 days. Further measurements were made after 28 days and longer periods in some cases.</td>
</tr>
<tr>
<td>12.</td>
<td>Each length measurement is made with respect to a standard invar bar.</td>
</tr>
</tbody>
</table>

have proposed a detailed procedure after carrying out a wide range of comparative tests and a detailed review of the literature. They found that expansion is influenced by the pressure and temperature employed, the water/cement ratio, the alkali concentration, and the cement type. The autoclave curing time is also significant. They recommend the following procedure, which can be completed in 3 days.

Mortar bar type: as ASTM C227
Water/cement ratio: fixed at 0.50
Alkali concentration: fixed at 3.5% Na₂O equivalent weight % of cement
Pre-autoclave curing: 2 days in water at 20°C
Steam curing: 5 h at 130°C and 0.17 MPa
Final measurement: at room temperature (25°C) after cooling over an hour.

The repeatability of this test was measured for one aggregate giving a mean expansion of 0.297% with a repeatability of 0.026 for seven determinations. Aggregate is considered to be innocuous at an expansion of 0.15% or less. The reproducibility, if all laboratories use precisely the same method, is therefore likely to be about 0.06% at 0.3% expansion.

A version of the autoclave method has also been studied by Saloman and Gallais. They used ultrasonic pulse velocity, dynamic elastic modulus, and length and weight changes to monitor the reaction. They recommended that the alcalies as Na₂O equivalent should be 4.0% by weight of cement, that the pressure should be 0.15 MPa, and that length measurement is more satisfactory than elastic modulus or USPV. They fixed the water/cement ratio at 0.5 and found that bars measuring 40 × 40 × 160 mm were more satisfactory than bars of a smaller cross-section. With these experimental conditions, discrimination between reactive and innocuous materials is made at 0.15% expansion. Criaud et al. have also studied this method, keeping close to the specification of Tang et al. The bars are immersed in 10% KOH solution and maintained in the autoclave at 150°C for 6 h. They found that the expansion was dependent among other things on the aggregate/cement ratio and have measured the repeatability and reproducibility of the method (see Table 3). An autoclave

Table 3. Example of relationship between mean and reproducibility for autoclave procedure

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Mean (% expansion)</th>
<th>Standard deviation (σ)</th>
<th>Reproducibility (% expansion)</th>
<th>Number of laboratories</th>
</tr>
</thead>
<tbody>
<tr>
<td>Limestone</td>
<td>0.026</td>
<td>0.008</td>
<td>0.022</td>
<td>4</td>
</tr>
<tr>
<td>Sand</td>
<td>0.053</td>
<td>0.011</td>
<td>0.030</td>
<td>2</td>
</tr>
<tr>
<td>Chert</td>
<td>0.146</td>
<td>0.008</td>
<td>0.022</td>
<td>4</td>
</tr>
<tr>
<td>Sand</td>
<td>0.227</td>
<td>0.079</td>
<td>0.219</td>
<td>5</td>
</tr>
<tr>
<td>Spratt</td>
<td>0.354</td>
<td>0.022</td>
<td>0.122</td>
<td>4</td>
</tr>
</tbody>
</table>
Concrete aggregates and alkali-aggregate reaction

procedure has also been applied to concrete samples. In the medium term, it seems likely that the obvious advantages of this type of test will mean that it supersedes those in common use at the moment and that the method will be very valuable as an engineering, as well as a research, tool. However, the poor reproducibility and wide range of working practice make it unreliable for practical purposes at the moment. Despite these variations, the tests demonstrate very rapidly that reaction occurs even where concrete is tested, and some field reactions requiring decades can be produced in a few hours. The problem of deciding an appropriate level of alkalis is, however, important. Almost any siliceous aggregate can be made to react if the alkali level is high enough. Dense flint from South East England can cause expansion in concrete prisms if the alkalis exceed 2.5% by weight of cement and in the field if the alkalis exceed 2%. French showed that concrete prism test results depend partly on the alkali level and partly on concrete quality. Hence, the manufacture of the bars requires strict definition of all parameters, including the alkali concentrations, and needs to be tested against a wide range of aggregate types of known field behaviour for discriminant criteria to be drawn reliably.

EXPERIMENTAL PROCEDURES

The two laboratories involved in the present testing used the same bulk sample of aggregate to make mortar bars of two sizes with three different cements and independent storage and measurement systems (Table 2). The sequence of testing and of making the bars was also left to the individual laboratories.

Aggregate

The aggregate for the tests was made up from innocuous quartz sands. These sands were carefully graded and blended to match that required by the ASTM C-227 specification. The coarsest fraction was then adjusted by replacing some of the original sand with fused silica so that the fused silica represented 10% of the total aggregate in the size range 2.36–4.75 mm and 5% of the total aggregate in the size fraction 1.18–2.36 mm so that the amount of fused silica in total made 15% of the aggregate. Sufficient material was prepared to make more than 100 mortar bars, and this carefully blended material was then divided by riffling and allocated to the two laboratories.

Cements

Most of the mortar bars were made using a single UK cement that was allocated to both laboratories. In addition, both laboratories used the same Norwegian cement, and one laboratory also made mortar bars using a rapid hardening Portland cement.

Mortar bar sizes and composition

Both laboratories made sets of four mortar bars with two different sizes: 25 × 25 × 250 mm and 40 × 40 × 160 mm. A total of five batches of four mortar bars were made of each size by both laboratories. These bars were all made with the same British ordinary Portland cement. Subsequently, three sets of four mortar bars in the two sizes were made with two other cements. For all mortar bars, a fixed aggregate/cement ratio of 2.25:1 and a fixed water/cement ratio of 0.44 were employed.

Moulding and storage

The mixture was placed in the moulds in two layers with compaction and vibration. The vibration at each stage was for 10 s. The surface was then trowelled smooth, and the mould was then cured for 24 h at 25°C in a closed polypropylene box containing water but not in contact with the water. The bars were then demoulded and cured in water at room temperature (25°C) for 48 h. They were then immersed in water in closed containers that were maintained at constant temperature in an oven at 80°C for 48 h. The lengths of the bars were then measured at a temperature of approximately 80°C. They were then placed in polypropylene containers with molar sodium hydroxide solution and, after sealing, were returned to the oven at 80°C. One laboratory used moulds in which ball-bearings were embedded at each end of each bar to act as the measuring points. The second laboratory used threaded bolts penetrating into the mortar for 15 mm at each end, and designed to take a ball-
bearing at each end for length measurement. Each set of mortar bars was enclosed in its own separate container. The moulds of two different sizes were filled from the same mixture, enough material having been prepared for both sets of bars. Also, both sets of bars were maintained under the same conditions and measured at various time intervals by both laboratories. One laboratory subsequently made a series of mortar bars of the two sizes that were maintained within various different volumes of alkaline solution.

Method of measurement

After 48 h, each set of mortar bars was removed from the oven in its container and placed in a water bath maintained in the range 80±2°C. The initial length for each bar was measured using a digital gauge which could be read to 0.001 mm. Each bar was read once only within 20 s of being removed from the hot water. This measurement was repeated for the other bars in the set. The bars were then allowed to regain the temperature of 80°C in the oven, and the measurements were then repeated in reverse order. All measurements were made a total of four times, with the order being changed on each occasion, after allowing the temperature to recover to 80°C each time. The bars were measured at various ages up to 56 days in the alkaline solution.

RESULTS

The results of measurements made by laboratories A and B are given in Tables 4–7. The means and repeatabilities for the 250- and 160-mm-long bars for each of the two laboratories are given in Table 8, for each time of measurement. The repeatability as given in this table treats the mean results for each batch of four bars as the individual test result and is defined as the expected random error associated with a single test operator obtaining successive results on identical material. It is the magnitude of the difference between two single results that would occur in only one instance in 20 measurements. The observations are discussed in detail in a later section. If the individual results for the two laboratories are taken together, the repeatability at 14 days is 0.150% for 160-mm bars and 0.077% for 250-mm bars. These figures are likely to compare closely with the interlaboratory reproducibility for the procedure.

DISCUSSION

Expansion criteria

Various proposals have been made for the interpretation of the magnitude of expansion in tests of this type. These proposals vary according to the correlation with field experience and with rock type. They may be influenced also by individual features of the test. In 1983, Oberholster defined divisions as follows: innocuous, <0.08%; slowly expansive, 0.08–0.20%; and rapidly expanding, >0.20%. These limits applied to various rock types and were based on a test of 10 days' duration and compared with the results of ASTM C-227 mortar bar and concrete prism tests. In 1986, Oberholster and Davies, again using the correlation with ASTM C-227 to interpret the potential for reactivity, defined a division between innocuous and potentially reactive at an expansion of 0.11% in 12 days. Davies and Oberholster, after an interlaboratory investigation, proposed boundaries after 12 days of testing as follows: innocuous, <0.10%; slowly expanding, 0.10–0.25%; and rapidly expanding, >0.25%. Again several aggregates were used in this test. Shayan et al. proposed that aggregate should be regarded as innocuous if the expansion was less than 0.10% in 22 days, slowly expansive if the expansion exceeded 0.10% in 22 days, and rapidly expanding if the expansion exceeded 0.10% in 10 days. Hooton and Rogers considered that an expansion of less than 0.10% at 14 days represented innocuous material, 0.10–0.25% expansion was ambiguous, and greater than 0.25% was considered to be reactive. Expansion of between 0.10 and 0.25% meant that further testing and evaluation were required. This test, according to these authors, appeared to be the most promising in making the distinction between reactive and non-reactive aggregates, and they were able to distinguish all 12 aggregates in their set that were known to be reactive. Grattan-Bellew, in 1989, again selected the boundary of innocuous as being less than 0.10% after 14 days but found that different magnitudes of expansion could be defined for differing potentially reactive rock
types. Hooton\cite{28} investigated various test methods using aggregates with known petrographic characteristics and field performance in concrete. He found the alkali immersion procedure to be the most promising in terms of distinguishing between reactive, marginal and non-reactive aggregates. He proposed a boundary of 0.15% expansion between innocuous and reactive 16 days after casting, 14 days in alkali, but ambiguous results suggested that the test should be continued for 28 days and 56 days with the boundary between innocuous and reactive being drawn at 0.33% and 0.48%, respectively. Hooton and Rogers\cite{30} used the same boundary but suggested a marginal zone below the discrimination boundary. This helped in the recognition of potential reactivity for some granites and gneisses containing micro-

<table>
<thead>
<tr>
<th>Table 4. Replicate length changes for standard bars 25 x 25 x 250 mm; laboratory A. % expansion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Days</td>
</tr>
<tr>
<td>Bar number</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>Days</td>
</tr>
<tr>
<td>Bar number</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>Days</td>
</tr>
<tr>
<td>Bar number</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
</tbody>
</table>

Grattan-Bellew\cite{19} considered that two discriminant criteria were required according to the field conditions to be expected. A general limit of 0.10% expansion after 14 days needed to be modified to 0.2% for greywackes and argillites, 0.15% for other aggregate types, while for limestones the limit was less than 0.10%. Berube et al.\cite{24} tested 142 aggregates. They detected all but one of the known reactive aggregates of Quebec but found the method too severe for many aggregates which have a good field record.

The recently introduced Canadian standard\cite{3} states that the maximum expansion value for alkali-aggregate reactivity is 0.15% at 14 days. Several aggregates that expand greater than 0.15% after 14 days have not caused deleterious

<table>
<thead>
<tr>
<th>Norwegian cement</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>Mean</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.30</td>
<td>0.20</td>
<td>0.30</td>
<td>0.30</td>
<td>0.28</td>
<td>0.028</td>
</tr>
<tr>
<td></td>
<td>0.110</td>
<td>0.130</td>
<td>0.120</td>
<td>0.140</td>
<td>0.125</td>
<td>0.013</td>
</tr>
<tr>
<td></td>
<td>0.360</td>
<td>0.370</td>
<td>0.350</td>
<td>0.340</td>
<td>0.355</td>
<td>0.013</td>
</tr>
<tr>
<td></td>
<td>0.410</td>
<td>0.340</td>
<td>0.380</td>
<td>0.380</td>
<td>0.378</td>
<td>0.029</td>
</tr>
<tr>
<td></td>
<td>0.460</td>
<td>0.360</td>
<td>0.440</td>
<td>0.480</td>
<td>0.440</td>
<td>0.043</td>
</tr>
</tbody>
</table>
expansion in field structures. Therefore, expansion in excess of the recommended limit calls for further testing of concrete specimens. A lower limit of 0.10% is recommended for quarried siliceous limestone. There are reports of field concrete made with aggregates containing granites, gneisses, and granodiorites, that exhibit less than 0.10% expansion at 14 days in the accelerated mortar bar test.

ASTM C1260\(^2\) states that an expansion of less than 0.10% at 16 days after casting (14 days in the NaOH solution) is indicative of innocuous behaviour in most cases. Some granitic gneisses and metabasalts have been found to be expansively reactive in field performance even though their expansion was less than 0.10% at 16 days after casting. Expansion of more than 0.20% at 16 days after casting is indicative of potentially deleterious expansion. Expansion between 0.10% and 0.20% at 16 days after casting includes both aggregates that are known to be innocuous and deleterious in field performance. For these aggregates, it is particularly important to develop supplemental information. In such a situation, it may also be helpful to take comparator readings until 28 days.

In Norway, at SINTEF — Structures and Concrete — a modified version of the alkali immersion procedure has been practised to investigate natural aggregates, including crushed rock and blended aggregates.\(^{41,42}\) The expansion is monitored by measuring length change of 40 × 40 × 160 mm bars after 4, 7, 12, 14, 28, and 56 days. The result at 14 days is used to evaluate the potential expansivity of the aggregate — less than or equal to 0.10% is considered innocuous, between 0.10% and 0.25% should be considered to be deleterious but slowly expanding, and 0.25% and above should be regarded as potentially deleteriously reactive and rapidly expanding. In Norway, further testing of the aggregate is recommended when petrographic examination indicates the presence of 20% or more of potentially reactive rock types in the aggregate, whereas aggregates with less than 20% are regarded as innocuous.\(^{28}\)

<table>
<thead>
<tr>
<th>Table 5. Replicate length changes for standard bars 25 × 25 × 250 mm; laboratory B % expansion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Days</td>
</tr>
<tr>
<td>Bar number</td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>2</td>
</tr>
<tr>
<td>3</td>
</tr>
<tr>
<td>4</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>5</td>
</tr>
<tr>
<td>6</td>
</tr>
<tr>
<td>7</td>
</tr>
<tr>
<td>8</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>9</td>
</tr>
<tr>
<td>10</td>
</tr>
<tr>
<td>11</td>
</tr>
<tr>
<td>12</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>13</td>
</tr>
<tr>
<td>14</td>
</tr>
<tr>
<td>15</td>
</tr>
<tr>
<td>16</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>17</td>
</tr>
<tr>
<td>18</td>
</tr>
<tr>
<td>19</td>
</tr>
<tr>
<td>20</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>Overall mean</td>
</tr>
<tr>
<td>Mean standard deviation</td>
</tr>
</tbody>
</table>
Many of the discriminant criteria therefore propose that aggregate should be considered innocuous if expansion is less than 0.10% after a period of about 14 days in 1 M NaOH solution at 80°C, though some propose more stringent conditions. This discriminant boundary is based on a comparison with field performance, where the aggregate has shown no reaction in service, and on a comparison with other test results, such as the ASTM C-227 mortar bar test. Neither of these comparisons can be regarded as wholly reliable. The conditions in the alkali immersion tests are much more severe than those found in the field, and if the alkali content of the cement paste is generally moderate in the field concrete or the conditions are not aggressive, then a reaction may not occur, even though in other circum-
stances, a deleterious reaction may take place. An expansion of 0.10% in the test is not necessarily comparable with the expansion of concrete containing the same aggregate, but if an expansion of 0.10% actually occurred in service then some cracking is a likely consequence. In addition, it is possible that some expansion may not be due to alkaliaggregate reaction and the value obtained must be interpreted also in terms of the reproducibility of the measurements. Thus, while significant expansion may be simple to interpret and require an aggregate to be rejected, or special conditions attached to its use, the diagnosis of innocuous material is less unambiguous. The cause of expansion therefore must be identified by microscopic examination of the test mortars. It may be possible for the expansion limit to the innocuous field to be

<table>
<thead>
<tr>
<th>Table 6. Replicate length changes for standard bars 40 × 40 × 160 mm; laboratory A % expansion</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Days</strong></td>
</tr>
<tr>
<td><strong>Bar number</strong></td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>2</td>
</tr>
<tr>
<td>3</td>
</tr>
<tr>
<td>4</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>5</td>
</tr>
<tr>
<td>6</td>
</tr>
<tr>
<td>7</td>
</tr>
<tr>
<td>8</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>9</td>
</tr>
<tr>
<td>10</td>
</tr>
<tr>
<td>11</td>
</tr>
<tr>
<td>12</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>13</td>
</tr>
<tr>
<td>14</td>
</tr>
<tr>
<td>15</td>
</tr>
<tr>
<td>16</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>17</td>
</tr>
<tr>
<td>18</td>
</tr>
<tr>
<td>19</td>
</tr>
<tr>
<td>20</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
<tr>
<td>Overall mean</td>
</tr>
<tr>
<td>Mean standard deviation</td>
</tr>
<tr>
<td><strong>Norwegian cement</strong></td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>2</td>
</tr>
<tr>
<td>3</td>
</tr>
<tr>
<td>4</td>
</tr>
<tr>
<td>Mean</td>
</tr>
<tr>
<td>Standard deviation</td>
</tr>
</tbody>
</table>
Table 7. Replicate length changes for standard bars 40 x 40 x 160 mm; laboratory B % expansion

<table>
<thead>
<tr>
<th>Days</th>
<th>4</th>
<th>7</th>
<th>14</th>
<th>56</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Bar number</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>0.206</td>
<td>0.279</td>
<td>0.392</td>
<td>0.650</td>
</tr>
<tr>
<td>3</td>
<td>0.186</td>
<td>0.258</td>
<td>0.361</td>
<td>0.619</td>
</tr>
<tr>
<td>4</td>
<td>0.186</td>
<td>0.258</td>
<td>0.382</td>
<td>0.640</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>0.193</td>
<td>0.265</td>
<td>0.378</td>
<td>0.637</td>
</tr>
<tr>
<td><strong>Standard deviation</strong></td>
<td>0.012</td>
<td>0.012</td>
<td>0.016</td>
<td>0.016</td>
</tr>
<tr>
<td>5</td>
<td>0.206</td>
<td>0.279</td>
<td>0.372</td>
<td>0.671</td>
</tr>
<tr>
<td>6</td>
<td>0.196</td>
<td>0.268</td>
<td>0.372</td>
<td>0.692</td>
</tr>
<tr>
<td>7</td>
<td>0.155</td>
<td>0.196</td>
<td>0.299</td>
<td>0.537</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>0.186</td>
<td>0.248</td>
<td>0.348</td>
<td>0.633</td>
</tr>
<tr>
<td><strong>Standard deviation</strong></td>
<td>0.027</td>
<td>0.045</td>
<td>0.042</td>
<td>0.084</td>
</tr>
<tr>
<td>9</td>
<td>0.258</td>
<td>0.320</td>
<td>0.434</td>
<td>0.754</td>
</tr>
<tr>
<td>10</td>
<td>0.268</td>
<td>0.330</td>
<td>0.423</td>
<td>0.723</td>
</tr>
<tr>
<td>12</td>
<td>0.310</td>
<td>0.392</td>
<td>0.495</td>
<td>0.805</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>0.279</td>
<td>0.348</td>
<td>0.451</td>
<td>0.760</td>
</tr>
<tr>
<td><strong>Standard deviation</strong></td>
<td>0.027</td>
<td>0.039</td>
<td>0.039</td>
<td>0.042</td>
</tr>
<tr>
<td>13</td>
<td>0.258</td>
<td>0.310</td>
<td>0.403</td>
<td>0.723</td>
</tr>
<tr>
<td>14</td>
<td>0.258</td>
<td>0.310</td>
<td>0.403</td>
<td>0.723</td>
</tr>
<tr>
<td>15</td>
<td>0.237</td>
<td>0.289</td>
<td>0.372</td>
<td>0.681</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>0.251</td>
<td>0.303</td>
<td>0.392</td>
<td>0.709</td>
</tr>
<tr>
<td><strong>Standard deviation</strong></td>
<td>0.012</td>
<td>0.012</td>
<td>0.018</td>
<td>0.024</td>
</tr>
<tr>
<td>17</td>
<td>0.235</td>
<td>0.299</td>
<td>0.413</td>
<td>0.630</td>
</tr>
<tr>
<td>19</td>
<td>0.240</td>
<td>0.310</td>
<td>0.413</td>
<td>0.709</td>
</tr>
<tr>
<td>20</td>
<td>0.236</td>
<td>0.289</td>
<td>0.403</td>
<td>0.709</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>0.237</td>
<td>0.299</td>
<td>0.409</td>
<td>0.709</td>
</tr>
<tr>
<td><strong>Standard deviation</strong></td>
<td>0.003</td>
<td>0.010</td>
<td>0.006</td>
<td>0.011</td>
</tr>
<tr>
<td><strong>Overall mean</strong></td>
<td>0.229</td>
<td>0.292</td>
<td>0.396</td>
<td>0.685</td>
</tr>
<tr>
<td><strong>Mean standard deviation</strong></td>
<td>0.016</td>
<td>0.024</td>
<td>0.024</td>
<td>0.041</td>
</tr>
</tbody>
</table>

Table 8. Mean percentage expansions and repeatability

<table>
<thead>
<tr>
<th>Days</th>
<th>250-mm bars</th>
<th>160-mm bars</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean expansion</td>
<td>Repeatability</td>
</tr>
<tr>
<td></td>
<td>Lab A</td>
<td>Lab B</td>
</tr>
<tr>
<td>1</td>
<td>0.023</td>
<td>0.023</td>
</tr>
<tr>
<td>3</td>
<td>0.169</td>
<td>0.137</td>
</tr>
<tr>
<td>4</td>
<td>0.402</td>
<td>0.039</td>
</tr>
<tr>
<td>7</td>
<td>0.426</td>
<td>0.044</td>
</tr>
<tr>
<td>11</td>
<td>0.519</td>
<td>0.048</td>
</tr>
<tr>
<td>14</td>
<td>0.053</td>
<td>0.039</td>
</tr>
<tr>
<td>28</td>
<td>0.116</td>
<td>0.050</td>
</tr>
<tr>
<td>56</td>
<td>0.479</td>
<td>0.199</td>
</tr>
</tbody>
</table>
defined from the data obtained from such examinations for the given aggregate type. For this purpose, qualitative recognition of the presence of alkali-silicate gel in the mortar bars coupled with damage to specific aggregate particles will be sufficient to demonstrate that an alkali-aggregate reaction has occurred.

It is evident from the foregoing paragraphs that there is little agreement or rigour concerning the values to be adopted for discriminating between reactive and innocuous materials. Consideration has also to be given to the repeatability of the test within the laboratory and the possible reproducibility in comparing laboratory results in order to interpret the discriminant criteria safely. The question to be answered should be either 'What is the probability of an aggregate being reactive in service, if it exhibits a particular level of expansion in the accelerated test?,' or 'What is the probability of an aggregate being innocuous in service if it exhibits a particular level of expansion in the test?'.

Influence of alkali volume

Some experiments have been carried out using differing ratios of 1 M NaOH to the surface area of the aggregate. The results of this work lead to a conclusion that the amount of expansion increases progressively as the ratio of the volume of alkali to the surface area of the aggregate increases. This relationship is shown in Fig. 1. The result obtained from this work shows that the percentage expansion for the aggregate under test is given by $10.5 (m/LD)^{0.25}$, where $m$ is the mass of alkaline solution in the vessel in kilograms, and $L$ and $D$ are the length and breadth of the mortar bar in millimetres. It is evident from this that once the ratio exceeds 4 g/cm$^2$, the change in expansion is small but continuing. It is essential, therefore, that this ratio is fixed, and it is prudent to ensure that individual sets of bars containing specific aggregates under test are placed in their own containers with a specific aliquot of freshly prepared sodium hydroxide solution.

Cement type

The specification for accelerated mortar bar tests gives no requirement for the type of cement to be employed. Davies and Oberholster$^{43}$ investigated the effect of the cement composition upon expansion, using three different types of cement, all with different alkali contents and with differing fineness. They concluded that the different cement types had little influence upon the measured expansion. Gratton-Bellew$^{13}$ found that the expansion made with the cement having an alkali content of 1.08% was identical with that of bars made with a cement having an alkali content of 0.66%. Fournier and Bérubé$^{44}$ investigated the effect of cement composition on expansion by using ten different types of cement. They found no clear correlation between the chemical composition of the cement and the expansion obtained. However, cements with higher fineness values exhibited a higher degree of expansion, even if they did not have the highest alkali content. In the experiments reported here, no significant differences were found for the three cement types employed.

Practical considerations

The manufacture and measurement of the bars measuring $160 \times 40 \times 40$ mm were found to be more convenient than the measurement of the longer bars. For the conventional ASTM C-227 mortar bar method, it has been found that the rate of expansion increases with increasing cross-sectional dimensions of the test specimens up to at least 100 mm.$^{15}$ In addition, Locker$^{16}$ reported a greater expansion for the $40 \times 40 \times 160$ mm bars than with ASTM C-490 bars ($25 \times 25 \times 285$ mm) in tests that otherwise followed the ASTM C-227 mortar bar method. The results of the experiments carried out in the present work show that there is little or no difference in the percentage expansion of the thicker shorter bars than for the longer bars if other factors are constant. However, the repeat-
ability of the measurements expressed as a percentage of the length of the bars is larger for 160-mm bars than it is for the 250-mm bars. Both systems of manufacture of the mortar bars proved satisfactory, but it was more difficult to ensure that the stainless-steel balls were attached to the mortar than it was for the inserted threaded measuring points. However, the threaded measurement points were found to be slightly more difficult to use with the loose ball-bearings than with the fixed bearings. Great care was taken in establishing the lengths by taking four readings at each reading interval. The time interval over which individual measurements were made was always only a few seconds following removal from the solution at 80°C. The effect of falling temperature was measured and is illustrated in Fig. 2. This shows the possible contribution of variation in time interval required for the measurement on the measurement error. The number of measurements carried out may have been excessive because the process takes a long time and hence gives a potential for continued expansion during the measurement period. This could influence the value of the repeatability at each time. Duplicate measurements for each bar might have sufficed.

**Pessimum aggregate proportions**

Shayan et al.\textsuperscript{38} showed that a sandstone exhibited the pessimum effect in the accelerated test, and Shayan\textsuperscript{37} has recorded a pessimum for the expansion of accelerated mortar bar tests using opal. The maximum expansion occurs where opal is 5% of the aggregate. This was the lowest opal level employed. The explanation for this is considered by Shayan to be due to the early generation of gel in the outer parts of the mortar bar. The larger amounts of reactive aggregate produce large amounts of gel that make the outer mortar an effective filter for NaOH. We have observed that the penetration of alkalies is in all cases slow and progressive and that bars of all aggregate types that we have tested react first in the outer zones (e.g. Wigum and French\textsuperscript{46}). The bars made by Shayan were described as ‘moisture-saturated’ before being placed in the alkaline solution. In our experiments, the bars are also kept in water or alkaline solution. This may be a very important aspect of the test because if the bars are allowed to dry fully, they will exert a suction pressure on the alkali solution, and alkalies will penetrate rapidly. If the bars are maintained in a saturated condition, the transference of the alkalies will be by diffusion along a concentration gradient and will be retarded by chemical fixation in cement hydration products. The distribution coefficient of Na\textsuperscript+ and OH\textsuperscript− ions for equilibria involving cement hydrates, reaction products and pore fluid, and the diffusion coefficient for the Na\textsuperscript+ and (OH)\textsuperscript− ions in the mortar system therefore will regulate the rate of reaction and the observed expansion. A large surface area for reaction will retard penetration of the diffusing species. It is therefore very important that

consideration be given to the amount of potentially reactive material in the mortar bar, which will influence the rate of ingress of alkalies, and

that the transference of the bars from the water to the alkalies be carried out under standard moisture conditions.

Further experimentation is required to confirm the magnitude of the difference in expansion created by allowing the bars to dry before placing them in the alkali solution relative to their remaining effectively saturated, but a variation in the rate of alkali penetration could have a very large effect on the reproducibility of the test.

With respect to the creation of a pessimum expansion, it is vital that the test be arranged to detect such possibilities. The reaction for most real aggregates involves only a small fraction of the rock with perhaps 1 or 2% of the rock actually taking part in reactions. Opal con-

![Fig. 2. Influence of falling temperature on length of mortar bars.](image-url)
versely is likely to be wholly reactive, and the presence of 5% opal certainly should be detected before testing. Hence, for many aggregates, it may be prudent to test the material alone and with 50% replacement with an inert material. If the expansion is changed by a significant amount, in terms of the measured repeatability, on dilution, then either the aggregate might be rejected or further testing undertaken to define the pessimum relationship more precisely. The significant change in expansion must be defined in the terms of the repeatability of the measurement, the test being repeated if, say, the expansion difference exceeds the repeatability.

STATISTICAL APPRAISAL OF TEST DATA

Variation of standard deviation with mean expansion

A graphical display of the data for both 250-mm and 160-mm bars shows that the variance changes with the mean, i.e. the data are heteroscedastic, and hence log-transformation stabilises the variance, and the plot of log_{10} [standard deviation] is found to increase linearly with the mean. Also, the trends for lab A conform with those for lab B for both sets of bars with the exception of two sets of data for 160-mm bars for lab B (see Fig. 3). Visually, it appears that no distinction can be made between 250- and 160-mm bars, and this was checked by calculating the Kolmogorov–Smirnov two sample goodness-of-fit statistic. This showed that no difference can be found between the two sets of data for the two laboratories. Using a robust least-squares regression method trimmed to downweight outliers, it was found that residuals were normally distributed, and a plot of residuals against estimated values showed no evidence of non-linearity. A linear model for log_{10}(s) variation with mean expansion is therefore satisfactory and gives

\[\log_{10}(s) = -1.8725 + 0.5867E,\]

where \(E\) is the percentage expansion.

The standard deviation (\(\sigma_0\)) for zero expansion is then a mean value of 0.013% expansion. The upper bounds for the standard deviation can be obtained from the distribution of the residuals of the regression equation. The 97.5th and 99.5th percentiles give multipliers of 2.600 and 2.745, respectively, which are upper bounds on the standard deviation for a given value of mean expansion based on the corresponding upper one-sided 95% and 99% prediction intervals. Hence

![Fig. 3. Observed spreads of bar-expansion as a function of mean expansion for laboratories A and B and for 160- and 250-mm bars.](image)
Table 9. Predicted variation in standard deviation with mean expansion

<table>
<thead>
<tr>
<th>Mean expansion %</th>
<th>( s_{0.05} ) expansion %</th>
<th>( s_{0.05} ) expansion %</th>
<th>( s_{0.05} ) expansion %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>0.013</td>
<td>0.035</td>
<td>0.037</td>
</tr>
<tr>
<td>0.10</td>
<td>0.015</td>
<td>0.040</td>
<td>0.042</td>
</tr>
<tr>
<td>0.15</td>
<td>0.016</td>
<td>0.043</td>
<td>0.045</td>
</tr>
<tr>
<td>0.20</td>
<td>0.018</td>
<td>0.046</td>
<td>0.048</td>
</tr>
<tr>
<td>0.25</td>
<td>0.019</td>
<td>0.048</td>
<td>0.052</td>
</tr>
</tbody>
</table>

\[
\log_{10} s_{0.05} = -1.8725 + 0.5867 E + 0.4150, \text{ and}
\]

\[
\log_{10} s_{0.09} = -1.8725 + 0.5867 E + 0.4386
\]

and the variations of the estimates of standard deviation with the mean expansion at various levels of confidence are as given in Table 9. Since it is common to consider that an expansion of <0.10% over a fixed period of time, as for example specified in ASTM C-1260, is not significant, this must represent the safe limit of measured expansion that could occur by chance or through some process of no significance for the durability of the concrete. Taking the safe estimate of \( s \) for zero expansion as 0.037% means that the limit of 0.10% is 2.70 standard deviations above the mean. As shown below, this figure (0.037%) is of general application, and the factor of 2.7 is close to the often-quoted safe limit of 2.75 standard deviations. The coefficient of variation given in ASTM C-1260 of 9.55% gives the variation with mean expansion summarized in Table 10.

The work of Oberholster\(^\text{15}\) on repeatability gives an upper bound to the expected standard deviation of 0.05% at 0.250% expansion. This is of the same order as found in the present work. The expansion of 0.10% corresponds to about three times the estimated upper bound of the standard deviation if the distribution of data of tests is similar to that of the present work. The ratio \( N = \text{mean expansion/0.037} \) therefore gives a measure of the potential for deleterious expansion. Expansion after the specified time giving a ratio of below \( N = 3 \) means that the test has failed to show up a potential for adverse alkali-aggregate reaction, whereas a ratio of, say, 8 might be taken to mean a potentially severe reaction. An expansion of, say, \( N = 4 \) may not always indicate a potential for deleterious alkali–aggregate reaction since it could represent a potential for expansion from some cause other than AAR combined with random error. Nevertheless, a value of \( N = 4 \) would make it unsafe to accept the aggregate. However, expansion cannot be used alone to identify AAR as the cause of expansion, and the cause of observed expansion must be found independently. Mortar bars therefore must be examined for evidence of adverse reaction using optical or electron microscopy of thin sections. Combining the test result with a petrographic examination leads to schemes for the interpretation of the likely behaviour of the aggregate such as that given in Table 11. This is particularly economical since the cost of the petrography can be less than half the cost of repeating the test and is also substantially quicker. It is also superior to the use of control tests because it provides different information and because the standard error in the measurement of the difference between a control and a test aggregate will be over 0.05%.

How many tests?

In the present work, three and four mortar bars were used to obtain a single result. ASTM C-1260 provides for the manufacture of three bars. Since random errors can occur in the manufacture and measurement of the sets of bars, it seems desirable that at least two sets should be made for each aggregate. However, the number of tests required depends largely on the precision required for the measured expansion. ASTM C-1260 says that two laboratories carrying out the same test properly should not differ by more than 27% of the mean expansion. This very large difference presumably represents three standard deviations. It could obviously mean that while one laboratory would find an aggregate reactive, another might find it innocuous by the simple criteria currently defined. It is also intuitively obvious that where a result is close to the discriminant criterion, a
### Table 11. Possible scheme for interpretation of test results

<table>
<thead>
<tr>
<th>Expansion after 14 days/0.037 system being reactive according to test</th>
<th>Probability of</th>
<th>Evidence of AAR in thin section of mortar*</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt; 4</td>
<td>&gt; 99.9% Reject</td>
<td>Reject</td>
</tr>
<tr>
<td>&gt; 3-4</td>
<td>&gt; 99-99.9% Establish cause of expansion, consider repeating the test, consider use of PFA, GGBS</td>
<td>Reject</td>
</tr>
<tr>
<td>2-3</td>
<td>&gt; 95-99% Establish cause of expansion, consider repeating the test, continue test to 28 days, consider use of low alkali cement, PFA, GGBS, etc.</td>
<td>Reject</td>
</tr>
<tr>
<td>&lt; 2</td>
<td>&lt;95% Accept aggregate</td>
<td>Consider repeating test or using low alkali cement, PFA, GGBS, etc.</td>
</tr>
</tbody>
</table>

*The qualitative recognition of gel in the thin section and of loss of aggregate or cracking within specific aggregate fragments is taken to be definitive. It may also be possible to assess the magnitude of reaction in terms of crack frequency/unit area and gel volume.

A larger number of measurements are necessary than if a large expansion is measured.

The risks involved in decision making are \( \alpha \) and \( \beta \), which are defined as follows:

Decision based on experimental mean \( \bar{x} \)

<table>
<thead>
<tr>
<th>Truth (which is unknown)</th>
<th>No expansion</th>
<th>Expansion</th>
</tr>
</thead>
<tbody>
<tr>
<td>0% occurs</td>
<td>( P_{\text{correct}} = (1-\alpha) )</td>
<td>( P_{\text{wrong}} = \alpha )</td>
</tr>
<tr>
<td>&gt; d% occurs</td>
<td>( P_{\text{wrong}} = \beta )</td>
<td>( P_{\text{correct}} = (1-\beta) )</td>
</tr>
</tbody>
</table>

For the purposes of illustration, suppose that \( \alpha = 0.01 \) and \( \beta = 0.001 \), i.e. 1% and 0.10% risk, respectively. The corresponding quantiles of the cumulative normal distribution are then \( Z_\alpha = 2.326 \) and \( Z_\beta = 3.090 \). Since \( \mu = 0.0 \), \( \sigma_0 = 0.037 \) and if the change \( \delta \) to be detected is +0.10%, the classical normal theory estimate of the required sample size is

\[
\hat{N} = \left( \frac{Z_x + Z_{\beta/2}}{\delta} \right)^2 \left( \frac{5.416 \times 0.0365}{0.10} \right)^2 = 3.9 \rightarrow 4 \text{ replicates.}
\]

However, Kupper and Haffner50 showed that since this traditional and widely used formula is based on large-sample approximation theory, it gives estimated sample sizes that are too small; an effect that becomes worse as \( N \) becomes smaller. Their tables show that in order to have a 95% tolerance probability that \( \mu \) is estimated with a confidence interval of 100\( \alpha \)%, the desired sample size is \( N_{\text{adj}} = 11 \) rather than \( N = 4 \). A working approximation to their one-sample table for a 95% tolerance is given by

\[
N_{\text{adj}} = 4.76 + 1.69 N^{0.926}.
\]

The corresponding critical value for the test criterion is given by

\[
\bar{x} = \mu_0 + \sigma_0 Z_\alpha / \sqrt{N_{\text{adj}}} = (\mu_0 + \delta) - \sigma_0 Z_{\beta/2} / \sqrt{N_{\text{adj}}}.
\]

Following the experiment, if \( \bar{x}_{\text{obs}} > \bar{x}^* \) then we accept the alternate hypothesis that \( \mu_1 > \mu_0 \) with at least a 100(1-\( \alpha \))% confidence that this decision is correct. If \( \bar{x}_{\text{obs}} < \bar{x}^* \), then we accept that \( \mu_1 = \mu_0 \) with at least a 100(1-\( \beta \))% confidence that the decision is correct, since the new population mean is \( < (\mu_0 + \delta) \). These lead to the following estimates of sample sizes and corresponding critical values.

\[
\begin{align*}
\mu_0 &= 0 & \alpha &= 0.01 & \beta &= 0.001 \\
\delta &= 0.10 & \delta &= 0.15 & \delta &= 0.20 & \delta &= 0.25 \\
N_{\text{adj}} &= 11 & 8 & 6 & 6 \\
\bar{x}^* &= 0.026 & 0.030 & 0.035 & 0.035.
\end{align*}
\]

If the limit of expansion is set lower than 0.10%, then the number of measurements required becomes large because of the large standard deviation. For example, if the limit is set at 0.05%, the number of tests required is 27. If expansion at an early age is high then the number of replicates can be low, but avoidance of ambiguity at lower expansion requires consideration of the number of extra tests to be started and the length of time for which the tests are to be continued.
Assessment at an early age

Expansions measured at 1, 3, and 7 days provide useful data on the eventual expansion and on the number of tests required and the length of time required for the test. We make use of the fast initial response cumulative sum (FIR CUSUM) method\textsuperscript{51} to detect drift of the mean expansion away from zero at as early a stage as possible. We are only interested here in positive drift, and the upper FIR CUSUM on the ith day is given by

\[ S(U)_i = \max\{0, \bar{x}_i - K + S(U)_{i-1}\}, \]

where

\[ S(U)_0 = K \]

and \( \bar{x}_i \) is the observed mean expansion; \( H \) is the critical threshold, usually defined in terms of either a number of standard deviations above the mean or a given value. Here, \( H \) is set at 0.10% (corresponding with a drift of 2.74\( \sigma_0 \)); and \( K = H/2 = 0.05\% \) is the headstart parameter, which ensures a rapid response of the monitoring process if the system has initially drifted away from the expected value of zero mean expansion. For example, consider the first batch of 250-mm bars of Table 4. These are listed in Table 12 along with the corresponding Cusum values. This list shows that the critical expansion level is exceeded after only 3 days, and that four bars will suffice. As an example, the second value in the list for these mortar bars is found from \( x_i = 0.034, \ K = 0.05, \ S(U)_{i-1} = 0.233 \). The second part of the table shows a slow rate of expansion that might lead to an ambiguous condition after 14 days so that at least two and preferably three batches need to be tested. Here, there is evidence of real expansion but at a rate that is ambiguous during the early stages but might reach the critical level after 28 days.

Contributions to variability

The contributions to the standard deviation of the expansion at any one time can derive from many sources. If the overall deviation increases with the mean then the deviation must include items that are related to the mortar itself rather than the system of measurement and storage. Consideration of the sets of 160-mm bars for laboratory A shows that the expansion is given by

\[ E\% = k t^{0.5}, \]

where \( t \) is the time in days. The slope of the straight line \( y = k t^{0.5} + c \) varies substantially (Figs 4 and 5). The range has been identified in this case as reflecting the ratio of the volume of alkali solution to the surface area of the prism. Other variables that would affect the slope include the proportion of reactive aggregate, the water/cement ratio, and the compaction of the mortar. Contributions to the measurement error include variations in the temperature of measurement, the limitations of the measurement system, wear and contamination of the end pieces and the precision with which the time of measurement is recorded. Consideration of the data for the five sets of 250-mm bars of laboratory A shows that for both measured results and the calculated expansion derived from a linear best-fit of expansion with root time, their standard deviation is close to 0.02%. However, the difference between measured and calculated expansion is large. This derives mainly from the measurements at 28 days where, for example, the mean expansion is 0.519\%, whereas the calculated expansion is 0.568\%. Examination of the data suggests that expansion is not linearly related to the square root of time after 14 days. If only expansions for up to 14 days are considered then the mean expansion and calculated expansions for 14 days are 0.427\% and 0.430\%, and the standard deviations are 0.016 and 0.011, respectively. The slope constants are very similar (0.1243, 0.1225, 0.1243, 0.1376, and 0.1246).

The best-fit straight lines drawn on Figs 4 and 5 intersect the expansion axis at negative values. This is interpreted as reflecting differences in the time of inception of the reaction due perhaps to differing diffusion rates for alka-

<table>
<thead>
<tr>
<th>Day number</th>
<th>High expansion</th>
<th>Intermediate expansion</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean expansion</td>
<td>FIR CUSUM</td>
</tr>
<tr>
<td></td>
<td>%</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>0.034</td>
<td>0.034</td>
</tr>
<tr>
<td>3</td>
<td>0.233</td>
<td>0.217</td>
</tr>
<tr>
<td>11</td>
<td>0.401</td>
<td>0.568</td>
</tr>
<tr>
<td>14</td>
<td>0.426</td>
<td>0.944</td>
</tr>
<tr>
<td>28</td>
<td>0.523</td>
<td>1.417</td>
</tr>
</tbody>
</table>

N = 4 bars, H = 0.10, and K = 0.05.
lies penetrating into the mortar. The 160-mm bars have a longer dormant period than the thinner 250-mm bars. The simple relationships also cannot be extrapolated to longer periods of time. However, two approaches have given excellent correspondence between modelled and actual expansion versus time. The first of these is

$$E\% = \beta_1 (t - \beta_2)^n,$$  \hspace{1cm} (1)

and is illustrated in Fig. 6 for the 250-mm bars of Table 4. This figure gives the three param-
Fig. 6. Fitted expansions as function of time; model (1); plotted example is series 1. \( R^2 \) is coefficient of determination.

Fig. 7. Fitted expansions as function of time; model (2); plotted example is series 1.

The parameters of the formula and the coefficient of determination \( (R^2) \). Equally useful is the equivalent relationship

\[
E\% = k \ln t + c, \tag{2}
\]

which is illustrated in Fig. 7. For both of these expressions, the residuals are very low with the main contributions to the residuals coming from expansion at early ages. The measurement system used to record the changes in length of the mortar bars allows each measurement to have a precision of \( \pm 2 \mu m \). If this is taken to be the standard deviation of each individual measurement, the standard deviation of a given length should be \( 4 \mu m \). An increase in this standard deviation will derive from slight variations in the temperature at which the bars are measured. This gives a measurement standard deviation approaching 0.001% where the measured expansion is 0.4%. Hence, the range found in each set of four bars is not due to measurement error. This range can be compared with a standard deviation of 0.048% for 41 laboratories in the interlaboratory study made by Rogers et al.\textsuperscript{53} for mortar with a mean expansion of 0.364% at 14 days. This is in keeping with the standard deviations obtained in the present work which are in the range of 0.02–0.03%.

All these figures evidently must include factors beyond those of the experimental measurements. The most likely cause of this large range is a variation in the composition of the mortar bars, and consideration must be given to the influence of the amount of reactive material in the mortar bars. We have carried out some experimental work on this. These influences will include a variation in the total amount of aggregate, a variation in the proportion of the reactive component in each given size range, and a variation in the grading curve of the aggregate. We have studied all three variables. The variation in the amount of reactive component in the individual size ranges has been found from Blyth's\textsuperscript{54} improved binomial confidence limits based on the inverse Beta distribution. The variation in the amount of each given particle size in a single bar has been measured experimentally, as has the variation in total mass of aggregate in each bar. The standard deviation of the total amount of aggregate in each bar was found to be in the range \( \pm 1.6–1.9\% \) of the amount expected. The variation in amounts falling in each grading division
for the sand used to make the specially graded aggregate was found to be as follows:

<table>
<thead>
<tr>
<th>Size range (mm)</th>
<th>Mass %</th>
<th>Standard deviation mass percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.36–5.0</td>
<td>7.1</td>
<td>0.62</td>
</tr>
<tr>
<td>1.18–2.36</td>
<td>8.4</td>
<td>0.60</td>
</tr>
<tr>
<td>0.60–1.18</td>
<td>16.9</td>
<td>0.49</td>
</tr>
<tr>
<td>0.30–0.60</td>
<td>51.3</td>
<td>1.27</td>
</tr>
<tr>
<td>0.15–0.30</td>
<td>16.3</td>
<td>0.97</td>
</tr>
</tbody>
</table>

The amount and standard deviation for the reactive blend, obtained by considering volume proportions for the grading curve of the mortar bars, were found to be as follows:

<table>
<thead>
<tr>
<th>Amount mass %</th>
<th>Standard deviation (calculated mass %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.3–5.0 mm</td>
<td>10% 0.54</td>
</tr>
<tr>
<td>1.18–2.36</td>
<td>25% 0.76</td>
</tr>
<tr>
<td>0.60–1.18</td>
<td>25% 0.76</td>
</tr>
<tr>
<td>0.30–0.60</td>
<td>25% 0.76</td>
</tr>
<tr>
<td>0.15–0.30</td>
<td>15% 0.63</td>
</tr>
</tbody>
</table>

The standard deviation of the glass in the two size ranges in our mortar bars was calculated to be 2.96 g in 37.5 g, and this, combined with the standard deviation for variation in the total amount of aggregate, gives a standard deviation of 1.2% to be attached to the 15% of glass.

These relationships were also modelled using a computer-based simulation. For the purposes of the simulation model, it is assumed that in any single trial, the total weight of the ith size fraction, \( w_i \), is drawn from a normal distribution \( N(\mu_i, \sigma_i) \), \( i = 1, 2 \), where parameters are defined by the experimental results. An equivalent number of grains in the ith fraction is estimated from 238.732\( r_i^3 \rho_i \), where \( r_i \) is the geometric mean radius (mm) of the size range, and \( \rho_i \) is the relative density. In the 1.18–2.36-mm fraction, which is the only one to have both glass and other sand, the number of glass grains expected to be present, \( n_g \), is modelled in each trial by a random sample from a binomial distribution \( B(n, p) \), where \( p \) is the expected proportion of occurrences of \( n \) grains. The diameters of these glass grains are then assigned by sampling from a uniform distribution in the range 1.18–2.36 mm. Since the density of the glass is known, the equivalent individual grain weights can be calculated, summed, and the weight of sand other than glass obtained by difference from the overall weight of the size fraction. The total sample weight is then given by summing over all the simulated size grades and the overall percent-age of glass is found. It was discovered that the simulated glass percentage had a small variable bias dependent on the magnitude of the difference between the glass and sand densities. This bias was corrected by setting \( z \) to 0.70. The results obtained from two independent sets of 10000 trials were then as follows:

<table>
<thead>
<tr>
<th></th>
<th>Run 1</th>
<th>Run 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>% glass</td>
<td>15%</td>
<td>15%</td>
</tr>
<tr>
<td>Standard deviation of glass</td>
<td>0.80%</td>
<td>0.78%</td>
</tr>
<tr>
<td>Total sample weight</td>
<td>248.89g</td>
<td>248.89g</td>
</tr>
<tr>
<td>Standard deviation (g)</td>
<td>4.82g</td>
<td>4.75g</td>
</tr>
</tbody>
</table>

The result obtained therefore corresponds reasonably with the experimental values, indicating that the standard deviation to be attached to the 15% of glass is 0.8%.

If it is assumed that the expansion is related to the glass content and the error bar in glass content is related to the variation in expansion, then for an expansion of 0.40%, the standard deviation would be 0.02% or 0.03%, depending on the value used for the standard deviation — in keeping with the actual standard deviation found experimentally. It is of course evident that the relationship between the chosen aggregate proportion and the pessimum will affect the relationship between aggregate amount and expansion. Variation will be highest on the limbs of the pessimum curve and should be minimal close to the pessimum proportion or at the extreme limits of the pessimum. For any given rock combination therefore the range will be dependent on the rock that is reactive and its pessimum proportion and the particular aggregate composition with respect to the pessimum. A high range would mean that the mixture is on the steepest part of the pessimum curve. Further work is therefore required to evaluate the effect of the pessimum on the range of results to be expected for a given test procedure. It can, however, be concluded that the range of results obtained will give an indication of whether the aggregate mixture is close to the pessimum. It is also clear that the inter-laboratory reproducibility will depend substantially on the pessimum composition and the statistics of the aggregate distribution between individual bars.

**Towards a reliable test**

Alkali immersion and autoclave methods offer excellent potential for development of a test but
at present the likely interlaboratory reproducibility means that great care is required in the interpretation of the result. Tighter specification is required to minimise random errors and to evaluate possible influences of the pessimum on the results obtained. It may prove necessary to blend aggregates in order to improve diagnostic quality. It is evident that performance of an aggregate in service is not alone a sufficient indicator of the reliability of the test unless this is accompanied by other details of the field conditions, mix design, and alkali levels in the field concrete and the proportions of lithologies in the concrete.

It is easier to reject than to accept since if expansion exceeds the reproducibility of the test or three times the bounding standard deviation (in the present case, 0.037) and the mortar bar contains obvious alkali silicate gel, then the aggregate must be regarded as unreliable. It may be used with given suitable selections of mix design and cement type. If the expansion approaches or slightly exceeds the reproducibility without gel formation, the cause of the expansion must be found. If the measured expansion is less than the expected reproducibility, then ambiguity may remain, and the test needs to be continued and repeated. If a test result of 0.14% expansion is observed then, although this is less than the discriminant value given by Hooton,25 the reproducibility would imply that the result is actually, say, 0.14 ± 0.08% and that there is about a 40% probability that the discriminant value would be exceeded in another test. Such a material could not be used with confidence. Clearly, the reproducibility of the test must be improved by an order of magnitude if it is to be used alone for engineering decisions involving the acceptance of aggregates that at present would be regarded as marginal. In view of the variation in aggregate proportions found for the present standard mortar bars, this does not mean an improvement in the system of mixing, measuring or storing, but an improvement in the representativeness of the aggregate in each bar. This could be achieved by the use of more bars or larger bars.

**Procedural suggestions**

1. The improvement in test result by replication is evident from Tables 4–7. If all five results were taken to give a single test result, after 14 days the expansion would be as follows:

<table>
<thead>
<tr>
<th>Laboratory</th>
<th>160 mm bars</th>
<th>250 mm bars</th>
<th>250 mm bars</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.318% ± 0.027</td>
<td>0.426% ± 0.020</td>
<td>0.397% ± 0.038</td>
</tr>
<tr>
<td>B</td>
<td>0.396% ± 0.047</td>
<td>0.397% ± 0.038</td>
<td></td>
</tr>
</tbody>
</table>

   However, the number of bars required increases as the expansion declines, and a method such as the FIR CUSUM calculation should be used to study the progress of the expansion, and, if necessary, the number of bars should be increased to give three sets of at least four bars.

2. The length of the bars is important, and marginally better results from the statistical point of view are to be obtained using 250-mm bars or longer than for the shorter bars. However, the thicker bars are easier to work with, have less tendency to cool during measurement and take the threaded end piece very well. Perhaps consideration should be given to the use of bars of, say, 300 × 40 × 40 mm, which would have substantially more aggregate and therefore reduce the effect of variation in aggregate amount and composition.

3. The storage conditions greatly influence the result obtained and the early age variability appears largely to reflect the ratio of the volume of alkali solution to the surface area of the bar. This should be fixed for each bar or individual set of four bars at a volume ratio of four, and bars should not be stored in previously used alkali or collectively in large tanks. The small difference between the shorter and longer bars found for laboratory A appears to be largely attributed to variations in the volume ratio of alkali solution to mortar.

4. The mortars should be kept wet at all stages since drying is likely to accelerate alkali ingestion.

5. In interpreting the result of the test, it is important that the aggregate tested matches that to be used in service. Separating individual rocks will be misleading. Opal obviously will give a strongly adverse reaction when present to a small extent but may appear innocuous at high concentrations. Tuffsite veins
may be reactive when tested alone but show no evidence of reaction when tested in an as-quarried mixture.

(6) It may be prudent to test certain aggregates in blends of 1:1 or other ratios with a known inert material with any significant difference in expansion between the pure aggregate and the blend being regarded as indicating a potential for reaction that requires further investigation.

(7) It is essential that a control aggregate and an inert aggregate should be available that can be tested along with the aggregate under investigation and with the same cement.

(8) Petrographic assessment of the cause of expansion is essential to the interpretation of the result obtained. Formation of recognisable amounts of gel coupled with damage to the aggregate in the test period should indicate rejection of the aggregate.

CONCLUSIONS

(1) Consistent results were obtained with a 14-day expansion of 0.4% which gave an upper bound standard deviation of 0.0365% expansion at zero expansion for both sizes of mortar bar. The figure for this upper bound of the standard deviation can be used as a unit of measurement of expansion with more than three times the standard deviation, indicating that significant expansion has occurred.

(2) Expansions less than 0.11% can arise from random errors, and expansion near this value requires the test to be conducted using at least three batches of four bars. The number of bars to be made and the duration of the test should be considered 3–4 days after the initiation of the first set of bars, but the current recommendations are inadequate for assessment of intermediate to low degrees of reaction. Consideration should then be given to making three sets of four bars as separate individual mixtures.

(3) In keeping with previous work, no significant differences were found with respect to the cement type or the details of the aggregate grading. The size of the mortar bar, however, is important, with large bars being easier to work with. The ratio of the volume of molar sodium hydroxide solution to the surface area of the mortar bar also critically affects the amount of expansion. This should be fixed at a ratio of 4:1, and a separate container should be used for each set of bars. It is possible that the rate of expansion is affected by the exchange of components with the ambient solution and hence will be influenced by the presence of bars of other mixtures in the solution or if the solutions employed have previously equilibrated with mortar.

(4) The accelerated mortar bar test such as the ASTM 1260-94 has considerable advantages over the slower and unreliable ASTM C-227 and over the unreliable ASTM C-289 for engineering purposes and, given appropriate quality control measures, should provide useable data. Experience is needed in interpreting the test result, particularly bearing in mind the differing styles of reaction that occur with different aggregates, and the differing rates at which expansion can develop. It is essential that the aggregate and the mortar bars should be examined petrographically in order to assess the nature of the material that is actually reactive and the style of reaction that is taking place, if any. Consideration of a scheme such as that given in Table 11 is required for the interpretation of the test result.

(5) The various forms of concrete prism test have the same problems of unknown repeatability and reproducibility as have affected the accelerated mortar bar tests. In addition, because the expansions are generally much smaller and require longer periods for their measurement, the concrete prism tests may be more difficult to interpret than the mortar bar tests. They have, of course, the advantage that the aggregate can be tested in a size closer to that in which it will be used in practice, and this may be particularly important where possibly slow, late expanding aggregates are involved. Proper correlation and close comparison with field experience and the observa-
tions of the bars themselves are the best guide to the validity of the results obtained in the accelerated mortar bar test, and, as was shown by Brown, the test can be carried out on aggregate removed from concrete damaged by the reaction if this is necessary. However, the use of field experience in interpreting both positive and negative test results may be unsatisfactory unless the full details of the field occurrence are available.

ACKNOWLEDGEMENTS

This study has been funded by the Norwegian Research Council, where it is part of the NORCON project. The paper is a part of B. J. Wigum's Dr.ing-study, which is supported financially by the Royal Norwegian Council for Scientific and Industrial Research.

REFERENCES


21. Fournier B. & Bérubé M.-A., A comparison of laboratory testing methods for evaluating potential alkali-reactivity in the St. Lawrence lowland (Quebec, Canada). Conference Papers of the 9th International Conference on Alkali–Aggregate Reaction in Concrete,
Concrete aggregates and alkali-aggregate reaction


27. CSA (Canadian Standards Association) 1990: Standards CAN/CSA A23.2-M90 (Concrete Materials and Methods of Concrete Construction), and CAN. CSA A23.2-M77 (Methods of Test for Concrete), and, CSA (Canadian Standards Association) Proposed Changes to CSA A23.1 and A23.2 as Regards Alkali-Aggregate Reactivity and Associated Test Methods. Cement-Aggregate Reactivity Sub-Committee CSA-A5 Task Group, Draft Document, October 1993, p. 80.


45. Bakker, R. F. M., The influence of test specimen dimensions on the expansion of alkali reactive aggregate in concrete. Proceedings of the 6th International Conference, Alkali in Concrete, Research and Practice,


Chapter 9

Examination of Microstructural Features of Norwegian Cataclastic Rocks and Their Use For Predicting The Alkali-Reactivity in Concrete

BØRGE JOHANNES WIGUM

Wigum, B.J. 1995: Examination of Microstructural Features of Norwegian Cataclastic Rocks and Their Use For Predicting The Alkali-Reactivity in Concrete. Accepted for publication in Engineering Geology, Amsterdam, The Nederlands.

B.J. Wigum, Department of Geology and Mineral Resources Engineering, University of Trondheim, The Norwegian Institute of Technology, 7034 Trondheim, Norway
Examination of microstructural features of Norwegian cataclastic rocks and their use for predicting alkali-reactivity in concrete

Børge Johannes Wigum

Department of Geology and Mineral Resources Engineering, University of Trondheim,
The Norwegian Institute of Technology, 7034 Trondheim, Norway

Received 17 March 1995; accepted 6 July 1995

Abstract

The microstructural features of various Norwegian rock samples have been studied and quantified by different techniques. The majority of the rocks studied were different types of cataclastic rocks. Expansion results from a modified version of the NBRI Mortar-Bar Test for alkali reactivity are used for correlation with the quantitative parameters. The aim is to identify microstructural features in the various rock types which promote the alkali-aggregate reaction, and to subsequently use these findings to improve and provide guidelines for engineering practice in order to predict and make more accurate determinations of potentially reactive aggregates. The deformation processes of rocks involve the general process of straining of quartz, grain size reduction and subgrain development. This is characteristic of mylonitisation. This study demonstrates that the grain size reduction of quartz enhances reactivity by increasing the surface area of quartz grain boundaries available for reaction, and thus giving an overall increase in surface energy. Subgrain development will, besides the high surface area, contribute even more to an enhanced reactivity, due to the high dislocation density associated with the quartz subgrain boundaries. The total grain boundary area of quartz, and the mean grain size of quartz appear to be the most favourable quantitative parameters related to the expansion of different rock types. The measurement of the total grain boundary area of quartz, will enhance the value and improve the effectiveness of the petrographical examination as an engineering tool to screen potentially reactive aggregates.

1. Introduction

It was suggested by Gogte (1973) that alkali-aggregate reactivity should be viewed from a new angle. Some common Indian crystalline rock types were evaluated for their alkali-reactivity, and it was concluded that the potential alkali-reactivity of some granites, granodiorites, gneisses, charnockites, quartzites and schists was related to the relative amount and straining effects of quartz. It is recognised that the reactivity of certain slow/late reactive rock types is dependent upon the microstructural features of the rock types, rather than the mineralogical composition and petrographic nomenclature. Quartz bearing aggregates that exhibit microstructural features such as undulatory extinction, deformation bands, lamellae and subgrain development, have been reported to have caused slow/late alkali-aggregate reaction in con-
crete structures. All these microstructural features can be observed and recognised by a variety of names, produced both during the deformation, annealing and recrystallisation period. However, all these microstructural features could in one way or another influence the reactivity of the quartz. The main question is which microstructural features are likely to have the most important influence upon the reactivity of the aggregate, and how best to identify these parameters.

Different terms are used for rock types exhibiting microstructural features associated with deformation, including granitic rocks, deformed granitic rocks, rocks that have been subjected to tectonic and dynamic deformation, and various sorts of cataclastic rocks (cataclasites and mylonites). However, the knowledge and quantification of the microstructural features of such rock types are still rather limited.

Some authors have made attempts to examine and assess microstructural features and other factors which influence the reactivity. However, insufficient attention has been given to quantifying the microstructural properties of the rock, and to relate this to the reactivity. There is a necessity for engineering practice to develop methods and procedures to enable more accurate determination of potentially reactive aggregates, based on examination and quantification of microstructural features. It is suggested by French (1991) that a full description of the rock sample is required, rather than the use of standard nomenclature. Also Jensen (1993) suggested that the reactivity of quartz-bearing, slowly expansive rocks should be evaluated by microstructural analyses and known field experience, rather than a list of rocks based on traditional classification.

The introduction of a new method to measure the undulatory extinction (UE) angle of strained quartz by Dolar-Mantuani (1981), was intended for measuring the amount of dislocation of optical arrangements in the crystal lattice, and to relate this to the reactivity of the material. The method has previously been used to a large extent as a tool for quantifying the reactivity of quartz-bearing rocks exhibiting strained quartz. However, many scientists have questioned the examination of the undulatory extinction (UE) angle of strained quartz and the value of the method. Because the principle of the method has limitations, the measuring of the undulatory extinction angle of strained quartz is no longer considered to be a reliable parameter for predicting alkali-reactivity (Grattan-Bellew, 1986, 1992; French, 1992).

Grattan-Bellew (1986, 1992) suggested that rather than the undulatory extinction in quartz, the reactivity may be due to the occurrence of microcrystalline quartz in aggregates. French (1992) claims also that wherever meta-quartzites or rocks containing strained quartz are reactive, the quartz grains exhibit either strain lamellae or are cataclasized or otherwise contain microcrystalline grains on larger grain boundaries or along fracture planes of various types.

Petrographic examination of potentially reactive aggregates, partially based on microstructural properties, has shown that a relationship does exists between the amount of reactive rocks in the aggregate and expansion obtained by the accelerated mortar-bar test (Wigum and Lindgård, 1994).

Zhang et al. (1990) investigated the microstructure of various reactive aggregates by using analytical transmission electron microscopy (TEM). The study suggested that the source of reactivity is in connection with large areas of grain boundary arising from a smaller quartz grain size, and the high density of dislocation present within many of these grains. In an investigation performed by Kerrick and Hooton (1992) mylonites were investigated for their reactivity. Besides the effect of microcrystalline quartz, it was shown that the reactivity depended upon the degree of foliation (schistosity) of the rock. The investigation concluded that the reaction primarily occurs at the grain boundaries of microcrystalline quartz formed by dynamic recrystallisation of strained quartz. In Sweden and Norway cataclastic rocks (especially mylonites) with strained quartz and subgrain development are found in structures in several areas exhibiting deleterious expansion due to alkali-reactivity (Jensen, 1990, 1993; Lagerblad and Trägårdh, 1992a). According to observations in Norwegian structures, cataclastic rocks are now considered the most common source of alkali-reactive rock types (Jensen, 1993). An interpretation of the reactivity based on the microstructural
features of these rocks has not yet been established. Thomson and Grattan-Bellew (1993) and Thomson et al. (1994) investigated the reactivity of various mineralogical and textural components of metamorphic deformed rocks. The study showed that the most reactive component appears to be the microcrystalline quartz that has undergone significant subgrain development, but not complete recrystallisation. It was also observed that zones with notable undulatory extinction were not significantly more reactive than the non-deformed quartz. However, the relative importance of grain size versus the degree of subgrain development remained unresolved. It was also emphasised that a simplified approach involving measurement of undulatory extinction angles may lead to an incorrect estimation of strain, and therefore an incorrect estimation of the potential reactivity.

The purpose of this work is to determine and quantify microstructural features, for certain rock types which are influenced by cataclasis, in an attempt to identify methods which could be adequately used for interpretation and prediction of the reactivity. The objective is to provide a better understanding of the reactive components in some selected reactive materials. Subsequently this might contribute to the development of reproducible methods and procedures for engineering practice. In time this could lead to better methods for prediction and determination of the potential reactivity of certain types of aggregates, based on examination and quantification of microstructural features.

2. Theoretical background

Before introducing the different results in this study, it is appropriate to first present a theoretical review of the various microstructural features which develop in quartz during the ductile deformation and recovery processes, and then discuss how these features might have an influence on the reactivity.

2.1. The important role of subgrain boundaries

Any understanding of the ductile deformation of rocks and the interpretation of possible factors contributing to the reactivity must primarily be based on the role that crystal defects and dislocations play in the deformation process. Information on this is found in the geological literature dealing with the topics of recrystallisation and recovery of metamorphic rocks. The dissolution and transformation of reactive material into gel during the alkali-reaction might be considered to follow a similar mechanism as the crystallisation and recrystallisation occurring in metamorphic rock.

When the stress of a specific rock exceeds a critical value, some phases will, depending on their mechanical properties, undergo plastic deformation while others deform in a brittle manner. In the case of quartz, however, deformation by crystal-plastic processes is characteristic over a broad range of conditions (Barker, 1990). The increase in stress required for continued deformation is called work hardening, a process well known in metals. The introduction of line defects, generally referred to as dislocations, are defects that largely influence the mechanical behaviour of crystals. Dislocations are present in virtually all crystalline solids, and typically form at low densities during crystal growth, whereas at very high densities they may be generated during plastic deformation of crystals. The strain energy associated with a dislocation increases the free energy of a crystal, and dislocations are therefore unstable defects (Hobbs et al., 1976).

Different terms have been used to describe the various optical features of planar defects observed in quartz, and different interpretations have been proposed for the origin of these features. Some authors interpret particular microstructural features to be the result of the deformation process, while others interpret them to be the result of the recovery processes. According to Suppe (1985) the permanent bending of a crystal lattice results in a change in lattice and optical directions across a crystal so that it does not extinguish as a whole when viewed between crossed polars under the microscope. A region of extinction sweeps across the crystal as the stage is rotated and the crystal is said to exhibit undulose, undulatory or strain extinction. Suppe regarded the detailed pattern of undulose extinction to reflect the degree of polygonization of a crystal. If the extinction sweeps
smoothly across a deformed crystal, then there is little or no polygonization, at least on an optical scale. However, if the extinction sweeps stepwise across a crystal, the crystal is polygonized at an optical scale. Each small area of homogeneous extinction is a subgrain, and their boundaries are subgrain boundaries. Smaller subgrains may occur inside the larger subgrains, causing segmented undulose extinction within the larger subgrains when viewed in the optical microscope (Vernon, 1976). The subgrain boundaries which exhibit planar orientation within grains, exhibit small lattice disorientation, of the order of 1 to 5°. In the optical microscope these separate regions of slightly differing extinction position are called low-angle boundaries (Hobbs et al., 1976). As the radius of lattice curvature becomes small (i.e. the crystal is more sharply bent) the undulose crystal may be composed of narrow regions of slightly different orientations referred to as deformation bands (Spry, 1976). Deformation lamellae are said to be narrow (of the order of 0.5 to 5 μm), approximately planar features developed within single grains that have slightly different refractive indexes at the host grain. They are common in quartzites deformed at low grades of metamorphism (Hobbs et al., 1976). Most deformation lamellae formed in naturally deformed quartz are dislocation walls forming narrow basal subgrains. White (1973b) investigated optical features in deformed quartz by studying identical microstructural features in both the optical and electron microscope in an attempt to determine which kind of lattice defects was responsible for these optical features, and to clarify the nomenclature ambiguities related to this. The investigation showed that all the microstructural features, including undulatory extinction, deformation bands, deformation lamellae and subgrain development, could be related to dislocation sub-structures, in particular to the development of subgrains. From this information, the interpretation was that the lattice is systematically bent at each subgrain wall, and the net result is seen in the optical microscope as undulatory extinction. Smaller subgrains are formed within the larger by low-angle boundaries of 1° or less. These cannot always be detected individually in the optical microscope, but their presence may be indicated by the development of segmented undulatory extinction within the large subgrains.

The exact size of quartz grains is difficult to determine. In the case of polygonization and subgrain development it is more difficult to distinguish between separate grains, where the grain boundaries might be quite ambiguous. Both the twodimensional limitation of the thin-section and the limitation of optical resolution will have a significant influence upon the results. Ord and Christie (1984) found the size of subgrains observed by optical microscopy to be an order of magnitude larger than those observed by TEM. The size of subgrains may be difficult to observe by optical methods because smaller subgrains may occur inside larger subgrains which cannot be resolved due to the limit of resolution of the microscope. Consequently, the quartz grain size measurements in this study do not represent an accurate distribution of the quartz grain size. However, based on the previous review regarding the origin of microstructural features in deformed quartz, it appears reasonable to consider the subgrain development as a parameter of great significance regarding the degree of deformation, and hence a rational parameter regarding the potential reactivity of an aggregate. Considering the undulatory extinction angles (UEA) alone, will only give an idea that the crystals are bent, hence the measurement cannot be regarded as a quantitative measurement of the properties of the material. The value of UEA will be dependent upon the orientation of the quartz grains, and more importantly, the size of the quartz grain itself (Rogers, 1990). Other studies have shown this method not to be reproducible (Andersen and Thaulow, 1989).

2.2. Activation energy, driving forces and preferential site of reaction

On the basis of the significance of the subgrain development, the remaining part of this paper will consider the influence of microcrystalline quartz and the subgrain development upon the reactivity of a material. To obtain a better understanding of the role that various factors play in the reaction, the different thermodynamic factors in the reaction
must be assessed. The following review considers the general studies on these factors regarding metamorphic rocks.

It is well established that a reaction must overcome a certain activation energy and take place at a certain rate under a certain driving force. The activation energy may be regarded as the barrier that must be overcome for a reaction to be able to take place (Lagerblad and Trägårdh, 1992b). This activation energy will be different depending on the form of silica. The changes and reactions are also accompanied by the nucleation of new phases. In the presence of impurities such as particles, grain boundaries, dislocations and strained areas, the nucleation will occur at much lower activation energies than in homogeneous crystals. Barker (1990) claims that the increase in dislocation density at subgrain margins raises the surface energy and provides more free bonds. The increase in surface energy is due to the activation energy for reaction and nucleation of new phases, and means that strained crystals will offer more favourable sites for nucleation. Blum et al. (1990) claim that the strain energy around dislocations promotes dissolution by a mechanism very similar to unassisted nucleation on a defect-free surface, but at an accelerated rate. In crystalline solids, nucleation commonly occurs along grain boundaries or deformation structures. The formation of a nucleus at a boundary or dislocation involves the destruction of part of an existing surface or dislocation, so that extra free energy is released to aid formation of the nucleus interface (Vernon, 1976). Nucleation is favoured at corners, triple points or high-angle boundaries and dislocations (Spry, 1976). It is also greatly assisted by the density of dislocations produced during deformation. Vernon (1976) found that the stored strain energy raises the free energy, which may permit metastable nucleation and growth outside its thermodynamically stable field. Wintsh and Dunning (1985) calculated the effect of dislocation density on the aqueous solubility of strained quartz. Their calculations showed that at a very high dislocation density the solubility of quartz is significantly enhanced. However, the total volume of quartz containing this very high dislocation density is rather small, mainly found in the region of the subgrain walls, consequently leading to increased solubility in these regions.

2.3. Other preferential sites of reaction

2.3.1. Inclusions

When dislocations group during deformation and recovery, the dislocations will pass through the crystal during strain. Consequently the OH− ions can become incorporated into the crystal structure, converting an Si–O–Si bridge to Si–OH:HO–Si. The protons can recombine with hydroxyls to form water and eventually create inclusions and bubbles containing not only water but other ions, for example, alkali ions. A build-up of inclusions, bubbles and impurities will occur at annihilation areas and preferentially at subgrain boundaries (White, 1973a). The formation of free water in micrometre-size inclusions, bubbles or voids from hydroxyl ions has been observed in various investigations (McLaren and Hobbs, 1972; Balakirev, 1981). Birkeland and Carstens (1969) observed the occurrence of bubbles of water formed by annealing of water-weakened quartz. Water enrichment is expected at the dislocation because these regions are charged. The interstitial ions, such as H+, Li+, Na+ and OH−, will be the most effective charge compensators in quartz because of their high diffusivities. Protons from water as well as alkali and hydroxyl ions will preferentially segregate to dislocations because of their mobility in the quartz lattice (White, 1973a).

2.3.2. Myrmekite

Myrmekite is the name of the symplectic intergrowth of vermicular quartz and plagioclase, resulting from the retrograde replacement of K-feldspar. Myrmekite is becoming commonly recognised in deformed rocks. The process of deformation appears to contribute indirectly to nucleation of myrmekite (Vernon, 1991). Concentrations of stress might destroy the K-feldspar-plagioclase-quartz equilibrium and induce replacement. Due to the unsteady state of myrmekite, it has been observed that myrmekite is one of the first constituents of a deformed rock to recrystallise (Vernon, 1991). The unsteady state of the quartz component in the myrmekite, and
the probably low crystallinity, have to be considered when assessing a potentially reactive aggregate. Rao and Sinha (1989) studied the mortar-bar expansion of various slow/late expansive Indian aggregates. The enhanced expansivity for one of the aggregates was considered to be due to the occurrence of myrmekite.

2.4. The accessibility of the reagent

Even with an enhanced rate of dissolution and lower activation energy, the alkali solution from the surrounding cement paste has to be able to penetrate into the dense crystalline material. In order to activate the reactive aggregates the reagent (alkalies and hydroxyl ions) must be able to reach the reaction site within the aggregate. It is a well-known fact that the various solid-state reactions, phase transformations and other microstructural changes occurring in solid-state materials are, in general, diffusion-controlled. The diffusion process itself, in turn, is strongly influenced by the structure of these materials. Imperfections such as solid-solid interface, and grain boundaries in particular, are the most important short-circuiting path. Diffusion along grain boundaries is orders of magnitude faster than through the bulk of the crystal, and plays a key role in many processes (Kaur and Gust, 1989). Along with various microstructural defects within crystals, defects such as cracks and cavities could also act as access paths for alkalies. Spry (1976) claims that diffusion through polycrystalline aggregates is aided by the presence of cleavages and cracks. Simons and Richter (1974) claimed that microcracks in rocks are very common and are important features of most rocks. An investigation by Sprunt and Brace (1974) observed by electron microscope strings of long, thin low-aspect ratio cavities which often followed grain boundaries in granites. The amount of grain boundaries occupied by cavities reached up to 90% in some granites; however, these boundaries are below the resolution of most optical equipment. Lagerblad and Trägårdh (1992a) suggested that the presence of initial cracks is needed in the aggregates to initiate the process of slow/late alkali-reaction. Jensen (1993) proposed a hypothesis on the sequential development of alkali-reaction in Norwegian slow/late reactive aggregates. He suggested that the access of alkalies into the aggregate occurs through interfacial cracks between detrital grains and/or mineral crystals which act as paths for the alkalies. French (1992) suggested that the accessibility of alkalies may occur along planes of weakness generated in the crushing or through a natural fracture, possibly occurring along a bedding plane or cleavage. White (1979) observed some cleavages in mylonites due to the preferential alignment of quartz grain boundaries and phyllosilicates parallel to the mylonitic foliation. Kerrick and Hooton (1992) found that mortar-bar expansion exhibited a marked dependence upon the foliated (schistose) structure of the rock.

3. Experimental procedure

3.1. Petrographic examination and grain size measurement

To classify the different rock types and quantify corresponding types of microstructural features, thin-sections (25 × 30 mm) of samples were examined under a petrographic microscope. Classification of the cataclastic rocks in thin-sections was done according to Higgins (1971). He divided cataclastic rocks into two main types; those without, and those with primary cohesion. Cataclastic rock is a general term for all rocks produced by cataclasism that are commonly felsic and/or silicic in composition. In this work the term cataclastic rock includes metamorphic rocks with primary cohesion due to a combination of crystalloblastic and cataclastic processes (Table 1).

Besides general petrographic examination, the quartz grain sizes, including subgrains, were measured for each rock type by point-counting in thin-section. By using an automatic point counter (Swift-model F), quartz grains along lines in the thin-section were measured. Approximately 200 points for each thin-section were counted. In the case of foliated rocks with elongated quartz grains, the lines were measured 45° to the parallel foliation of the rock to obtain an average grain size diameter. As a measurement of the grain size, the length
Table 1
Classification of cataclastic rocks with primary cohesion; all rocks are gradational (after Higgins, 1971)

<table>
<thead>
<tr>
<th>Rocks with primary cohesion</th>
<th>Approximate size of most porphyroclasts (visible to naked eye)</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Cataclasite dominant over neominalisation - recrystallisation</em></td>
<td><em>Mylonite gneiss (mylonite schist)</em></td>
</tr>
<tr>
<td>Rocks without fusion structure</td>
<td>Rocks with fusion structure</td>
</tr>
<tr>
<td><em>50%</em></td>
<td><em>&lt; 0.2 mm</em></td>
</tr>
<tr>
<td><em>Microbreccia</em></td>
<td><em>Mylonite</em></td>
</tr>
<tr>
<td><em>50%</em></td>
<td><em>&lt; 0.2 mm</em></td>
</tr>
<tr>
<td><em>Cataclasite</em></td>
<td><em>Blastomylonite</em></td>
</tr>
<tr>
<td><em>10%</em></td>
<td><em>&lt; 0.2 mm</em></td>
</tr>
<tr>
<td><em>Ultramysonite</em></td>
<td></td>
</tr>
</tbody>
</table>

of a quartz grain lying at the line was measured. For measurement of the mean grain size of quartz, the d<sub>50</sub> was used.

In order to estimate the grain boundary area of quartz, the following assumption was made. Each quartz grain, including subgrains, was assumed to be cubic in shape. The average grain size between two main selected fractions was used to calculate an area for this specific part of the grading. The area obtained was multiplied with the proportion of the quartz grains within these two main fractions. This was done for all the selected fractions in the grading. By multiplying this grain boundary area with the quartz amount in the rock type, obtained by the X-ray diffraction analysis (XRDA), an estimate for the total available grain boundary area of quartz (m<sup>2</sup>/cm<sup>3</sup>) in each sample was achieved.

3.2. Additional measurements related to the reactivity

Along with the petrographic examination and the point-counting of quartz grain sizes, other techniques were used in an attempt to obtain quantitative parameters which mirror the microstructural properties of quartz in the various rock samples.

Florke (1961) has shown by experimental work that the finer quartz grain sizes (30 to 60 μm) are not detectable by differential thermal analysis (DTA), while it is detected by XRDA. On the basis of this knowledge, both DTA analyses and XRDA were used to determine the quartz content, in an attempt to possibly observe differences in quartz amount as a measurement of the amount of microcrystalline quartz. In addition to the quartz amount, XRDA also gave the amount of associated minerals in the samples.

A method to determine the index of crystallinity (CI) of quartz was introduced by Murata and Norman (1976). According to them, the CI appears to be largely a function of the quartz grain size (up to about 1 μm diameter) and will also be affected by lattice distortions induced by mechanical stress. The method involves measuring the relative heights of the (2132)z<sub>0</sub> peak and the (2132)z<sub>1</sub> peak which occur between the 2θ angles of 67° to 68° in XRD traces. The CI is divided on a scale from <1 to 10, where 10 represents well-crystallised euhedral quartz. The 2θ interval of 60° to 70° was scanned at 1/4 degree/min. A sample of a clear single crystal euhedral quartz was used to calibrate the scale of crystallinity.

In order to activate reactive aggregates, the reagent must be able to reach the reaction site within the aggregate particles. By determining the water absorption of the rock samples according to a modified version of the ASTM C97-90 (1990),
an indication of the accessibility of reagents to permeate into the material could be obtained. The water absorption was measured on cubes (30 × 30 × 30 mm) of the different rock samples which were dried for 24 h in a ventilated oven at a temperature of 105°C. After drying, the specimens were cooled at room temperature for 30 min and weighed to the nearest 0.02 g. The specimens were then immersed in distilled water at 20°C for 48 h. At the end of this period the specimens were removed from the water bath one at a time, surface-dried with a damp cloth, and weighed to the nearest 0.02 g. The mass percentage absorption was calculated as follows:

\[
\text{Absorption}_{\text{mass}} \% = \left[ \frac{(B - A)}{A} \right] \times 100
\]

where \( A \) = mass of the dried specimen, and \( B \) = mass of the specimen after immersion.

3.3. Test procedure to determine potential alkali-reactivity

A Norwegian modified version of the NBRI Mortar-Bar Test was used to measure the reactivity and expansion of different samples. The test, introduced by Oberholster and Davies (1986), has been slightly modified in Norway. This involves demoulding the mortar-bars first after 48 hours, and using mortar-bars with dimensions according to the RILEM size; 40 × 40 × 160 mm (Jensen, 1993). After de-moulding, the bars are immersed in water in closed containers, and kept at a constant temperature of 80°C in an oven. After 48 h, the lengths of the mortar-bars are measured hot as a zero reading. Then the bars are immersed in an 80°C 1 N NaOH solution, and subsequent expansion is measured after 4, 7, 14, 28, 42, and 56 days, respectively.

4. Results

4.1. Geological sampling and petrographic description of rock samples

Twelve different rock samples were collected from five different geological areas in Norway (Fig. 1). The petrographic classification and mineralogical composition of the samples are presented in Table 2. When performing the petrographic classification and description of the different rock types, the microstructural properties of quartz were the primary consideration.

A collection of six rock types were obtained from the Aunvatnet and the Namsskogan areas. Both areas are part of the Helgeland Nappe Complex, located in the Nord-Trøndelag and Nordland county of Norway. Rocks in the first area, Aunvatnet, are part of the Bindal Massif, where Kollung (1967) was first to report about the mylonites along the western boundary of the Bindal Massif. Roberts et al. (1983) carried out tectonic studies in the area, and located an extensive zone of mylonitisation. The thrust was reported to contain series of mylonite horizons exhibiting largely ductile deformation at least 200 m, and possible up to 600 m thick. The horizons show variable and repetitive transformation from massive porphyritic granites into blastomylonites and
### Table 2
Petrographic classification and major mineralogical composition

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Rock names</th>
<th>Mineralogical composition (%) obtained by:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>DTA Excel</td>
</tr>
<tr>
<td>1.1</td>
<td>Mylonite</td>
<td>29</td>
</tr>
<tr>
<td>1.2</td>
<td>Ultramylonite</td>
<td>29</td>
</tr>
<tr>
<td>1.3a</td>
<td>Mylonite</td>
<td>33</td>
</tr>
<tr>
<td>1.3b</td>
<td>Mylonite,</td>
<td>26</td>
</tr>
<tr>
<td>1.4</td>
<td>Porphyritic granite</td>
<td>16</td>
</tr>
<tr>
<td>2.1</td>
<td>Cataclasite</td>
<td>80</td>
</tr>
<tr>
<td>3.1</td>
<td>Blastomylonite</td>
<td>29</td>
</tr>
<tr>
<td>3.2</td>
<td>Micro-granite</td>
<td>29</td>
</tr>
<tr>
<td>3.3</td>
<td>Mylonite</td>
<td>40</td>
</tr>
<tr>
<td>4.1</td>
<td>Ultramylonite</td>
<td>6</td>
</tr>
<tr>
<td>4.2</td>
<td>Gneiss</td>
<td>26</td>
</tr>
<tr>
<td>5.1</td>
<td>Mylonite</td>
<td>23</td>
</tr>
</tbody>
</table>

Ultramylonites. A gradual increase of quartz- and SiO₂-content due to the mylonitisation at the expense of the feldspar was also reported.

Four of the samples from Aunvatnet (samples 1.1, 1.2, 1.3a, and 1.3b) belong to the group of mylonites, with typical foliation structures, and where cataclasism dominates over neo-mineralisation. Three of the rock types (samples 1.1, 1.3a, and 1.3b) are classified as mylonites, with cataclasism dominant over neomineralisation, while sample 1.2 is classified as an ultramylonite, where relative small porphyroclasts of feldspar are surrounded by a subgrained quartz matrix (Fig. 2). All the mylonites are typical with fractured feldspar surrounded by foliation structures of subgrained and elongated quartz and lenses of recrystallised quartz (Fig. 2, Fig. 3 and Fig. 4). The reason for selecting samples 1.3a and 1.3b was to study the potential differences in microstructures and reactivity between the two samples, which were located only a few decimetres from each other. The porphyritic granite (sample 1.4) represents the parent rock close to the extensive zone of mylonitisation. The porphyritic granite has maintained the original coarse quartz grains. However, most of the quartz

---

**Fig. 2.** Ultramylonite (sample 1.2). Subhedral feldspars are surrounded by a matrix consisting of quartz and mica exhibiting foliation structures (crossed polars).

**Fig. 3.** Mylonite (sample 1.1). Feldspar grains are rigid and deform brittle. Note foliation structure in the matrix of microcrystalline and subgrain quartz, but also lenses of recrystallised quartz (crossed polars).
grains exhibit strong undulatory extinction, and many of them contain parallel strain lamellae indicating some ductile deformation (Fig. 5). The occurrence of myrmekite could also be observed in some of the feldspars in the porphyritic granite.

The rock sample from the second area, at Namsskogan (sample 2.1), is classified as a cataclasite, deformed at a brittle regime. The brittle deformation has crushed the quartz to a microcrystalline matrix. However, the microcrystalline quartz should not be regarded as subgrains. The rock shows a lack of foliation structure.

Three rock types (samples 3.1, 3.2 and 3.3) were collected from the third area, in the southeastern Precambrian region, between the Permian Oslo rocks to the west and the Swedish boundary to the east. They were all collected from a second major mylonite zone, Precambrian of age, which separates the Romerike gneisses from the Østfold grey gneisses. This zone stretches from Lake Øyern to the Swedish border (Oftedahl, 1980). Sample 3.1 was collected at a location south within the mylonite zone. This rock sample is classified as a blastomylonite due to the dominance of recrystallisation of quartz grains. Even though sample 3.2 is located within the main mapped mylonite zone, the rock appears in thin-section to show no signs of deformation and was classified as a microgranite. It might be the case that the rock originally was a mylonite, where the quartz on a later stage has been subjected to complete recrystallisation. The quartz grains exhibit no sign of deformation, while the feldspar exhibits some sericitization. From within the same area sample 3.3 was collected north in the mylonitic zone, which borders with the rocks belonging to the Oslo region. This rock is classified as a mylonite (Fig. 6). Elongated quartz grains show commonly microfolding. Some of the quartz grains show sutured grain boundaries and the beginnings of subgrain development, while some areas are granulated into small subgrains of quartz. It also contains a relatively high content of finely distributed micas and chlorite.

At the fourth area, two rock types were collected
from the Fosen Peninsula. Sample 4.1 was located along the shore, north of Stjørdalsfjorden, which is part of the Hitra-Snåsa Fault (Gronlie et al., 1991). This rock is classified as an ultramylonite due to relatively small porphyroclasts of feldspar surrounded by a subgrained quartz matrix. Sample 4.2 was located close to the valley of Skaudalen, which is part of the Verran Fault. Petrographic examination of the rock showed it was a typical gneiss, with alternating bands of biotite and quartz/feldspar, with some minor amount of garnet.

Only one rock type (sample 5.1) was collected from the fifth area, located in the southwest part of the Norway. This rock is part of the Hardanger-Ryfylke Nappe Complex. The main thrust zone in this nappe complex lies just above, or directly upon the sub-Cambrian peneplain (Ofteedahl, 1980). From petrographic examination the rock was classified as a mylonite with significant subgrain development (Fig. 7).

4.2. Mineralogical composition and other experimental data

The amounts of quartz, obtained by both XRDA and DTA, are presented in Table 2. In addition this table also outlines the main mineralogical composition for the different samples, as obtained by XRDA.

In Table 3 other additional parameters are presented. Most of the samples exhibited a higher quartz content from DTA than from XRDA, which gives a typical XRDA/DTA ratio less than 1.00. However, some samples show significantly higher values for the quartz content obtained by XRDA than by DTA. The index of crystallinity varies from 6.58 to 9.54. The measured mean grain size of quartz (d50), and the estimated total grain boundary area of quartz (m²/cm²) are also presented in Table 3. The total grain boundary area of quartz is generally a function of the grain size distribution. However, the values of the grain boundary area are strongly influenced by the quartz content, since it is included in the estimation of the total grain boundary area. Table 3 also includes the results of the water absorption.

4.3. Mortar-bar expansion

The development of expansion for different aggregates is presented in Fig. 8, based on an average from three mortar-bars. These results show that the ultramylonite (sample 1.2) has the highest expansion. However, all the different cataclastic rocks show an expansion greater than 0.1% after 14 days, and are classified as reactive according to the Norwegian 14-days expansion limit (Norsk Betongforenings Publikasjon Nr. 19, 1991). The micro-granite (sample 3.2) and the gneiss (sample 4.2) are both slightly below the 14-days limit, while the porphyritic granite (sample 1.4) just reaches the 0.10% limit. Note, the expansion development for the mylonite (sample 3.3) which is relatively low up to 7 days, but thereafter this aggregate exhibited a much higher rate of expansion than the rest of the samples, and continued to do so even after 14 days.

5. Discussion

When evaluating all the different parameters possibly mirroring the grain size of quartz, and the degree of deformation, it was of interest to correlate these parameters with the average expansion of the different samples. The average expansion after 14 days was chosen as the response variable for the various parameters. The purpose of this study is to determine which of the micro-

Fig. 7. Mylonite (sample 4.1). Subgrain development in quartz (crossed polars).
Table 3
Various parameters measured for different samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Quartz ratio XRD/DTA</th>
<th>Index of Crystallinity</th>
<th>Quartz grain size $d_{50}$ (mm)</th>
<th>Total grain boundary area of quartz (m$^2$/cm$^3$)</th>
<th>Water absorption (mass %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>0.90</td>
<td>6.58</td>
<td>0.02</td>
<td>0.130</td>
<td>0.22</td>
</tr>
<tr>
<td>1.2</td>
<td>1.10</td>
<td>7.72</td>
<td>0.01</td>
<td>0.160</td>
<td>0.18</td>
</tr>
<tr>
<td>1.3_a</td>
<td>0.79</td>
<td>7.45</td>
<td>0.04</td>
<td>0.057</td>
<td>0.33</td>
</tr>
<tr>
<td>1.3_b</td>
<td>0.85</td>
<td>7.65</td>
<td>0.04</td>
<td>0.046</td>
<td>0.27</td>
</tr>
<tr>
<td>1.4</td>
<td>0.63</td>
<td>7.94</td>
<td>0.35</td>
<td>0.003</td>
<td>0.39</td>
</tr>
<tr>
<td>2.1</td>
<td>1.13</td>
<td>7.88</td>
<td>0.33</td>
<td>0.297</td>
<td>0.93</td>
</tr>
<tr>
<td>3.1</td>
<td>0.55</td>
<td>7.87</td>
<td>0.05</td>
<td>0.022</td>
<td>0.19</td>
</tr>
<tr>
<td>3.2</td>
<td>0.48</td>
<td>8.26</td>
<td>0.17</td>
<td>0.007</td>
<td>0.25</td>
</tr>
<tr>
<td>3.3</td>
<td>0.78</td>
<td>9.54</td>
<td>0.04</td>
<td>0.112</td>
<td>0.26</td>
</tr>
<tr>
<td>4.1</td>
<td>4.00</td>
<td>6.13</td>
<td>0.02</td>
<td>0.110</td>
<td>0.18</td>
</tr>
<tr>
<td>4.2</td>
<td>0.96</td>
<td>8.59</td>
<td>0.09</td>
<td>0.020</td>
<td>0.28</td>
</tr>
<tr>
<td>5.1</td>
<td>0.83</td>
<td>8.25</td>
<td>0.04</td>
<td>0.042</td>
<td>--</td>
</tr>
</tbody>
</table>

Fig. 8. Average mortar-bar expansion for all the twelve aggregates.

Structural parameters play an important role in controlling the reactivity. It was decided to use the average expansion results after 14 days obtained from the accelerated mortar-bar test, in order to establish the reactivity of each aggregate. When using the average expansion results, it is important to be aware of the accuracy of the expansion data. The procedure for the accelerated mortar-bar test might differ slightly between laboratories, i.e. the time of curing and the size of moulds. This should be taken into consideration when assessing the total expansion of different materials, and the expansion results should not directly be transferred and compared with expansion results elsewhere. However, the expansion results between each rock type in this study have been obtained under the
same conditions, so the comparison should be satisfactory. When it is appropriate, statistical regression analysis has been performed to help establish the correlation between different parameters and the average expansion results. A multivariable analysis which included all the different parameters would have been beneficial. However, the reason why such an analysis is not included in this study is because many of the parameters are based on interrelated assumptions, and hence hold a lack of independence.

It is always important to be aware of the errors and limitations involved in the measurements and results of such a study. When dealing with cataelastic rocks in particular, the properties of the rock might differ significantly even when sampled from within relatively small areas. However, it is interesting to observe that the two samples 1.3a and 1.3b, which were collected from within a few decimetres of each other, showed a similarity, both in the quantification of the various microstructural parameters, and in the measured average expansion.

5.1. The quartz XRDA/DTA ratio

The graph showing the correlation between the quartz ratio (XRDA/DTA) and the average mortar-bar expansion after 14 days, is presented in Fig. 9. A total of ten rock samples is included in the graph. Two outliers, the ultramylonite (sample 4.1) and the gneiss (sample 4.2), are not included in the graph.

According to Flörke (1961), quartz grains below 30 to 60 μm are not detectable by DTA, while they are by XRDA. It was observed that samples with a higher content of microcrystalline quartz gave a lower quartz content by the DTA, and hence a higher quartz XRDA/DTA ratio. However, the question why most of the samples show a ratio below 1.00 remains unanswered. Many ambiguities still exist for the quantification of quartz from these two methods, and it appears that the ratio is not ideal for determining accurately the grain size distribution of quartz. For instance a high content of chlorite will give uncertain results for the quantification of quartz in XRDA. A logarithmic relationship is apparent when assessing the correlation between the quartz XRDA/DTA ratio and the average expansion after 14 days in Fig. 9. An increase in the ratio shows a corresponding increase in the expansion value. However, the coefficient of determination ($R^2 = 0.61$) is low, even when two outliers were removed from the correlation. One of the outliers exhibited a very high XRDA/DTA ratio of 4.0, while the other outlier exhibited a high XRDA/DTA ratio with a relatively low expansion value. The reasons for the anomaly in the data of these outliers are not known.

5.2. The index of crystallinity (CI)

The graph showing the correlation between the index of crystallinity (CI), obtained by XRDA, and the average expansion after 14 days, is presented in Fig. 10. One outlier, a mylonite (sample 3.3), is removed from the graph. The reason for removing this outlier will be discussed later.

The measure of the index of crystallinity (CI) has previously been used in a few cases to assess the alkali-reactivity of aggregates. Morino (1986) investigated the crystallinity of cherty rocks and observed a good correlation with experimentally determined reactivity, obtained by the ASTM C289 chemical test method. Katayama and Futagawa (1989) were able to distinguish between reactive, poorly crystallised chaledonic quartz from innocuous well-crystallised stable quartz, on the basis of the CI. Thomson and Grattan-Bellew (1993) did not succeed in finding a relationship between the CI and the reactivity. They concluded that the outcome could be a result of a number of different factors, leaving the case with CI unresolved. However, in a later study, by Thomson et al. (1994), a better relationship between the CI and the expansion was found, in spite of some exceptions.

Results in this study do not show a strong relationship between the CI and the average expansion after 14 days. As evidenced from the scatter in Fig. 10, these results could, therefore, not be used with any great accuracy to predict the reactivity of these samples. The mylonite (sample 3.3) showed a high CI, while showing a high average expansion. The reason for this outlier showing
Fig. 9. Graph showing correlation between the quartz ratio (XRDA/DTA) and the average expansion after 14 days.

Fig. 10. Graph showing correlation between the index of crystallinity (CI), obtained by XRDA, and the average expansion after 14 days.
high expansion could be due to the occurrence of veins of euhedral quartz, which were observed in hand specimens of the rock. Euhedral quartz with a near perfect crystallinity, will probably even in small amounts overwhelm the intensity of the refraction values. This could obviously be a problem, which might be a factor in the results obtained with some of the other samples. A certain amount of recrystallised or secondary quartz in veins might overwhelm the intensity of the refraction. Hence, the CI might be unusable to predict the reactivity for heterogeneous materials, such as cataclasite rocks; however, for homogeneous or certain selected materials, the method may be beneficial.

5.3. Water absorption

The graph showing the correlation between water absorption (mass %) and the average expansion after 14 days is presented in Fig. 11. The result for the cataclasite (sample 2.1) showed an abnormal value and is removed from the graph, while the result from the mylonite (sample 5.1) was not available.

The relationship between the water absorption and the average mortar-bar expansion after 14 days shows a great deal of scatter (Fig. 11). These results, however, seem to suggest that samples with high values of water absorption, exhibit lower mortar-bar expansion. This contradicts the expected behaviour, that samples which absorb the most water would also show favourable tendencies to absorb alkali solution, and hence enhance the reactivity. The cataclasite (sample 2.1) exhibited a high absorption value, and is not included in Fig. 11. The reason for this high value is not known. These results seem to indicate that water absorption alone cannot be used to predict the accessibility of reagents to reactive aggregates.

5.4. The mean quartz grain size ($d_{50}$), and the total grain boundary area of quartz

The graph showing the correlation between the inverse of the mean grain size of quartz ($d_{50}$) and the average expansion after 14 days is presented in Fig. 12. All twelve samples are included in the graph. In addition, existing results from two

![Graph showing correlation between the water absorption (mass %) and the average expansion after 14 days.](image-url)

Fig. 11. Graph showing correlation between the water absorption (mass %) and the average expansion after 14 days.
supplementary crushed rock samples (black dots) are included in this graph. These rocks, a mylonite gneiss and a gneiss, were included to obtain more information of rock types exhibiting low expansion (0.03–0.05%) after 14 days.

The graph showing the correlation between the total grain boundary area of quartz (m²/cm³) and the average expansion after 14 days is presented in Fig. 13. Existing results from the two supplementary crushed rock samples (black dots) are also included in this graph. Two of the rock samples, the porphyritic granite (1.4) and the cataclasite (sample 2.1) are not included in this graph. The reason for not including these two outliers will be discussed later.

A logarithmic relationship is evident for both the inverse of the mean grain size of quartz (d₅₀) and the total grain boundary area of quartz, when correlating with the average result of the mortar-bar expansion after 14 days (Figs. 12 and 13). Based on the best fit line, aggregates with a measured mean grain size of quartz less than about 0.12 mm would be expected to exceed 0.10% mortar-bar expansion. This is in agreement with what has been suggested by Grattan-Bellew (1992), who claimed that an enhanced solubility of quartz occurs when the mean grain size of quartz is less than 0.10 mm. The mean grain size of quartz and the total grain boundary area of quartz are both based on the grain size distribution of quartz grains obtained from the thin-section examination. However, in the case of the mean quartz grain size, the proportion and the distribution of the finer fraction, and the total amount of quartz in the rock sample, are not considered. It is evident that when comparing two rock samples with the same mean grain size, the sample with a higher amount of quartz and a higher proportion of microcrystalline quartz, will give a significantly higher available total surface area, and thus enhanced reactivity. Hence the total grain boundary area of quartz, gives a better relationship, with a higher coefficient of determination ($R^2=0.89$), than when only considering the mean grain size ($R^2=0.72$).

When assessing the relationship between the
total grain boundary area of quartz and the average expansion after 14 days, two samples showed unexpected behaviour. These two rock samples, the porphyritic granite (sample 1.4) and the cataclasite (sample 2.1), were regarded as outliers and were not included in the statistical model. The anomalous data for these samples could be explained as follows. The porphyritic granite contains relatively coarse quartz grains, which contribute little to the total grain boundary area of quartz. However, the mortar-bar test for the aggregate showed 0.10% expansion after 14 days. The reason for this relatively high expansion might be due to the occurrence of strain lamellae which are observed in some of the quartz crystals. If these lamellae were regarded as subgrain boundaries, then the proportion of microcrystalline quartz would increase, and consequently the total grain boundary area would increase significantly. This would have led to a better agreement with the model. However, the occurrence of myrmekite with thin rods of quartz within the feldspar, might also contribute to an enhanced solubility of the porphyritic granite. The roles of strain lamellae and myrmekite must be investigated further before such types of granite are included into the model.

In the case of the cataclasite (sample 2.1), a high content of microcrystalline quartz was observed which resulted in a very high value for the grain boundary area. During the formation of cataclasites, which are deformed under a brittle regime, the microcrystalline quartz is a product of the crushing process, and should not be regarded as real subgrains with a high dislocation density. Opposite the ductile deformed rocks, where the applied deformation energy is stored in the material as high dislocation density, the energy for cataclasites is used in the crushing processes. Additionally the extremely high amount of quartz (90%) in the sample gives a very high value for the total grain boundary area, when multiplied with the quartz amount. This high amount of quartz might result in unpredictable behaviour.

On the basis of the theoretical knowledge of the properties of subgrains, it is expected that these grains will give a higher reactivity than ordinary grains of similar size. However, the enhanced reactivity of subgrains versus other quartz grains
of equivalent size has not yet been established. No attempt was made to distinguish between subgrains and other recrystallised microcrystalline grains of quartz in this study. The reason was due to practical limitations in the thin-section examination, and to the uncertain effect of reactivity of subgrains versus other quartz grains. However, most of the observed microcrystalline quartz was due to subgrain development. Consequently, in most cases, the major contribution to the grain boundary area was due to the subgrain development. In the case of microcrystalline material without subgrain development (i.e. gneiss, blastomylonite and cataclasite), this might give incorrect estimations. Further research should include the enhanced reactivity of subgrains into the model, hence making a better basis for prediction. By using techniques such as electron- and transmission electron-microscopy, which give better resolution, more information about specific microstructural features, including subgrain development, may be obtained. The dilemma is that reactive aggregates are polymorphic materials with complex, heterogeneous structures, and it would be very difficult to obtain a quantitative assessment of the material as a whole with these advanced techniques. Even within the area of examination in a thin-section (25 × 30 mm), which is only a small part of the whole rock, microstructural features may differ significantly.

The measurement of the total grain boundary area of quartz will enhance the value of the petrographical examination as an engineering tool to screen potentially reactive aggregates. By using advanced image analysis systems, it would be possible to establish standard procedures, hence making the method more reproducible. For other types of slow/late reactive aggregates, including various types of greywackes, sandstones and argilites, further investigations have to be carried out.

6. Conclusion

The most probable mechanism for enhanced reactivity of cataclastic rocks is due to the presence of microcrystalline quartz, and in particular the high strain energy surrounding dislocations in subgrains which provide preferential sites of dissolution on the surface. An enhanced content of subgrains will promote the ability of the alkali-rich fluid to permeate to the reaction site.

In this study it is demonstrated, by theoretical approach and experimental work, that the alkali-reactivity of cataclastic rocks is clearly related to the total grain boundary area of quartz. This parameter is strongly influenced by the subgrain development.

The measurement of the total grain boundary area of quartz will enhance the value and improve the effectiveness of the petrographical examination as an engineering tool to screen potentially reactive aggregates.

The recrystallisation of the quartz will decrease the reactivity of the rocks due to the amalgamation of smaller grains into larger ones, leading to a smaller total grain boundary area of quartz. Considering only the mean grain sizes of quartz might give unpredictable results, in particular for materials containing a high or a low amount of quartz.

Acknowledgment

The staff at the Department of Geology and Mineral Resources Engineering, University of Trondheim and the staff at the SINTEF-Structures and Concrete are thanked for their technical assistance. I am grateful to Dr. W.J. French and all colleagues at Geomaterials, Unit, Queen Mary and Westfield College, University of London, for their support during the course of my work. Mr. A.L. Nissen’s assistance is appreciated during the course of collecting some of the samples. I also wish to thank my supervisor, Dr. S.W. Danielsen, for helpful advice and discussions during the work, and my co-supervisor, Dr. B. Brattli for the introduction into the theory of cataclastic rocks. This work is a part of the author’s PhD study, which has been funded by the Royal Norwegian Council for Scientific and Industrial Research. Special thanks to Dr. H.H. Patel for his help in the editing of this manuscript.
References


gate Reaction, Proceedings of the 8th International Conference, Kyoto, pp. 495–499.


Chapter 10

Summary

The main purpose of the present work has been to study the properties, classification and testing of certain Norwegian aggregates, in particular various types of cataclastic rocks. The aim has been to achieve a better understanding of the microstructural properties of these rocks in relation to the alkali-reactivity, in order to improve existing test procedures and guidelines for engineering practise. The following chapter summarises the main content of this work, both the theoretical section (chapters 2-4) and the experimental section (chapters 5-9).

Chapter 2: The level and effects of alkalies in the concrete system are reviewed in this chapter. The so-called "safe" minimum levels of alkalis are considered on a wide basis, and it has been shown that many different factors have to be considered when assessing the effects of the amount of alkalis. It is also suggested that other damaging mechanisms should be considered when investigating AAR, particularly when there is doubts about which damaging process is likely to be the initial mechanism in deterioration of the concrete.

Chapter 3: The petrographic properties of various types of alkali-reactive aggregates are considered in this chapter. The description of reaction patterns and microstructural features of various types of alkali-reactive aggregates are exemplified with various micrographs of thin-sections. The petrographic properties of slow/late alkali-reactive aggregates as the classification and nomenclature of certain rocks subjected to deformation are introduced, and the frequently used petrographic method of measuring the undulatory extinction angle of quartz is disputed. Various aggregate properties affecting the onset and severity of the AAR are sited, particularly the effect of reactive particle sizes. It is suggested that the grading is an important characteristic of the aggregate and influences many important properties of fresh concrete. With respect to AAR different grading curves can lead to different amounts of alkalies being made available for a given particle. The critical amounts of slow/late alkali-reactive aggregates are introduced and discussed. Possible differences between natural aggregates versus crushed aggregates with regards the alkali-reactivity are discussed.
Finally in this chapter, a new approach is introduced based on the quantity of reactive constituents in an aggregate, rather than the volume of potentially- and alkali-reactive particles.

**Chapter 4:** The necessity of reliable test methods to determine the alkali-reactivity of aggregates is covered in this chapter. Both the method of petrographic examination, including the Norwegian method, and the method of accelerated mortar bar testing are discussed, in light of results obtained in this study.

It is suggested that the only way to make proper classification of slow/late alkali-reactive rock types is by point counting of aggregate particles in thin sections. This examination should include a full quantitative description of the microstructural features. The semi-quantitative petrographic method applied in Norway appeared to give suitable results regarding the amount of potentially- and alkali-reactive aggregates. However, it is claimed that the method requires experienced petro-graphers, and it was found that test results variability can be associated with the methodology. It is also emphasised that the method ignores the properties of the innocuous particles, and potential differences in reactivity between various types of alkali-reactive rocks. A new method of petrographic examination is introduced, based on a quantitative determination of the total grain boundary area of quartz.

The development and use of accelerated mortar bar test both internationally and in Norway, along with various factors affecting expansion of mortar bars are also covered in this chapter.

**Chapter 5:** The experimental part of the thesis starts with the first paper which deals with the occurrence and distribution of cataclastic rocks in Norwegian glaciofluvial aggregates. These aggregates are assessed in light of various mechanisms and processes which were thought to be the primary cause of the high amount of cataclastic rocks observed. It has been suggested that as a result of the intense thrusting and faulting, cataclastic rocks are widely distributed in Norway and should thereby be expected to occur in many glaciofluvial deposits. It is also believed that during comminution and transportation of glaciofluvial materials, more fragile materials would be more quickly abraded into finer fractions, leading to an enrichment (or maintenance of a high level) of quartz bearing rocks in other fractions in the deposits. In particular this was thought to be the case for cataclastic rocks, which have been reported to show very high abrasion resistance.

The data processing of results of petrographic examination of glaciofluvial aggregates, gave guidelines that cataclastic rocks are a common constituent in the majority of the investigated glaciofluvial sediments. However the amounts of cataclastic rocks vary between different areas, governed by the local geology.
It was evident that glaciofluvial materials near mylonite zones showed a higher content of mylonitic rocks in the 2 to 4 mm fraction than in the 1 to 2 mm fraction. The opposite trend was observed for samples located more remote, in the direction of downstream ice-movement. Relatively high amounts of mylonitic rocks, were found in the fine fraction (1 to 2 mm) in samples taken up to 30 to 40 km downstream of the parent rock. The occurrence of particles of cataclasite in glaciofluvial materials was shown to follow similar trends as for the mylonite. However, the cataclasite appeared to be enriched in the 1 to 2 mm fraction in relation to the 2 to 4 mm fraction, for regions closer to the parent rock (fault zone) than for mylonites. This suggested that cataclasite is less durable to mechanical abrasion than mylonite, over such particularly long distances.

Chapter 6: In the second paper, relationships between the amount of reactive- and potentially alkali-reactive aggregates, obtained by the Norwegian petrographic examination, and the expansion results obtained by the accelerated mortar-bar test are discussed. Natural aggregates, crushed stones and blends of aggregates were included in the investigation. The established limit of critical amount of reactive rocks in aggregates was assessed in light of the knowledge of field performance of different Norwegian aggregate types.

It was demonstrated that the mortar-bar expansion increased with increasing amount of reactive- and potentially alkali-reactive rocks in the aggregate. However, no linear correlation by statistical analyses was found. It was observed a "marginal" amount of approximately 40 percent reactive rocks in the aggregates producing an upper limit of 0.25 to 0.30 percent mortar bar expansion. No additional expansion was found with higher content of reactive rocks in the aggregates. However as mentioned in chapter 3, later considerations give reason to believe that a logarithmic relationship will give a better explanation of the distribution.

The established lower limit of 20 percent reactive rocks in aggregate appeared sufficient in most cases, however, the established lower limit was not statistically verified.

Chapter 7: The third paper investigated the sequential development of AAR by accelerated mortar testing, in an attempt to identify possible differences in the behaviour of different types of slow/late expanding reactive aggregate. In association with the expansion development, the characteristics and the sequential development of the products at the reaction site were examined, along with the evaluation of the formation, properties and amount of the gel reaction product.

It was found that the accelerated mortar bar test appeared to reflect satisfactorily the naturally occurring alkali-aggregate reaction, in terms of distinguishing between different types of reactive aggregate, and properties of the gel reaction products.
However, for extremely slow reactive aggregates, the accelerated test might show some limitations regarding the predictions of service life for structures.

Results implied that AAR could continue in old structures, provided that the supply of alkalies is not exhausted or alkalies are added to the structure. Coarser aggregate particles appeared to contribute more to the total expansion. The rate of expansion was observed to differ in time and extension for different types of reactive aggregate.

Two main processes were observed during the reaction; a process of dissolution, and a process of cracking. Even though the process of dissolution appeared to produce the greater quantity of the gel reaction products, the process of cracking appears to contribute most to the expansion.

The reaction appeared to be associated with microcrystalline quartz, in particular with the occurrence of subgrain development. No significant signs of reaction could be associated with coarser quartz grains dominated by extensive undulatory extinction.

Na-rich gel reaction products were observed in 14 days specimens, however, a high CaO content gives indications of the phenomenon of regeneration of alkalies. No relation was found between the amount of gel reaction products and the total expansion.

Chapter 8: The fourth paper started by reviewing the literature relating to three commonly used test, the ASTM C-289 Rapid Chemical Test, the ASTM C-227 Mortar Bar Test, and what has become the ASTM C1260-94 Accelerated Mortar Bar Expansion Test. The experimental work in essence represented a determination of the errors to be expected from application of the accelerated mortar bar test to a single aggregate and the factors that influenced these errors. These factors were examined independently by two separate laboratories, and the precision and variation of the results obtained were discussed. The variables examined were the size of the mortar bars, the type of measurement points employed, the precision of the aggregate grading, the ratio of the volume of alkali to the surface area of the mortar, and the cement type employed.

The work showed that the repeatability of the test suggested an upper bound standard deviation of 0.0365% expansion. This figure was used to established discriminant criteria for the recognition of the potentially damaging materials. It was evident that diagnosis of potentially highly reactive aggregate can be made with confidence, but that a large number of tests are required to assess marginal types. No significant differences were found with respect to the cement type or the details of the aggregate grading. The storage conditions were found to greatly influence the result obtained and the early age variability appeared largely to reflect the ratio of the volume of alkali solution to the surface area of the mortar bar. Petrographic evaluation of the mortar following the test was assessed as essential, particularly
where the results are marginal. Much of the uncertainty in the test results was found to be derived from inherent aggregate variability.

**Chapter 9:** In the last paper, the background knowledge of cataclastic rocks from the various test methods were considered. The microstructural features, for certain rock types as influenced by cataclasis were determined and quantified in an attempt to identify methods which could be adequately used for the interpretation and prediction of the alkali-reactivity.

According to an extensive literature review, it was proposed that the most probable mechanism for enhanced reactivity of cataclastic rocks is due to the presence of microcrystalline quartz, and in particular to the high strain energy (dislocations) surrounding subgrain boundaries.

The experimental results demonstrated that the alkali-reactivity for cataclastic rocks is clearly related to the value of the total grain boundary area of quartz. This parameter which is strongly influenced by the subgrain development, appeared to provide a better understanding of the reactive components in these selected materials, and it was suggested that it should be used to improve the quantitative aspect of petrographic examination. It was also suggested that considering only the mean grain sizes of quartz, might give unpredictable results, in particular for materials containing a high or a low amount of quartz.
Chapter 11

Conclusion

The main conclusions regarding the properties, classification and testing of certain Norwegian cataclastic rocks are as follows:

Petrographic examination

- Cataclastic rocks are commonly found in glaciofluvial aggregates in southern Norway, where the amounts of cataclastic rocks are governed by the geology in the area. Particles of mylonitic rocks might however be found in relatively high amounts in certain aggregate fractions tens of kilometres from their origin.

- It is proposed that the petrographic examination of slow/late alkali-reactive rocks includes a full quantitative determination of the microstructural features by thin-section examination. When naming alkali-reactive rock types subjected to deformation, nomenclatures associated with fault rocks should be used rather than standard mineralogical nomenclatures.

- A new method of petrographic examination is introduced based on the quantitative determination of the total grain boundary area of quartz.

The accelerated mortar bar test

- It is found that the accelerated mortar bar test echoes the naturally occurring alkali-aggregate reaction, in terms of distinguishing between different types of reactive aggregate, and the properties of the gel reaction products. However, for extremely slow reactive aggregate, the accelerated test might show some limitations regarding the predictions of service life for structures, and that a large number of tests are required to assess marginal types of reactive aggregate.
The enhanced expansivity of cataclastic rocks in the test is due to the presence of microcrystalline quartz, and in particular to the high strain energy (dislocations) surrounding subgrain boundaries.

The rate of expansion was observed to differ in time and extension for different types of reactive aggregate. Coarser aggregate particles appeared to contribute more to the total expansion.

Two main processes were observed during the reaction; a process of dissolution, and a process of cracking. Even though the process of dissolution appeared to produce greatest amount of the gel reaction products, the process of cracking appears to contribute most to the expansion.

No relation was found between the amount of gel reaction products and the total expansion.

The repeatability of the test suggested an upper bound standard deviation of 0.0365% expansion.

Petrographic evaluation of the mortar following the test was assessed as essential, particularly where the results were marginal. Much of the uncertainty in the test results was found to be derived from inherent aggregate variability.

No significant differences were found with respect to the cement type or the details of the aggregate grading. The size of the mortar bar however is critical and the ratio of the NaOH solution to the surface area of the mortar bars is critical to the amount of expansion.

Petrographic examination and the accelerated mortar bar test

Relationships between results from the currently used Norwegian petrographic examination and accelerated mortar bar expansion, show an increasing expansion with increasing amount of reactive- and potentially alkali-reactive rocks in the aggregate. Later considerations suggest the data to follow a logarithmic distribution. The use of a critical lower limit of amount of alkali-reactive particles in an aggregate might be adequate as a temporary assessment of aggregate's reactivity. However an improved classification of aggregate's reactivity should be based upon the quantitative determination of reactive constituents rather than the volume of potentially- and alkali-reactive particles.
• A logarithmic relationship is observed between the total grain boundary area of quartz, and the 14 days mortar bar expansion. The measurement of the total grain boundary area of quartz, enhances accuracy and improves the effectiveness of the petrographical examination as an engineering tool to screen potentially reactive aggregates. Considering only the mean grain sizes of quartz, might give unpredictable results, in particular for materials containing a high or a low amount of quartz.

• No significant signs of reaction were observed associated with undulatory extinction of quartz. The undulatory extinction of quartz is not regarded as an adequate parameter for assessing alkali-aggregate reactivity.
INDEX

A
AAR 1, 7, 13, 22, 24, 32, 88, 106
Accelerated mortar bar tests 32, 36, 37, 50, 135, 140
- aggregate 144
- alkali immersion method 140, 171
- assessment at an early age 165
- blending of aggregate 54, 92, 93
- cement 51, 141, 145, 155
- critical amount of aggregates 95
- crushed stones 93
- expansion criteria 91, 141, 152,
- grading 51, 122
- magnitude of expansion 121,
- measurement 146
- measurements points employed 51
- modified accelerated mortar bar
testing 51, 153, 191, 193
- mortar bar expansion 110, 198
- mortar bar sizes 145, 156
- moulding 145
- NaOH concentration 55
- natural aggregates 92
- particle size 53, 122
- pessimum effect 157
- precision 51
- procedure 142, 144, 193
- sequential development 112,
- South African NBRI-mortar bar test 51,
  87, 91, 106, 125, 140, 193
- standard deviation 160
- storage 145, 172
- surface area 51
- temperature 55, 157
- variation 51
- volume of alkali 51, 141, 154
- water/cement ratio 52, 167
Acid volcanic rocks 69
Activation energy 188
Abrasion 66
  resistance 63, 64, 79
Additives 12
- condensed silica fume 12
- ground granulated blast furnace slag 12
- pulverised fly ash 12
Aggregate 11, 22, 24
- alkali release from 10
- blends of 54, 92, 93
- concrete- vs. mortar-bars 111
- crack patterns 16, 113, 118
- critical amount 36
- crushed aggregates 36, 51
- grading 33, 122
- grain size 10
- leaching of alkalies 10,
- maximum density 34
- microstructure 27
- natural aggregates 35, 51
- properties of 21, 32
- rapid reactive aggregate 1, 22, 24
- reactive constituent 26, 36, 37, 46, 127,
  136
- size of reactive particles 32, 122
- slow/late alkali reactive aggregates 21,
  24, 26, 29, 33, 46, 105
- volume of alkalies 51, 154
- water/cement ratio 52,
Air voids 14, 118
Alkalies 8, 9, 51
- amount 8, 33
- concentrations 9,
- effect 8
- enhancements 12, 16
- enrichment 9,
- external 12
- leaching 10, 16
- migration 9, 14
- movement 12,
- other sources 10
- pore solution 23
- ratio 51
- recycling 9
- sea water/dredged aggregates 11
Alkali-Aggregate Reactions see AAR
Alkali-Carbonate Reaction 7, 21
Alkali immersion method see Accelerated
mortar bar tests
Alkali-Silica Reaction 7
Alkali-Silicate Reaction 7
Annealing 31
Argillaceous rocks 69, 94
Arkose 36
ASTM C-1260, 51, 52, 106, 136, 153, 161
ASTM C-227 Mortar Bar Method 50, 106, 138
ASTM C-289 Chemical Test 50, 139
ASTM C-295, 46, 136
ASTM C-33, 138
ASTM C-97, 192
Aunvatnet 194
Aust-Agder 76
Australia 51
Autoclave 143, 171

B
Belgium 51
Bindal Massif 194
Binder 9
Binocular microscope 47
Blastomylonite 37, 63, 191, 194
Breccia 28
Brittle deformation 63, 79
BS 812, draft Appendix C, 46

C
Calcium 9, 35, 118
Calcium hydroxide 118
Caledonian nappes 64
Canada 10, 22, 36, 51
Carbonation 107
Carbonates 139
Cataclastic rocks 21, 25, 27, 28, 63, 65, 69, 94, 184
- classification 1, 28, 109, 191
- properties 1, 28
Cataclasite 191, 194, 28
Cation exchange 10, 24
Cement 51, 141, 145, 155
Charnockites 29, 183
Chemical Test see ASTM C-289
Chert 21, 23, 27, 32
Chloride 11, 12, 15
Clinker 8
Coal ash 8
Concrete bar 11, 119, 122
Cores 106
Corrosion see Secondary processes
Cracking 16, 113, 118
- micro-cracking 15, 118
Cracks 10, 14, 23, 36, 119, 123
Criteria see Expansion
Crushed aggregate see Aggregate
CSA A23.2-14A Concrete Prism Test, 50, 88, 136, 141
CSA A23.2-94, 52, 136, 141, 153

D
Dam 10, 34, 47, 106
Deformed granitic rocks 29
Deformation 31, 110
Deformed rocks 28
- classification of 191
Deformation lamellae see Quartz
Deformation bands see Quartz
De-icing salts 12
Delayed expansion 21
Denmark 1
DGB 2, 37, 62, 76, 88
Dislocations see Quartz
Disordered crystal see Quartz
Dissolution 113, 118, 127
Dissolved silica 120, 127
Driving forces 188
DTA 46, 192, 197
Ductile deformation 31, 63, 79, 186

E
EDAX analysis 119
Enhanced alkali-method 143
Ettringite see Secondary processes
Expansion 12, 23, 33, 37, 91, 95, 110, 121, 139, 152, 159, 167, 198, 199
- criteria 91, 141, 152
- development 123
- limits 37, 91, 95, 96, 121, 139
- magnitude 121
- pattern of 123
- rapid 91
- rate of 33, 124
- slowly 91

F
Fault rocks 28, 75
Field performance 97
FIR CUSUM 165
Flint 21, 27, 144
Fluorescent dye 107
Fluvial aggregates 35
Foliated rocks 49
Fosen Peninsula 69, 196
Freeze/thaw 14
Frost resistance 14
Fused silica 144

G
Gabro 69
Gel see Reaction product
Geomaterials Research Service Ltd. 22, 23
Glacier 66
Glaciifluvial Deposits 35, 64, 92
- aqueous environments 66
- comminution 66
- deposition 68
- modification during transportation 66
- provenance 66
- transportation distance 67, 77
Glass 21, 23, 32, 34, 106, 118,
Gneiss 29, 183, 194, 69, 79
Gouge 28
Grading see Aggregate grading
Grain boundaries 23, 27
Grain size 10
Granitic rocks 29
Granite 29, 69, 79, 196
- micro-granite 106, 110, 115, 117, 183, 194
- porphyritic granite 194, 205
Granodiorites 29, 183
Greywacke 27, 32, 36, 69, 94, 122, 140
- meta-greywacke 21, 106, 110, 115
Ground-water 16

H
Hardanger-Ryfylke nappe Complex 197
Helgeland Nappe Complex 194
High strength concrete 14
Hitra-Snåsa Fault 196
Hydroxyl ions 7, 189

I
Iceland 11, 12, 23
Icelandic Building Research Institute 22, 23
Inclusions 66, 189
Index of Crystallinity 192, 198, 201

J
Japan 32

K
K-feldspar 189
K₂O 118

L
Leaching 16
Lime 16
Limestone 69, 106, 115, 120
- dolomitic limestone 21

M
Maentwrog dam 106, 110
Mafic rocks 69
Magnesium 35
Maximum density 34
Mechanisms see Reaction mechanisms
Meta-argillite see Argillite
Meta-greywacke see Greywacke
Meta-sandstone 21
Mica 109
Microgranite see Granite
Microscopic examination see Petrographic examination
Microstructures 27, 185

Microstructural features see Quartz
Microcracks see Cracks
Microcrystalline Quartz see Quartz
Microbreccia 191
Microscopic investigations see Petrographic examination
Mjøsa - Vtnern mylonite zone 64, 69, 70
Modified accelerated mortar-bar test see Accelerated mortar bar testing
Mortar-bar see Accelerated mortar bar testing
Mortar bar expansion see Expansion
Mylonite 25, 27, 30, 63, 106, 109, 115, 116, 184, 191, 194, 195, 196
Mylonite gneiss 63, 191
Mylonite zone 77
Myrmekite see Quartz
Møre og Romsdal 76

N
NaCl see Chloride
Namsskogan areas 194
NaOH see Alkalies
Natural aggregate see Aggregate
NBRI Mortar Bar Test see Accelerated mortar bar testing
Norway 9, 37, 46, 47, 51, 61, 64, 65, 76, 153, 185, 193
Nova Scotia 53
Nucleation 188

O
Odal granites 69
Opal 21, 172
Oppland 76, 80
Optical microscope 32
Oslo Region 64, 70

P
Permeability 36
Pessimum proportions 157
Petrographic examination 37, 46, 47, 61,
- 68,76, 90, 162, 190
- grain size measurement 190
- point-counting 49, 68, 91, 192
- properties 26
- subgrain boundaries 38, 66, 186, 205
- total grain boundary area 49, 191, 198, 203
Phyllite 21
Plagioclase 189
Plastic deformation see Ductile deformation
Plucking 66
Polygonization see Quartz
Index

Pore solution 8
Porosity 26
Porphyritic volcanic glass 23
Porphyroblasts 76
Potassium 7, 35
Pozzolan 12, 97
Precambrian 196
Precision 140
 Preferential site of reaction 188
Protomylonite 63, 191

Q
Quartz
- c-axes 30
- defects 26, 106
- deformation bands 32, 187
- deformation lamellae 32, 187
- dislocations 31, 38, 66, 110, 125, 183, 188
- inclusions 66, 189
- low angle boundaries 31
- mean grain size (d50) 27, 49, 109, 191, 198, 203
- microcrystalline 21, 26, 69, 76, 94, 96, 106, 125
- myrmekite 189, 205
- polygonization 31, 186
- strain energy 125, 188
- strain lamellae 27, 110, 205
- strained 1, 21, 26, 96, 106, 140
- subgrain boundaries 38, 66, 186, 205
- subgrain development 28, 32, 49, 109, 125, 185, 187
- total grain boundary area 49, 191, 198, 203
- triple points 188
- undulatory extinction 26, 29, 32, 66, 126, 184, 186
- XRDA/DTA ratio 198, 200
Quartz arenite 36
Quartzite 27, 69, 183, 187
meta-quartzite 26, 32

R
Railway sleepers 15
Reaction mechanisms 7
- activation energy 188
- driving forces 188
- preferential site 188
- quantity of silica involved 120, 127
- sequential development 112
Reaction products 14, 23, 118, 126
- composition 118, 126
- dissolved silica 120, 127
- microstructure 127
- regeneration of alkalies 126
- silica 7, 26, 120
Realkalization 12
Recrystallisation 31, 110
Repeatability 137, 143, 162
Reproducibility 137, 143
Replacement materials 12
Rhyolite 23, 37, 91, 94
RILEM 141, 192
Rogaland 76

S
Salt water see Sea water
Sandstone 32, 36, 69, 94
Scanning electron microscopy 46, 107, 119, 127
Schist 25, 29, 183
Schistosity 27
Sea water 11
Secondary processes 13
- corrosion of reinforcing steel 11, 15
- freeze/thaw 14
- leaching 16
- secondary ettringite formation 14
SEM see Scanning electron microscopy
Sericitization 110
Silica 7, 26
- fused 144
- glass 106, 118
- quantity 120, 127
Silica fume see Additives
Silicon-hydroxyl bonds 189
Silicon-oxygen bonds 189
Siltstone 36, 94
SINTEF Structures and Concrete 2, 22, 51, 62, 63, 69, 89, 153
SiO2 see silica
Skaidalen 197
Slow/late alkali-reactive aggregate 21, 24, 26, 29, 46, 105
Sodium 7, 35
Sodium oxide equivalent 8
Solubility 205
Soluble silica 108
Solar gneisses 69
South Africa 51
Southeastern Precambrian province 69, 74, 76, 196
Standard deviation 137, 159, 160
Standard error 137
Strain lamellae see Quartz
Strained quartz see Quartz
Sub-grain boundaries see Quartz
Sub-grain development see Quartz
Sweden 185
Sør-Trøndelag 76, 80

T
Telemark 76
Test methods 46
- accelerated test methods see Accelerated mortar bar test
Test procedure 142, 144, 193
Thin-section 47, 68, 90, 107, 206
Thrust zones 64
Transmission electron microscopy 27, 185

U
Ultratymonite 25, 37, 63, 65, 113, 114, 191, 194, 195
Undulatory extinction angle 1, 28, 29, 184
Undulatory extinction see Quartz
Universal stage 30
UK 9
USA 51

V
Vermiculite 21
Verrabotnen - the Verran Fault system 70, 75, 79
Voids 34, 118
Volcanic glass 21, 23, 32, 34
Volcanic rocks 69, 94

W
Wales 106
Water absorption 192, 198, 202
Water/cement ratio 52, 167
Water soluble sulphates 8
Workability 34

X
XRD -analysis 46, 49, 191, 197
XRD/DTA ratio 198, 200

Y
Younger Dryas 70