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Application of Electron Backscatter Diffraction to evaluate the ASR risk of concrete aggregates

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ABSTRACT

Alkali-Silica Reaction (ASR) is a frequent cause of reduced concrete durability. Eliminating the application of alkali reactive aggregates would reduce the quantity of ASR concrete deterioration in the field.

This study introduces an Electron Backscatter Diffraction (EBSD) technique to distinguish the ASR risk of slow-late reacting aggregates by measuring microstructural properties of quartz. Quantifying the amount of quartz grain boundaries and the associated misorientation of grains can thereby be used to differentiate microstructures bearing an ASR risk. It is also shown that dissolution of quartz in high pH environments occurs along quartz grain and subgrain boundaries.

Results of EBSD analysis are compared with ASR performance testing on concrete prisms and optical light microscopy characterization of quartz microstructure. EBSD opens new possibilities to quantitatively characterize microstructure of quartz in concrete aggregates with respect to ASR. This leads to a better understanding on the actual cause of ASR.

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1. Introduction

Despite long and frequent applications of concrete as building material, concrete durability is often a challenge. This fact becomes more obvious with increasing demands of today's society for improved durability and thus sustainability of materials, including concrete. Durable design requires in-depth understanding of concrete deterioration mechanisms, as much as reliable methods for material assessment prior to use.

Among several known mechanisms that reduce concrete durability, ASR is a severe one especially for applications in highway concrete pavements, airfields and bridges [1–3]. Risk of concrete deterioration by ASR is increased in temperate and cold climate zones [2,4]. Also concrete exposed to seawater in humid climates tends to develop ASR deterioration [5,6]. Lack of durability of concrete in highway pavements and bridge structures is a severe problem that leads to reduced service life and with it to reduced overall sustainability of the material.

ASR begins with dissolution of silica present in concrete aggregates initiated by high pH value of the pore solution of concrete [1]. This can be followed by precipitation of an alkali-silicate gel (e.g. [1,7–9]). Deterioration of concrete may therefore be a result of dissolution and/or precipitation processes [2,9,10]. Macroscopically ASR damage is detected as cracking, expansion and abrasion of concrete [10–12]. Investigations to determine exact attributes of concrete deterioration are ongoing [12,13].

Two factors that increase ASR risks for concrete deterioration are freeze thaw cycles and the application of de-icing agents (NaCl, alkali acetates and formiates etc. [14,15]). The freeze thaw cycling is inevitably imposed by weather and climate conditions. ASR damage could be limited by using alternative deicers [16–18], but none (cost-) effective have been found yet. Further factors for increased ASR risk include high alkali content of cementitious materials and reactive concrete aggregates with increased solubility in alkaline aqueous environments [1,10]. Alkali content of cementitious materials can be limited but alkalis from external sources can enter concrete pore systems as de-icing agents or sea water. A more promising approach may be effective testing of aggregates for their ability to withstand dissolution in high pH environments [19–21].

Thus, reliable characterization of concrete aggregate microstructure [22–24] is one step to secure design of pavement concretes that are durable, even under conditions known to promote ASR. Commonly used aggregates for concrete production are river sands and gravel but recently, crushed aggregates such as granites, greywackes etc. are increasingly used. Among different rock forming minerals that contain silica (i.e. quartz, feldspar, mica), quartz and amorphous silica are known to be implicated in ASR [1,10,23,25]. ASR involving feldspar minerals have also been reported [26]. Thus, most investigations of ASR risk in aggregates used for concrete production have been focused on investigating the influence of quartz microstructure [25,26,27]. It is well is known that silicate rocks and gravel containing flint, micro- or cryptocrystalline

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quartz (chert, chalcedony) or even amorphous silica (opal) are very prone to ASR and thus, to concrete deterioration [22,25,27,28]. Increasing solubility of quartz and silica is one factor known to increase risk of ASR [25].

Recent studies have shown that decreasing quartz crystal size [27,22,29] and increasing dislocation density increase quartz solubility [29,30]. This could partly be correlated with increased expansion caused by ASR [29,30]. Also it is known that dislocations arising from tectonic deformation of quartz [31] increase the ASR risk. Other results indicate that dissolution rate of quartz in diluted aqueous condition is not controlled by dislocation density, because overall dissolution rate of quartz exceeds the effect of dislocations [32]. There is an open discussion if increasing rock deformation as experienced by gneiss, schist and mylonite is always correlated with increased ASR expansion of concrete [22,33,34].

The standard procedure for evaluation of ASR risk of concrete starts with a petrographic evaluation of aggregates [35,36] using optical light microscopy petrography followed by mortar and concrete tests (ASTM C1260, C1293, C1567 [37–39], RILEM Recommendations TC106 and AAR-0 – AAR3 [40–42] and DAfStb "German guideline for accelerated ASR mortar bar test" [43]). Identification of aggregates known to induce slow-late expansion of concrete is generally more difficult [35,44]. To identify such slower alkali-reactive aggregates, a special ASR climate simulation concrete prism test (CS-CPT) can be applied [15]. The drawback of the test is that it is relatively time consuming (test durations of several months are always necessary, as typical for ASR concrete tests). Thus, a fast-track method for identification of slow-late expanding aggregates is still desirable.

A more complete evaluation of aggregates would emphasize quartz microstructures such as grain size, specification of grain boundaries, dislocation density, twinning etc. [34,36]. Quantities of amorphous silica and other sources of silicate (feldspars, mica and other minerals) should also be assessed [35]. Microstructure of aggregates is typically described by optical petrography [22,9, 40,45] and quantitative X-ray diffraction (QXRD) analysis [28,46]. Light microscopy can resolve spatial information (grain sizes, recrystallisation structures), but resolution and quantification of crystal structures and defects are limited [2,34]. Results of QXRD can give quantitative information on phase assemblage of ground, bulk samples [28,47]. But often analytical error of grain size, strain and phase analysis for multiphase rock samples (such as deformed granites, granodiorites and rhyolites for example) is larger than variation between samples. Thus, it is often the case that aggregates possessing different ASR potentials cannot be differentiated by microstructural investigations [28,47].

In other fields of research analysis of quartz deformation by optical petrography is now usually complemented by Electron Backscatter Diffraction (EBSD) analysis performed in the Scanning Electron Microscope (SEM) [48,49]. Thereby the focused electrons from the electron gun enter the sample and some are scattered [49]. When these primary electrons are diffracted on the crystallographic planes of the sample material and the Bragg's law is fulfilled a strong reinforcement of reflected beam occurs [49]. Diffracted electrons emerge as so called Kossel cones that are detected with a phosphor screen as Kikuchi lines [49]. The distance between Kikuchi lines is correlated with interplanar spacing of atoms in crystal structure [49]. These Kikuchi diffraction patterns allow for mineral identification and analyses of crystal lattice orientations [48]. Furthermore, orientation imaging microscopy (OIM), based on automated analyses of EBSD patterns, can resolve microstructural parameters including crystal orientation, crystallographic texture, grain size and shape as well as grain boundary characteristics [50].

EBSD is a surface analysis technique – with diffraction signal coming from the top few nanometers of sample surfaces [49]. Therefore, a prerequisite for obtaining EBSD pattern is a polished surface of aggregates that is virtually free of defects caused by sample preparation (sectioning, mechanical polishing) [49]. A good indication for quality of EBSD patterns is the image quality value [49,51]. This quality metric is calculated from maximum diffraction intensities [51]. Thus image quality maps show the quality/contrast of electron diffraction patterns as a grey scale image. Keeping the microscope settings constant, the image quality of the grain interior (not grain boundary) is proportional to lattice orientation and deformation [51]. Lattice deformations may arise from mechanical sample preparations or from deformational states of rock samples [49,51]. After mechanical sample polishing of up to 0.25 μm (diamond paste), the EBSD pattern quality of quartz is too poor for effective indexing [52]. High quality indexing of EBSD pattern (using interband angles) is indicated by value of confidence index [53]. At confidence index (CI) > 0.1 approximately 95% of indexing solutions are correct if at least six Kikuchi bands are used for indexing [53]. Once high EBSD pattern quality is assured, EBSD mapping can not only be used to identify crystal orientation and size, but also to discriminate between subgrain and grain boundaries [50,54]. Grain boundaries are specified by the rotation angle that is needed to bring the crystal lattice of one grain into coincidence with a neighbouring grain. Typically rotation angle smaller than 15° (slightly material dependent) depicts low-angle or subgrain boundaries, whereas angles above 15° reveal high-angle boundaries (including twin boundaries, [55]). OIM Analysis of EBSD data can resolve differing lengths of grain boundaries that are correlated with crystal misorientation angles [49]. High resolution EBSD information (angular resolution of 0.2°, step size of 0.1 μm, [56,57]) can also be used to differentiate micro- and cryptocrystalline quartz and amorphous areas. Perhaps more importantly, preferred orientation of minerals (caused by deformation) can thereby also be quantified [58].

The present study shows how EBSD-OIM analysis can be used to characterize quartz microstructure in concrete aggregates. The aim is to get a basic understanding which microstructural features of quartz induce an increase in ASR risk of concrete.

2. Materials and methods

2.1. Aggregates

Mineralogical compositions of aggregates and rock types were determined by QXRD analysis. Results are shown in Table 1. Obviously granodiorite B contains less quartz but more biotite and muscovite and also some pyroxenes and hornblende. Both aggregates are used in commercial concrete production. The aggregates have been chosen for investigation because of their mineralogical close similarity (i.e. grain size and phase assemblage is very similar). This excludes other effects than quartz microstructure as cause for differences in concrete expansion as measured during ASR testing.

For reference a resin embedded, polished section of undeformed druse quartz was used for investigation.

2.2. ASR testing

According to specified testing of aggregates [43], both granodiorites are assessed to be non-reactive (i.e., as useable for production). In field

Table 1
Mineral assemblage of aggregates (in wt%) as determined by QXRD Rietveld analysis.

	Quartz	Alkali-feldspar	Plagioclase	Muscovite	Biotite	Chlorite	Pyroxene	Horn-blende
Grano-diorite A	33	15	45	2	2	1	–	–
Grano-diorite B	25	11	44	5	6	4	4	<2

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