



Application of micro X-ray diffraction to investigate the reaction products formed by the alkali–silica reaction in concrete structures



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ABSTRACT

Alkali–silica reaction (ASR) is one of the most important deterioration mechanisms in concrete leading to substantial damages of structures worldwide. Synchrotron-based micro-X-ray diffraction (micro-XRD) was employed to characterize the mineral phases formed in micro-cracks of concrete aggregates as a consequence of ASR. This high spatial resolution technique enables to directly gain structural information on ASR products formed in a 40-year old motorway bridge damaged due to ASR.

Micro-X-ray-fluorescence was applied on thin sections to locate the reaction products formed in veins within concrete aggregates. Micro-XRD pattern were collected at selected points of interest along a vein by rotating the sample. Rietveld refinement determined the structure of the ASR product consisting of a new layered framework similar to mountainite and rhodesite.

It is conceivable that understanding the structure of the ASR product may help developing new technical treatments inhibiting ASR.

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1. Introduction

Alkali–silica reaction (ASR) is one of the most important deterioration processes in concrete leading to substantial damages of structures worldwide [1,2]. A wide variety of structures, such as supporting walls, bridges or dams, have been affected worldwide and ASR-induced damage has caused significant costs for society. A preliminary survey in Switzerland identified over 400 affected structures, and it is expected that several major structures will have to be repaired or rebuilt in the coming years. The exact mechanisms leading to concrete damage by ASR are unclear, but what is known is that the reaction proceeds relatively slowly, i.e. damages appear 5 to 20 years after the structures have been built. The reaction between amorphous or crystalline SiO₂ in aggregates and the alkaline pore solution of concrete (pH 13.0–13.5) results in secondary products that can lead to expansion. The stresses which this expansion generates may eventually cause cracks in the concrete which further increases the risk of damage to the structures.

Our understanding of the mechanisms leading to the expansion caused by ASR is limited by the difficulties associated with the in situ analysis of the reaction products in concrete on the micro-scale.

Investigations on synthetic reaction products generated by the interaction of high pH water with aggregates and silica in laboratory experiments have shown that the products could consist of polymeric silica chains with water being either incorporated in relatively large interlayers, as is typical for kanemite (NaHSi₂O₅·3H₂O, [3]), or between nano-sized silicate particles [4]. The presence of divalent ions (e.g. Ca) instead of monovalent alkalis (e.g. Na, K) was shown to generate lower repulsive forces and reduced water adsorption, thus causing less expansive pressure [5–7].

Cole and Lancucki [8] used XRD to identify crystalline ASR products from a dam built in Australia as okenite, CaSi₂O₅·2H₂O or its precursor, CaSi₂O₅·4H₂O, in which some Ca atoms are replaced by K and Na. On the other hand, de Ceukelaire [9] concluded that mountainite, KNa₂Ca₂[Si₈O₁₉(OH)]·6H₂O, or rhodesite, KHCa₂[Si₈O₁₉]·5H₂O, are likely candidates for ASR phases formed in a Belgian bridge (based on scanning electron microscopy compared with XRD pattern of other studies). Based on XRD measurements Peterson et al. [10] found similarities to nekoite and okenite in the structure of the ASR product. In general the identification of ASR phases by XRD has been hampered due to the small size of the crystals (~1–10 μm), which has prevented the isolation of the pure compound from the matrix material. Attempts have been made to determine the crystal structures of the reaction products in concrete, which had been collected from outer layers of dam concrete [11]. However, these results are of limited use because the material may have been altered by calcium

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uptake from the cement paste or CO₂ ingress from the air during surface exposure (carbonation) and consequently, the transferability of such results to real concrete is presently uncertain.

However, it was recognized that knowledge of the composition and crystal structure of the reaction products is required to further understand ASR. While the chemical composition of ASR products is well documented from studies using scanning electron microscopy coupled to quantitative microanalysis (SEM/EDX), the mineralogy is only poorly known as the structures are difficult to study in situ due to the very small crystal size (μm range). In this study, samples were prepared from a 40-year old motorway bridge showing damage due to ASR.

To the best of our knowledge this study reports the first use of synchrotron-based micro-XRD measurements directly on ASR products formed in an intact vein in a concrete aggregate. It is conceivable that knowledge of the structure and the initial stage of the formation of the ASR product can stimulate new technical routes which could prevent or reduce ASR damage in concrete structures in the future.

2. Experimental

2.1. Sample preparation

The concrete samples (density of 2450 kg/m³) were collected from the abutment of a bridge built in 1969. The abutment is damaged by cracks caused by ASR. Several pieces of concrete cores were cut to a size of 15 × 18 × 23 mm³. They were dried in an oven at 50 °C for three days, impregnated with epoxy resin and polished on one side. The polished side was fixed with a thermo-glue (melting point of about 60–80 °C) on a glass carrier. Subsequently, polished thin sections (thickness of ~20 μm) were produced. The thin sections were studied using a polarized light microscope. One sample with an aggregate showing a vein filled with ASR products was chosen for further investigation. The vein in a predominantly limestone aggregate (that additionally contains detrital quartz and feldspar minerals) is 15–40 μm wide (Fig. 1a). Preliminary microXRD measurements were performed at the beamline 2-ID-D at the Advanced Photon Source (APS) with the aim of assessing the sample preparation. The data exhibited a large background produced by the high-purity glass support (Suprasil), which hampered the data analyses significantly. Therefore, the thin section was shortly heated to 70 °C with the aim of liquefying the thermo-glue. During the heating the sample was carefully sheared off the glass support and fixed onto an acrylic glass carrier (thickness of 1.5 mm) which had an 8 mm wide hole in the center. The area of interest was located directly above this opening. This set-up meant that the X-ray beam did not interfere with the glass support, and only interacted with the sample, thus minimizing the background intensity. Other samples from the same structure were used for measuring the E-modulus of the ASR products [12].

2.2. Scanning electron microscopy (SEM)

After coating the sample with carbon it was studied with an environmental scanning electron microscope (ESEM-FEG XL30) in the high-vacuum mode (3.0–5.0 × 10⁻⁶ Torr) with an accelerating voltage of 12 kV and a beam current of 200–240 mA. The chemical composition of the ASR product was determined with energy dispersive X-ray spectroscopy (EDX). An EDAX 194 UTW detector, a Philips digital controller, and Genesis Spectrum Software (Version 4.6.1) with corrections for atomic number, absorption and fluorescence (ZAF) were used.

2.3. Micro XRD measurements and data analysis

MicroXRD measurements were performed at the microXAS beamline of the Swiss Light Source (SLS) using a Dectris PILATUS 100 K detector (487 × 195 pixels). The X-ray beam with a wavelength of 0.6963 Å (17.8 keV) was focused down to 1 × 1 μm² using Kirkpatrick-Baez mirrors. The measurements resulted in 2θ data down to 2.64° (15.1 Å d-spacing).

The identification of crystals required rotational scans, which were collected on selected points of interest on the sample [13]. The rotation and high resolution x/y-table stages were mounted on the top of the sample manipulator. The eccentricity of this setup was ~1 μm. The sample alignment was done using an optical microscope, which was tilted by 45° with respect to the X-ray beam. A series of approximately 20 experimental data sets was measured at different points of interest across the vein. The beam size of ~1 μm allowed us to focus on a vein with ASR reaction products, which had a width of ~25 μm and an extension of a few thousand micrometers into the aggregate.

The collected microXRD frames of the rotated sample were summed up into a composite pattern and then integrated from a two-dimensional image into a classical one-dimensional powder pattern using the program Fit2d [14]. The integration of the powder pattern was performed from the half part of each frame, where the lower angle diffraction is well resolved. The powder pattern, integrated from the frames of the best available quality, was used for the structure determination and Rietveld refinements (Fig. 2). Rietveld refinements were performed with the JANA2006 program package [15]. A Pseudo-Voigt peak shape function and anisotropic strain line broadening were applied for the profile approximation [16].

3. Results

3.1. Elemental analysis

The ASR product shows small shrinkage cracks (Fig. 1b) that are caused by drying during sample preparation. The distance between

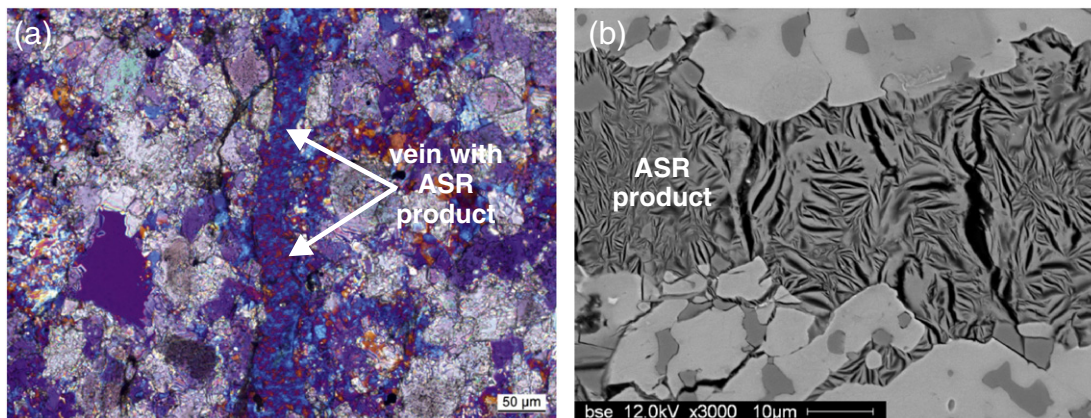


Fig. 1. (a) Optical image of the ASR product in the concrete aggregate obtained with a polarization microscope. Crossed polarizers with inserted gypsum plate. (b) SEM image of the vein with ASR product in the concrete aggregate. The light gray mineral of the aggregate is calcite, the dark gray ones are quartz and feldspar.

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