ALKALI-SILICA REACTION OF AGGREGATES IN REAL CONCRETE AND MORTAR SPECIMEN

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ABSTRACT

Alkali-silica reactivity potential of aggregates was investigated in concrete samples and experimental mortar bar specimens employing petrographic methods, such as polarising microscopy, petrographic image analysis, and scanning electron microscopy combined with an energy dispersive spectrometer (SEM/EDS method). Chert-rich limestone is regarded to be the most reactive rock type among the investigated aggregates. Quartz-rich aggregates (e.g. quartz-rich sediments, meta-sediments and other metamorphic rocks) show medium ASR potential. A very low degree of ASR was observed in connection with magmatic rocks, especially with basaltic rocks, serpentinites and granitoids. Comparison of those alkali-silica gels in concrete samples with those from mortar bar specimens pointed-out several important factors, such as the time factor and external parameters, affecting the ASR. This study also aimed to the application of main conclusions in a practice. A modification of the conventionally used mortar bar method (following the ASTM C1260 standard) by an after-test petrographic investigation of mortar bar specimens was incorporated into the updated version of the Czech Technical Regulation TP 137 (the complex of methods regulating aggregates testing for ASR in the Czech Republic).

KEYWORDS: alkali-silica reaction, concrete, microscopy, SEM/EDS

STRUCTURE OF THE THESIS

The thesis comprises main text body including six chapters discussing: (1) problem of alkali-silica reaction (ASR) generally, (2) investigation of concrete samples, (3) experimental testing of ASR potential of natural aggregates, (4) ASR products and the factors affecting the reaction, (5) the main conclusions and applications and (6) references. The main conclusions of the thesis were summarised in five papers. Two of them were published in conference proceedings (Lukschová and Přikryl, 2006, Lukschová et al., 2008), two papers were accepted and/or published in the impacted journals (Lukschová et al., 2009; Lukschová et al., in print) and one paper is still under review (Lukschová et al., under review). All five papers are in appendix of the thesis.

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INTRODUCTION

The alkali-silica reaction (ASR) represents one of the most important damaging mechanisms in concrete. ASR is based on the dissolution of silicarich aggregates in highly alkaline solutions in concrete constructions (e.g. Hobbs, 1988; St John et al., 1998). Three important alkalis (Na⁺, K⁺ and Ca²⁺) release from cement into pore liquids and form $Na^+ + OH^-$, $K^+ + OH^-$, and $Ca^{2+} + OH^-$. If these constituents are not incorporated in the cement's hydration products, they accumulate in the pore solution, increasing the concentration of hydroxyl ions in the cement's pore fluids. Thus, it is the hydroxyl ion, not the alkali ion, which initiates the chemical reaction of silica. First, the oxygen Si-O bonds on the aggregate surface are hydrated; and then replaced by silanol groups. Silanol groups react with hydroxyls. The increasing number of siloxane groups replaced by silanol groups creates a gel-like layer covering the aggregate surface, containing monomers of $H_2SiO_4^{2-}$ and $H_3SiO_4^{-}$. Their negative charges attract positive ions of K, Na and Ca present in the pore solution, forming alkali-silica gels with variable proportions of alkalis (e.g. Diamond et al., 1975; Chatterji et al., 1989; Vestraete et al., 2004; Chatterji, 2005; Ichikawa and Miura, 2007; Poyet et al., 2007). These alkali-silica gels tend to absorb water molecules, expand, and cause mechanical damage in



Fig. 1 Macroscopically observed ASR cracks – floor framing arch (Tuřice, part a), construction beam (Trutnov, part b), landing front (Lysá nad Labem, part c), crash barrier (Prague, parts d, e, accepted from Horský et al. 2007, modified) and face side (Dolany, part f, accepted from Míčka and Klier 2006, modified).

concrete by increasing tensile stress (e.g. St John et al., 1998; Struble and Diamond, 1981; Garcia-Diaz et al., 2006). Macroscopically, the ASR is visible by the network of ASR cracks covering the concrete surface, partially filled by white and/or transparent alkali-silica gels (e.g. Clark et al., 1992; Ponce and Batic, 2006). The ASR can be mitigated by: the elimination of alkali-silica reactive aggregates, the application of chemical additives mitigating the ASR in the concrete, and the use of coatings for the reconstruction of concrete constructions affected by ASR. The first mentioned, with the elimination of alkali-silica reactive aggregates, seems to be the most effective (e.g. Xu et al., 1995; Kurtis and Monteiro, 2003; Feng et al., 2005; Bektas et al., 2006). Aggregates are tested using petrographic (e.g. ASTM C295, RILEM TC 191-ARP), dilatation (e.g. ASTM C227, ASTM C1260, ASTM C9) and chemical methods (e.g. ASTM C289). Also detailed petrographic inspection of ASR-affected concrete structures produces important information about the ASR mechanisms and alkali-reactive aggregates (e.g. Hobbs, 1988; French, 1991; Clark et al., 1992; St John et al., 1998; Eden, 2004; Jensen, 2004; Fernandes et al., 2007).

This PhD thesis represents a pilot study, carried out in the Czech Republic, focused on the employment of the petrographic methods (such as optical and electron microscopy, and petrographic image analysis) on the investigation of ASR in both real concrete structures and experimental mortar specimens.

EXPERIMENTAL

The concrete samples, taken from 13 different bridge constructions and one crash barrier, from various places within the Czech Republic, were investigated in three different steps: on the macroscale, based on the in-situ inspection (realized in cooperation with Pontex Ltd. and Horský Ltd.); on the macro-scale, based on laboratory methods (e.g. colouring agents); and on the micro-scale using optical microscopy (polarising microscope Leica DMLP, Department of Geochemistry, Mineralogy and Mineral resources, Faculty of Science, Charles University in Prague), petrographic image analysis, as well as electron microscopy combined with energy dispersive spectrometry (Cambridge Cam Scan S4 instrument with energy dispersive analytical system, Oxford Instruments LINK ISIS 300, Laboratory of Electron Microscopy and Microanalysis, Laboratories of the Geological Institutes, Faculty of Science, Charles University in Prague).

Alkali-silica reactivity potential of quartz sands and gravels was tested using modified mortar bar and gel pat tests (ASTM C1260, BS 7943, see detailed description in Lukschová et al., 2008). The microscopic methods, similar to those employed in the investigation of the concrete, were also applied on the experimental mortar specimens containing natural aggregates (quartz sands and gravels from the areas around the Cidlina, Dyje, Ohře, Sázava rivers; although primarily in the Labe river area), with the objective to determine the ASR potential of aggregates, as well as to identify the reactive components in the aggregates (Lukschová et al., 2008; Lukschová et al., in print).

RESULTS

In-situ inspection pointed-out possible ASR degradation affecting all of the concrete constructions



Fig. 2 Alkali-silica gels (indicated by black arrow) and ASR cracks (indicated by white arrow) intruding quartz-rich aggregates. Optical microscope, parallel (parts a, c) and crossed (parts b, d) nicols. Concrete sample no. X604 (parts a, b) and mortar bar specimen no. 208a (parts c, d). Scale marker bar – 500 μm. CP – cement paste, Q – quartz or quartz-rich aggregate.

(Fig. 1). Additionally, other concrete degradation mechanisms were observed (e.g. steel reinforcement corrosion and in-leakage into construction joints). Microscopically, ASR was only confirmed in 9 out of the 14 constructions investigated (Lukschová et al., 2009).

In micro-scale, alkali-silica reactive aggregates were detected according to their spatial relationship with the ASR products. The ASR cracks (Fig. 2, part a, b), alkali-silica gels (Fig. 2, part c, d), and aggregate boundaries attacked by cement paste are the most important indicators of ASR.

The aggregates investigated in the concrete samples, as well as in the experimental mortar specimens, could be divided into four main groups, depending on their ASR potential:

I. The most reactive aggregates, composed mainly of chert-rich limestones, were detected in concrete samples. A very fine-grained siliceous (chert) component is regarded to be responsible for extensive ASR. On the macro-scale, the ASR is exhibited by intensive ASR cracks, partially filled by alkali-silica gels. On the micro-scale, high volumes of alkali-silica gels were detected, reaching 2.3 - 4.9 vol. %. Also, an intensive network of ASR cracks was observed, intruding into the aggregates and cement paste. This type of ASR is regarded to be very rapid.

- II. That aggregates showing a medium degree of ASR, such as quartz-rich aggregates. These aggregates were investigated in concrete samples, as well as in experimental mortar specimens. On the macro-scale, the ASR exhibit a network of ASR cracks, partially filled by alkali-silica gels. On the micro-scale, the volume of alkali-silica gels does not exceed 0.9 vol. % and ASR cracks are well-developed. The degree of ASR seems to be strongly affected by the time factor.
- III. A low degree of ASR was observed in connection with some types of aggregates (similar to those mentioned in Group II) contained in young concrete samples, taken from concrete constructions from the second half of



Fig. 3 Comparison between amount of alkali-silica gel in concrete and the year of the bridge construction.

the 20th century. The volume of alkali-silica gels does not exceed 0.3 vol. %. ASR cracks are hardly visible on either the macro-scale or micro-scale. The characteristic ASR indication, typical of the initial degree of ASR, is observed to be found in the aggregate boundaries, attracted by the cement paste.

IV. Non-reactive aggregates composed of magmatic rocks (such as granitoids, serpentinite, and basic volcanic rocks) show no or very low ASR. The volume of alkali-silica gels does not exceed 0.1 vol. %. Aggregate boundaries are unaffected by ASR. The ASR degree is independent from the time factor (Lukschová et al., 2008).

DISCUSSION

FACTORS AFFECTING ASR

Volume of alkali-silica gels and their chemical composition and morphology are affected by several parameters including type of specimen (real concrete sample or experimental mortar specimen), hardening period, composition and concentration of accelerating solution, temperature during test period, length of test period, type of cement, w/c ratio and/or aggregate petrographic characteristics (Wang and Gillot, 1991; Cong et al., 1993; Thaulow et al., 1996; Kurtis et al., 1998; St John et al., 1998; Jensen, 2004; Peterson et al., 2006; Fernandes et al., 2007; Lukschová et al., in print). A comparison of alkali-silica gels observed in concrete samples, with those from experimental mortar specimens, pointed to two the most important factors influencing the ASR:

- Time factor affecting especially concrete samples containing quartz rich aggregates (Group 2 in Fig. 3). Increasing age of the concrete increase volume of alkali-silica gels in concrete samples. Time factor also affects the morphology of alkali-silica gels. Alkali-silica gels from more than 50 years old concrete begin to "crystallize" (Fig. 4, part a). In contrast, alkali-silica gels from young concrete samples as well as mortar bar specimens show typical amorphous structure (Fig. 4, part b, c).
- Chemical composition of the surrounding environment (resp. chemical composition of accelerating solutions affecting the ASR in experimental mortar specimens). Alkali-silica gels from concrete samples contain high CaO/(Na₂O + K₂O) ratio. Alkali-silica gels originating in mortar bars (accelerated in NaOH solution) contain higher amount of Na₂O and alkali-silica gels originating in gel pats (accelerated in NaOH + KOH solution) contain higher amount of Na₂O and K₂O.

Both factors also affect the extent of ASR cracks and presence of aggregate boundaries attacked by cement paste (Lukschová et al., under review).

ASR OF QUARTZ-RICH AGGREGATES – INFLUENCE OF DEFORMATION AND RECRYSTALLIZATION CHARACTERISTICS

Part of this PhD study is focused upon the assessment of quartz-rich aggregates, with respect to their ASR potential. It was possible to describe three



Fig. 4 Alkali-silica gels. Crystallized alkali-silica gel filling microcracks in quartzite aggregate (part a, concrete sample no.180-010, image in SE, SEM/EDS method), amorphous alkali-silica gel overlaying quartz fragment (part b, gel pat specimen no. 208-GP1, image in SE, SEM/EDS method), amorphous alkali-silica gel filling pore voids and ASR cracks intruding chert-rich limestone and cement paste (part a, concrete sample no.V11B, image in BSE, SEM/EDS method). Scale marker bar – 500 μm. ASG – alkali-silica gel, CP – cement paste, C.-L. – chert-rich limestone, Q – quartz, Qtz. – quartzite.

different recrystallization mechanisms, according to the deformation, recrystallization, and grain size characteristics of quartz-rich aggregates: 1) a bulging recrystallization mechanism (BLG, typical of low temperature recrystallization and characterized by newly-formed fine quartz grains originating alongside of the old large quartz grains); 2) a subgrain rotation recrystallization mechanism (SGR, typical of medium temperature and medium-sized quartz particles); and 3) a grain boundary recrystallization mechanism (GBM, typical of high temperature and coarse grained quartz particles, see more detailed description of recrystallization mechanisms in Hobbs, 1968; White, 1977; Hirth and Tullis, 1992; Stipp et al., 2002). The ASR potential was assessed to be highest in the case of quartz-rich aggregates recrystallized by the BLG recrystallization mechanism; and decreasing in association with those quartz-rich aggregates recrystallized by the SGR and GBM recrystallization mechanisms. The origin of grains fine within the verv quartz BLG recrystallization mechanism seems to be responsible for the increase of the ASR potential of those aggregates that were initially of low alkali reactivity. The small number of analyzed samples decreases the statistical significance of the results. Consequently, an increase in the number of aggregates analyzed in both concrete samples, as well as mortar bar specimens, is required as a part of any future research (Pertold et al., 2008).

CONCLUSIONS

The contribution of this study is regarded to be the identification of alkali-reactive aggregates, spatially connected with ASR products, by employing petrographic techniques in real concrete and experimental mortar specimens. Optical microscopy enabled petrographic classification of the aggregates, investigation of their structural characteristics, determination of the volumes of the cement paste and pore voids, and identification of the ASR products. Comparing the methodologies, petrographic image analysis is the only technique allowing quantification of the data obtained during optical microscopy. Conventional optical microscopy is, however, necessary for identification of the phases present. The SEM/EDS method enhances detailed identification of both the chemical composition and morphology of the ASR products.

Alkali-silica gels, ASR cracks, and aggregate boundaries showing an interaction with the cement paste are the most important indicators of ASR. The intensity of individual signs is variable, dependent on both the type of sample and type of reactive aggregates.

The investigation of quartz rich aggregates is regarded to be a pilot study in the Czech Republic. The volume of quartz fragments and aggregates in the investigated samples showed poor or no correlation with the results from the different experimental methods. Detailed petrographic observations of the structural parameters of quartz aggregates, as well as petrographic inspections of these aggregates in concrete samples, showed the following important aspects: (1) very small new quartz grains, originating during a low degree recrystallization mechanism, exhibit important ASR; (2) well recrystallized coarse aggregates exhibit low or no ASR; (3) the ASR potential of quartz-rich aggregates is probably not only dependent on the degree of quartz deformation, but also on the grain size parameters. Both of these factors can be related. This approach to the reactivity



Fig. 5 Variability of individual types of alkalies in alkali-silica gels (ASG) in mortar bar specimens (mortar bar), gel pat specimens (gel pat) and concrete samples (concrete).

of quartz-rich aggregates seems to be very suitable for future research. Repeated comparisons of recrystallization characteristics of aggregates with their ASR potential can also increase the statistic rigor of the methodology.

The main conclusions of this PhD study regard possible application in practice in the Czech Republic. The petrographic analysis of experimental mortar bar specimens was summarised and amended into an updated version of Technical Regulation TP 137 (TP 137, 2003), which regulates the testing of aggregates in the Czech Republic with respect to ASR.

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ALKALICKO-SILIKÁTOVÁ REAKCE KAMENIVA V BETONU A V MALTOVÝCH TĚLESECH

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ABSTRAKT:

Náchylnost kameniva k alkalicko-silikátové reakci (ASR) byla zkoumána ve vzorcích betonu a v experimentálních maltových tělesech. Při výzkumu byly využity metody polarizační mikroskopie, petrografické analýzy obrazu a skenovací elektronové mikroskopie kombinované s energiově disperzním spektrometrem (SEM/EDS metoda). Kamenivo s maximální náchylností k ASR obsahovalo především rohovcem bohaté vápence. Jako středně reaktivní byly klasifikovány křemenem bohaté typy kameniva (křemenem bohaté sedimenty, meta-sedimenty a metamorfované horniny). Velmi nízký nebo nulový stupeň ASR byl pozorován ve spojitosti s některými magmatickými horninami (bazické vulkanity, serpentinit a některé typy granitoidů). Porovnání produktů ASR vzniklých v betonových vzorcích s produkty ASR v maltových tělesech pomohlo k určení hlavních faktorů ovlivňujících vznik ASR. Jedním z hlavních cílů dizertační práce bylo vyvinout (případně modifikovat) postupy používané k identifikaci ASR tak, aby byly použitelné v praxi. Běžně používaná urychlená trámečková metoda (podle ASTM C1260) byla doplněna o mikroskopický výzkum maltových těles a zahrnuta v návrhu aktualizované verze technického předpisu TP 137 regulujícího zkoušení ASR kameniva v České republice).