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Reaction products of densified silica fume agglomerates in concrete

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The writers are pleased to dedicate this small contribution to the memory of Professor H.F.W. Taylor, who over so many years contributed so much to the field of cement chemistry

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Abstract

Most silica fume currently used in concrete is in the dry densified form and consists of agglomerates of sizes between 10 μm and several millimeters. Many of these agglomerates may break down only partially in normal concrete mixing. Examination of various mature silica-fume-bearing concretes using backscatter mode scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) analysis shows that such agglomerates have reacted in situ and given rise to recognizable types of reaction products filling the space within the original outline of the agglomerate. One type is "quiescent," and usually shows no evidence of volume instability. EDX spectra indicate that the product formed within such grains is C-S-H of very low Ca/Si ratio, with modest alkali contents. Other silica fume agglomerates may undergo a distinct alkali–silica-type reaction (ASR), with the reaction product found within the original outline of the agglomerate having significantly less calcium and usually much higher alkali contents than the quiescent type. Such reacted agglomerates show evidence of local expansion, shrinkage cracking (on drying), and other features common to ASR. Both types may be found within the same concrete, sometimes in close proximity. It further appears that exposure to seawater may convert previously formed reaction products of silica fume agglomerates to magnesium silicate hydrates.

Keywords: Silica fume; Alkali–aggregate reaction; SEM; Calcium-silicate-hydrate (C-S-H); Microstructure

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1. Introduction

It is now well established that silica fume (or microsilica), in whatever form supplied, exists almost completely in the form of fine spheres linked together into chains or clusters, rather than as isolated spheres [1–3]. These clusters of spheres are formed in the cooler part of the production furnace, as the air-suspended droplets of molten SiO_2 contact each other in the process of solidifying. Multiple contacts between the solidifying droplets seem to form permanent fused bonds, which appear to be difficult to break. Transmission electron microscope images of such clusters have been published by various authors [1,2,4,5], and most recently by Diamond and Sahu [3]. The latter authors pointed out that isolated spheres are rare, and that typical overall clusters may contain anywhere from a few to several hundred spheres. The sizes of the clusters seem to range between about 0.3 μm and slightly over 1 μm.

Silica fume was originally supplied for use in concrete in the "as-recovered" form, i.e., as loose assemblages of such clusters and whatever individual spheres remained unattached. However, the extreme difficulty in handling such material led to the development of more easily handled forms, i.e., slurried and dry densified silica fume products. The slurried product is now rarely used, and may no longer be available at all in the United States. Except for silica fume interground in blended cements by cement manufacturers, it appears that almost all silica fumes currently used is incorporated in concrete as the dry densified product.

Dry densified silica fume is normally produced by air flotation within silos. The densification process is carried out at temperatures very much lower than the melting point of silica; thus, it presumably does not result in any additional permanent bonding between individual spheres, or enlargement of previously formed clusters. However, the tumbling action induces progressive entanglement of clus-

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ters into dense agglomerates [2]. The densification process is relatively slow, and air flotation may often require 24 h or more to generate commercially acceptable bulk densities in the order of 600–700 kg/m^2.

The end result of this process is the formation of agglomerates of various shapes and of sizes much larger than the original clusters. Agglomerates present in dry densified silica fumes range in size from perhaps 10 μm to as much as several thousand microns. The material constitutes a dry powder that flows easily and is readily incorporated in concrete. Fig. 1 provides a secondary electron mode scanning electron microscope (SEM) image of a commercial fume supplied by a major American silica fume supplier and used in research at Purdue University.

Secondary electron mode SEM images that show the sizes and shapes of intact agglomerates in three dimensions, such as Fig. 1, have not been commonly published. However, a number of authors, for example [6–8], have published backscatter electron mode SEM images like that of Fig. 2, which shows silica fume agglomerates as they appear in cross section.

To produce such images, the powdered silica fume is normally “potted” in epoxy resin, and after the epoxy is hardened, a random cross section is produced by low-speed diamond sawing. The surfaces so created intersect various agglomerates. One of these surfaces is then ground, polished, and coated using the normal methods of specimen preparation for backscatter mode SEM imaging.

As seen in Fig. 2, the agglomerates appear to be fairly dense. The larger ones are roughly spherical and an onion-like internal structure is commonly detected.

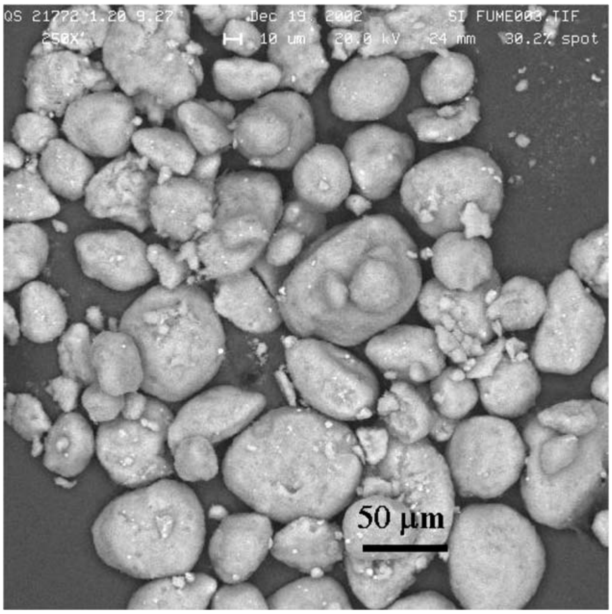

Fig. 1. Secondary electron mode SEM image of a commercial dry densified silica fume. Note the range of sizes and shapes of the various agglomerates present.

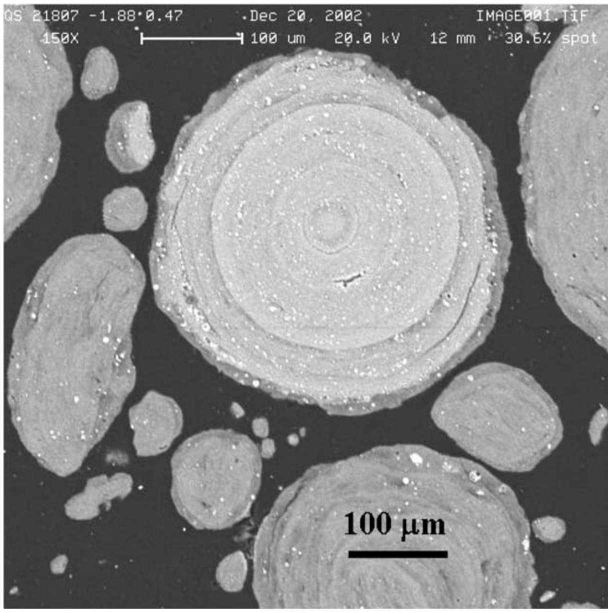

Fig. 2. Backscatter mode SEM of a commercial dry densified silica fume potted in an epoxy matrix.

While it is sometimes stated that the agglomeration process is somehow “reversible” [9], most of the available evidence suggests that this is generally not so.

The breakdown of agglomerates in actual concrete mixing depends on the effectiveness of the crushing and shearing actions transmitted to them during the mixing process. Extensive evidence has been supplied by Lagerblad and Utkin [6,10] and more recently by Baweja et al. [11] that, in conventionally mixed concrete, the breakdown of densified silica fume agglomerates is quite incomplete. While published data are fragmentary, it appears that in many field concretes, a considerable portion of the agglomerates remain at least partly intact. It appears that smaller agglomerates and fragments tend to be protected from crushing by the action of sand grains that surround them and cushion them against impact. It was indicated by both Lagerblad and Utkin [6] and Baweja et al. [11] that the agglomerates undergo even less effective breakdown in mortars than they do in concretes.

Undispersed agglomerates in mortars and concretes have been reported by various authors to be capable of participating in alkali–silica reaction (ASR) by acting as a form of reactive silica [7,10,12–15]. Some details of the process were investigated by Shayan et al. [12], using secondary electron mode SEM and energy-dispersive X-ray (EDX) analysis in examining concrete fracture surfaces. They indicated that the composition and characteristics of the reaction products varied with the alkali content of the concrete. In mature laboratory concretes, they found that both alkali and calcium had penetrated throughout the reacted agglomerates (“globules”); but only in high-alkali-content concrete were evidences of partial dissolution and formation of ASR gel seen.

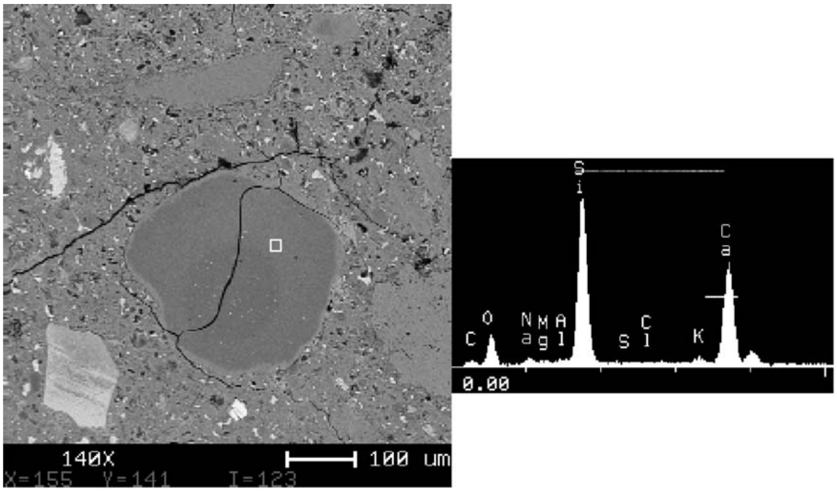

Fig. 3. Appearance and composition of a typical agglomerate that has reacted to produce low-calcium C-S-H.

In the present article, the writers provide evidence that silica fume agglomerates found in concrete after prolonged exposure have undergone reactions to produce two distinct classes of mature reaction products replacing the silica within the boundary of the agglomerate. Under sufficient alkaline exposure, ASR-type gel is produced, but some agglomerates within the same concrete may produce an apparently stable low-calcium C-S-H gel product entirely confined within the boundary of the original agglomerate. While such internally generated C-S-H products contain appreciable calcium, they are of much lower Ca/Si ratio than the C-S-H gel developed in cement hydration.

2. Agglomerates generating low-calcium C-S-H

Silica fume agglomerates that have remained undispersed after conventional mixing in various field and laboratory concretes have been examined in backscatter-mode SEM. All of the concretes involved are mature and nearly fully hydrated; most have been exposed to moist environments for several years.

The relicts of silica fume agglomerates are readily found and easily recognizable but are quite different in textural appearance and composition from the original unreacted silica fume agglomerates, such as are shown in Figs. 1 and 2.

One type of reacted agglomerate found in most concretes is exemplified in Fig. 3. In this and succeeding figures, a backscatter mode SEM image is provided on the left, and an EDX spectrum taken at a point within it is presented on the right. The specific location of the spot from which the EDX spectrum displayed was secured is indicated by a white open square marker in the SEM image.

The reacted silica fume agglomerate shown in Fig. 3 is about 300 μm in size. The gray level is uniform across the grain, suggesting a uniform composition. The accompanying EDX spectrum indicates the reaction product is a calcium silicate hydrate with small contents of both sodium and potassium. The Ca/Si peak height ratio is about 0.6, which is very much less than the usual value of about 1.5 for

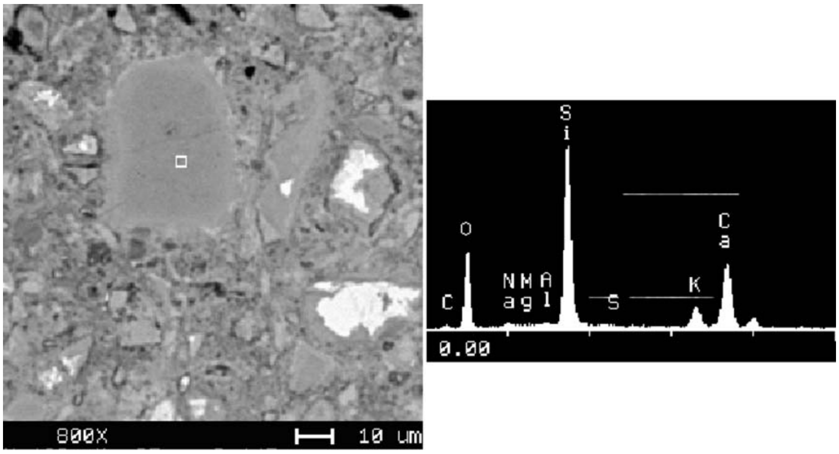

Fig. 4. Appearance and composition of another example of a silica fume agglomerate that has reacted to produce low-calcium C-S-H.

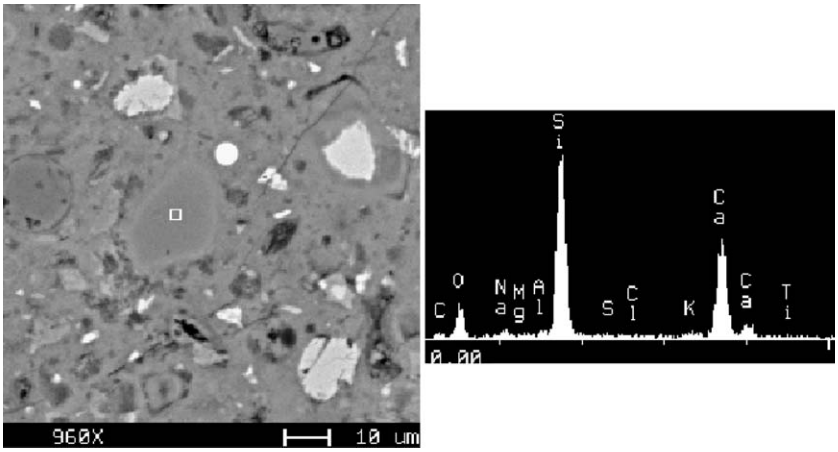

Fig. 5. Appearance and composition of a small agglomerate in a fly-ash-bearing concrete. Note the difficulty of identifying it purely by appearance.

normal C-S-H in concrete. EDX examinations in different areas across the reacted grain showed sensibly uniform composition, except for the bright rim, which is enriched in calcium. Such bright rims are characteristic of essentially all reacted silica fume agglomerates and have been reported by Shayan et al. [12], among others.

A smaller in situ reacted silica fume agglomerate, taken from a different concrete, is shown in Fig. 4. The agglomerate depicted here is about 50 μm in size. As indicated by the accompanying EDX spectrum, its composition is similar to that shown in Fig. 3, except that there is slightly less calcium, slightly more potassium, and almost no sodium. Even smaller, but otherwise similar, reacted silica fume agglomerates occur in many concretes, many of them presumably representing agglomerates partly broken down during mixing.

Very small agglomerates or fragments become increasingly difficult to detect in backscatter SEM because of their close resemblance to "inner product" C-S-H, both in gray level and general appearance. However, they appear to retain the low Ca/Si ratio characteristic of larger reacted agglomerates of this class. An illustration of such a small reacted agglomerate is shown in Fig. 5, taken from a concrete that contains fly ash as well as silica fume. The reacted agglomerate, about 25 μm in size, blends in very well with the surrounding C-S-H, and the surrounding rim is not well marked. In such cases, recourse to chemical composition information by EDX is usually needed for identification.

Fig. 6 shows the composition of a bright rim surrounding a reacted silica fume agglomerate in a concrete exposed to seawater. The Ca/Si peak height ratio of the rim is slightly more than 1, a value quite characteristic of most distinct rims that we have examined. The area is visibly microcracked, but petrographic study indicated that the cracking was not induced by the reacted agglomerate. There is a substantial chloride peak displayed in the EDX spectrum. Such chloride peaks are unusual, and in this case, reflects the penetration of chloride derived from seawater exposure.

It is apparent that within-agglomerate reaction products described here are produced when the calcium, alkalis, and occasionally other ions diffuse from the surrounding cement paste into the agglomerates. Low-calcium C-S-H reaction products of this type appear to be dimensionally stable, and there is usually little or no apparent disturbance of the surrounding paste. Microcracks are occasionally seen, but

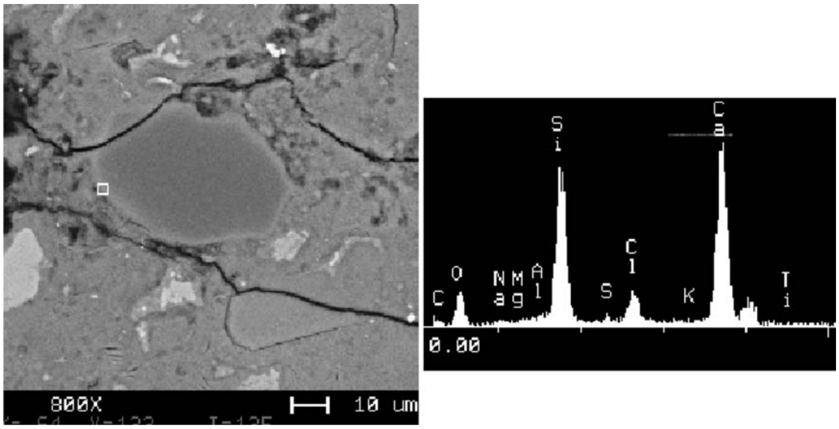

Fig. 6. A low-calcium C-S-H-type-reacted agglomerate in a seawater-exposed concrete showing the composition of the bright rim.

can usually be traced to other causes, as in Fig. 6. These reacted agglomerate grains are dense and appear to be well bonded to the surrounding cement paste. No visible internal porosity is detectable at the comparatively low backscatter SEM resolution usually employed. Mechanical probing of some larger reacted grains of this type with a fine-pointed pick under an optical microscope indicated that they were quite hard.

In the present article, the writers will generally refer to low-calcium C-S-H in situ reaction products as quiescent, in distinction to the ASR-type reaction products discussed subsequently. The latter, in contrast, usually show strong evidence of volume change activity.

3. Agglomerates generating ASR-type reaction products

As has been mentioned previously, a number of authors have reported that undispersed silica fume agglomerates can react as alkali–silica reactive aggregates in concrete. Fig. 7 provides a typical illustration.

While the gray level of the reacted agglomerate in Fig. 7 seems roughly similar to those of the quiescent-reacted agglomerates shown previously, the composition and appearance of the reaction product is quite different. The ASR reaction products typically show much lower calcium contents than those found in the low-calcium C-S-H products described previously, and the alkali peaks are usually higher. In the example shown in Fig. 7, substantial contents of both potassium and sodium are present. In other examples found in various concretes, one or the other alkali metal cation may predominate. The calcium contents are often even lower than that shown in Fig. 7, but a little calcium is always present.

However, one characteristic feature that differentiates the ASR class of in situ reacted agglomerates is the presence of indications of physical changes that have taken place subsequent to the formation of the reaction product. Most agglomerate grains showing compositions like that of Fig. 7 are multiply cracked, with the largest cracks often extending into the surrounding paste. Such cracking usually indicates that the grains expanded while still mechanically fairly competent. The reacted agglomerate also may have taken up water during expansion, and subsequent drying–shrinkage cracking may have taken place. Apparent dissolution of parts of the reacted grains is a common feature. Actually, the microstructural appearance and range of chemical composition of such ASR-type-reacted silica fume agglomerates are essentially identical to those found when similarly sized opal sand grains undergo ASR. Examples of the latter have been illustrated, for example, in Ref. [16].

A second illustration of this kind of reacted agglomerate grain is shown in Fig. 8. Here, the light-colored rim is more clearly marked and definite. The composition of the reacted agglomerate product is similar to that in Fig. 7, except that the contents of both calcium and potassium are lower. Similar features in ASR-reacted silica fume agglomerates were depicted in secondary electron SEM examinations by Shayan et al. [12].

It is important to note that both the quiescent-reacted agglomerate grains and the ASR-reacted agglomerate grains often occur within the same concrete, and are sometimes physically quite close to each other. Examples have been found where grains of the two different types were less than a millimeter apart and presumably were exposed to similar, if not necessarily, identical pore solution compositions. The specific factors that predispose a given agglomerate to produce one or the other form of product are not evident, although we have noticed that, in general, the quiescent product grains tend to be smaller on average than the ASR product grains.

An absolute distinction between the two types of grain is sometimes difficult to maintain. Nearly all of the grains that show low-calcium C-S-H compositions, like those illustrated in Figs. 3–5, are visibly quiescent, but occasionally, a grain is found with a calcium content that would tend to place it in this class, but nevertheless, that

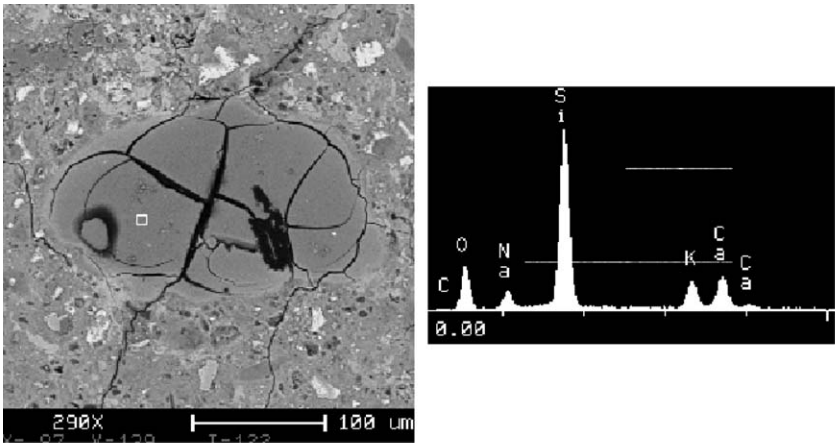

Fig. 7. Appearance and composition of a silica fume agglomerate that has reacted to produce a typical ASR-type reaction product.

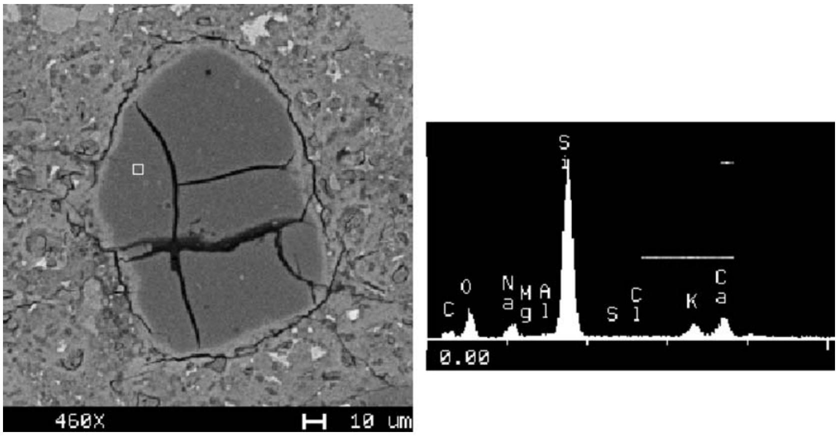

Fig. 8. Appearance and composition of a second silica fume agglomerate that has reacted to produce the ASR-type reaction product.

shows some indication of the cracking characteristic of ASR-type behavior.

4. Secondary magnesium silicate hydrate reaction products

An unusual feature was detected in the course of examining certain silica-fume-bearing concretes exposed to seawater. Grains were found of the general size and overall appearance of other reacted silica fume agglomerates, but of unexpected compositions. Some showed only Mg and Si peaks; others showed Mg and Si peaks with smaller Ca peaks; still, others had small peaks for aluminum as well. All of these grains were found within a very narrow outer layer of the concrete, typically within a millimeter or so from the exposed outer surface. Reacted agglomerates found further into the concrete showed either the quiescent low-calcium C-S-H or the ASR compositions discussed previously.

The thin concrete layer in which these products were found showed evidences of carbonation, but the carbonated layer extended much below the narrow surface zone within which they were confined.

An example of such a grain is shown in Fig. 9. The grain appears quite dark in the backscatter image, due to the high Mg content and perhaps also partly to its porosity, and is extensively cracked.

It was considered that the Mg-rich reaction products found in these agglomerates are secondary alteration products of earlier quiescent and ASR-type products, with magnesium replacing calcium and alkalis. This conclusion is supported by the observation, in another concrete, of the unusual reaction product grain shown in Fig. 10.

This grain was also very close to the surface of a concrete exposed to seawater. The composition of the dark outer portion, which is shown in the upper EDX spectrum, is virtually identical to that of the grain in Fig. 9. The white open square denotes the location of this spectrum. The lower EDX spectrum, for the lighter inner portion of the grain and taken at the small filled square near the center, is very different. The light-colored inner part of the grain shows no Mg at all, and comparatively little calcium and

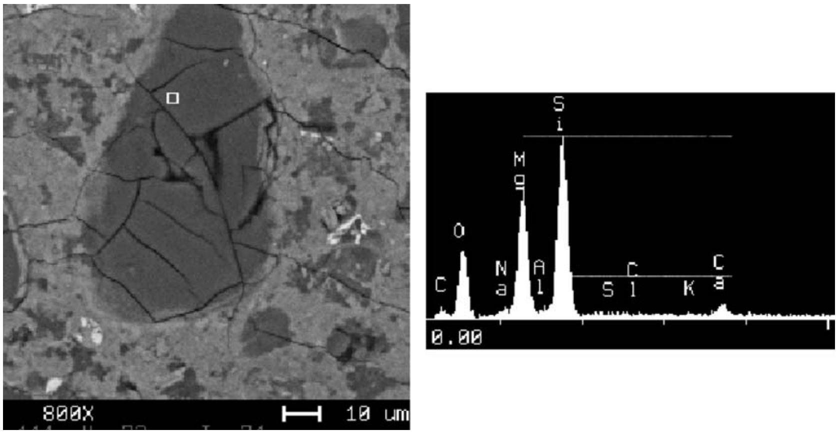

Fig. 9. Appearance and composition of a magnesium silicate hydrate secondary reaction product.

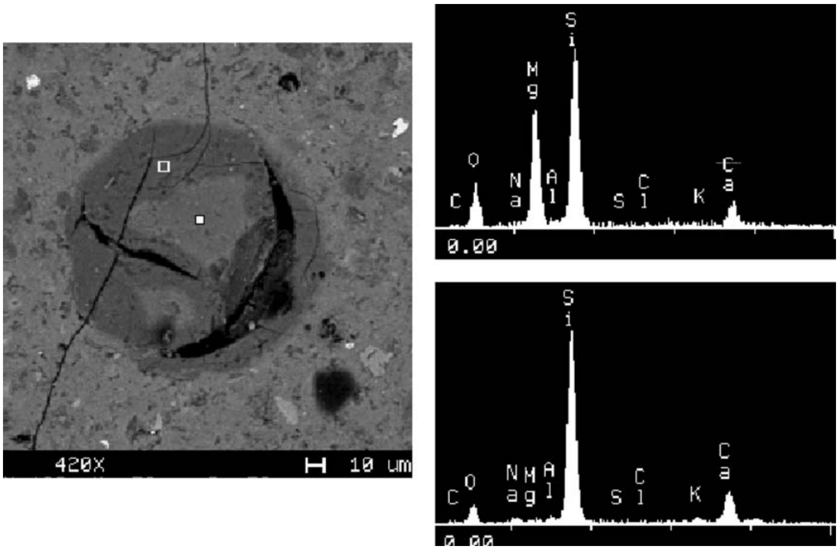

Fig. 10. Appearance and compositions of a reacted silica fume agglomerate apparently undergoing conversion to magnesium silicate hydrate. Upper EDX spectrum taken in outer (dark) zone. Lower EDX spectrum taken in central (somewhat brighter) zone.

alkali. The spectrum appears to represent that of a leached ASR-type reaction product.

It appears that magnesium has intruded into grains containing a previously established ASR-type reaction product and, by replacing the existing cations, has generated a secondary magnesium silicate hydrate reaction product. The magnesium is obviously associated with the seawater contact, although no brucite was found in the immediate vicinity. Local replacement of C-S-H by M-S-H in concrete exposed to seawater has been reported by various authors, including Shayan and Morris [18].

5. Discussion

A number of years ago, Bonen and Diamond [17] recorded the occurrence of several incompletely reacted but apparently quiescent-reacted silica fume agglomerate in a laboratory-mixed cement paste. As mentioned previously, Shayan et al. [12] appear to have found such products in laboratory concretes of low alkali content and also appear to have considered that they were incompletely reacted. However, most of the published investigations of reactions of agglomerated silica fume in concrete have been focused on ASR effects. It appears that alternative cases, that is, cases of agglomerates converting in situ to mechanically competent quiescent low-calcium C-S-H products, seem not to have been extensively reported.

Various factors may be responsible for this. In contrast to agglomerates that react in the ASR mode, agglomerates that react in the quiescent mode lack the familiar attention-drawing microscopic features stemming from expansion, dissolution, and drying–shrinkage characteristics of ASR. Moreover, as shown previously, small agglomerates of this type (approximately 40 μm or less) may appear to be very much like larger reacted cement grains of similar sizes that have undergone complete inner product hydration, except for the much lower Ca/Si ratio of the silica-fume-derived product.

While the ASR mode of reaction produces obvious attention-drawing features, local ASR-type behavior of individual agglomerates does not necessarily imply sufficient ASR reaction to produce distress in concrete. Serious ASR-induced distress certainly can happen, as has been indicated by various workers, but as indicated by Shayan et al. [12], it is likely to develop only if a sufficiently large population of reactive undispersed agglomerates undergoes ASR reaction.

Questions arise as to the permanent chemical stability of the quiescent low-calcium C-S-H reaction products formed in undispersed agglomerates. It has often been noted that expansive ASR reaction products in concrete may eventually be converted to more stable calcium-rich forms. Whether the low-calcium C-S-H products described here may eventually accumulate additional calcium and over time grow more nearly like cement-derived C-S-H is not known for certain; however, the writers have seen no evidence of this. On the other hand, whether these low-calcium C-S-H grains might conceivably be converted to unstable ASR-type grains on being exposed to fresh sources of alkali also remains to be determined. In particular, seawater penetration into exposed concrete may possibly produce such an effect.

Finally, the very presence of partly undispersed silica fume agglomerates in concrete raises another question. It is conventionally considered that one of the primary benefits of silica fume in concrete derives from the so-called fine filler effect. In this effect, the interfacial transition zone is thought to be densified by the incorporation within it of submicron-size spheres of silica fume. It is obvious that whatever densified silica fume remains as multimicron-sized agglomerates after mixing, cannot contribute to such an effect.

6. Conclusions

1. In most modern silica-fume-bearing concretes, the silica fume is introduced in densified (agglomerated) form, and the agglomerates are likely to remain at least partly undispersed in normal concrete mixing.
2. Such agglomerates react in situ to produce distinctive dense reaction products that appear to fill the entire space within the original boundaries of the agglomerates. Silica fume agglomerates do not remain unaltered in concrete.
3. Reacted agglomerates that have been found in various concretes show compositions and characteristics that fall into two distinct classes. One consists of C-S-H gel of much lower calcium content than usual in concrete. They typically show Ca/Si EDX peak height ratios of about 0.6, compared to about 1.5 for ordinary C-S-H. The second class consists of ASR-type reaction products of even lower Ca/Si ratios, and with variable but usually substantial, contents of alkali.
4. Both types of grain appear to develop surrounding rims of augmented calcium content.
5. The low-calcium C-S-H products are usually quiescent in that no visible effects of volume change are found; they appear to be hard and firmly bound to the surrounding hardened cement paste.
6. In contrast, the ASR-type reaction product agglomerates usually show local evidences of volumetric instability associated with ongoing ASR, i.e., swelling, shrinkage cracking on drying, and partial dissolution.
7. Both types of reacted agglomerates often appear in the same concretes, and may be in close proximity. Thus, differences in their development do not seem to reflect differences in alkali content of the concrete.
8. A secondary reaction may convert reacted agglomerate grains directly exposed to seawater to magnesium silicate hydrate.
9. The existence of ASR-type-reacted silica fume agglomerates in a given concrete may, but does not necessarily, indicate that sufficient ASR damage will occur to produce deterioration to the concrete as a whole.

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