# METHODS OF EXAMINATION

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ABSTRACT

Light microscopy, X-ray diffraction and electron optical methods like scanning electron microscopy, electron microprobe analysis, X-ray photoelectron spectometry are used in the examination of damaged concretes or in the mineralogical determination of potentially reactive aggregates.

Mechanism of concrete deterioration have been related to the localization of expansive gels, their elementary composition and evolution versus time, the coexistence of amorphous and crystalline products due to the diffusion of various ions through the pore solution. The potential reactivity of aggregates to alkalis has been connected with the presence in polycrystalline rocks of amorphous and cryptocrystalline silica, dolomitic grains, strained quartz, degraded feldspars and clay like materials.

# 1. INTRODUCTION

Light microscopy, X-ray diffraction and electron optical methods i.e. scanning electron microscopy, electron probe microanalysis, X-ray photoelectrons spectroscopy have already been used in the examination of deteriorated concretes. Such techniques showed recently the complexity of the alkali-aggregate reaction due to the ionic diffusion through the pore solution and to the formation of secondary compounds beside the expansive gel of alkaline silicate. This communication will present the microstructure of altered concretes and the mineralogical composition of aggregates used as a preventive measure. Most of the results which will be reported have been published since the Fifth International Conference held at Cape Town in 1981.

## 2. MICROSTRUCTURE OF DAMAGED CONCRETES

The understanding of the alkali-aggregate reaction has been approached through the examination of laboratory specimens and core samples extracted from concrete structures.

### 2.1. Laboratory specimens

The formation and composition of expansive gels were at first studied on Beltane opal or Pyrex glass immersed in soda or potash solutions.
A comprehensive work on the reactivity of Beltane opaline rock was carried out by Gutteridge and Hobbs /1/. As determined by X-ray diffraction, Beltane rock contained microcrystalline quartz, calcedonic silica, cristobalite, tridymite and clay-like material. The residue of a 3M NaOH solution attack was composed of the $\alpha$ quartz and clay material only. The reaction product from silicas occurred as a thin gelatinous layer around the opal grains. X-ray photoelectron spectometry, a surface analysis technique /2/, showed a change in the binding energies of $Na_{1s}$, $O_{1s}$, $Ca_{2p}$, $Si_{2p}$ electrons (Table 1). When a 3M NaOH solution saturated with $Ca(OH)_2$ was used, XPS data suggested that the reaction rim was composed of two silicates: a sodium silicate and a sodium-calcium silicate.

Table 1. Binding energies obtained from Beltane rock immersed in different alkaline solutions for 3 days. /1/

| Specimen | Binding Energie (eV $\pm$ 0.3) | | | | | | |
|----------|-------------|-----|-------|-----------|---------|---------|---------|
| | $Si_{2p}$ | | $O_{1S}$ | | | $Na_{1S}$ | $Ca_{2p}^{3/2}$ |
| Standard | 103.9 | | | 553.1 | | | |
| 1 | 103.0 | 100.9 | | 531.6 | | 1071.2 | 347.0 |
| 2 | 102.6 | 101.0 | 532.5 | 531.7 | | 1071.7 1070.2 | |
| 3 | 102.2 | 100.6 | 533.0 | 531.6 | 530.1 | | 347.3 |
| 4 | 101.4 | | | 531.8 | | 1070.8 | |

-1- 3 days in 3M NaOH solution at 20 °C.
-2- 3 days in 3M NaOH solution saturated with $Ca(OH)_2$ at 20 °C.
-3- Gel scraped from mortar bar containing Beltane opal and high alkali cement.
-4- Crystalline sodium metasilicate.

The results from sample 2 are very close to those from sample 3 which was the gel formed in the presence of a high alkali cement in a mortar containing Beltane opal as fine aggregate. The ratio of sodium to potassium (characterized by the XPS $K_{15}$ peak) in sample 3 was similar to the ratio of soluble sodium to potassium in the Portland cement used, respectively $0.11 \pm 0.03$ and $0.08 \pm 0.03$.

Gillott and Beddoes /3/ developed a Fourier method in order to follow the evolution of the shape and texture of a grain immersed in alkaline solutions. The shape of opal and quartzite grains has been expressed as the total roughness $T = P_c + T_c$ if $P_c$ is the shape contribution factor and $T_c$ the surface texture contribution factor. The boundaries of aggregates have been traced from the enlarged image on the screen of a Vickers projection microscope, before and after attack by a 1.6 M NaOH solution at 38 °C. Changes in shape coefficients, although significant in some cases, were difficult to interpret for opal. On the contrary, no shape change was observed after three months in the soda solution for quartzite even if quartz crystals were highly strained. Microstructure modifications were more pronounced than shape changes i.e. at the smooth and glassy fracture surface of opal grains appeared microfibers the existence of which has been explained as the outgrowth of gel products.

Diamond, Barneyback and Struble /4/ followed, by scanning electron microscopy SEM and energy dispersive analysis of X-ray EDAX, the diffusion of potassium and calcium into reacting opal aggregates. Two kinds of samples were studied : cylindrical opal specimen surrounded by a 1M KOH solution with solid $Ca(OH)_2$ particules and mortars where 10 % sand were replaced by an equal weight of opal and where the cement was a moderately high alkali cement. In both cases, K and Ca have spread from the pore solution into the opal grains. Although the calcium was low in the solution, its diffusion is clearly observed and could be related to a process of surface diffusion as already postulated by Powers and Steinour/5/.

Among commercial glass composition, Pyrex glass gives the highest expansion in alkaline solutions. As reported by Figg /6/, hydroxyl ions attack the silica network and leach alkali metal and borate ions into the solution giving rise to expansions even if Portland cements are low alcali.

These examples of laboratory studies have thus brought important results related to the mechanism of the alkali-silica reaction. However they represent simplified systems and neither Pyrex glass nor Beltane opal rock completely model the behaviour of real alkali-susceptible aggregates in concrete /6/. Reactions in concrete are more complex due to the heterogeneous composition of aggregates, different sources of alkalis simultaneous action of ions like $Ca^{2+}$, $SO_4^{2-}$, $CO_3^{2-}$ coexisting with $Na^+$, $K^+$, $OH^-$ in the pore solution.

### 2.2. Concrete specimens

The examination, on polished sections under light microscope, of core samples extracted from damaged concrete scructures shows that aggregates can be surrounded by a reaction rim : white rim around calcareous grains, dark rim around siliceous grains. Both were found in a concrete 30 year old /7/ (Fig. 1 and 2).
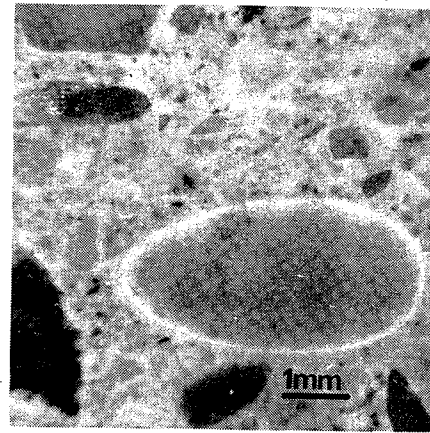
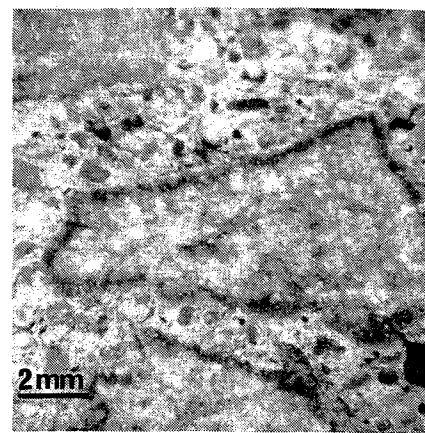Fig. 1.
White rim around a dolomitic aggregat^.



Fig. 2.
Dark rim around a quartzite grain.

The microstructure of these rims as revealed by electron optical methods, SEM and EDAX or EPMA (electron probe microanalysis) and WDS (wavelength dispersive spectrometer), is very often composed of several layers of different reaction products. A gel is also clearly observed in pores and cracks of the cement paste /6, 8, 9, 10, 11/. Alkali-carbonate reaction and alkali-silica (or silicate) reaction proceed somewhat differently and have been studied separately.

### 2.2.1. Dedolomitization

Dolomitie calcareous aggregates dissolve in the alkaline pore solution forming brucite $Mg(OH)_2$ and alkaline carbonate which reacts with $Ca(OH)_2$ and regenerates the alkali hydroxide. Poole /12/ detected by EPMA three zones of reaction around dolomite. At the contact with the aggregate, brucite characterizes the first zone. Calcite and the alkaline carbonate constitute the second zone. In the third zone carboaluminates and magnesium silicate are formed respectively by reaction of the alkaline carbonate with sulfoaluminates and by reaction of silica with brucite. $Mg(OH)_2$ is also found away from aggregates.

Carboaluminates $C_3A$. $CaCO_3$. $11H_2O$, calcite, brucite, ettringite $C_3A$. $3CaSO_4$. $31H_2O$ and thaumasite $CaCO_3$. $CaSO_4$. $CaSiO_3$. $15H_2O$ have been identified in the concrete containing calcareous /Fig. 1/ aggregates /7/. A richer zone in potassium at the interface cement paste–aggregate is shown by EPMA (Fig.3). These calcareous dolomitic aggregates were finely crystalline and microporous with inclusions of clay minerals. Though there was no expansive gel around dolomite, the weakening of the cement-aggregate bond could be partly related to the presence of clays due to their swelling behaviour and their capability to liberate alkalies. However, a gel was observed surrounding siliceous grains in the concrete made with mixed aggregates.
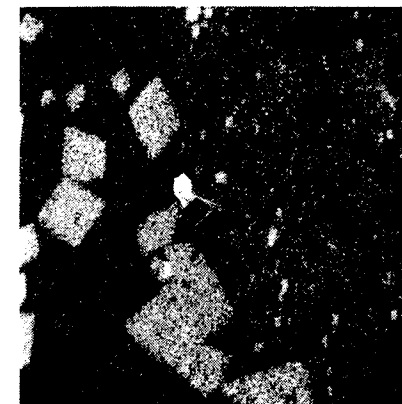
### 2.2.2. Alkali-silica and alkali-silicate reaction

As already seen in the case of reactive opal, the alkali-silica reaction manifests itself by the formation of a gel of alkaline silicate. The pressure
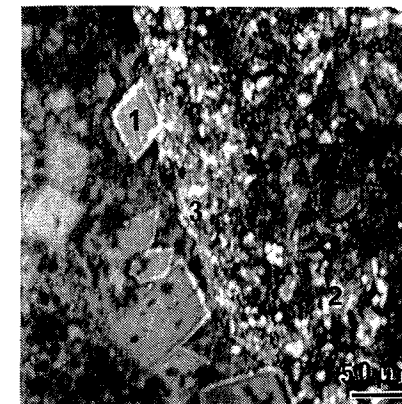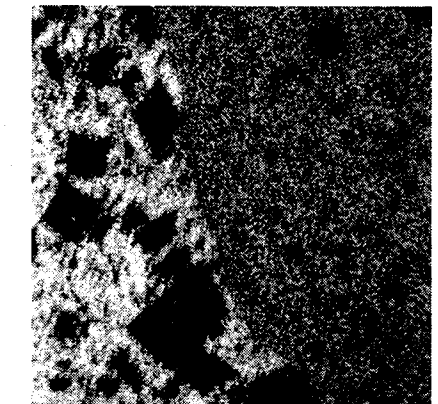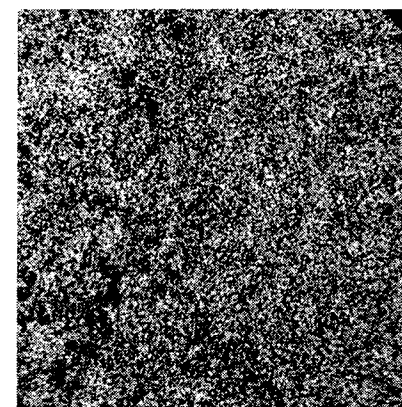
Absorbed electrons

Fig. 3.
EPMA. Damaged concrete.

1. Dolomite and siliceous aggregate
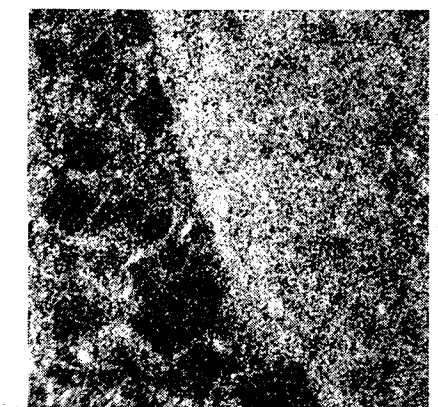2. Cement paste
3. Zone enriched in K



Mg



Si



Ca



K

exerted by this gel leads to the cracking of concrete structures. Many examples of this kind of deterioration have been published in the proceeding of the previous international conferences. One more example concerns a concrete structure built five years ago in a hot and humid country /7/. Granodiorites and quartzites were the reactive parts of the aggregates used in the concrete.

Granodiorites included strained quartz coexisting with plagioclases and feldspars, both minerals altered with many cracks and cleavage planes. Strained quartz crystals presented in transmitted light between crossed nicols, a high undulatory extinction angle (31 to 39°) after Dolar Mantuani procedure /13/. Quartzite aggregates were composed of microcrystalline silica as calcedony and amorphous silica as opal, both embedding quartz grains. Pyrite and calcite were minor compounds. In the damaged concrete, a gel is observed around each quartzite grain (Fig. 4) at the cement paste-aggregate interface. In the cracks within aggregates or between the cleavage planes appear groups of lamellar crystals richer in potassium but lower in calcium than the gel (Fig. 5). Massive ettringite deposits are localized either at the aggregate boarder or in pores and cracks of the cement paste (Fig. 6).

The same flaty silicates as those shown in Fig. 5 have also been observed by Cole, Lancucki and Sandy /14/ in a dam built 30 years ago. The crystalline product within the aggregates gave an X-ray diffraction pattern close to those of artificial zeolites (Na Al $SiO_4$)$_{12}$. 27H$_2$O or ( Ca$_6$ Al $SiO_4$ )$_{12}$. 30H$_2$O. The aggregates used in the concrete were sandstones (70 % quartz, 25 % chlorite, 20 % mica, feldspar and organic matter) and silstones (55% quartz, 25 % chlorite, 20 % mica, feldspar and organic matter). The quartz grains were embedded in a matrix of fine micaceous and argillaceous minerals. X-ray diffraction data /14/ confirm the results obtained by Buck and Mather /15/ and Regourd, Hornain and Poitevin /11/. They can characterize either a product different from the gel or a transformation of the gel. The second hypothesis could be emphasized by the not very different elemental composition of gel and crystals given by EDAX /14/ or EPMA /11/ (Table 2) and by an intermediate semicrystalline zone between gel and crystals as observed by SEM /11/.

Table 2. Chemical composition of alkali-aggegate reaction products.

| | $SiO_2$ | $K_2O$ | $Na_2O$ | $CaO$ | $Al_2O_3$ |
|---|---|---|---|---|---|
| **EPMA /11/** | | | | | |
| 1) Gel. | | | | | |
| – Around hornfels aggregates | 51.1±0.5 | 4.7±0.1 | 0.4±0.1 | 21.5±0.4 | – |
| – In the cement paste | 27.9±0.4 | 0.67±0.03 | – | 35.2±0.6 | |
| 2) Crystals within aggregates | 43.1±0.5 | 6.6±0.2 | 2.1±0.3 | 9.1±0.3 | |
| **EDAX /14/** | | | | | |
| Zeolite like material | | | | | |
| – Within aggregates (sandstone, silstone) | 83.7 | 5.6 | < 0.3 | 9.9 | – |
| – In the cement paste | 62.9 | 11.3 | 6.9 | 17.6 | 1.2 |



GEL
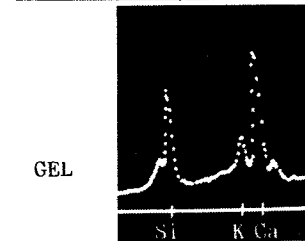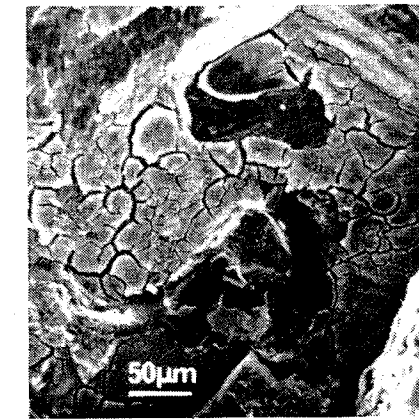
Fig. 4.
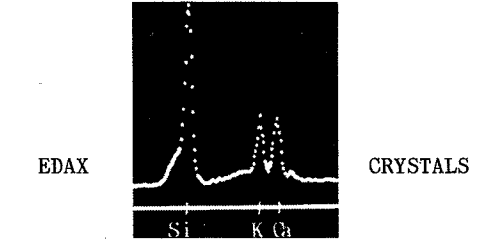Gel around aggregates at the contact of cement paste.
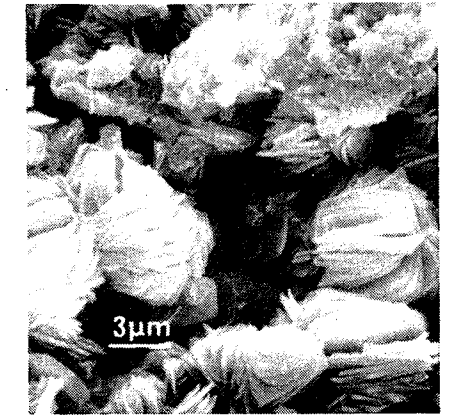


EDAX    CRYSTALS

Fig. 5.
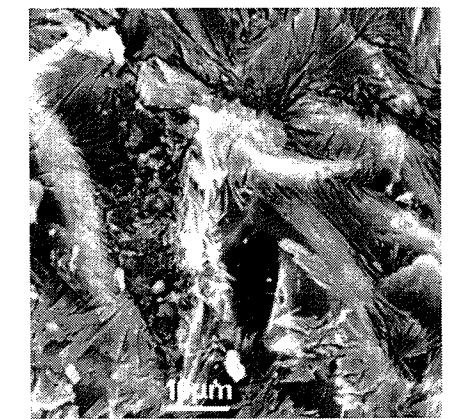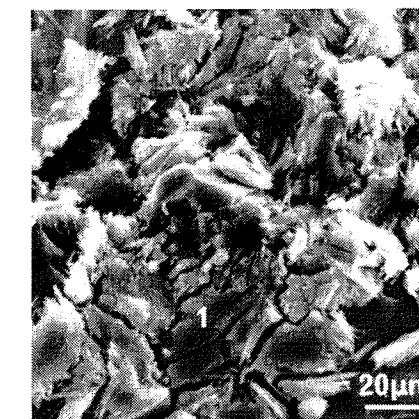Crystals on the aggregate.



a



b

Fig. 6.
Ettringite deposit at the surface of aggregates (a) and sand (b).

In their aged concrete, ·Cole, Lancucki and Sandy /14/ identified also the mineral trona $NaHCO_3$ . $Na_2CO_3$ . $2H_2O$, in pores filled with transparent or opaque gel. Cœxisting with calcite, this sodium hydrated carbonate occurred as fine interlocking needles and resembled to that found by Figg, Moore and Gutteridge /16/. Trona could have been formed by the carbonation of metastable silicate gels and might have been partly decomposed in zeolite-like material. Calcium hydroxyde reacted at the aggregate interface leaching out Al, Si, K, Mg and Fe. The action of $Ca(OH)_2$ would be the same as that studied in laboratory by Van Aardt and Visser /17/ on feldspars.

## 2.2.3. Discussion

The examination of concrete samples from deteriorated concretes shows not only an expansive gel around aggregates or in the pores of the cement paste but also crystalline products i.e zeolite-like material, carbonated minerals. as hydrated carboaluminates, trona, thaumasite. If all these compounds prove the importance of dissolved ions such as $Na^+$, $K^+$, $OH^-$ always considered in the mechanism of the alkali aggregate·reaction, they also emphasize the role of other ions like $CO_3^{2-}$, $SO_4^{2-}$, $Al^{3+}$.

The evolution of the reaction products versus time as the structuration of gels has to be considered regarding the life of the damaged concretes. Concrete must be carefully inspected in laboratory before recommending to demolish, to repair or to wait as it was done in USA /18/. Damaged dams were regulary inspected during forty years. The reaction ended after thirty years. Petrographic examinations of core samples showed that all the reactive parts of aggregates had reacted at that time. The gel was solidified, filling up the cracks.

One more signifiant point is the composition of polycrystalline aggregates. Beside reactive silicas, amorphous or cryptocrystalline, and strained quartz, altered minerals like feldspars or clays have a non negligible action, i.e releasing alkalis or leading to swelling. In consequence a petrographic examination of aggregates is highly recommended before using them in concrete.

## 3. PETROGRAPHIC EXAMINATION OF AGGREGATES

As we have seen in the observation of damaged concretes, reactive aggregates were very often constituted of both alkali reactive compounds and minerals with crystal defects. A petrographic examination can be done using at first a polarizing microscope, then a scanning electron microscope for more details of the microstructure.

### 3.1. Light microscope

Thin sections of polycrystalline rocks examined between crossed nicols using polarized light show the mineral distribution and in particular the suspect compounds. Altered feldspars appear striated with fine deposits of phyllitic material and spangled mica (Fig. 7). Quartz can occur vermiculated in myrmekite (Fig. 8) or strained with undulatory extinction (Fig.9). Crystalline quartz is easily distinguished from amorphous opal or fibrous calcedony (Fig. 10).

### 3.2. SEM

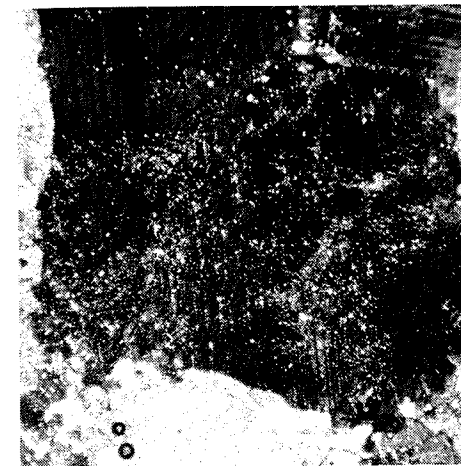More details on the surface or microtexture of aggregates are provided

Fig. 7.
Altered striated feldspar with phyllitic deposits and splangled mica in a granodiorite aggregate.
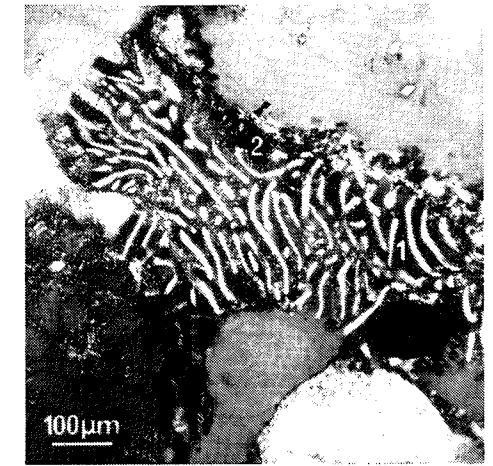


Fig. 8.
Myrmekite (1) and vermiculated quartz in an altered feldspar (2). This granodiorite aggregate also contains large quartz grains (3).
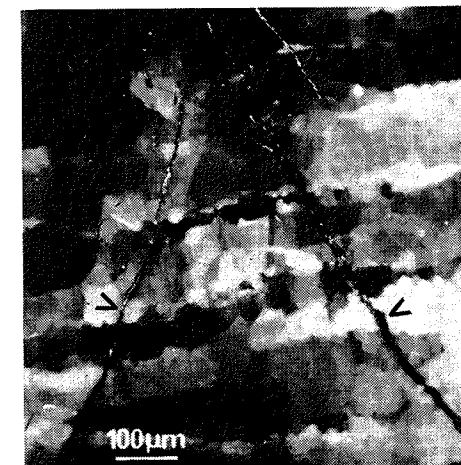


Fig. 9.
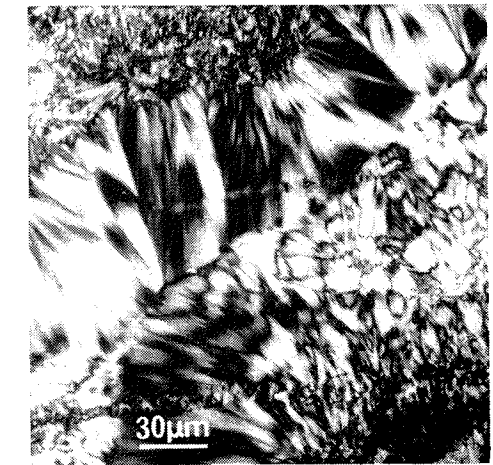Strained quartz with undulatory extinction and fine diaclases (↘) in a granite aggregate.



Fig. 10.
Fibrous calcedony in a flint aggregate.

by the SEM examination of rough surface fractures i.e. fibrous silica on a sand grain (Fig. 11), porous quartz boundaries in a granodiorite (Fig. 12), cleavage planes in a muscovite mica (Fig. 13), microcrack in a calcareous dolomitie aggregate (Fig. 14). Clay inclusions can be identified by their elementary analysis : Al, Si, K.

### 3.3. Reactivity indicators

Grattan-Bellew /19/, Sims /9/, Oberholster /20/ discussed the significance of accelerated tests. The optimum test depends on the type of aggregate (silica, carbonate, siliceous rock). The ASTM C 289-71 chemical test has given either pessimistic results for flint /9/ or optimistic results for deleterious shales /20/. The ASTM C 227-71 physical mortar bar test can be difficult to interpret due to unexplained contraction or only progressive and late expansion /19/. A poor correlation has been found between the expansion of rock cylinders and concrete prisms. If more research is needed to establish the best tests, scientists agree with a preliminary petrographic examination which detects the suspect compounds. Each country with cases of alkali aggregate reaction has undertaken a detailed analysis of characteristics and service record of used aggregates /8-21-22/.

The measure of the undulatory extinction angle UE is an adequate reactivity indicator for quartz. In this test elaborated by Dolar Mantuani /13/, three paramaters have to be determined :
(i) The amount of quartz in the rock
(ii) The number of quartz grains with undulatory extinction
(iii) The size of the UE angle.
At least ten grains are measured in the thin section. An average UE angle greater than 25° indicates an alkali reactivity of the aggregate.

The SEM examination of large polished sections of aggregates immersed in alkaline solutions at 80°C for three days could also be considered as a possible accelerated test. After treatment some grains are either simply corroded (Fig. 15) or partly or fully covered with a foliated reaction product (Fig. 16). In the presence of $Ca(OH)_2$, a thicker layer of calcium alkaline silicate is evident, XPS surface analysis of some of these polished sections shows a significant enlargment of the $Si_{2p}$ peak after treatment (11). The value of the $CaO/SiO_2$ molar ratio obtained via a calibration curve of $Ca_{2p}/Si_{2p}$ against the $Ca/Si$ atomic ratio was
$C/S = 0.11$ in the standard sample non treated,
$C/S = 0.12$ in the KOH treated aggregate,
$C/S = 0.61$ in the KOH and $Ca(OH)_2$.

## 4. ALKALIS DISTRIBUTION IN CEMENT PASTE

The solubilized alkalis which react with aggregates can be released both by the cement and aggregates. In a Portland cement, alkalis which are distributed in $C_2S$, $C_3A$ and sulfates go through solution rapidly. A concrete can be damaged even if the Portland cement used is low in alkali when feldspars, mica, clays liberate alkaline ions. In the presence of reactive aggregate, the best cement choice is a blended cement either a slag cement with more than 65 % slag /22-23/ or a pozzolanic cement with 30 % reactive pozzolan /24/. Slags and pozzolans give hydrated silicates C-S-H lower in calcium but richer in alkalis than the Portland cement C-S-H. The microporosity is also different : slag and pozzolan give a like amorphous C-S-H denser than the fibrous or reticulated Portland
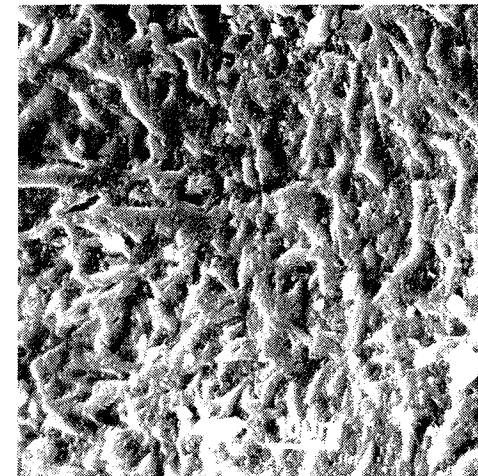
SEM. Fracture surface of reactive aggrgate.



**Fig. 11.**
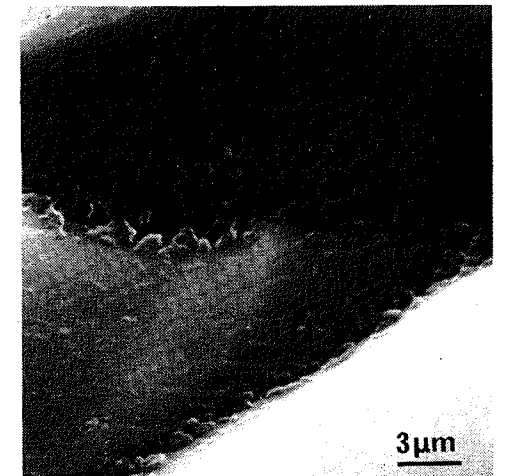Microfibrous silica at the surface of a sand grain.



**Fig. 12.**
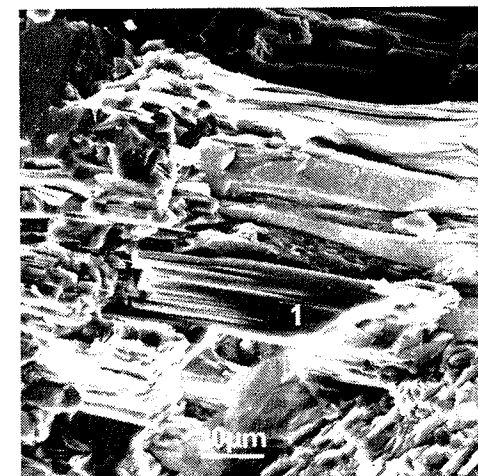Porous boundaries of quartz grains in a granodiorite aggregate.



**Fig. 13.**
Cleavage planes of a mica muscovite (1) in a granitic aggregate.



**Fig. 14.**
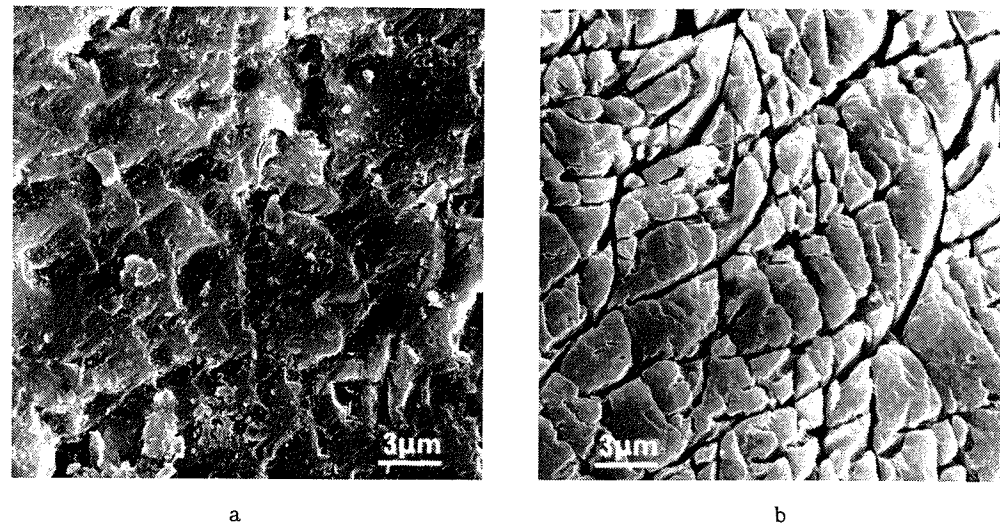Microcrystalline and microcracked dolomitic aggregate.

**Fig. 15.**
(a) Polished section of a quartz grain before treatment (b) after three days immersion in 1N NaOH, corroded surface but free of reaction product.
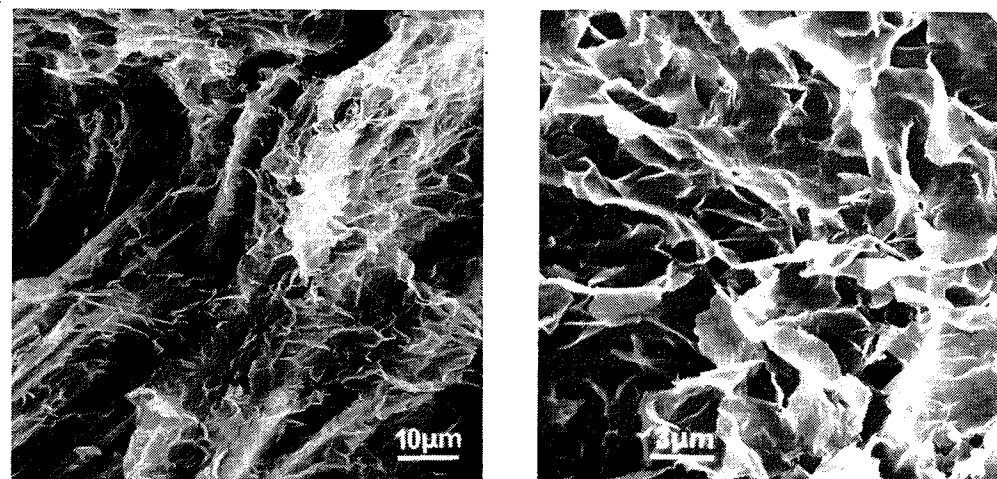


**Fig. 16.**
Foliated reaction product at the surface of a phyllosilicate after three days immersion in 1N NaOH.
There is no reaction product on the large quartz grains in this granite aggregate.

cement C-S-H. As an example in a pozzolanic cement containing 30 % silica fume at 2.8 % $K_2O$ and 70 % Portland cement at 0.8 % $K_2O$, the C-S-H keeps 1.1 % $K_2O$ and the C/S ratio is 0.9. In the same conditions the C/S ratio of the Portland cement C-S-H is 1.55 and the amount of $K_2O$ is 0.1 % $K_2O$ only, all values determined by EPMA /25/. Same results have been obtained with a pozzolanic cement containing tuff by Tang Ming She et al/26/.

## 5. CONCLUSION

Due to their complexity, alkali-aggregate reactions are not yet well understood and more research is needed for elucidating their mechanisms.
The microstructure study of core samples extracted from damaged constructions has shown that the source of alkalis can be cement and/or aggregates. The reactive parts of polyphased aggregates are not only dolomite and amorphous or crypto crystalline silica but also strained quartz, altered feldspars and micas, clay-like materials.

Beside the well known gel of alkaline silicate coexist, around or within aggregates, crystalline alkaline zeolite-like silicates and carbonated hydrates such as trona, carboaluminates and thaumasite. Ettringite very often in large amount is nearly omorphous. These coexisting compounds result from the diffusion of various ions $Ca^{2+}$, $SO_4^{2-}$, $CO_3^{2-}$, to be add to $Na^+$, $K^+$ and $OH^-$ ions always considered in the alkali-aggregate reaction which may thus be looked upon as an hydration reaction as IDORN /27/ suggested in his general Report to the Paris Congress on the Chemistry of Cement.

Accelerated tests at first elaborated for sand are not always well adapted to calcareous and siliceous rocks. Today scientists can however help civil engineers in the choice of concrete constituents. If aggregates are potentially reactive as determined by a petrographic examination, the cement will be a blended cement which keeps alkali in its C.S.H. If a concrete structure is damaged by the alkali-aggregate reaction, microscopists should follow the evolution of the process by examining core specimens at determined intervals correlated with the evolution of the map surface cracking.

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