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# Expansion mechanism of alkali–dolomite and alkali–magnesite reaction

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**Abstract**

The aim of this paper is to give a better understanding of the mechanism of alkali–carbonate reaction, which is essential to solve this durability problem in concrete structures. After studying the microstructural and compositional changes of alkali–dolomite and magnesite reactions both in KOH solution and in cement paste under 150°C autoclaving condition, the authors proposed that the formation and growth of reaction products in a confined space generated the expansion force, which was responsible for alkali–carbonate reaction expansion. © 1999 Elsevier Science Ltd. All rights reserved.

*Keywords:* Expansion mechanism; Alkali–carbonate reaction; Alkali–dolomite reaction; Alkali–magnesite reaction; Expansion of concrete; Microstructure

## 1. Introduction

The expansion mechanism of alkali–carbonate reaction (ACR) is still uncertain even the reaction was first recognised in 1957 [1]. One of the main discussion arguments is the source of the expansion force causing the damage of concrete structures. According to the literature, the expansion force may be due to water absorption by the clays of newly exposed surfaces [1,2], the swell of colloidal materials by absorbing water [3], the osmotic pressure by the membrane of clay around the individual dolomite rhombs [4], or the crystallisation pressure generated by brucite and calcite [5]. Katayama [6] suggested that ACR should be categorised into alkali–silica reaction, because the reactive cryptocrystalline quartz hiding in the matrix of carbonate rocks causes expansion. Besides, the location of the expansion force generated in concrete is not well understood. For some alkali reactive aggregates, reaction rims develop at the margin aggregate particles [7]. However, no definite relationship has been found between rim formation and expansion of concrete [8]. Carles-Gibergues et al. [9] proposed that secondary calcite formation in external rims was responsible for ACR expansion.

The reaction between dolomite and alkaline solution is usually called dedolomitization. In fact, dolomite is not the only mineral that can react with alkali solution to produce a solid product [10]. Magnesite has similar reaction as well.

Alkali–dolomite reaction (ADR):

$$CaMg(CO_3)_2 + 2MOH = Mg(OH)_2 + CaCO_3 + M_2CO_3 \quad (1)$$

Alkali–magnesite reaction (AMR):

$$MgCO_3 + 2MOH = Mg(OH)_2 + M_2CO_3 \quad (2)$$

where M represents Na, K or Li. If ADR or AMR occurs in the presence of portlandite ($Ca(OH)_2$) from cement hydration, $CO_3^{2-}$ may convert into calcite due to the following reaction:

$$M_2CO_3 + Ca(OH)_2 = CaCO_3 + 2MOH \quad (3)$$

In order to avoid the effects of clays and microcrystalline quartz, which are usually found in most reactive carbonate rocks, pure dolomite and magnesite rock were chosen in this study.

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In a previous study [11], we found that ADR and AMR can cause considerable expansion if the reaction products were highly confined in a limited space. The reactivity of pure and coarse-grained dolomite (NM-0) and magnesite (LM) rocks were tested by a newly developed autoclave method [12]. The results showed that NM-0 is non-reactive, while LM belongs to the intermediate reactive group. In order to simulate reactive carbonate rocks, laboratory experiments by use of the rock–cement compacted cylinder method described in the next section was carried out [13]. Test results showed that both NM-0 and LM compacts expanded. The expansion of compacts increases with the decrease of the particle size of dolomite and magnesite. There is a good correlation between the degree of the reaction and the expansion of compacts [13].

The purpose of the present paper is to provide microstructural evidence of ADR and AMR, the condition for generating expansion force and the basis for the previous hypothesis [5,11], namely that the formation and growth of brucite and calcite in reactive aggregates are the source of expansion.

## 2. Experimental

### 2.1. Materials

High purity coarse-grain dolomite (NM-0) and magnesite (LM) rocks were used in this study. They are the same as in the previous investigation [11]. The chemical compositions are shown in Table 1.

### 2.2. Surface reaction

Rock slices with the dimension 30 × 30 × 5 mm were cut from dolomite (NM-0) and magnesite (LM) rock samples. After being polished on one side, the slices were immersed in 10% KOH solution and autoclaved at 150°C for 10 h. The reacted rock surfaces were then washed carefully with distilled water and analysed by X-ray diffraction, scanning electron microscope (SEM) together with energy dispersive X-ray analysis (EDXA).

### 2.3. Interfacial reaction

Cement paste was cast on the top of the polished surface of NM-0 and LM rocks (Fig. 1). After seven days of curing in a moist container, the specimens were autoclaved at 150°C for 24 h and broken along the interface as marked in the figure. Both rock and cement paste sides were investigated by SEM-EDXA as well as XRD. In addition, the interfaces between aggregate and cement paste in concrete microbars were studied after 20 and 50 h of autoclave.

### 2.4. Reactions inside aggregate

Rock samples were cut into the shape of prisms about 10 × 10 × 40 mm. Measurement studs were mounted on both ends using Portland cement. After reaching a constant length in water, the prisms were autoclaved at 150°C in 10% KOH solution for different periods of time. The expansion was monitored for 50–60 h in the autoclave, and the results have been reported [11]. After

Table 1
The chemical composition of the rocks (wt%)

| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | Loss on ignition | $\Sigma$ |
|---|---|---|---|---|---|---|---|---|
| Magnesite (LM) | 0.54 | 0.15 | 0.24 | 0.20 | 47.07 | – | 51.66 | 99.86 |
| Dolomite (NM-0) | 0.36 | 0.16 | 0.16 | 31.38 | 20.98 | – | 46.64 | 99.68 |

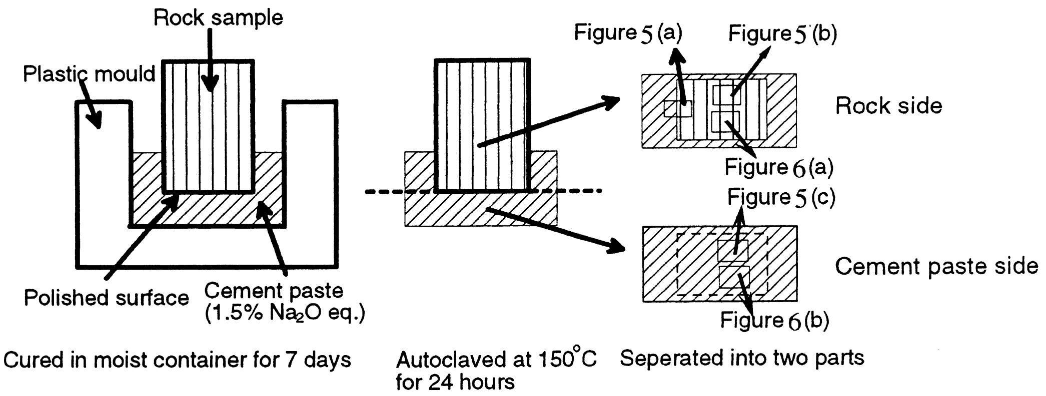

Fig. 1. Preparation of the reaction interface between cement paste and rock surfaces, and location of the SEM examination.

20 h of autoclaving in alkali solution, several prisms were taken out and broken across their length. Both optical and electronic microscopy analyses were carried out to detect the changes after autoclaving. Both NM-0 and LM showed reaction rims. The rim part and the internal part of the autoclaved prisms were sampled carefully for XRD analysis.

SEM-EDXA and XRD analysis were also carried out for the so-called concrete microbars. This autoclave method was proposed as a rapid method for the evaluation of alkali reactive carbonate rocks. The concrete microbar, 20 × 20 × 60 mm, was cast with the aggregate 0.5 ~ 1.0 cm in size at cement/aggregate ratio of 1:1 and water/cement ratio of 0.3. The alkali content in the cement was adjusted to 1.5% $Na_2O$ equivalent (per mass of cement) by adding KOH. The bars were demoulded following one day moist-chamber curing, and their original lengths were measured. The concrete microbars were then steamed at 100°C for 4 h and autoclaved at 150°C for different periods of time in 10% KOH solution. The length changes were measured after the bars had cooled down to 20°C. The results have already been published [11]. In parallel, after 20 h, some bars were taken out and analysed by microscopy and XRD.

### *2.5. Reactions in rock–cement compacted cylinders*

Rock samples were crushed into several size ranges (0.15–0.80 mm) and mixed with Portland cement with the ratio of 4:1. The alkali content in the cement was 1.5% $Na_2O$ equivalent (per mass of cement) adjusted by KOH, and the water/solid ratio was 0.1, the mixture was compacted under 300 MPa, which was maintained for about one minute. The compacted cylinders were usually 9 mm diameter and 30 mm length. Studs were then cemented on both ends as rock prisms for length measurement. The cylinders were cured in a moist container for 7 days and then autoclaved at 150°C in 10% KOH solution for 20 h. After this, the cylinders were broken and studied by SEM and XRD. Both dolomite and magnesite as well as corundum ($\alpha$-$Al_2O_3$ used as a control group) compacts were included in the present study. The compacted cylinders were proved to be an efficient way of simulating the reactive carbonate aggregates and reactions occurred between dolomite crystals and surrounding matrix [11,13,14].

## 3. Results and Discussion

### *3.1. Surface reaction*

The microstructural changes of the polished surfaces of rock slices after reacting in KOH solution are the first step towards understanding the chemical process. Fig. 2(a) is the SEM image of the unreacted dolomite (NM-0) after a slight of etching by HCl. EDXA (Fig. 2(d), Point "1") showed that the dolomite has an ideal chemical composition (Ca/Mg $\approx$ 1). After 10 h of autoclaving, it was found by EDXA that high calcium-bearing (Fig. 2(d), Point "3"), polyhedral particles "floated" on a film of the high magnesium-bearing products (Fig. 2(d), Point "2"). XRD analysis indicated the present of calcite and brucite ($Mg(OH)_2$). The large floating particles, several microns in diameter, were calcite, while the high magnesium film consisted mainly of brucite whose particle size was about twenty times smaller than that of calcite. The morphology of the reacted surface and the enlargement of the brucite layer are shown in Fig. 2(b) and (c) respectively. Because of the penetration of electron beams, the tested composition of the brucite film was influenced by the presence of other minerals beneath. Thus, no detecting point containing only magnesium was observed because of the presence of dolomite under brucite layer as shown in Fig. 2(d), point "2".

Dolomite reacted with NaOH solution at 23°C and 80°C were reported by Choquette et al. [10]. It can be concluded that the reaction of dolomite in alkaline solution and the morphology of calcite and brucite are very similar under the 150°C autoclave condition and at the lower temperatures.

Alkali–magnesite reaction was observed on magnesite rock (LM) surface. Fig. 3 shows the microstructure of LM surface before and after the autoclaving treatment. There is no visible deposit between magnesite cleavage planes before treatment. After autoclave in KOH solution, the surface was covered with very tiny (0.1 μm) brucite flakes. The X-ray diffraction spectrums of dolomite and magnesite surface before and after 150°C autoclaving treatment are shown in Fig. 4.

Obviously, magnesite can react intensively with alkali solution under the autoclave condition as described in Eq. (2). Different from ADR, the product of AMR is only brucite.

### *3.2. Interfacial reactions between cement paste and aggregate*

The interface of dolomite and cement paste is shown in Fig. 5. It was found that $Ca(OH)_2$ tended to deposit near the interface (Fig. 5(a)) as usually observed. At the dolomite side, a large amount of calcite was observed while $Mg(OH)_2$ was observed under the calcite particles clinging to the dolomite surface. Small amount of gel-like product containing Ca, Mg, Si was also recognised (Fig. 5(b) and (d), point "2"). However, it was not possible to determine whether it was C–M–S–H gel, i.e., $Mg^{2+}$ was combined into C–S–H gel or C–S–H gel mixed with a certain amount of $Mg(OH)_2$. Thus, C–M–

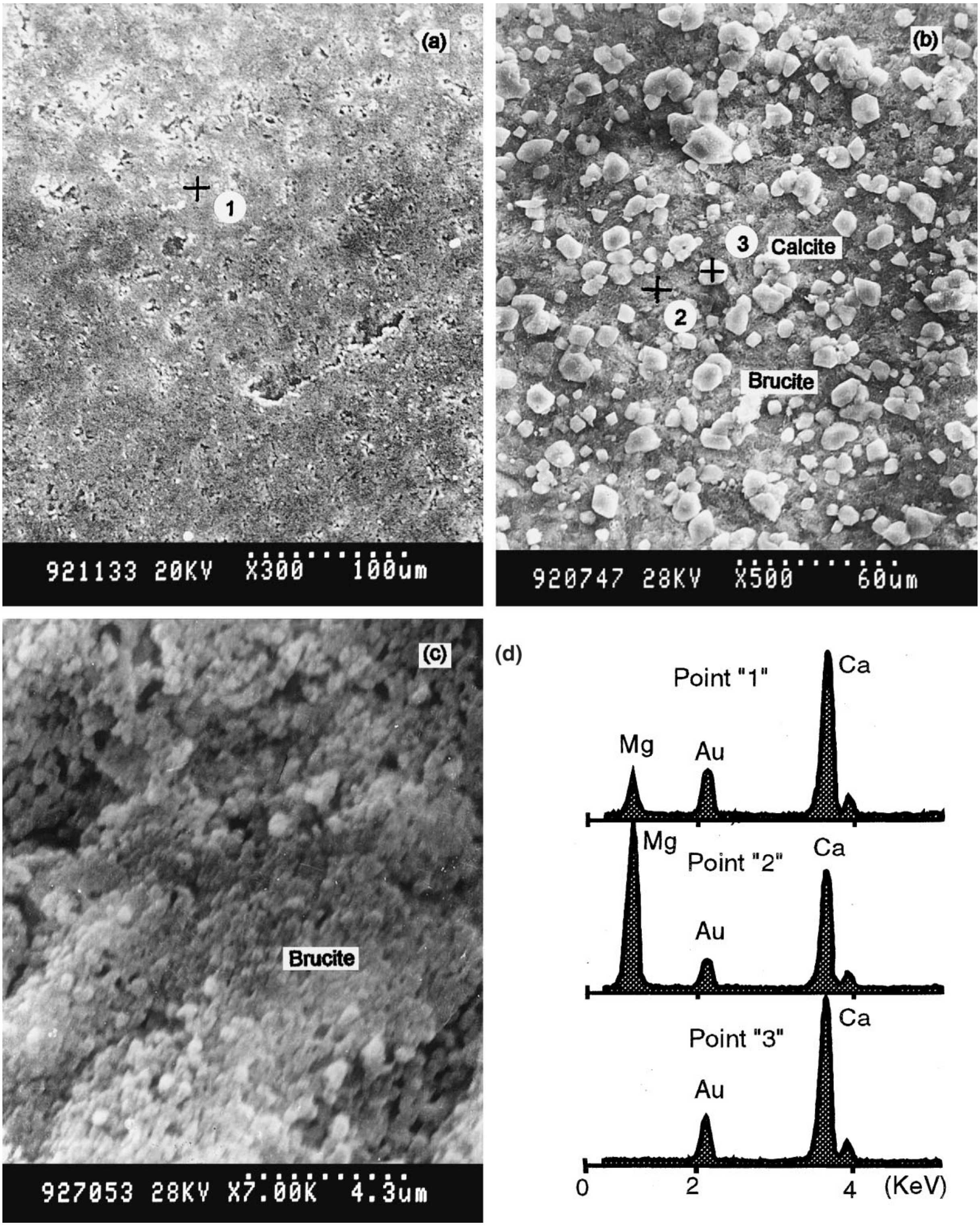

Fig. 2. SEM images of dolomite (NM-0) surface and EDXA results: (a) the original smooth surface of dolomite (NM-0) after slightly etching by dilute HCl solution; (b) the rock surface dolomite (NM-0) after autoclaving at 150°C in 10% KOH solution for 10 h showing calcite particles floating on a layer of brucite film; (c) the enlargement of the central part of the photo; (b) showing extremely fine brucite particles; (d) EDXA results of the marked points.

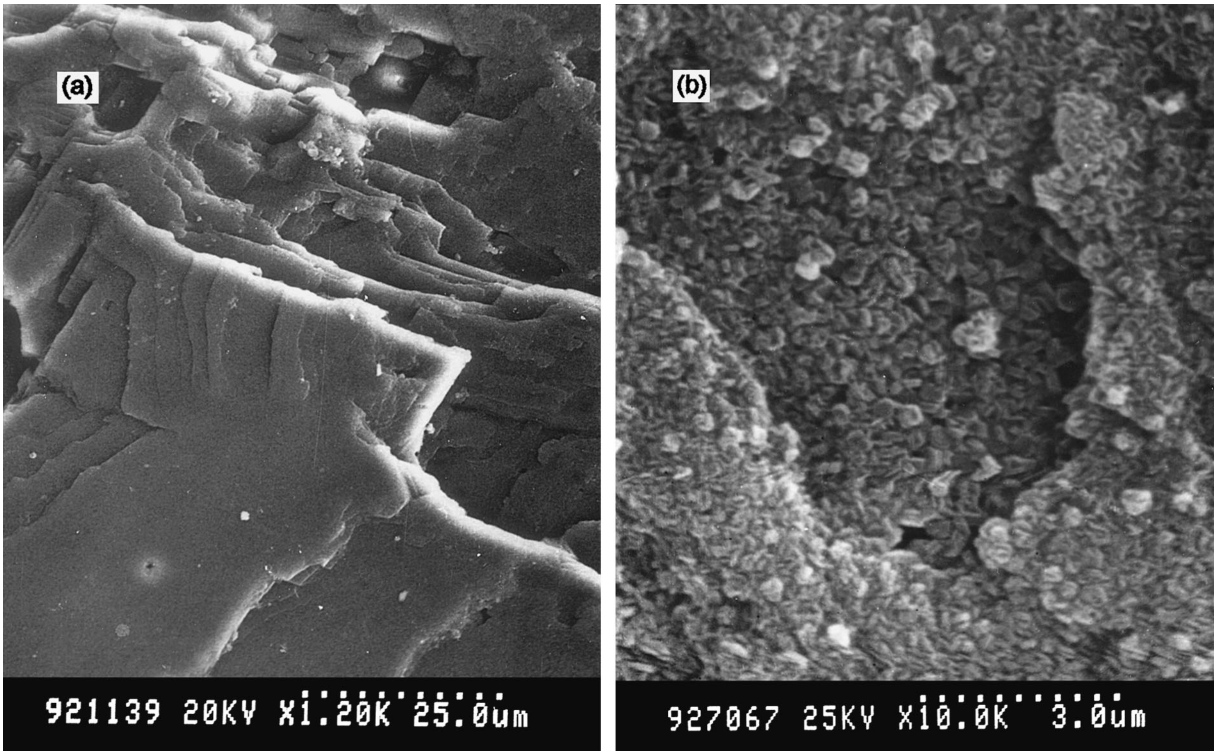

Fig. 3. SEM image of magnesite (LM) broken surface before and after the autoclave curing: (a) the original surface of the magnesite rock; (b) brucite flakes deposited on the surface of magnesite after 10 h autoclaving at 150°C in 10% KOH solution.

S–H is indicated on Fig. 5(b). There were evidences [14] that silica can react with dolomite or brucite in alkaline solution. On the cement paste side, the surface was mainly covered by calcite (as described in Eq. (2)) though its morphology was different from the calcite observed on the dolomite side, as shown in Fig. 5(c).

Similarly, on the magnesite rock side, brucite flakes were produced as in the surface reaction (Fig. 6). On the cement paste side, semi-cubic crystals containing high proportion of calcium were observed as well as a small amount of brucite crystals and possible C–M–S–H product as mentioned above. The interfacial reactions between dolomite or magnesite and cement paste detected by SEM-EDXA agreed with the XRD data (Fig. 7). It was also noted that the interface between the polished rock surface and cement paste as described in Fig. 1 was almost the same as the interface between aggregate and surrounding cement paste in concrete microbars.

Based on the previous autoclave test [11], NM-0 is non-reactive dolomite and LM is a reactive magnesite aggregate. But, microstructural changes were quite the same on the rock surface and on the interface of aggregate and cement paste. Obviously, the different expansion behaviour of these two aggregates in alkaline environment is difficult to explain only from the above study.

### 3.3. Reactions inside aggregate

Microcracks due to ACR were observed often starting inside the aggregate particles and extending into the surrounding cement paste of affected concrete. This indicates that internal reactions, i.e., reactions in the margin of reactive aggregate particles instead of the interfacial reactions as studied above, may be responsible for the expansion of concrete.

After 60 h of autoclaving in 150°C in 10% KOH solution, dolomite NM-0 concrete microbars and rock prisms expanded negligibly. When the concrete microbars and the prisms were broken, reaction rims (10 ~ 20 μm in width) were found at the margin of aggregate particles and the rock prism respectively. SEM observed that dedolomitization occurred within the rims where reaction products were formed (Fig. 8(a)). However, no reaction was observed beyond this rim zone (Fig. 8(b)). XRD analysis confirmed that dedolomitisation only occurred in the rims (Fig. 4(c) and (d)).

Similarly, when magnesite rock prisms were broken after 20 h autoclave, white reaction rims with the width of 100–300 μm were observed under an optical microscope. The SEM morphology within the rim is showed in Fig. 9(a). Platy $Mg(OH)_2$ crystals were formed indicating the occurrence of AMR. However, unlike non-reactive dolomite MN-0, the reaction

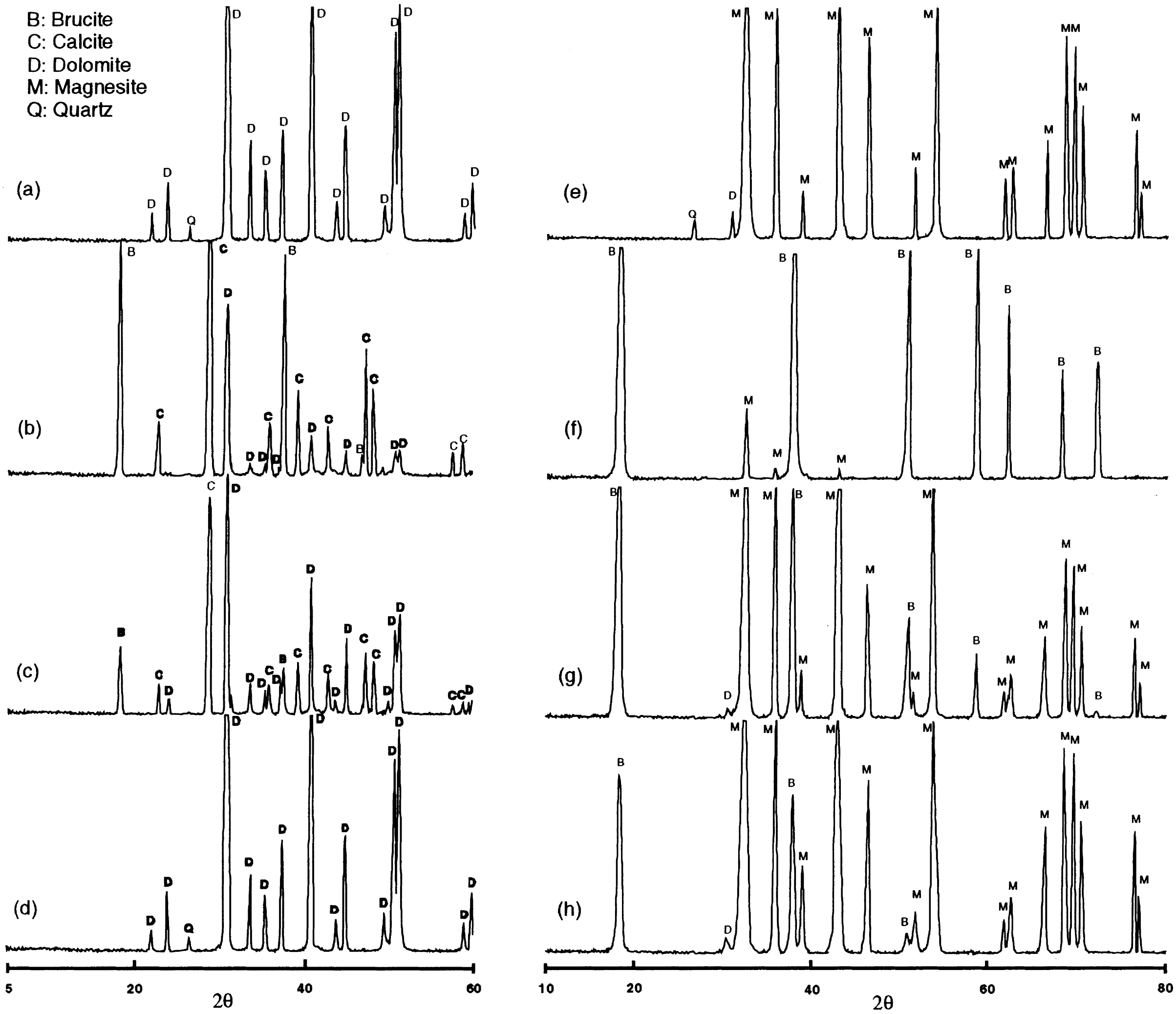

Fig. 4. XRD patterns of dolomite and magnesite rock at different locations before and after autoclaving at 150°C in 10% KOH solution: (a) the unreacted dolomite rock surface; (b) the dolomite surface after autoclaving for 10 h; (c) the reaction rim at the margin of a dolomite prism after autoclaving for 60 h; (d) the central part of the dolomite rock prism after autoclaving for 60 h; (e) the unreacted magnesite rock surface; (f) the magnesite surface after autoclaving for 10 h; (g) the reaction rim at the margin of a magnesite prism after autoclaving for 20 h; (h) the central part of the magnesite rock prism after autoclaving for 20 h.

occurred far beyond the rim zone. At the centre of LM prisms, the reaction product was observed as shown in Fig. 9(b). Brucite crystals, similar to those found near surface, grew along the cleavage planes. XRD data showed that brucite had been produced both in the reaction rims and in the internal region (Fig. 4(g) and (h)).

The difference between non-reactive dolomite (NM-0) and reactive magnesite (LM) is therefore documented. The reaction in NM-0 was restricted to within 20 μm in depth, while for LM, the reaction occurred in much wider reaction rims and alone cleavage cracks because of alkali solution penetrating alone cleavage planes.

### *3.4. Reactions in rock–cement compacted cylinders*

The above results indicated that the location where the reaction occurs is very important for ACR expansion. The limited space in which the reaction products grow could be the key reason of the expansion. If the products grow in a porous interface, it can hardly generate enough expansion force. In the rock–cement compacted cylinders, therefore, a highly compacted interface between rock powders and cement was made, and both the dolomite (NM-0) and magnesite (LM) compacts expand [11].

XRD analysis showed that reactions occurring in the compacts were the same as in the interface between ce-

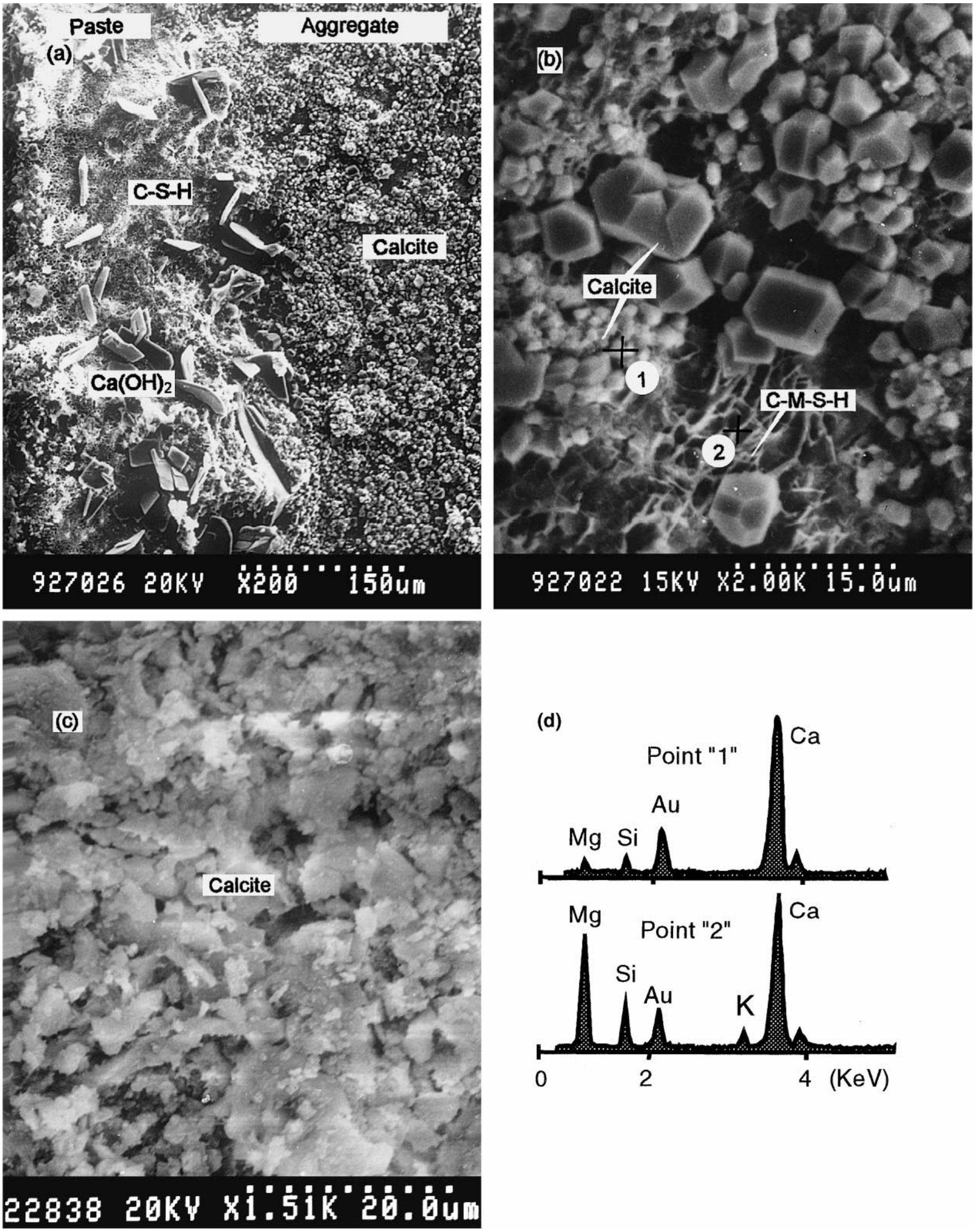

Fig. 5. SEM images of the interface between dolomite and cement paste, and EDXA results: (a) interfacial zone of dolomite and cement paste; (b) the interface between dolomite and cement paste at the dolomite side; (c) the interface between dolomite and cement paste at the cement paste side; (d) the EDXA results of the marked points.

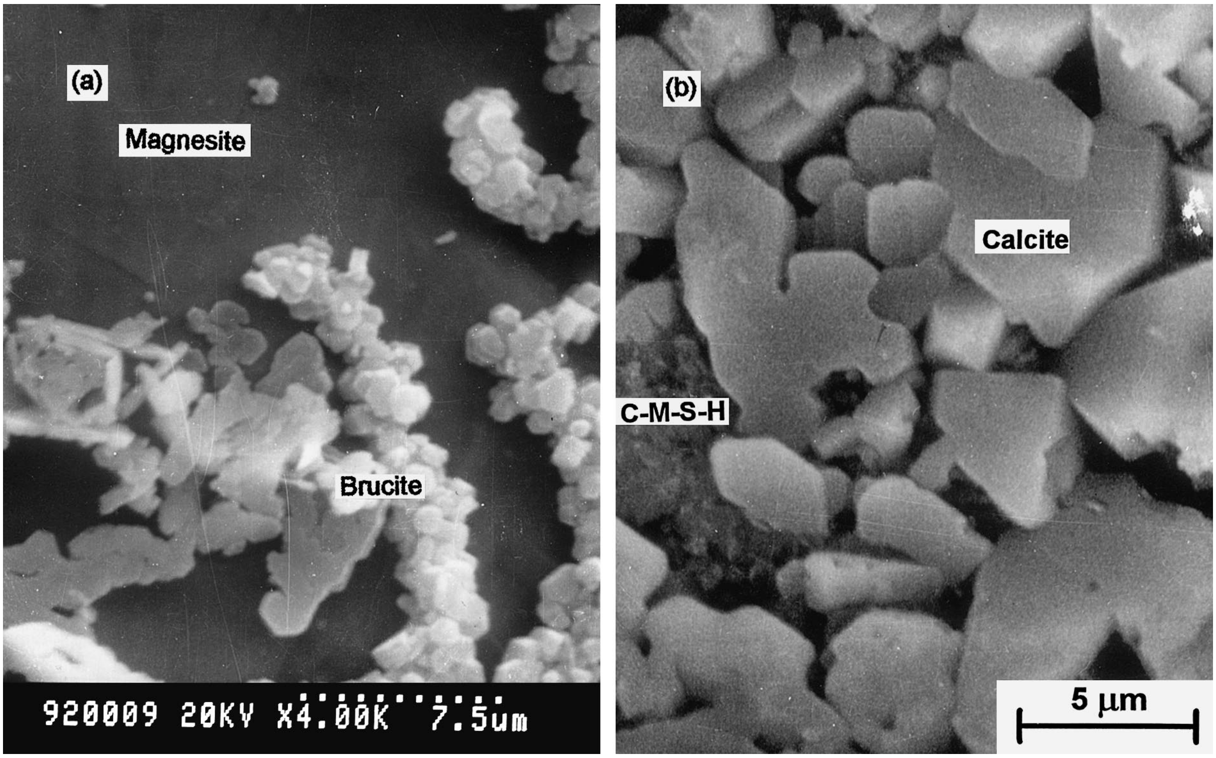

Fig. 6. SEM image of the interface between magnesite and cement paste: (a) the interface between magnesite and cement paste at magnesite the side showing brucite flakes and (b) the interface between magnesite and cement paste at the cement paste side.

ment paste and aggregate (Fig. 7(c) and (f)). SEM-EDXA studies showed that before the NM-0 compacted cylinders were autoclaved at 150°C in 10% KOH solution, small amount of $Ca(OH)_2$ formed in pores (Fig. 10(a) and (b)). After 50 h of autoclaving, the compact became less porous. Pores were filled with calcite and brucite as well as some C–S–H gel (Fig. 10(c)). Extremely fine-grained brucite formed on the surface of dolomite (Fig. 10(d)). The brucite and calcite were much smaller in size than that formed in the interface between dolomite and cement paste in concrete microbars.

Before autoclaving, the magnesite particles in compacts showed almost no sign of attack by alkaline solution after two weeks of curing under 25°C. Similar to dolomite compacts, $Ca(OH)_2$ crystals formed between magnesite particles (Fig. 11(a)). After autoclaving in 150°C in 10% KOH solution for 50 h, calcite and brucite were produced (Fig. 7(f)). The products closely compacted on the surface of magnesite (Fig. 11(c)). Based on XRD and EDXA results (Fig. 11(d)), it was concluded that the flake crystals were brucite and the large ones were calcite, respectively. Because calcite, brucite and magnesite compacted closely, EDXA gave a mixed composition. The outer layer (point "3" on Fig. 11(c)) probably was C–S–H gel mixed with brucite or C–M–S–H product.

Similar to dolomite, the size of the reaction products produced was much smaller in magnesite compacts than in the interface between magnesite and cement paste in concrete microbars. Limited space in compacts could be an important reason.

For corundum compacts (the control group), however, after 50 h of autoclaving, $\alpha$-$Al_2O_3$ particles were not attacked by alkaline solution and no reaction products were found excluding the cement hydration products. The compacts did not expand [11]. This indicated that chemical reactions, as in dolomite and magnesite compacts, were the direct reason for the expansion.

### *3.5. Expansion mechanism*

The expansion mechanism of ADR and AMR is suggested as follows: the expansion of rock prisms, concrete microbars and rock–cement compacts is due to the formation and growth of the solid product or products in a highly confined space.

In dolomite (NM-0) rock prisms and concrete microbars, ADR takes place only on rock surface and in a very thin reaction rim around rock particles. It can neither lead to the dilation of rock particles, nor generate sufficient expansion force in the interface of aggregate and cement paste in concrete microbars.

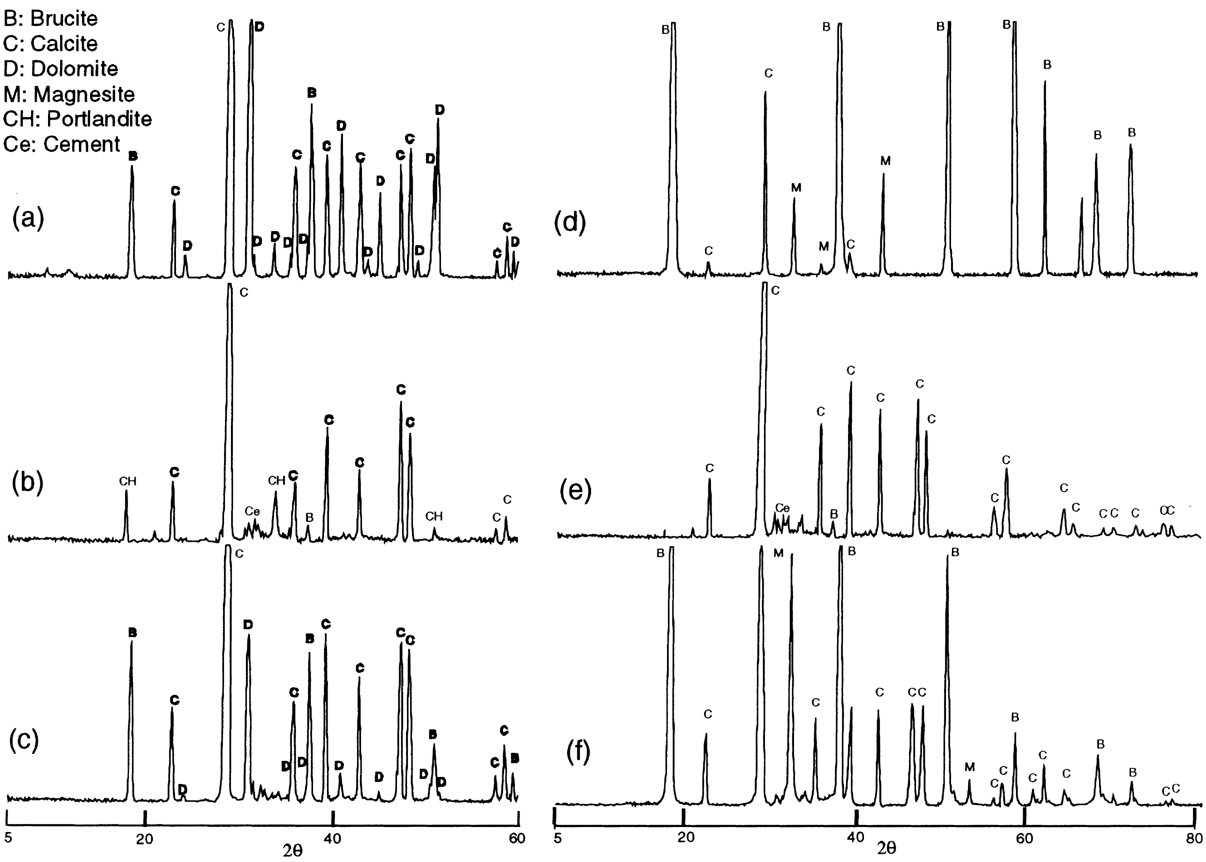

Fig. 7. XRD patterns of the interfacial reaction between dolomite or magnesite and cement paste: (a) the interface of dolomite and cement paste on the rock side after autoclaving for 18 h; (b) the interface of dolomite and cement paste on the cement paste side after autoclaving for 18 h; (c) a dolomite compact after autoclaving for 50 h; (d) the interface of magnesite and cement paste on the magnesite side after autoclaving for 18 h; (e) the interface of magnesite and cement paste on the cement paste side after autoclaving for 18 h; (f) a magnesite compact cylinder after autoclaving for 50 h.

Therefore, no obvious expansion was observed. As in the case of magnesite, however, in addition to the reaction (AMR) occurred on the rock surface, the reaction occurred in much thicker reaction rims and in cleavage planes. Brucite formed in a confined space and generated the force to cause the expansion of rock prisms and aggregate particles in concrete microbars.

In rock–cement compacts, both dolomite and magnesite particles were compacted and cemented together in a way as fine-grained dolomite rhombs in reactive carbonate aggregate. When intensive reactions occurred, the products grew in a confined space and caused the expansion of the compacts. Although the same reactions occurred in the dolomite (NM-0) concrete microbars and in NM-0 compacts, NM-0 concrete microbars did not expand because the reactions were not sufficient enough and the reaction products grew in a porous interface, not in a highly confined space. Furthermore, because more reaction products produced, the finer the dolomite or magnesite particles, the higher expansion observed. Without any reaction between α-$Al_2O_3$ particles and the surrounding pore solution, α-$Al_2O_3$ compacts do not expand [13].

The above expansion mechanism indicate that alkali–dolomite and alkali–magnesite reaction can cause expansion, though both reactions are solid-volume-reducing reaction. Compared with the space occupied by reactants, the reaction products deposited in a larger space together with pores. Without considering the pores in reaction products, the net-volume-change calculation can not be applied for the explanation of ACR expansion. Besides, the expansion mechanism also pointed out the location of the reaction is an important factor for the expansion. Reaction in the interface of aggregates and cement paste usually can not cause expansion because the reaction products grow relatively free. However, if reaction occurs in cleavages, in rock–cement compacts or in the interface between fine-grained reactive particles (e.g., dolomite rhombs) and surrounding matrix as in reactive carbonate aggregate, the formation and growth of reaction products in a confined space can generate sufficient expansion force to cause the whole body expansion.

Another possibility, as pointed out by Carles-Gibergues et al. [9], is that calcite formed around aggregate particles in cement paste can be the reason. However, we

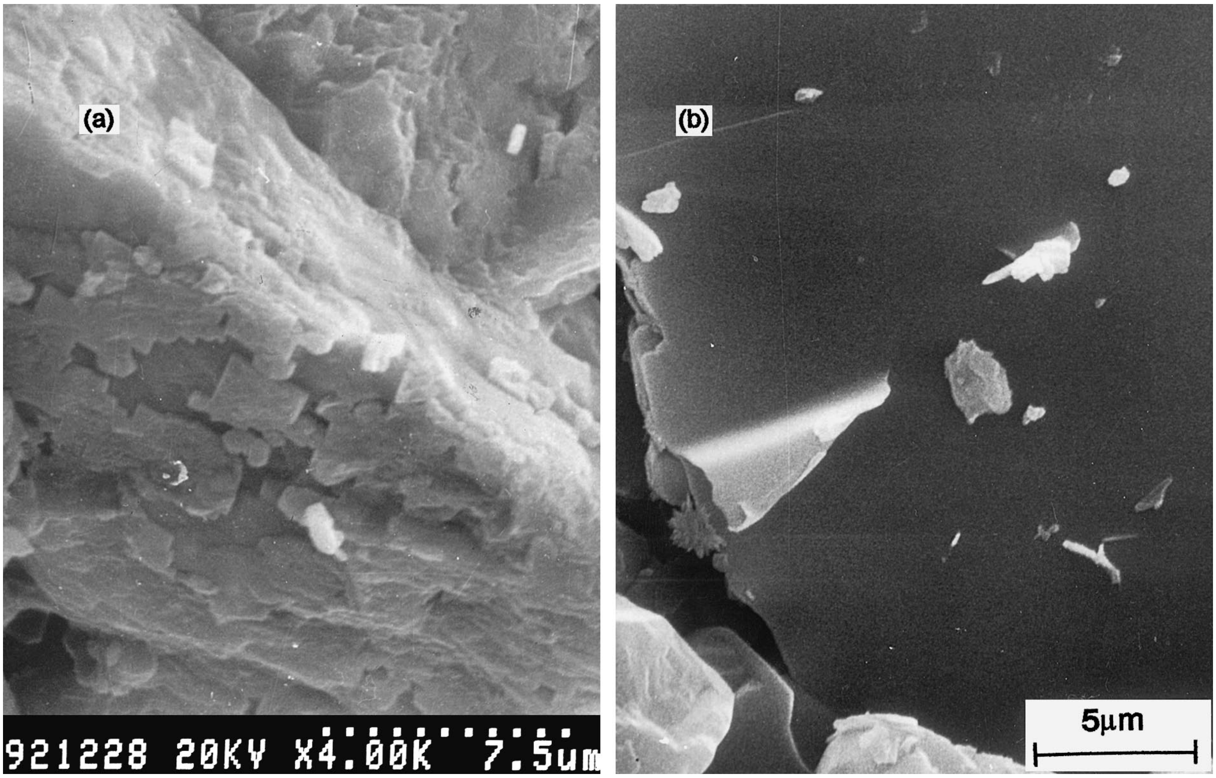

Fig. 8. SEM image of a dolomite rock prism after 60 h of autoclaving at 150°C in 10% KOH solution showing dedolomitization only occurred within the reaction rims: (a) inside a reaction rim and (b) beyond the rim zone.

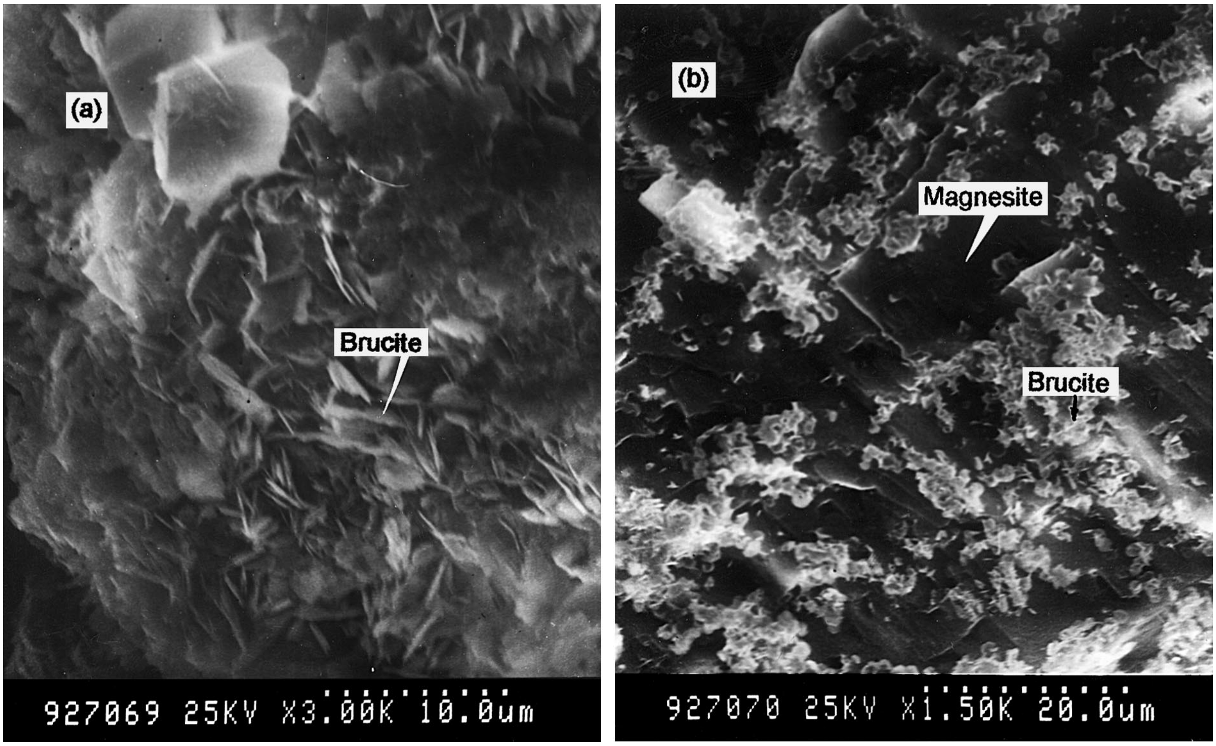

Fig. 9. SEM image of a magnesite rock prism after 20 h of autoclaving at 150°C in 10% KOH solution showing brucite formation area in the central part of the prism: (a) brucite deposits in the reaction rims and (b) brucite occurs between cleavages at the center of the rock prism.

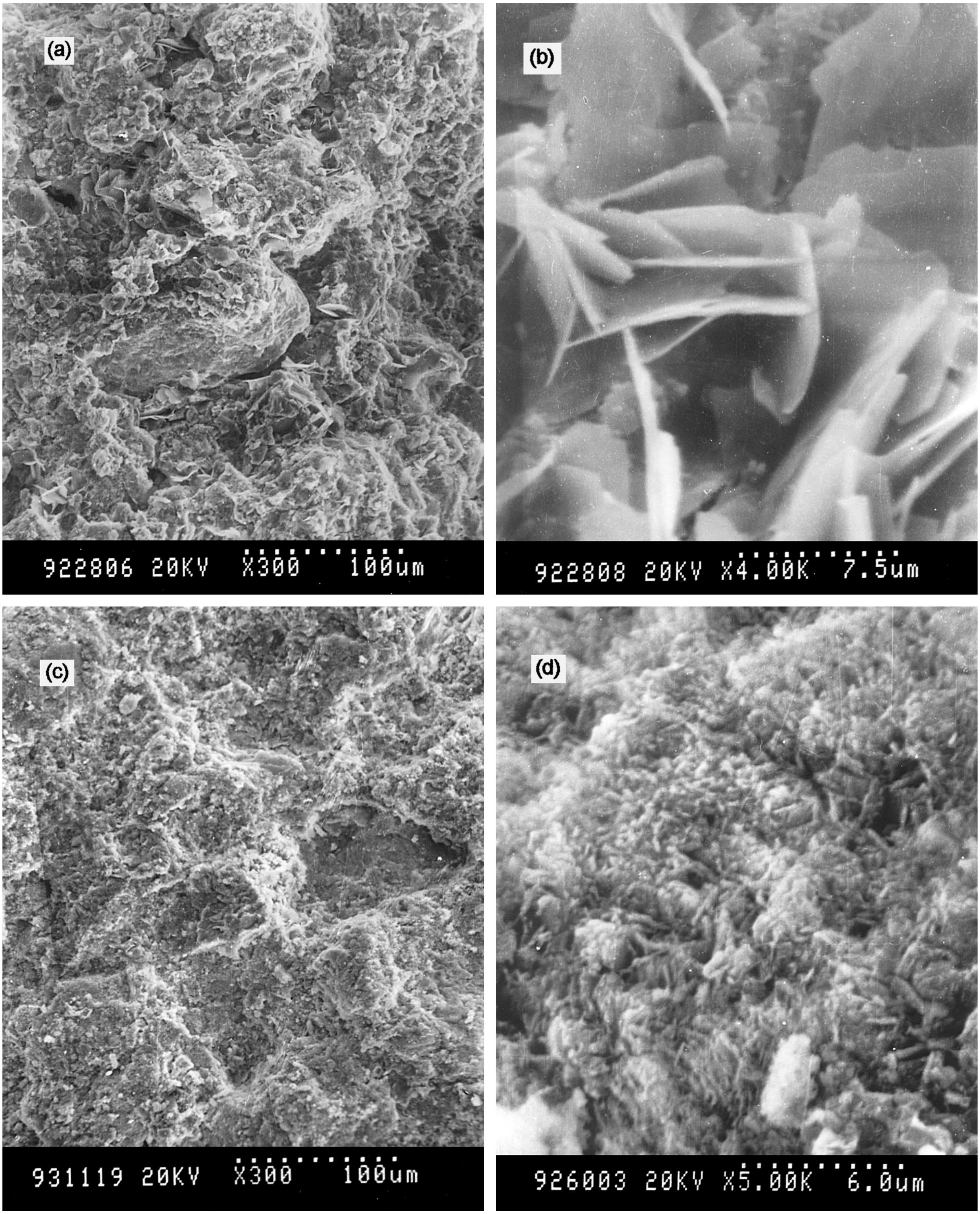

Fig. 10. SEM image of a dolomite–cement compact before and after autoclaving at 150°C in 10% KOH solution for 50 h: (a) fractured surface of a dolomite compact before autoclave; (b) enlargement of the central part of the photo (a); (c) broken surface of dolomite compacts after autoclave; (d) enlargement of the central part of the photo (c).

have not found obvious difference of the outer rims between non-expansive NM-0 concrete microbars and the expansive LM concrete microbars as well as some other reactive aggregates. More work is needed to justify the contribution of reaction in aggregates and in outer rims.

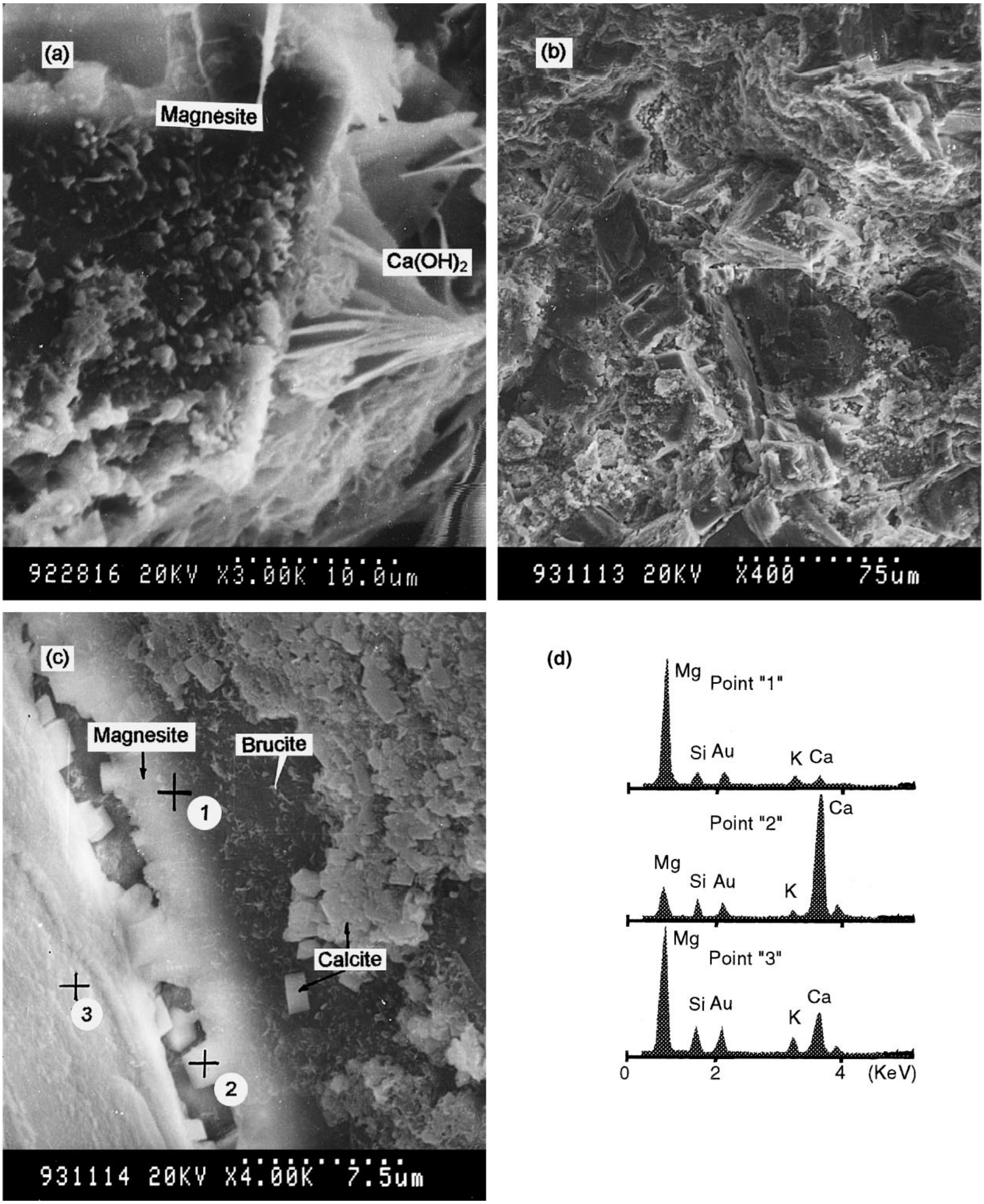

Fig. 11. SEM image of a magnesite compact before and after autoclaving at 150°C in 10% KOH solution for 50 h: (a) $Ca(OH)_2$ flakes in the pores between magnesite particles in a compact before autoclave; (b) microstructure of a magnesite compact after autoclave showing obvious reaction of magnesite particles; (c) enlargement of the central part of the photo (b); (d) EDXA results of the marked points.

Researches on alkali reactivity of carbonate aggregates, which will be reported later, showed that the above mechanism could also explain most of the observations of ACR expansion since the early 50s [14]. One of the interesting facts is that when the smaller the reactive aggregates in concrete, the less expansion observed. Although more reaction may be found in the cement-aggregate interface, the relative percentage of volume expansion of smaller aggregate particles may be higher than that of larger particles, but the absolute expansion can be opposite. Surrounded by the flexible, porous interface, smaller aggregate particles will contribute less expansion to the whole concrete than larger aggregates. If the porosity in the interface is considerably reduced, a reversed grain-size effect results as found in the expansion of rock–cement compacts.

Generally, for some other expansion processes in concrete, a similar expansion mechanism can be expressed as that for a certain reaction product (in solid state), its formation and growth in a confined space can generate an expansion force. Sufficient force will lead to the whole system expand. The microenvironment where a reaction product forms and grows and the characteristics of the product are critical determiners rather than the net solid-volume-increase of the reaction. For free-CaO and MgO hydration and sulphate expansion, there are some evidences show that reaction insitu may be the main reason of the expansion [15,16]. However, it is well documented that the hydration of cement can not cause any expansion of the cement paste. More work is needed to the general expansion mechanism.

## 4. Conclusions

Both dolomite and magnesite can react with alkali solutions to produce crystalline products in 150°C autoclave condition and normal temperatures. The products usually formed and grew insitu. Brucite deposited adjacent to the reacted surface, and the calcite tended to produce in the outer layer.

The expansion of magnesite rock prisms and concrete microbars was caused by the formation and growth of brucite along cleavages and in wide reaction rims. The expansion of dolomite and magnesite compacts is due to the reaction occurred. Based on these results, it is further suggested that generally, the formation and growth of some crystalline reaction products in a confined space can lead to the expansion of a system, even when the reaction is solid-volume-reducing. This mechanism may be the main reason for alkali–carbonate reaction expansion, and valuable for understanding some other expansion processes such as CaO and MgO hydration and ettringite formation in concrete as well.

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