Pergamon

Cement and Concrete Research, Vol. 26, No. 11, pp. 1661-1672, 1996

0008-8846/96 $15.00 + .00

PII S0008-8846(96)00160-3

HOT ALKALI CARBONATION OF SODIUM METAPHOSPHATE MODIFIED FLY ASH/CALCIUM ALUMINATE BLEND HYDROTHERMAL CEMENTS

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(Refereed)
(Received July 2, 1996; in final form September 11, 1996)

ABSTRACT

Sodium metaphosphate-modified fly ash/calcium aluminate blend (SFCB) cements were prepared by autoclaving for 1 day at 300°C and their resistance was evaluated in a highly concentrated Na_2CO_3 solution at 300°C. The hydroxyapatite and analcime phases formed in the autoclaved SFCB cements played an essential role in conferring resistance to the degradation of cements caused by alkali carbonation. Although the carbonating reaction of the analcime phase led to the formation of cancrinite, this analcime cancrinite transformation did not show any influence on the changes in the mechanical and physical properties of the cements. Additionally, there was no formation of the water-soluble calcium bicarbonate in the cements exposed for 28 days. Contrarily, the conventional class G cement systems were very vulnerable to a hot alkali carbonation. The major reason for the damage caused by carbonation of the cements was the fact that the xonotlite phase formed in the 300° autoclaved cements was converted into two carbonation products, calcite and pectolite. Furthermore, the reaction between calcite and carbonic acid derived from Na_2CO_3 led to the formation of water-soluble calcium bicarbonate, thereby causing the alteration of dense structures into porous ones and the loss of strength of cements.

Introduction

One of the criteria needed for formulating geothermal cementitious materials is that these materials must be a resistant to alkali carbonation, especially Na_2CO_3-induced carbonation, at temperatures up to 300°C. In our previous work aimed at developing the alkali carbonation-resistant hydrothermal cements[1,2], we reported that two reactants, carbonic acid (H_2CO_3) and sodium hydroxide (NaOH), produced by the hydrolysis of Na_2CO_3 in hot water, had a strong chemical affinity for the $CaO-SiO_2-H_2O$ and $CaO-Al_2O_3-SiO_2-H_2O$ cement systems. The Na-catalyzed carbonation of these systems led to the formation of calcite ($CaCO_3$) which was converted into water-soluble calcium bicarbonate [$Ca(CaCO_3)_2$] by the following reaction: $CaCO_3 + H_2CO_3 \rightarrow Ca(CaCO_3)_2$. Furthermore, the leaching of such a water-soluble salt from the carbonated cement bodies caused the development of a cavernous and porous microstructure, thereby lowering their mechanical strength.

To overcome the deficiencies of conventional cement systems, our emphasis was centered on hydrothermal synthesis and the development of three cement systems, which minimized the rate of carbonation: $CaO-P_2O_5-H_2O$[3-5], $Al_2O_3-P_2O_5-H_2O$[6], and $Na_2O-Al_2O_3-SiO_2-H_2O$[7]. The first cement system, calcium phosphate cement (CPC), was prepared by the acid-base reactions between sodium metaphosphate as an acid liquid reactant and calcium aluminate cement as a base powder reactant, followed by the hydrothermal treatments at temperatures up to 300°C. The phase composition of CPCs produced by two-step reaction routes, acid-base and hydrothermal reactions consisted of hydroxyapatite (HOAp) as the major phase and boehmite as the minor one. Although some $CaCO_3$ was formed by the carbonation of unreacted calcium aluminate cements after exposing them for 120 days to Na_2CO_3-laden water at 250°C, the loss of strength for the exposed CPC specimens depended on the degree of crystallinity of the HOAp phase growing in the amorphous phases, but was independent of the rate of carbonation. The second system ($Al_2O_3-P_2O_5-H_2O$) defined as the aluminum phosphate cement (APC) was formed by the hydrothermal-catalyzed acid-base reaction between alumina as a base reactant and ammonium phosphate as an acid reactant. Of course, this system in the absence of CaO did not form any $CaCO_3$ after a long-term exposure in a hot Na_2CO_3 solution. Unfortunately, the 300°C-autoclaved APCs consisting of $Al_2PO_4(OH)_3$, boehmite, and $AlPO_4$ as the major crystalline phases had a very low strength, compared with those of CPC specimens. The hydrothermal treatments of $Na_2O-Al_2O_3-SiO_2-H_2O$ cement systems introduced the zeolite and boehmite phases which were responsible for strengthening neat cement pastes into the cement bodies. However, no attempt was made to assess the susceptibility of zeolite phase to alkali carbonation.

As part of our study on alkali carbonation-resistant cements, the current work was aimed at identifying the phase compositions presence in the cements produced by hydrothermal reaction between the calcium aluminate-blended class F fly ash as solid reactant and the sodium metaphospahte solution reactant, and to determine their mechanical properties[8]. As a result, the combination of the analcime in a zeolitic compound and HOAp contributed significantly to strengthening and densifying the specimens. This interesting information urged us to further investigate the characteristics of this cement for use in the alkali carbonation-resistant geothermal cementitious material systems. The factors to be investigated were as follows: (1) the thickening properties of fast- and slow-setting neat cement slurries at an isothermal temperature of 50°C; (2) the rate of $CaCO_3$-related carbonation as a function of exposure time, in a highly concentrated Na_2CO_3-laden water at 300°C; (3) the identification of phase formed in carbonated cements; and (4) the changes in compressive strength and porosity of the specimens after exposure. An extremely high Na_2CO_3 concentration of 4.0 wt% was used to accelerate the rate of carbonating reaction with the cements. For the purpose of comparison with this blend cement, the conventional American Petroleum Institute (API) class G cement, which is commonly used in the geothermal wells, was included in this study.

Experimental

Materials. Class F fly ash with a Blaine fineness of 10,585 cm^2/g was supplied by Pozament Corp; a typical analysis made in accordance with Spec. ASTM 618, showed 38.57 wt% silica, 38.57 wt% aluminum oxide, 11.95 wt% iron oxide, 1.36 wt% magnesium oxide, 1.94 wt% sulfur trioxide, 1.52 wt% sodium oxide, 0.47 wt% calcium oxide, and 1.94 wt% potassium oxide, with a 3.68 wt% loss on ignition at 816°C. The x-ray powder diffraction (XRD) data showed that the crystalline components of fly ash consist of two major phases, mullite and quartz. Commercial calcium aluminate cement (CAC, Refcon) obtained from Lehigh Cement Company, was used to blend with the fly ash. The chemical constituents of this CAC are as follows; 57.4 wt% Al_2O_3, 1.2 wt% Fe_2O_3, 34.2 wt% CaO, 5.7 wt% SiO_2, 0.36 wt% SO_3, and 1.14 wt% other. CAC had two major phases, gehlenite ($2CaO \cdot Al_2O_3 \cdot SiO_2$, C_2AS), and monocalcium aluminate ($CaO \cdot Al_2O_3$, CA). A 40 wt% sodium metaphosphate [$-(-NaPO_3-)-_n$, SMP] supplied by the Aldrich Chemical Company, Inc., was employed as the cement-forming aqueous reactant to modify the CAC-blended fly ash solid reactants. In this study, the composition of the solid reactants made by Twin Shell Dry Blender had the ratio of fly ash/CAC of 60/40 by weight. On the other hand, three ingredients, class G cement from Unocal Corporation, silica flour (SiO_2) having a particle size of < 60 µm, and water were used in preparing the class G cement systems as a comparative material.

Cement slurries were prepared by handmixing 60 wt% CAC-blended fly ash, and 40 wt% $-(-NaPO_3-)-_n$ solution for the SMP-modified blending cement systems, and 45 wt% class G cement, 20 wt% SiO_2, and 35 wt% water for the class G cement systems at room temperature. These cement slurries were then cast into cylindrical molds (30 mm diam × 60 mm long) and allowed them to harden for 24 hrs at 25°C in air. These aged specimens were preautoclaved for 24 hrs in plain water without the Na_2CO_3 at 300°C, to prepare the control specimens, and subsequently exposed for up to 28 days in a 4 wt% Na_2CO_3-laden water at a temperature of 300°C. The specimens were then examined to determine the rate of carbonation and to identify the crystalline phase composition and transition of the carbonated cements. All data were correlated directly with the compressive strength and porosity of the specimens.

Measurements. The consistencies of the slurries were measured in accordance with API schedule 4G Specification. This schedule corresponds to a simulated 1830 m deep well at 50°C under a pressure of 26.9 MPa. The test was completed when the consistency (Bc value) of the slurry exceeded 70. Quantitative data on the amount of $CaCO_3$ formed in the cements were obtained from thermogravimetric analysis (TGA), by the weight loss at which thermal decomposition of $CaCO_3$ occurs over the range 600 to 700°C. This information was supported by investigating the changes in intensity of the band near 1430 cm^{-1} , corresponding to the carbonates (CO_3^{2-}) obtained from the Foruier Transform Infrared Spectroscopy (FT-IR) analysis. XRD was used to identify the phases formed in these hydrothermal cement pastes, and to gain information on the transformation of phases caused by carbonation after exposure in a 4 wt% Na_2CO_3 solution at 300°C. Compressive strength tests were performed on neat cement specimens having a diameter of 30 mm and a length of 60 mm; the result given is the average value of three specimens. The porosity of carbonated cement pastes was determined by helium comparison pyconometry.

Results and Discussion

When cementitious materials were used to complete geothermal wells, their slurries were required to maintain pumpability for at least 3 hours at a temperature of 50°C. Thus, it was indispensable to gain data on the thickening time of cement slurries. Figure 1 shows the comparison between the consistency-thickening time relations of the class G cement and the SMP-modified fly ash/CAC blend (SFCB) cement slurries at 50°C. As seen, the feature of the curve for the SFCB cement slurry is characterized by a rapid rise in consistency after ≈

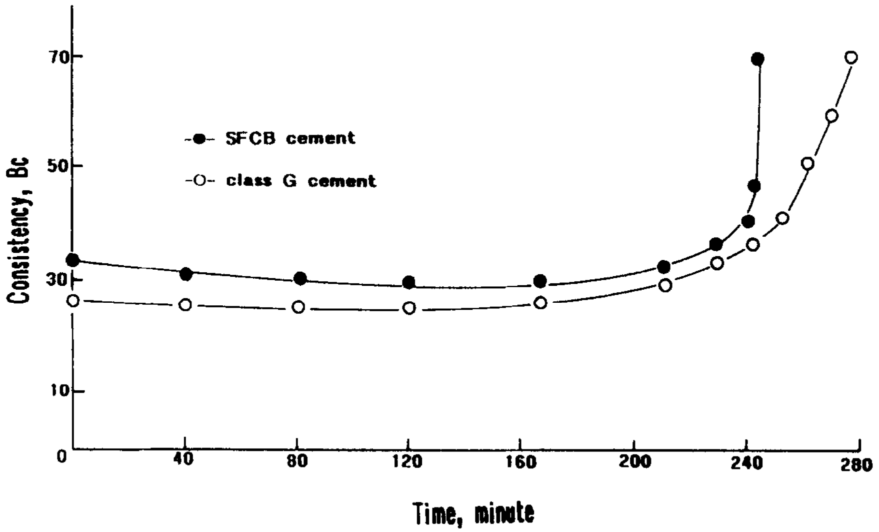

FIG. 1.

Changes in consistency of SFCB and class G cement slurries as a function of time at isothermal temperature of 50°C.

230 min. Since the thickening time is recorded as the elapsed time before the consistency reached to a Bc value of 70, this slurry corresponded to a thickening time of ≈ 240 min which fully satisfies the criterion of pumpability for 180 min. In contrast, the class G cement showed that although the curve's feature in an elapsed time of ≈ 230 min was almost the same as that of the SFCB cement, beyond this time, the consistency gradually rises with increasing time. As a result, the thickening time of this slurry was ≈ 280 min, corresponding to the extension of ≈ 40 min, compared with that of SFCB cement slurries.

Table 1 gives the changes in compressive strength and porosity of these cements after exposure for up to 28 days in a 4 wt% Na_2CO_3-laden water at 300°C. The SFCB control specimens had a compressive strength of 34.86 MPa and porosity of 29.59 %. Although the class G control specimens had a porosity of > 40 %, their strength was ≈ 10 % higher than that of SFCB controls. The data for the carbonated SFCB specimens show that their porosity

TABLE 1

Changes in Porosity and Compressive Strength for SFCB and Class G Cements as a Function of Exposure Time in 4 % Na_2CO_3-Laden Water at 300°C

Cement	Exposure time, day	Porosity, %	Compressive strength, MPa
SFCB	0	29.59	34.86
SFCB	7	29.13	35.28
SFCB	14	27.71	35.35
SFCB	28	23.43	35.67
Class G	0	43.51	40.03
Class G	7	42.63	40.10
Class G	14	39.04	39.96
Class G	28	42.01	32.87

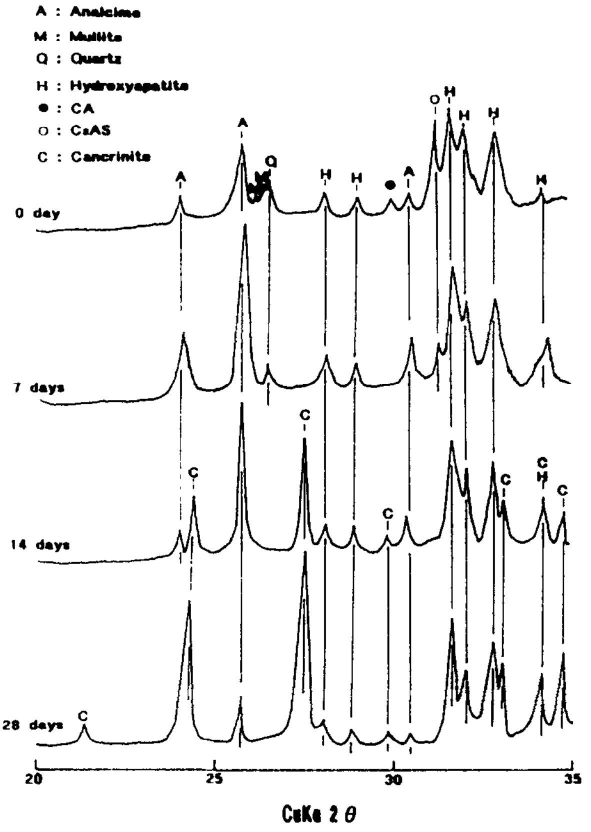

FIG. 2.

XRD patterns for SFCB cements before and after exposure for 7, 14, and 28 days in 4 % Na_2CO_3 solution at 300°C.

tends to decrease with increasing time of exposure, suggesting that a long-term exposed specimen becomes a dense structure; correspondingly, there is an increase in the strength of specimens. Thus, the strength of 35.67 MPa developed after exposure for 28 days was 0.81 MPa higher than that of the control. As a result, SFCB cements might be expected to display a great resistance in a concentrated Na_2CO_3 solution at 300°C. On the other hand, the porosity of the class G cements decreased in the first 14 day exposures, but an prolonged exposure to 28 days caused the increasing porosity. The increase in porosity directly reflected the loss of compressive strength of the specimens. Thus, class G cements seem to be vulnerable to the attack of a hot Na_2CO_3 solution.

To better understand and rationalize these findings, our attention was focused on identifying the phase composition of the carbonation products in the carbonated cement bodies by XRD. An XRD trace, ranging from 0.256 to 0.444 nm, was made for both the 300°C/1-day-autoclaved SFCB and class G cement pastes after exposing for 7, 14, and 28 days in a hot Na_2CO_3 solution. For the SFCB cements (Figure 2), the diffraction pattern of the control (0 day) reveals that although the four unreacted components, quartz and mullite belonging to the fly ash, and C_2AS and CA for CAC, remain in the cement body, the two crystalline reaction products, analcime ($NaAlSi_2O_6 \cdot H_2O$, AN) and hydroxyapatite [$Ca_5(PO_4)_3(OH)$, HOAp],

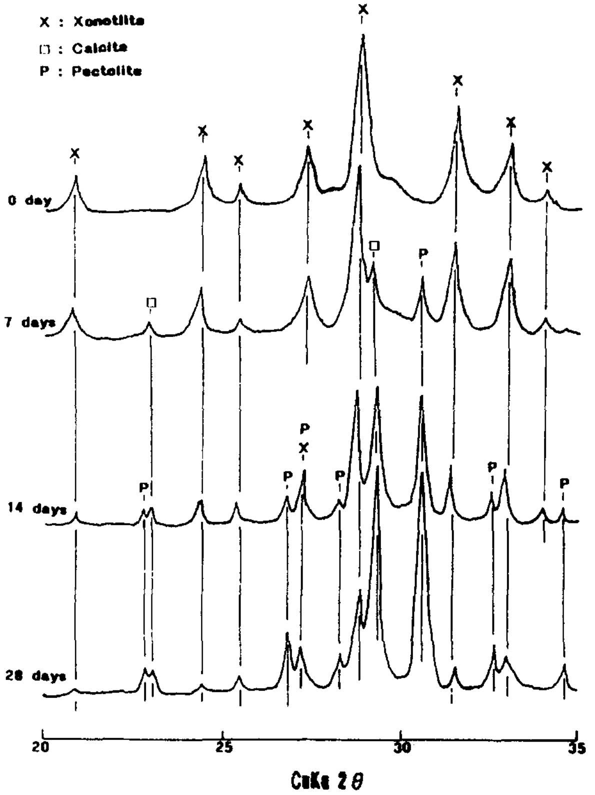

FIG. 3.
XRD tracings of 0, 7, 14, and 28-day-carbonated class G cements.

are formed by a hydrothermal reaction between the SMP and fly ash/CAC blend reactants. As described in our previous paper[8], we assume that HOAp phase was perhaps precipitated by an interaction between the Ca ions liberated from hydrolysis of CA and C_2AS of CAC, and the phosphate ions from the SMP; meanwhile the Na as a counter-ion of phosphate anion reacted with the Ca-depleted CAC and fly ash reactants to form AN. When this sample was exposed to Na_2CO_3 solution for 7 days, the XRD pattern in comparison to that of control changed in the following ways: an increase in the line intensities of AN and HOAp; a considerable reduction in those of quartz- and C_2AS-related peaks; and a disappearance of the mullite and CA phases. These results suggest that exposure for 7 days promotes the rates of reaction of SMP with fly ash and CAC to yield a well-formed AN and HOAp. There were striking changes in the diffraction pattern in the 14-day-carbonated samples, in which new spacings had developed, and the AN-related line intensities had decreased, while intensive signals of HOAp remained, and also the fly ash and CAC reactants-related x-ray lines had vanished. The new lines reveal the formation of the cancrinite [$Na_8(Al\ Si)_{12}O_{24}CO_3 \cdot 3H_2O$, CAN] phase. Relating the formation of this new phase to a declining line intensity of AN, the AN phase seems to be susceptible to a reaction with the Na_2CO_3 solution, thereby resulting in the AN → CAN phase transformation. Increasing the exposure time to 28 days led to enhancing the extent of such a phase transformation. In fact, the XRD pattern of this

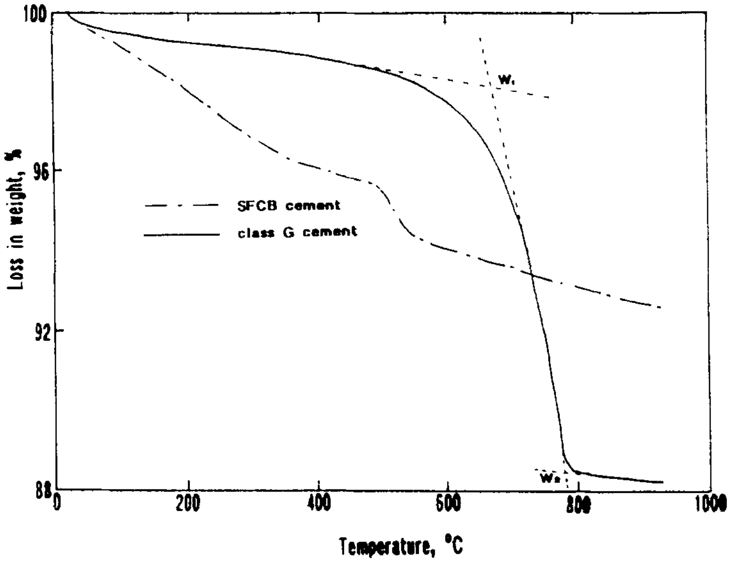

FIG. 4.

Comparison of TGA curve's features for carbonated class G and SFCB cements in temperature range 25 to 950°C.

sample indicated two peculiar features; one referred to a conspicuous growth of CAN peaks and the other was a striking decrease in the signal related to AN. There were no x-ray lines from the $CaCO_3$-related carbonation products. Also, the pattern indicated the presence of a strong signal of HOAp. Thus, the phase composition of the 28-day-carbonated SFCB cements consists of CAN and HOAp as the major crystalline components and AN as minor one. Relating this finding to the results of compressive strength, we believe that although AN undergoes the Na_2CO_3-induced carbonation, CAN phase formed by this carbonation has no influence on lowering the strength of the cements.

Figure 3 depicts the XRD traces for the unexposed and exposed class G cement samples. As seen, the 300°C-autoclaved control samples produced xonotolite [$Ca_6Si_6O_{17}(OH)_2$, XO] as a single phase. No major reactants such as C_3S phase in class G cements and quartz phase in a silica flour additive, were identified from this pattern, implying that the hydrothermal reaction between these reactants to form XO was completed after autoclaving for 1 day in pure water at 300°C. By comparison, an interesting pattern emerged from the 7-day-carbonated samples; two additional phases, calcite ($CaCO_3$) and pectolite ($NaCa_3HSi_3O_9$, PEC), were also detected. The identification of these newly developed phases seems to verify that XO phase is reactive with the Na_2CO_3. This information was strongly supported by the pattern of the 14-day-carbonated samples; the calcite- and PEC-related peaks markedly intensified, while a pronounced decrease in intensity of XO signals was seen. Because the hydrolysis of Na_2CO_3 liberates carbonic acid (H_2CO_3) and sodium hydroxide (NaOH), these reactants react with XO to form calcite and PEC. This carbonation reaction may proceed as follows: $Ca_6Si_6O_{17}(OH)_2 + 2NaOH + H_2CO_3 \rightarrow 2NaCa_3HSi_3O_9 + CaCO_3 + Ca(OH)_2 + H_2O$. Moreover, the calcite formed may be converted into a water-soluble calcium bicarbonate [$Ca(CaCO_3)_2$] by the following reaction: $CaCO_3 + H_2CO_3 \rightarrow Ca(CaCO_3)_2$. In addition, considering the reactivity of $Ca(OH)_2$ with H_2CO_3, it is possible to assume that the acid-base reaction between them leads to the formation of $CaCO_3$, according to the following

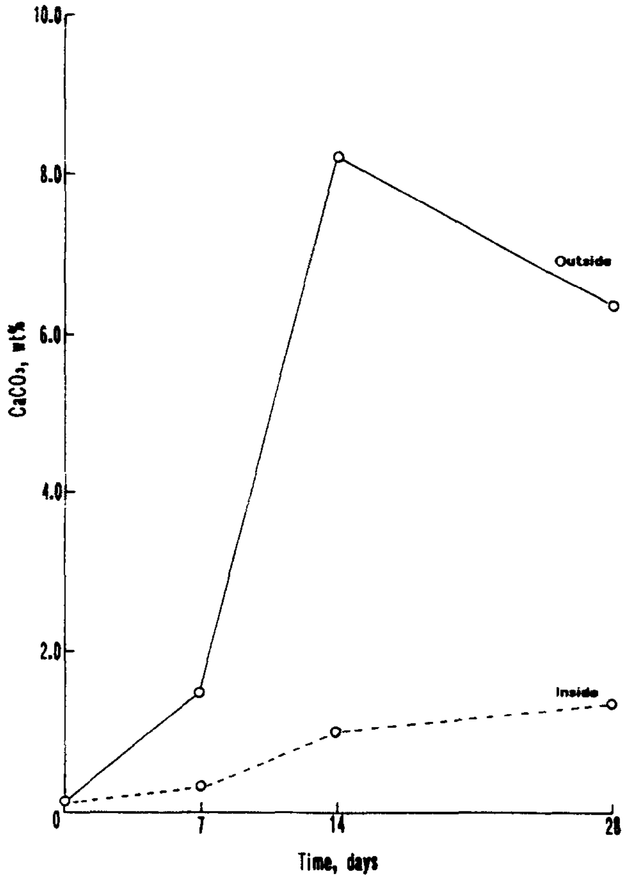

FIG. 5.

Concentrations of $CaCO_3$ formed on the outside and in the inside of class G cements after exposure for 0, 7, 14, and 28 days to 4 % Na_2CO_3-laden water at 300°C.

formula: $Ca(OH)2 + H_2CO_3 \rightarrow CaCO_3 + 2H_2O$. Again, when $CaCO_3$ came in contact with H_2CO_3, it is conceivable that the $CaCO_3 \rightarrow Ca(CaCO_3)_2$ phase conversion took place. If this interpretation is correct, then such alkali carbonating reactions repeatedly occur within XO until its conversion into $CaCO_3$ and PEC is completed. In fact, extending the carbonation time to 28 days revealed that the x-ray lines of these conversion products became dominant in XRD pattern, meanwhile a further reduction of XO line intensities had occurred.

To substantiate further the information on alkali carbonation described above, we reexamined the same samples used in the XRD analysis by the combined techniques of TGA and FT-IR, which gave us a quantitative estimation of the $CaCO_3$-related carbonation rates. Using the TGA, the amount of $CaCO_3$ was measured by the loss in weight at which the thermal decomposition of $CaCO_3$ occurred over the range 600 to 770°C. Figure 4 shows a typical relationship of weight loss-temperature for both the 14-day-carbonated SFCB and class G cement samples. From the TGA curve of class G cements, estimation of $CaCO_3$ was achieved from the difference between the losses in weight at the onset (W_1) and end (W_2) of the curve generated by the thermal decomposition of $CaCO_3$. These W_1 and W_2 values were

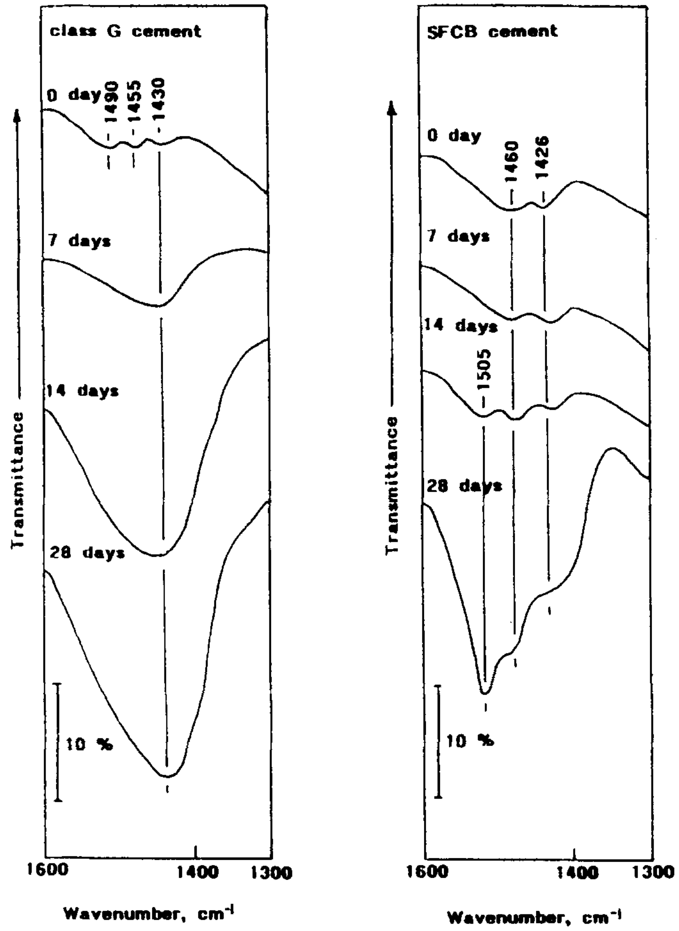

FIG. 6.

FT-IR spectra over the frequency range 1600 to 1300 cm^{-1} for class G and SFCB cements before and after exposure for 7, 14, and 28 days in Na_2CO_3 solution at 300°C.

obtained from the curve by finding the intersection point of two linear extrapolations. By comparison, there was no clear identity of $CaCO_3$-related decomposition in the curve for the SFCB cements, implying that the amount of $CaCO_3$ formed in the cements after carbonation for 14 days might be very small or negligible. The TGA curve of the 28-day-carbonated SFCB cements (not shown) closely resembled that of the 14-day ones. These findings agreed with the results from XRD study. Thus, no attempt for the SFCB cements was made to further investigate the rate of $CaCO_3$-related carbonation.

Based upon this information, we determined the amount of $CaCO_3$ for only the class G cement samples. In this determination, the samples were taken from two different locations of the specimens; one was at a depth of ≈ 3 mm from the surfaces of cylindrical specimens with 30-mm diam. × 60-mm size and the other was the midpoint sections located at a depth of ≈ 15 mm from the surfaces. The former and latter samples were defined as the outside and inside, respectively. Figure 5 depicts the changes up to 28 days in the amount of $CaCO_3$ at

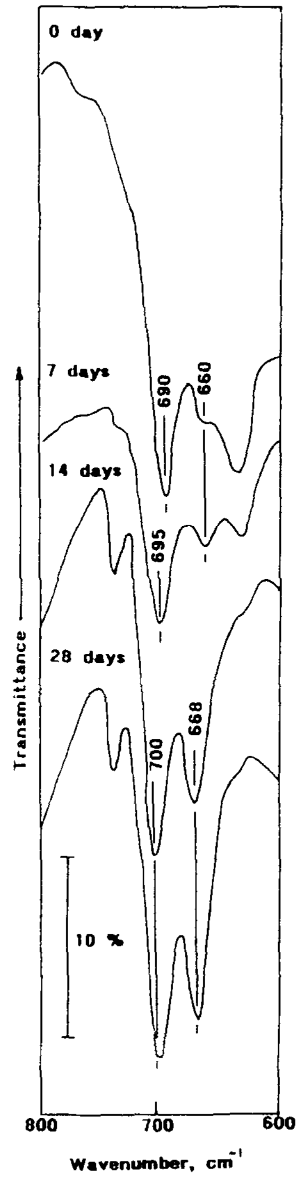

FIG. 7.
FT-IR spectra in the range 800 to 600 cm^{-1} for 0, 7, 14, and 28-day-carbonated class G cements.

both the locations as a function of time. A small amount of $CaCO_3$, ranging from 0.1 to 0.2 wt%, had already been formed in the untreated control specimens. As expected, the extent of carbonation for the outside samples increasingly rose in the first 14 days of exposure, and then declined to ≈ 6.4 wt% after 28 days. The reason for this decrease may be due to the conversion of $CaCO_3$ into the $Ca(CaCO_3)_2$ salt which is very susceptible to hydrolysis. The data also indicated that the inside samples had a considerably lower amount of $CaCO_3$ than those of the outside samples.

Figure 6 compares the spectral features of the samples taken from the outside of 0, 7, 14, and 28-day-carbonated class G and SFCB cements. The spectrum (left in Fig. 6) for the control class G cements indicated the presence of three weak bands at 1490, 1455, and 1430 cm^{-1}. The band at 1430 cm^{-1} reveals the stretching vibration of CO_3 in the calcite[9]. The remaining two bands may be due to the carbonates different from calcite. After carbonation for 7 days, the band at 1430 cm^{-1} became a principal peak. The intensity of this peak increased with the time of carbonation, suggesting that the in-situ XO $\rightarrow$ $CaCO_3$ phase conversion in class G cements progressively occurred as a function of time. For the SFCB cements, the spectrum of the control had two bands at 1460 and 1426 cm^{-1}. Assuming that the latter peak originates from $CaCO_3$, some $CaCO_3$ is present in the 300°C-autoclaved control samples. The spectral feature similar to that of control can be seen in the 7-day-carbonated samples. Extending the carbonation to 14 days produced an additional band at 1505 cm^{-1} in the spectrum. In our previous study of the carbonation of HOAp[1], we reported that the bands near 1460 and 1500 cm^{-1} were due to the formation of CO_3-incorporated HOAp structure generated by substituting the CO_3^{2-} for OH^-. Since CAN phase formed by the carbonation of AN contains the CO_3^{2-} in its structure, it is also possible to assume that these bands may belong to the CAN. The spectrum of the 28-day-carbonated samples was characterized by a striking growth of the bands at 1505 and 1460 cm^{-1}, especially the former one; meanwhile the band at 1426 cm^{-1}, relating to $CaCO_3$, changed in the shoulder peak. Thus, the amount of $CaCO_3$ formed in the SFCB cements after 28 days is very small. However, there is no evidence as to whether the growing peaks at 1505 and 1460 cm^{-1} were due to an increase in the rate of AN $\rightarrow$ CAN phase transformation or the formation of CO_3-incorporated HOAp, or both.

To obtain information on the water-soluble $Ca(CaCO_3)_2$ phase, we inspected the FT-IR absorption bands in a particular frequency range 800 to 600 cm^{-1}, for the carbonated class G and SFCB cements. As is well known[10], the bicarbonates can be identified by the pair of bands at $\approx$ 700 cm^{-1} as a strong absorption and at $\approx$ 665 cm^{-1} as a weak one. The IR spectra from the class G cements after carbonating for 0, 7, 14, and 28 days are given in Figure 7. The spectrum of control samples seems to demonstrate that, even though they were not exposed to Na_2CO_3, some calcium bicarbonate is present in the 300°C-autoclaved cements because of the presence of a strong band at 690 cm^{-1} and shoulder band at 660 cm^{-1}. When this sample was exposed for 7 days in Na_2CO_3, the band at 690 cm^{-1} appreciably shifted to a high wavenumber at 695 cm^{-1}, while the shoulder band at 660 cm^{-1} becomes a prominent peak. Further shift in the wavenumbers of these bands from 695 to 700 cm^{-1} and 660 to 668 cm^{-1} occurred in the 14-day-carbonated samples. Also, the spectrum showed that the peak intensities of these bands were much stronger than those of the 7 day samples, seeming to suggest that more $Ca(CaCO_3)_2$ salt was produced in a carbonating period of 14 days. Increasing the exposure time to 28 days led to the further intensification of these bands, thereby indicating a large amount of $Ca(CaCO_3)_2$ salt in the cements. The data seem to account for the increasing rate of in-situ $CaCO_3$ $\rightarrow$ $Ca(CaCO_3)_2$ phase conversion as a function of time. Relating this finding to the porosity and compressive strength of the specimens, a high rate of such a phase conversion appears to be one of the major factors for altering the dense structures into porous ones and weakening the strength of cements. No salt compound like $Ca(CaCO_3)_2$ was identified in any of the carbonated SFCB cements. Thus, we believe that SFCB cements have a high potential for use as alkali carbonation-resistant hydrothermal cementitious materials in the geothermal wells at temperatures up to 300°C.

Conclusion

Sodium metaphosphate-modified fly ash/calcium aluminate blend (SFCB) hydrothermal cements displayed a great resistance in a highly concentrated Na_2CO_3 solution at 300°C. The major reason for the alkali carbonation resistance of SFCB cements was due to the crystalline phase composition consisting of hydroxyapatite (HOAp) and analcime (AN) formed hydrothermally in the cement bodies. Although the AN phase was susceptible to a reaction with the Na_2CO_3 to convert into the cancrinite (CAN) phase, such in-situ phase conversion did not show any influence upon the degradation of cements caused by alkali carbonation, which led directly to the increase in porosity and loss of strength of the specimens. The 28-day-carbonated SFCB cements included two principal phases, HOAp and CAN, and also AN as a minor one.

In contrast, the xonotlite (XO) phase formed hydrothermally in the conventional class G cement systems at 300°C reacted with Na_2CO_3 to produce three major carbonation products; calcite and pectolite (PEC) as crystalline phases, and calcium bicarbonate phase. The rate of this carbonating reaction increased with exposure times, resulting in the well-formed calcite, PEC, and calcium bicarbonate phases in the bodies exposed for 28 days. There was no information on whether in-situ XO → PEC phase transformation caused the changes in the mechanical and physical properties of cements.

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