Energy, Mines and
Resources Canada

Énergie, Mines et
Ressources Canada

CANMET
Canada Centre
for Mineral
and Energy
Technology

Centre canadien
de la technologie
des minéraux
et de l'énergie

EFFECT OF ELEVATED TEMPERATURES ON COMPRESSIVE STRENGTH, PULSE VELOCITY AND CONVERSION OF HIGH ALUMINA CEMENT CONCRETE

D.H.H. Quon and V.M. Malhotra

MAY 1977

MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
CANMET REPORT 77-49

Available by mail from:

Printing and Publishing
Supply and Services Canada,
Ottawa, Canada K1A 0S9

CANMET
Energy, Mines and Resources Canada,
555 Booth St.,
Ottawa, Canada K1A 0G1

or through your bookseller.

Catalogue No. M38-13/77-49 Price: Canada: $2.25
ISBN 0-660-01436-X Other countries: $2.70

Price subject to change without notice.

En vente par la poste:

Imprimerie et Édition
Approvisionnements et Services Canada,
Ottawa, Canada K1A 0S9

CANMET
Énergie, Mines et Ressources Canada,
555, rue Booth
Ottawa, Canada K1A 0G1

ou chez votre libraire.

N^{o} de catalogue M38-13/77-49 Prix: Canada: $2.25
ISBN 0-660-01436-X Autres pays: $2.70

Prix sujet à changement sans avis préalable.

MINERAL SCIENCES LABORATORIES
CANMET 77-49

EFFECT OF ELEVATED TEMPERATURES ON COMPRESSIVE STRENGTH, PULSE VELOCITY AND CONVERSION OF HIGH ALUMINA CEMENT CONCRETE

by

D.H.H. Quon* and V.M. Malhotra**

* * *

ABSTRACT

High alumina cement has achieved considerable attention in recent years due to structural failures associated with the use of this cement in England. Canadian Standards Association Committee A23.1, Concrete Materials and Methods of Concrete Construction, is currently debating the use of high alumina cement in structural concrete for reference in the standard. This investigation was therefore undertaken to obtain data on the performance of test specimens cured at temperatures from 21 to 66°C under both humid and dry conditions.

A series of 2-ft^3 (0.056-m^3) concrete mixes was made in the laboratory using crushed gravel and natural sand as coarse and fine aggregate respectively. Thirty 4 x 8-in. (102 x 203 mm) cylinders were cast from each mix. Following the initial moist curing period of 24 hours at 18°C, one third of the cylinders were subjected to standard moist curing at 21°C, one third were cured at 30 and 38°C in water and the remaining one third were cured at 30, 38 and 66°C under dry heat conditions. The curing period varied from 1 day to 180 days for each condition of curing. At each age, weight and pulse velocity of cylinders were determined before testing the specimens in compression. Small samples of mortar from the tested specimens were subjected to X-ray diffraction (XRD) and differential thermal analysis (DTA) studies to determine the nature of hydrates and the degree of conversion.

The change in compressive strength of high alumina cement concrete with age followed the established pattern. The

---

* Research Scientist, Ceramic Section, and ** Head, Construction Materials Section, Mineral Sciences Laboratories, CANMET, Department of Energy, Mines and Resources, Ottawa, Canada.

strength reached a maximum value followed by a minimum, which in turn was followed by a slow increase in strength with age. However, there was on exception: the cylinders exposed to dry heat at 66°C, after following the usual pattern of strength development with age as mentioned above, indicated a trend towards further loss in compressive strength.

The rate of conversion of high alumina cement concrete, i.e., the conversion of hexagonal hydrates ($CaO.Al_2O_3.10H_2O$) to cubic hydrates ($3CaO.Al_2O_3.6H_2O$), is a function of the temperature to which test cylinders are exposed, irrespective of the water/cement ratio of the concrete. The rate of conversion also appears to be independent of the type of exposure, i.e., whether it is dry heat or immersion in water.

There is no correlation between the degree of conversion and compressive strength of high alumina cement concrete so that the former cannot be used as a measure of compressive strength.

The pulse velocity technique appears to be an excellent means of monitoring the long-term changes in compressive strength due to conversion in high alumina cement concrete. This is especially so for test specimens exposed to dry heat, in which case this technique is superior to the determination of the degree of conversion by DTA. However its use to determine absolute values of strength cannot be recommended.

X-ray diffraction studies and scanning electron photomicrographs were shown to provide useful supporting data in the investigation of conversion phenomena but these studies are not considered essential for this purpose.

EFFETS DES HAUTES TEMPERATURES SUR LA RESISTANCE A LA COMPRESSION, SUR LA VITESSE D'IMPULSION ET SUR LA CONVERSION DU BETON A HAUTE TENEUR EN CIMENT ALUMINEUX

par

D.H.H. Quon* et V.M. Malhotra**

* * *

RESUME

Le ciment à haute teneur en alumine a fait l'objet d'une attention particulière, au cours des dernières années, en raison des défaillances dans l'utilisation de ce ciment en Angleterre. Le Comité A23.1 sur les matériaux en béton et les méthodes de construction en béton, de l'Association canadienne de normalisation, étudie actuellement l'utilisation du ciment à haute teneur en alumine dans le béton de charpente, pour fins de normalisation. Ces recherches on donc été entreprises pour obtenir des données sur la performance d'échantillons exposés à des températures variant de 21 à 66°C en atmosphère humide et sèche.

Une série de mélanges de béton de 2-pi^3 (0.056-m^3) a été effectuée en laboratoire en utilisant du gravier broyé et du sable naturel, respectivement, comme agrégat grossier et fin. Trente cylindres de 4 x 8-po. (102 x 203 mm) ont été coulés à partir de chaque mélange. Après la période initiale d'étuvage de 24 heures à à 18°C, un tiers des cylindres ont été soumis à l'étuvage standard à 21°C, un tiers à 30 et 38°C dans l'eau et le troisième tiers à 32, 38 et 66°C en atmosphère sèche. La période d'étuvage variait de 1 à 180 jours pour chaque milieu ambiant. Pour chaque période, on a déterminé le poids et la vitesse d'impulsion des cylindres avant de tester les échantillons sous compression. De petites quantités de mortier, prélevées sur les échantillons, ont été soumises à des études sur la diffraction moléculaire aux rayons-X et sur l'analyse thermique différentielle, afin de déterminer la nature des hydrates et le degré de conversion.

Le changement, dû au vieillissement, de la résistance à la compression du béton à haute teneur en ciment alumineux, suivait le modèle établi. La résistance a atteint une valeur maximale suivie

---

*Chercheur, Section de la céramique, et **Chef de la section des matériaux de construction, Laboratoires des sciences minérales, CANMET, Ministère de l'Energie, des Mines et des Ressources, Ottawa, Canada.

d'une valeur minimale, elle-même suivie à son tour d'une légère augmentation de la résistance due au vieillissement. Cependant, il y avait une exception: on a constaté qu'après avoir suivi le modèle habituel susmentionné de l'évolution de la résistance avec le vieillissement, les cylindres exposés à une chaleur sèche de 66°C avaient tendance à subir une diminution de la résistance à la compression.

Le taux de conversion du béton à haute teneur en ciment alumineux, c.-à-d., la conversion des hydrates hexagonaux ($CaO.Al_2O_3.10H_2O$) en hydrates cubiques ($3CaO.Al_2O_3.6H_2O$), est fonction de la température à laquelle sont exposés les cylindres, quel que soit le rapport eau/ciment du béton. Le taux de conversion semble également être indépendant du type d'exposition, c.-à-d., soit en atmosphère sèche soit en immersion dans l'eau.

Il n'ya aucune corrélation entre le degré de conversion et la résistance à l'écrasement du béton à forte teneur en ciment alumineux, ce qui empêche d'utiliser le degré de conversion comme mesure de résistance à la compression.

La technique de la vitesse d'impulsion semble un excellent moyen de surveiller les changements à long terme de la résistance à la compression, en raison de la conversion en béton à forte teneur en ciment alumineux. Ceci s'applique spécialement aux échantillons exposés à la chaleur sèche, et, dans ce cas, cette technique prévaut contre la détermination du degré de conversion par l'analyse thermique différentielle. Cependant, on ne peut recommander de l'utiliser pour déterminer des valeurs absolues de résistance.

Les études sur la diffraction moléculaire aux rayons-X et les photomicrographies électroniques par balayage ont fourni des données utiles à l'appui des recherches sur les phénomènes de conversion, mais on estime que ces études ne sont pas essentielles dans le cas des présentes recherches.

CONTENTS

LIST OF TABLES

LIST OF FIGURES

INTRODUCTION

High alumina cement is substantially different from normal portland cement in terms of composition. The main compounds in normal portland cement are tricalcium silicates and dicalcium silicates, whereas in high alumina cement the main compounds are the calcium aluminates ($CaO.Al_2O_3$ and $5CaO.3Al_2O_3$). These differences in composition are due to the differences in the raw materials used for the two types of cements. In the production of normal portland cement, limestone and clay are used as the raw materials, whereas limestone and bauxite are used in the manufacture of high alumina cement.

CANMET (formerly Mines Branch) has been investigating the properties of high alumina cement concretes for some time. Earlier investigations in 1962-1963 dealt with the effect of temperatures ranging from 100 to 1000°C on the properties of concrete made with high alumina cement.(1) In 1963, Neville(2) published a paper on the deterioration of structural concrete made with high alumina cement, which attracted considerable attention(3). During the last four years, high alumina cement has again become a topic of discussion due to the structural failures associated with this cement in England (4-8). The Canadian Standards Association Committee A23.1, Concrete Materials and Methods of Concrete Construction, is currently debating the use of high alumina cement in structural concrete for reference in the standard(9).

This investigation was therefore undertaken to obtain data on the performance of high alumina cement concrete test specimens cured at temperatures ranging from 21 to 66°C with and without the presence of humidity.

SCOPE OF INVESTIGATION

A series of nine 2-ft^3 (0.056-m^3) concrete mixes were made in the laboratory using crushed gravel and natural sand as coarse and fine aggregate respectively. Thirty 4 x 8-in. (102 x 203-mm) cylinders were cast from each mix. Following the initial moist curing period of 24 hours at 18°C, the test specimens were treated as follows: one third were subjected to standard moist curing at 21°C, one third were cured at 30 and 38°C in water and the remaining one third were cured at 30, 38 and 66°C under dry-heat conditions. Curing ages varied from 1 to 180 days. At each age, the weight and pulse velocity of test specimens were determined before testing the specimens in compression. Small samples of mortar from the tested specimens were subjected to X-ray diffraction (XRD) and differential thermal analyses (DTA) to identify the various hydrates and to determine the degree of conversion.

CONVERSION PHENOMENON AND DEGREE OF CONVERSION

The hydration of high alumina cement concrete is a complex phenomenon and its detailed discussion is beyond the scope of this report. However, Lea(10), Robson(11) and Midgley(12,13) have discussed the hydration phenomenon at considerable length. Briefly, the hydration of high alumina cement results in the formation of monocalcium aluminate decahydrate ($CaO.Al_2O_3.10H_2O$) together with a small quantity of $2CaO.Al_2O_3.8H_2O$ and alumina gel ($Al_2O_3.nH_2O$).

The CAH_{10}* and C_2AH_8** are hexagonal hydrates and of these CAH_{10} is of prime importance. Both are unstable at room and higher temperatures and convert to more stable cubic hydrate $3CaO.Al_2O_3.6H_2O$*** and either alumina gel or gibbsite (AH_3)****. Application of heat accelerates the process. The conversion reaction takes the following form:

$$3(CaO.Al_2O_3.10H_2O) \rightarrow 3CaO.Al_2O_3.6H_2O + 2(Al_2O_3.3H_2O) + 18H_2O$$

$$\text{or} \quad 3CAH_{10} \rightarrow C_3AH_6 + 2AH_3 + 18H{*}{*}{*}{*}{*}$$

It has been pointed out that, although water is liberated in this reaction, the conversion can take place only in the presence of water (4,5).

It is generally accepted(10) that the conversion of hydrates from the hexagonal to the cubic form results in an increased porosity of the cement paste because the specific gravities of C_3AH_6 and AH_3 are higher than those of CAH_{10} and C_2AH_8 as shown below:

CAH_{10} = 1.72
C_2AH_8 = 1.95
C_3AH_6 = 2.53
AH_3 = 2.44

---

* CAH_{10} = $CaO.Al_2O_3.10H_2O$
** C_2AH_8 = $2CaO.Al_2O_3.8H_2O$
*** C_3AH_6 = $3CaO.Al_2O_3.6H_2O$
**** AH_3 = $Al_2O_3.3H_2O$ or Gibbsite
***** H = H_2O

As the overall dimensions of the cement paste or concrete remain constant, the change in specific gravities results in porosity developing in the cement paste and this, in turn, probably causes loss in strength of high alumina cement concrete. Mehta and Lesnikoff (14) point out that the low adhesive capacity of C_3AH_6 and its low specific area also contribute to a loss in strength during conversion.

Notwithstanding the above, it should be stressed that the exact mechanism of the conversion is still not clear and needs further research.

According to Midgley the degree of conversion expressed as a percentage can be defined as follows (12,13):

$$\text{Degree of conversion} \quad \frac{\text{Weight of } C_3AH_6}{\text{Weight of } C_3AH_6 + \text{Weight of } CAH_{10}} \times 100$$

The relative weights of C_3AH_6 and CAH_{10} are determined from the measurements of the endothermic peaks recorded in DTA

SOME TYPICAL PROPERTIES OF HIGH ALUMINA CEMENT

Physical Properties

Some typical physical properties of high alumina cement are as follows:

Colour: Grey black

Specific gravity: 3.20 to 3.75

Surface area fineness (by Blaine method): 2250 to 3000 cm^2/g

Setting time: Initial set: 2 hrs 30 min
Final set: 3 hrs 20 min

Compressive strength of 2 in. (51 mm) cubes,
(w/c=0.40): 6100 psi (42 MPa) at one day
7120 psi (49 MPa) at three days

Chemical Composition

The chemical compositions of the aluminous cements used in the U.S.A. and England are given in Table 1, together with the typical chemical composition of a normal portland cement.

MATERIALS USED

Cement

A well-known commercially available brand of high alumina cement was used in this investigation. The oxide analysis and composition of this cement are given in Table 1.

High alumina cement, like portland cement, is a hydraulic material; its setting and hardening is achieved by chemical reactions with water. The most important compounds present in high alumina cement are calcium mono-aluminate, $CaO.Al_2O_3$, and calcium di-aluminate, $CaO.2Al_2O_3$. In addition to these principal compounds, other compounds present are: $2CaO.Fe_2O_3$, β-$2CaO.SiO_2$, FeO and pleochroite (a quaternary compound of CaO, $SiO_2.Al_2O_3$ and FeO).

There is no Canadian standard for the use of this cement.

Aggregates

Minus 1-in. (25-mm) river gravel was used as coarse aggregate and local natural sand as fine aggregate. To keep the grading uniform for each mix, the sand was separated into various size fractions which were then combined to a specified grading.

The grading and physical properties of both the coarse and fine aggregates are given in Tables 2 and 3.

Air-Entraining Agent

Apart from a commercially available air-entraining agent, no other admixtures, such as plasticizers, were used in the concrete mixes because little is known about their effect on high alumina cement concrete.

CONCRETE MIXES

A total of 9 concrete mixes was made in the CANMET laboratory between December 10, 1975 and April 8, 1976. A 2.5-ft^3 (0.067-m^3) laboratory counter-current mixer was used for preparing the concrete batches.

Mix Proportioning

Mix proportioning data for the concrete mixes are given in Table 4. The room-dry coarse and fine aggregates were soaked in water for 24 hours prior to use and the amount of mixing water was adjusted according to the water absorbed. A total of three batches was made for each water/cement ratio of 0.37, 0.47 and 0.60. The water content and the ratio of coarse to fine aggregates were kept constant for each series. An attempt was made to maintain the temperature of fresh concrete at 60 ± 5°F (15.6 ± 2.8°C) however, in three instances this was not possible.

Properties of Fresh Concrete

The properties of the freshly mixed concrete, i.e. temperature, slump, unit weight and air content are given in Table 4.

PREPARATION AND TESTING OF SPECIMENS

Preparation

Thirty 4 x 8-in. (102 x 203 mm) cylinders were cast from each mix. The cylinders were cast by filling steel moulds in approximately two equal layers, each layer being compacted by vibrating the moulds on a vibrating table in accordance with ASTM Standard C 31-69. After casting, the moulded cylinders were covered with glass plate which in turn was covered with water-saturated burlap kept wet by a water spray and were left in the casting room for 24 hours. During this period the temperature in the casting room was maintained at 18 ± 2°C. Subsequently, all the specimens were demoulded. Immediately after demoulding, density and pulse velocity measurements were made. Finally, three cylinders were capped and tested in compression in accordance with ASTM Standard C 39-72. The remaining cylinders were then treated as follows:

Compression Testing

Mix Series A - Batch 1

1. Nine of the cylinders were transferred to a moist curing room maintained at 21 ± 3°C. At the ages of 2, 7, 28 and 180 days, two* of the cylinders were removed from the curing room and

---

* At 28 days, three cylinders were tested for each curing condition.

the densities and pulse velocities* were determined. The cylinders were subsequently capped with a sulphur-flint mixture and tested in compression in accordance with ASTM Standard C39-72.

2. Nine of the cylinders were transferred to a water bath maintained at 30°C. At the ages of 2, 7, 28 and 180 days, two** of the cylinders were removed from the water bath, cooled to room temperature and the densities and pulse velocities* were determined. The cylinders were finally tested in compression as above.

3. Nine of the cylinders were transferred to a cabinet maintained at 30°C (dry heat). At the ages of 2, 7, 28 and 180 days, two** of the cylinders were removed from the heating cabinet, cooled to room temperature and pulse velocities determined. The cylinders were then tested in compression as above.

Mix Series A - Batch 2

The test specimens were treated as for Batch 1 except that the temperature of exposure in both the water bath and the heating cabinets was increased to 38°C.

Mix Series A - Batch 3

The test specimens were treated as for Batch 1 except that the temperature of exposure in the heating cabinets was increased to 66°C. No cylinders were immersed in water at 66°C.

---

* Pulse velocity was measured using a portable type ultrasonic tester of British origin.

** At 28 days, three cylinders were tested for each curing condition

Mix Series B and Mix Series C

The test specimens from the batches of these mixes were treated in a manner identical to that employed for the specimens from the concrete batches of Mix Series A.

X-ray Diffraction Studies

Small samples of concrete were secured from the broken test cylinders. The cementitious fraction was separated from the aggregate and ground to minus 100 mesh for XRD studies. X-ray photographs were taken with a Guinier-de Wolff focussing camera using Co Kα radiation. Subsequently, a diffractogram was obtained from each diffraction pattern using a Joyce Loebel double beam recording microdensitometer, thus enabling the relative intensity of each diffraction peak to be measured.

Differential Thermal Analysis (DTA)

DTA was used to study conversion of the high alumina cement concrete and to identify the hydrate phases present. As for the X-ray studies, very small samples of the cementitious component from the broken test cylinders were obtained for each exposure condition, great care being taken to remove aggregate particles from the samples.

One-gram aliquots of the cementitious component, ground to minus 100 mesh, were used for the DTA studies. The DTA examinations were conducted in air using a nichrome resistance furnace and a heating rate of 12°C per minute, alpha-alumina being used as the reference material.

The sample was held in a nickel holder and chromel-alumel thermocouples were used for both the sample and differential temperature measurements. The differential temperature and the reference material temperature were simultaneously recorded on a two-pen recorder, the former after amplification and the latter directly.

The presence of various hydrate phases, such as alumina gel, CAH_{10}, C_2AH_8, AH_3 and C_3AH_6, is shown in a DTA thermogram by endothermic reactions occurring at 120, 160, 195, 290 and 300°C respectively. The peak height recorded in the thermogram can be used to estimate the quantity of each hydrate phase present in the test sample. By the use of pure synthetic compounds of alumina gel, CAH_{10}, C_2AH_8, AH_3 and C_3AH_6, the DTA apparatus can be calibrated and used to estimate the percentage of each compound from the proportion of the peak heights in the thermogram. However, because synthetic compounds were not available for this study, samples having a known degree of conversion were obtained from the Building Research Establishment, U.K., and were used as standards for the calibration of the DTA instrument. The technique of estimating the degree of conversion was that used by Midgley$^{(13)}$ and is as follows:

$$\frac{\text{Peak height of } AH_3 \times 100}{\text{Peak height of } AH_3 + \text{Peak height of } CAH_{10}} \times k$$

where k is a calibration factor.

Scanning Electron Microscopy

No attempt was made in this study to characterize the high alumina cement concrete using scanning electron microscopy (SEM). Only two samples were examined by this technique; one sample was an unconverted high alumina cement paste and the other the converted high alumina cement concrete.

A conventional sample preparation technique for SEM studies was used. The samples were held on the specimen stub by a conducting silver paste and subsequently coated with carbon using a vacuum evaporator.

TEST RESULTS AND THEIR ANALYSIS

A total of 243 concrete cylinders were tested in this investigation. The test results for densities, pulse velocities, compressive strengths and degree of conversion are given in Tables 5, 6, 7 and 8. The between-batch coefficients of variation for the test results of three series of concrete mixes are shown in Table 9. The relationships between water/cement ratio and compressive strength of test cylinders are shown in Fig. 1 to 5. Plots of test results showing relationships between age and changes in weight, pulse velocity and compressive strengths are shown in Fig. 6-14. The relationships between age and degree of conversion for each temperature of exposure are shown in Fig. 15.

Typical examples of XRD patterns of the unconverted and converted test samples of high alumina cement concrete are shown in Fig. 16. The XRD peaks used for the identification of hydrated compounds in test samples are given in Table 10 and peak intensity for C_3AH_6 for water/cement ratio = 0.37 in various storage conditions is shown in Fig. 17.

A total of 120 samples of concrete were tested by DTA. Typical thermograms of the converted and unconverted test samples stored in different conditions at various temperatures with various water to cement ratios at 2 days age are shown in Fig. 18-20.

Scanning electron micrographs of converted concrete and unconverted cement paste are shown in Fig. 21 to 23.

DISCUSSION OF RESULTS

Changes in Weight and Density of Test Specimens

The changes in weight of the test specimens under various storage conditions are shown in Fig. 6-8. There is a small but steady increase in the weight of moist-cured test specimens. This is true for all water/cement ratios and the increases are less than 1.5% at 180 days.

The gain in weight of test specimens stored in water at 30°C is generally somewhat higher than those of moist-cured specimens, except for those with a water/cement ratio of 0.47 where the converse is true. This anomaly is unexplained.

For test specimens stored in water at 38°C the percentage gain in weight is considerably higher than for those stored in moist conditions. This is true for all concretes investigated, the maximum percentage gain being 2.25% for test specimens with a water/cement ratio of 0.37. These values are comparable with values reported by Neville(4).

The rate of weight increase, as well as its magnitude is greater for test specimens having a lower water/cement ratio and exposed to higher temperatures. The rationale is that in richer mixes there is a larger volume of cement paste and in a converted state there is a larger volume of pores, which in turn allow more water to be held.

The test cylinders stored in dry heat at 30, 38 and 66°C show steady loss in weight with age, the maximum percentage loss being 3.15 at 180 days for concrete with a water/cement ratio of 0.60. The loss in weight is due to the uncombined water being driven out by the drying process and porosity being created by the conversion process.

The density of the test cylinders at various ages is shown in Table 5. These results seem to be less meaningful because there are no definite patterns as to the increase or decrease in the density.

Degree of Conversion and Associated Strength Loss

Moist Curing Regime

The degree of conversion data in Fig. 15 show that for test specimens stored in the moist-during room at 21°C, the conversion had started at one day, even for concrete having a water/cement ratio of 0.37, the degree of conversion being about 15 %. Apart from one instance where it reaches as high as 37% at 180 days (for concrete mixes with a water to cement ratio of 0.60), the degree of conversion does not significantly increase with age up to 180 days and no changes in compressive strength were apparent.

The data by Teychenné(5) showed that, even for test specimens cured in water at 18°C, the conversion continued at least up to 8.5 years, the value rarely exceeding 60%. There was no loss in compressive strength with time associated with this conversion except when the water/cement ratio exceeded 0.45. When this occurred, there was significant loss in compressive strength of concrete.

It is emphasized that in normal structural concrete, water/cement ratios are usually higher than 0.45 and temperature of exposure generally exceeds 20°C. These conditions would invariably lead to conversion with substantial strength losses at later ages.

The absolute values of the compressive strengths of test cylinders are shown in Table 6. The strengths at ages from one to 180 days expressed as a percentage of one-day strength are shown in Fig. 12 to 14.

It may be seen in Fig. 13 that, at 180 days, test specimens for water/cement ratio of 0.47 show the maximum strength loss, the values being 70 % of the one-day strength. The plots in Figures 12 to 14 suggest continuing strength loss would have occurred if the tests had been continued.

Curing Regime - in Water and Dry Heat at 30°C

The exposure of test specimens to water at 30°C and to dry heat at 30°C showed that, at ages up to 28 days, the degree of conversion was between 20 and 24% (Fig.15). In spite of this low degree of conversion, the test specimens had lost strength between

7 and 28 days. This was true for all water/cement ratios used in this investigation.

Beyond 28 days there was a sharp increase in the degree of conversion. At 180 days, when the tests were discontinued, the conversion had reached between 65 and 72% for dry-heat exposure and between 56 and 65% for test specimens stored in water. The conversion trends in Fig. 15 show that if the tests had been continued beyond 180 days, there would probably have been further increases in the degree of conversion of the high alumina cement concrete.

Curing Regime - in Water and Dry Heat at 38°C

The degree of conversion was generally less than 20 percent at 7 days for all test cylinders cured at 38°C regardless of the water/cement ratio (Fig.15). However, starting at 7 days, there was a sharp increase in conversion continuing up to 28 days, following which the rate of conversion increase slowed down. For cylinders with a water/cement ratio of 0.60 and stored in water and in dry heat at 38°C, degree of conversion values of 85 and 67% were reached at 180 days respectively.

As regards compressive strength, the cylinders generally reached maximum values at 2 days following which they started losing strength with minimum values being reached at 28 days (Fig.12-14). Between 28 and 180 days there was some gain in strength, the amount being dependent on the strength level of the concrete. For test cylinders with a water/cement ratio of 0.37, stored in water, the compressive strength at 2 days was 115% of the one-day value. It reached a low of 75% at 28 days following which there was some recovery, with a value of 90% being reached at 180 days.

The above phenomenon of recovery of strength following the decrease associated with conversion has also been reported by others(4,5). The maximum recovery in strength was observed for low water/cement ratio and the least gain was noted for those concretes having a high water/cement ratio (Fig.12-14). Neville(4) has tried to explain this recovery in strength as follows:

> "It seems thus that when rapid conversion occurs, a large proportion of the cement remains unhydrated. Further gradual hydration takes place and this produces new hydrates which occupy some of the voids induced by hydration. As a result, a denser hydrated cement paste is obtained and this naturally has a higher strength than at the time of completion (or nearly so) of conversion. We confirmed this by boiling concrete with a water/cement ratio of 0.45 at the age of 24 hours and then storing it in cold water. There was a recovery of strength of about 3.4 MPa* during the next few days and in the following eight months there was a further gain of 1.7 MPa in some cases."

Curing Regime - Dry Heat at 66°C

In this curing regime, cylinders were stored in dry heat at 66°C only; no specimens were stored in water at 66°C. At two days, the degree of conversion varied from 66% for cylinders with a water/cement ratio of 0.47 to 76% for cylinders with a water/cement ratio of 0.37 (Fig.15). A maximum value of about 80% was reached at 7 days. Beyond 7 days, the degree of conversion did not change significantly with age and remained at about 80% at 180 days. This was true for all water/cement ratios investigated.

---

* Megapascal (MPa) = 145.04 psi

As to compressive strengths, a minimum value was reached at 2 days (Fig. 12-14). This varied from 35% of the one-day compressive strength for cylinders with a water/cement ratio of 0.47, to 58% for cylinders with a water/cement ratio of 0.37. Following this, there was a sharp recovery in strength. This lasted up to 7 days, after which the rate of recovery in strength decreased markedly but continued up to 28 days. At this age the strengths of cylinders, irrespective of the water/cement ratio, reached about 90% of the one-day strength. Beyond 28 days the strengths showed a small drop. A low value of 75% was reached at 180 days for cylinders with a water/cement ratio of 0.47.

The recovery in strength after 2 days may be explained according to a hypothesis advanced by Neville(4) but the indicated loss in strength after 28 days is unexplained. This anomaly needs further investigation. If the phenomenon of regression in strength is confirmed, then the statements by various researchers(15,16) that high-strength concrete can be made with high alumina cement by exposing low water/cement ratio concretes to high temperatures, may be open to question.

Pulse Velocity Measurements

The ultrasonic pulse velocity results are shown in Table 7 and Fig. 9 to 11. Briefly, the ultrasonic pulse velocity method involves measuring time of travel of electronically generated mechanical pulses through concrete, the time interval being measured by a digital meter and/or cathode ray oscilloscope.

The distance travelled through concrete divided by time gives the pulse velocity.

The plots in Fig. 9 to 11 follow the same general pattern as those for strength and conversion, showing decreases in pulse velocity with increasing age. This is true for all water/cement ratios and all storage conditions, indicating that pulse velocity is an excellent means of monitoring the progression and retrogression of strength in high alumina cement concrete. However, it is cautioned that pulse velocity measurements should not be used for estimating absolute values of strength of concrete because of poor correlations between compressive strength of concrete and its pulse velocity (17,18).

The following statement by Malhotra (17), best sums up the relationship between the pulse velocity and the strength of concrete:

> "Inasmuch as a large number of variables affect the relations between the strength parameters of concrete and its pulse velocity, the use of the latter to predict the compressive and/or flexural strengths of concrete is not recommended. Indeed, serious consideration should be given to the use of pulse velocity as a control test in its own right, and perennial attempts to correlate pulse velocity with strength parameters should be discouraged."

The above statement is well supported by the data in Fig. 9 to 11. The pulse velocity at various ages for concrete made with a water/cement ratio of 0.37 and cured under standard moist-room conditions is of the same order of magnitude as that of concrete made with water/cement ratios of 0.47 (Fig. 10) and 0.60 (Fig. 11). This is especially true for values at 28 days.

Another interesting feature about the pulse velocity technique is that for each water/cement ratio there is a marked difference between the pulse velocity values of cylinders stored in water at elevated temperature and those of cylinders exposed to dry heat. The cylinders exposed to dry heat show relatively lower pulse velocity because, in addition to porosity caused by conversion, they contain microcracks caused by exposure to heat. On the other hand, the higher pulse velocity of cylinders stored in water at elevated temperatures is due to the fact that water fills the pores caused by conversion thus resulting in shorter pulse travel time. Compression strength testing and determination of the degree of conversion fail to detect the presence of microcracks as effectively as the pulse velocity technique.

X-ray Diffraction Studies

The peak intensity data obtained for the various phases were considered for quantitatively estimating the relative proportion of each phase present in the samples of converted high alumina cement concrete. However, it was found that the presence of fine quartz, feldspar and other minerals in the fine aggregate interfered with some of the diffraction peaks for CAH_{10}, C_2AH_8, C_3AH_6 and gamma AH_3. In addition, at early ages the hydrated phases formed were generally of poor crystallinity and as a result some of the diffraction peaks observed were too weak and less distinct at high diffraction angles.

The unconverted concrete showed a strong diffraction peak at 7.16Å, which was due to the presence of CAH_{10} (Table 10). In the converted concrete, the diffraction peaks at 4.37 and 5.13Å were due to the presence of AH_3 (gibbsite) and C_3AH_6.

In view of the difficulties encountered in using XRD for quantitatively determining the hydrate phases, XRD patterns were used only for phase indentification and as a guide for determining the products during hydration and conversion.

The principal phases detected by XRD analysis (Fig.16) in the high alumina cement concrete were CAH_{10},C_3AH_6 and AH_3. The phase C_2AH_8 was not detected, the reason probably being that only a small quantity of this phase was present.

Based on the qualitative peak-intensity measurements for CAH_{10} and C_3AH_6, the results indicated that, for concretes stored in dry heat at 66°C for 7 days, the peak intensity for CAH_{10} gradually decreased with age, while there was an increase in peak intensity for the C_3AH_6 and AH_3 phases. At 28 days the peak intensity for C_3AH_6 and AH_3 reached a maximum and was maintained for a storage period of six months.

A rapid increase in X-ray intensity for peaks ascribed to C_3AH_6 and a decrease in peak intensity for CAH_{10} was observed for high alumina concretes after 28 days of storage in both dry heat and in water at 38°C.

In test specimens obtained from concretes stored in water or dry heat at 30°C, a very gradual decrease in peak intensity for CAH_{10} was observed; both C_3AH_6 and AH_3 peaks showed gradual increases in intensity after 28 days.

In test specimens obtained from moist-cured concretes, the peak intensity for CAH_{10} showed no significant change with age. Fig. 17 shows typical changes with age in the relative intensity of 5.13Å diffraction peak for C_3AH_6 for high alumina cement concrete (W/C = 0.37) after various storage conditions. The plot indicated that the amount of the C_3AH_6 phase increases with increasing conversion. The minimum compressive strength of concrete corresponds with the maximum intensity of the C_3AH_6 peak, indicating that at this stage the unstable hexagonal CAH_{10} is largely converted to C_3AH_6.

Differential Thermal Analysis Results

Phase Identification

Differential thermal analysis was used for the phase identification of high alumina cement concrete having various water/cement ratios and stored in different environmental conditions.

DTA thermograms for 2-day-old high alumina cement concrete with a water/cement ratio = 0.37 after various storage conditions are shown in Fig. 18. The DTA thermogram for this concrete moist cured at 21°C for two days showed two strong endothermic peaks at 120 and 160°C which was ascribed to the removal of water from alumina gel and the formation of CAH_{10}. The weak endothermic peak appearing in this curve at 290°C was attributed to the crystallization of gibbsite from the alumina gel. The DTA curves obtained for concretes stored in water and in dry heat at

30°C for two days showed similar characteristics. However, the DTA curves obtained for concretes under similar storage conditions but at a temperature of 38°C showed broad endothermic dips between 290 and 300°C. The weak endothermic peak in these curves at about 300°C was due to the presence of C_3AH_6, a conversion product of CAH_{10}. The DTA curve for high alumina cement concrete after two days of storage in dry heat at 66°C contained two strong endothermic peaks at 290 and 300°C which were ascribed to the presence of gibbsite and C_3AH_6.

An additional thermal effect which may be observed at 570°C in these DTA curves was due to the transformation of quartz from the α to β phase, while the weak endothermic shoulder occurring at 195°C in some of the DTA curves was ascribed to the presence of C_2AH_8.

DTA thermograms for high alumina cement concretes with increasing water/cement ratio and stored in different environmental conditions are given in Fig. 19 and 20. These thermograms are essentially similar to those obtained for high alumina cement concrete having a water/cement ratio of 0.37.

Scanning Electron Microscopy

The converted high alumina cement concrete sample examined had a water/cement ratio of 0.37 and had been stored at a temperature of 66°C for 28 days. This sample was estimated by the DTA to be about 80% converted.

An SEM micrograph of the fracture surface of this sample is shown in Fig. 21a. The surface consists of fine shperical-shaped grains measuring from 2 to 7 microns in diameter.

At higher magnification (Fig. 21b) the presence of polyhedral crystals and traces of tubular crystals can be seen. The polyhedral crystals, though poorly developed, show characteristics of the trapezohedron outline and this morphological characteristic indicates the presence of C_3AH_6. The tabular crystal shown in the SEM micrograph reveals the presence of AH_3.

An SEM micrograph of the fracture surface of the unconverted cement paste prepared with a water/cement ratio of 0.25 and stored at $25^{o}C$ for 28 days is shown in Fig. 22a. Examination of the fracture surface does not reveal any information regarding microstructure or morphology of the material. Close examination of the pores in the fracture surface reveal that in these areas unrestricted crystal growth has occurred. Fig. 22b shows the inner surface of a pore viewed at a relatively low magnification; the entire surface is covered with a very fine-grained material. Under increasing magnification (Fig.23a) the presence of platy crystals is revealed. Although the micrograph appears to show the presence of both 'needles' and 'platelets', this apparent difference in morphology arises due to the random orientation of the crystals with respect to the direction of view. The morphology of this material is more clearly seen at higher magnification in Fig. 23b where the random orientation of the hexagonal crystals is shown and it can be seen that the needle-like crystals observed at lower magnification in Figure 23a are only the edges of the hexagonal crystals. The needle shaped crystals are seen when the crystal is in the prismatic direction $[10\bar{1}0]$, whereas the hexagonal outline is seen when the crystal is viewed in the basal direction [0001]. XRD analysis confirmed that the hexagonal crystals are CAH_{10}.

CONCLUSIONS

The change in strength with time of high alumina cement concrete cured at various temperatures under both humid and dry conditions, in general, follows an established pattern: The strength reaches a maximum value followed by a minimum which, in turn, is followed by a slow increase in strength with age. However, the test specimens exposed to dry heat at 66°C, after following the usual pattern of strength change with time as mentioned above, indicate a trend towards further loss in strength. This is explained and needs further investigation.

The pulse velocity technique appears to be an excellent means of monitoring the long-term changes in compressive strength due to conversion of high alumina cement concrete. This is especially so for test specimens exposed to dry heat, in which case this technique is superior to others for determining degree of conversion. However, its use to determine absolute values of strength of concrete cannot be recommended.

The degree of conversion should not be used as a measure of compressive strength of high alumina cement concrete because of lack of correlation between the two parameters.

The changes in weight of test specimens appear to be more meaningful than their densities. The rate of increase in weight as well as its magnitude are greater for test specimens having a lower water/cement ratio and exposed to higher temperatures.

During continuous exposure to moist curing conditions at 21°C, the degree of conversion increases with age, regardless of the water/cement ratio of concrete. A value of about 20% is reached after 6 months. The tests were not continued long enough to obtain maximum values. For continuous exposure at 66°C, a value of about 75% is reached at 2 days and this does not change significantly with time.

The rate of conversion is a function of the temperature to which the concrete test specimens are exposed - irrespective of the water/cement ratio of the concrete. The rate also appears, in general, to be independent of the type of exposure, i.e., whether it is dry heat or immersion in water.

After continuous exposure to water or dry heat at 30°C and above, a minimum strength is developed as the degree of conversion rises to 65 to 70%. However, for continuous exposure to moist curing conditions at 21°C, minimum values were not reached at 6 months when the degree of conversion was about 20%. If the test had been continued, it is probable that, for the water/cement ratio used in this investigation, minimum strength values may have been reached with a degree of conversion of less than 50%.

ACKNOWLEDGEMENTS

Acknowledgement is made to K. Painter, D. Malanka, L.K. Zemgals and C.H.J. Childe, all of Mineral Sciences Laboratories, CANMET for performing the laboratory work associated with this investigation.

Thanks are extended to H.G. Midgley of Building Research Station, U.K., for supplying standard samples for use in DTA calibrations.

REFERENCES

1. Zoldners, N.G., Malhotra, V.M. and Wilson, H.S. "High temperature behaviour of aluminous cement concretes containing different aggregates". Proc., Am. Soc. Testing Mater. v. 63, pp 966-991; 1963.

2. Neville, A.M. "A study of deterioration of structural concrete made with high alumina cement". Proc. Inst. Civil Engineers; v. 25, paper 6652, pp 287-324; July 1963.

3. Zoldners, N.G. and Malhotra, V.M. "Discussion of reference 2" Proc. Inst. Civil Engrs.; v.28, pp 72-73; May 1964.

4. Neville, A. and Wainwright, P.J. "High alumina cement concrete". John Wiley & Sons, New York; 198pp; 1975.

5. Teychenné, D.C. "Long-term research into the characteristics of high alumina cement concrete". Mag. of Concrete Research; v. 27, no. 91, pp 78-102; 1975.

6. Neville, A.M. "Is high alumina cement a satisfactory structural material?". Can. Civil Eng. v. 2, no. 4, pp 373-380. Dec. 1975.

7. Building Research Establishment. "High alumina cement concrete in buildings". Current paper CP 34-75, Department of the Environment, Garston U.K.; 50 pp; May 1975.

8. George, C.M. "The structural use of high alumina cement concrete". Undated paper available from Lafarge Fondu International, Neuilly-Sur-Seine, France.

9. Malhotra, V.M. "Discussion of reference 6"; Can. Civil Eng.; v. 3, no 3, pp 47-75; Sept. 1976.

10. Lea, F.M. and Desch, E.H. "Chemistry of cement and concrete". Revised Edition of the Chemistry of Cement and Concrete, Arnold, London, 1956, Revised Edition; 727 pp; 1970.

11. Robson, T.D. "High alumina cement and concretes". John Wiley and Sons, New York; 1962.

12. Midgley, H.G. "The conversion of high alumina cement". Magazine of Concrete Research; v. 27, no. 91, pp 59-77; June 1975.

13. Midgley, H.G. "The determination of the degree of conversion of high alumina cements". Unpublished data, Building Research Establishment, U.K.; 1975.

14. Mehta, P.K. and Lesnikoff, G. "Conversion of $CaO.Al_2O_3.10H_2O$ to $3CaO.Al_2O_3.6H_2O$"; Am. Ceramic Soc.; v. 54, no. 4, pp 210-212; April 1971.

15. French, P.J., Montgomery, R.G.J. and Robson, T.D. "High concrete strength within the hour"; Concrete; v. 5, no. 8, pp 253-258; August 1971.

16. Ramachandran, V.S. and Feldman, R.F. "Hydration characteristics of monocalcium aluminate at a low water/cement ratio"; Cement and Concrete Research; v. 3, no. 6, pp 729-750; 1973.

17. Malhotra, V.M. "Testing hardened concrete: nondestructive methods"; American Concrete Institute; Monograph no. 9, p. 188; 1976.

18. Mayfield, B. and Bettison, M. "Ultrasonic pulse testing of high alumina cement concrete: in the laboratory"; Concrete; v. 8, no. 9, pp 36-38; Sept. 1974.

TABLE 1

Chemical Compositions of High Aluminous Cements and a Typical Normal Portland Cement

Oxide	High Aluminous Cement Used in This Investigation	Typical Aluminous Cements in England and U.S.A.		Typical Portland Cement
		England	U.S.A.	
SiO_2	4.40	4-5	8-9	21.88
Al_2O_3	41.98	38-40	40-41	4.50
Fe_2O_3	12.62	8-10	5-6	2.16
CaO	36.96	36-39	36-37	62.67
MgO	0.83	1.0	1.0	2.50
SO_3	0.00	0.1	0.2	3.24
FeO	3.82	5-7	5-6	-
TiO_2	not reported	2.0	2.0	-

TABLE 2

Grading of Aggregates

Coarse Aggregate		Fine Aggregate	
Sieve size	Cumulative percentage retained	Sieve size	Cumulative percentage retained
3/4 in. (19 mm)	33.3	No. 4 (4.75 mm)	0
		No. 8 (2.38 mm)	10.0
3/8 in. (9.5 mm)	66.6	No. 16 (1.19 mm)	32.5
		No. 30 (600 μm)	57.5
No. 4 (4.75 mm)	100.0	No. 50 (300 μm)	80.0
		No. 100(150 μm)	94.0
		Pan	100.0

TABLE 3

Physical Properties of Coarse and Fine Aggregates

	Gravel Stone	Natural Sand
Specific Gravity	2.68	2.70
Absorption, %	0.40	0.50

TABLE 4

Mix Proportions and Properties of Fresh Concrete

Mix Series	Mix No.	Mix Proportions		Temp. °C	Properties of Fresh Concrete				
					Slump		Unit Weight		Entrained Air Content, %
		W/C*	A/C**		in.	mm	lb/ft^3	kg/m^3	
A	1	0.37	4.29	15.0	2.25	60	153.2	2279	2.7
	2	0.37	4.29	14.4	4.0	100	154.4	2297	2.2
	3	0.37	4.29	18.0	3.75	95	154.8	2303	2.0
B	4	0.47	5.42	11.0	4.25	110	152.4	2267	2.4
	5	0.47	5.42	10.0	5.00	125	153.2	2279	2.4
	6	0.47	5.42	16.0	3.00	75	152.4	2267	2.4
C	7	0.60	6.97	16.7	1.75	45	150.4	2238	3.2
	8	0.60	6.97	15.0	1.75	45	149.8	2229	2.4
	9	0.60	6.97	20.0	1.50	40	155.2	2309	3.7

* Water/cement ratio (by weight)

** Aggregate/cement ratio (by weight)

TABLE 5

Density of Test Cylinders at Various Ages

W/C*	Age, days	Curing Regime									
		A		B				C			
		Moist Curing at 21°C		In Water at 30°C		Moist Curing at 21°C		In Water at 38°C		Moist Curing at 21°C	
		lb/ft^3	kg/m^3	lb/ft^3	kg/m^3	lb/ft^3	kg/m^3	lb/ft^3	kg/m^3	lb/ft^3	kg/m^3
0.37	1	154.8	2480	-	-	155.7	2494	-	-	155.5	2491
	2	153.5	2457	153.8	2464	155.0	2483	154.7	2479	155.3	2489
	7	154.4	2475	153.7	2463	154.7	2479	154.1	2468	155.0	2483
	28	153.1	2452	154.6	2477	153.4	2458	152.3	2440	153.5	2461
	180	154.0	2467	155.1	2485	151.8	2431	152.9	2451	152.2	2438
0.47	1	154.5	2475	-	-	154.3	2472	-	-	153.9	2466
	2	153.0	2450	154.0	2464	154.7	2479	153.5	2459	154.5	2331
	7	154.4	2475	153.7	2463	154.7	2479	154.1	2468	155.0	2483
	28	152.5	2442	153.9	2465	150.0	2403	152.4	2441	153.7	2462
	180	150.8	2416	151.6	2477	152.2	2438	150.9	2418	151.8	2432
0.60	1	153.7	2462	-	-	153.3	2456	-	-	152.4	2442
	2	152.1	2437	153.3	2456	153.2	2454	151.8	2432	151.6	2429
	7	152.9	2449	151.2	2422	151.5	2427	152.6	2445	151.1	2421
	28	153.1	2453	150.2	2406	151.8	2432	153.4	2457	151.1	2421
	180	153.9	2465	152.1	2437	149.7	2398	149.3	2392	149.4	2393

* Water/Cement ratio by weight.

TABLE 6

Strength Development of Concretes Stored under Different Temperature and Humidity Regimes

W/C	Age, days	Compressive Strength															
		A						B						C			
		Moist Curing at 21°C		In Water at 30°C		In Dry Heat at 30°C		Moist Curing at 21°C		In Water at 38°C		In Dry Heat at 38°C		Moist Curing at 21°C		In Dry Heat at 66°C	
		psi	MPa	psi	MPa	psi	MPa	psi	MPa	psi	MPa	psi	MPa	psi	MPa	psi	MPa
0.37	1	7290	50.2	-	-	-	-	7820	53.9	-	-	-	-	7890	54.4	-	-
	2	8120	55.9	8080	55.7	7850	54.1	7650	52.7	9080	62.6	8280	57.1	7920	54.6	4580	31.6
	7	10410	71.7	9380	64.6	4320	64.2	8740	60.3	7110	48.9	7300	50.3	8460	58.3	6670	45.3
	28	9800	67.5	8540	58.8	8460	58.3	9260	63.8	5850	40.3	4310	36.6	10050	69.2	7460	51.4
	180	9560	65.9	6310	43.5	5470	37.7	11000	75.9	7220	49.8	6050	41.7	10370	74.1	7180	49.6
0.47	1	6020	41.5	-	-	-	-	5960	41.0	-	-	-	-	5930	40.9	-	-
	2	6150	42.4	7130	49.1	7080	48.8	6350	43.8	6290	43.4	7000	48.3	6630	45.7	2080	14.3
	7	8180	56.4	8240	56.8	7570	52.2	7180	49.5	6170	42.5	6690	56.1	7260	50.0	5020	34.6
	28	7980	55.1	8750	60.3	6460	44.5	7540	51.9	3770	76.0	3920	27.0	8030	55.2	5120	35.3
	180	9050	62.4	4340	29.9	4160	28.7	8220	56.7	4740	32.7	4170	28.8	7480	55.0	4450	30.6
0.60	1	4350	30.0	-	-	-	-	4700	32.4	-	-	-	-	4460	30.8	-	-
	2	4660	32.1	5110	35.3	4970	34.2	5990	41.3	5460	37.6	7580	52.3	4520	31.1	2290	15.8
	7	6890	47.5	6200	42.7	5850	40.4	6800	46.9	6530	45.0	6840	47.2	5830	40.2	3880	26.7
	28	8030	55.3	6180	42.6	5890	40.6	7450	51.3	3180	21.9	3840	26.4	6230	42.9	4020	27.7
	180	7830	54.1	3290	22.7	3600	24.8	7350	50.8	3240	22.4	4050	27.9	6160	42.5	3790	26.1

TABLE 7

Pulse Velocity of Cylinders Stored under Different Temperature and Humidity Regimes

W/C Ratio	Age, days	Pulse Velocity															
		A						B						C			
		Moist Curing at 21°C		In Water at 30°C		In Dry Heat at 30°C		Moist Curing at 21°C		In Water at 38°C		In Dry Heat at 38°C		Moist Curing at 21°C		In Dry Heat at 66°C	
		ft/sec	m/sec	ft/sec	m/sec	ft/sec	m/sec	ft/sec	m/sec	ft/sec	m/sec	ft/sec	m/sec	ft/sec	m/sec	ft/sec	m/sec
0.37	1													14652	4466	–	–
	2													14981	4566	14035	4278
	7	–	–	–	–	–	–	–	–	–	–	–	–	15670	4593	14035	4278
	28													14981	4566	13988	4263
	180													15591	4752	13621	4151
0.47	1	–	–	–	–	–	–	–	–	–	–	–	–	13657	4162	–	–
	2	14035	4297	14335	4369	14335	4369	14570	4441	14335	4369	14735	4491	14341	4371	12758	3888
	7	14814	4515	15151	4617	14652	4466	14801	4511	13900	4237	14650	4465	15109	4605	13011	3966
	28	15094	4600	15564	4744	14547	4434	15044	4585	14400	4389	13650	4160	15038	4583	12875	3924
	180	16862	4895	14335	4369	12822	3908	15875	4838	14667	4470	12580	3834	15875	4838	13265	4043
0.6	1	13831	4215	–	–	–	–	13655	4162	–	–	–	–	13831	4215	–	–
	2	14335	4369	14335	4369	14037	4278	14582	4444	13816	4211	14033	4277	14335	4369	12641	3853
	7	14979	4565	14814	4515	14184	4323	14502	4419	14269	4349	14194	4326	14808	4513	13071	3984
	28	15810	4819	15099	4602	13927	4244	15810	4819	14120	4304	13510	4118	15152	4618	12731	3880
	180	15595	4752	14035	4278	12189	3715	15684	4780	13674	4164	12115	3692	15895	5117	12346	4050

TABLE 8

Degree of Conversion, Per Cent, of High Alumina Cement Concrete for Different Water/Cement Ratios at Various Ages

Mix Series	W/C*	Age, days	Curing Regime								
			A	B	C	A	D	E	A	F	G
A	0.37	1	-	-	-	11	-	13	13	No specimen were available for this exposure	
		2	-	19	4	14	23	-	16		76
		7	17	19	23	12	38	21	12		80
		28	13	22	24	19	66	71	18		80
		180	15	56	70	19	78	75	18		80
B	0.47	1	16	-	-	9	-	-	8		
		2	15	11	6	9	15	7	14		66
		7	12	10	12	14	21	13	10		75
		28	14	17	15	17	71	65	16		78
		180	14	65	72	18	79	70	17		79
C	0.60	1	15	-	-	9	-	-	-		-
		2	18	18	18	7	12	10	24		73
		7	19	18	16	16	17	14	19		82
		28	20	18	24	17	66	62	24		83
		180	20	63	65	16	85	67	37		82

* Water/Cement ratio by weight

**A: Stored for 24 hours in moist air at $18^{\circ}C$, then in moist curing room at $21^{\circ}C$;

B: Stored for 24 hours in moist air at $18^{\circ}C$, then in water at $30^{\circ}C$;

C: Stored for 24 hours in moist air at $18^{\circ}C$, then in dry heat at $30^{\circ}C$;

D: Stored for 24 hours in moist air at $18^{\circ}C$, then in water at $38^{\circ}C$;

E: Stored for 24 hours in moist air at $18^{\circ}C$, then in dry heat at $38^{\circ}C$;

F: Stored for 24 hours in moist air at $18^{\circ}C$, then in water at $66^{\circ}C$;

G: Stored for 24 hours in moist air at $18^{\circ}C$, then in dry heat at $66^{\circ}C$.

TABLE 9

Between-Batch Coefficient of Variation for Strength Test Results

Mix Series	No. of Batches	Average 28-Day Compressive Strength psi	Standard Deviations, psi	Coefficient of Variation, %
A	3	9700	401	4.1
B	3	7850	269	3.4
C	3	7230	919	12.7*

* High value due to one batch having a low 28-day compressive strength.

TABLE 10

X-ray Diffraction Peaks Used for the Identification of Hydrated Compounds in High Alumina Cement Concretes

CAH_{10}		C_3AH_6		AH_3		C_2AH_8	
d(Å)	I	d(Å)	I	d(Å)	I	d(Å)	I
14.3	100	5.13	80	4.85	100	12.6	80
7.16	100	4.45	80	4.37	100	6.3	60
5.39	40	3.36	80	4.32	80	4.98	40
4.75	40			3.30	60	4.39	20
4.52	20					4.28	40
4.16	20					4.18	80

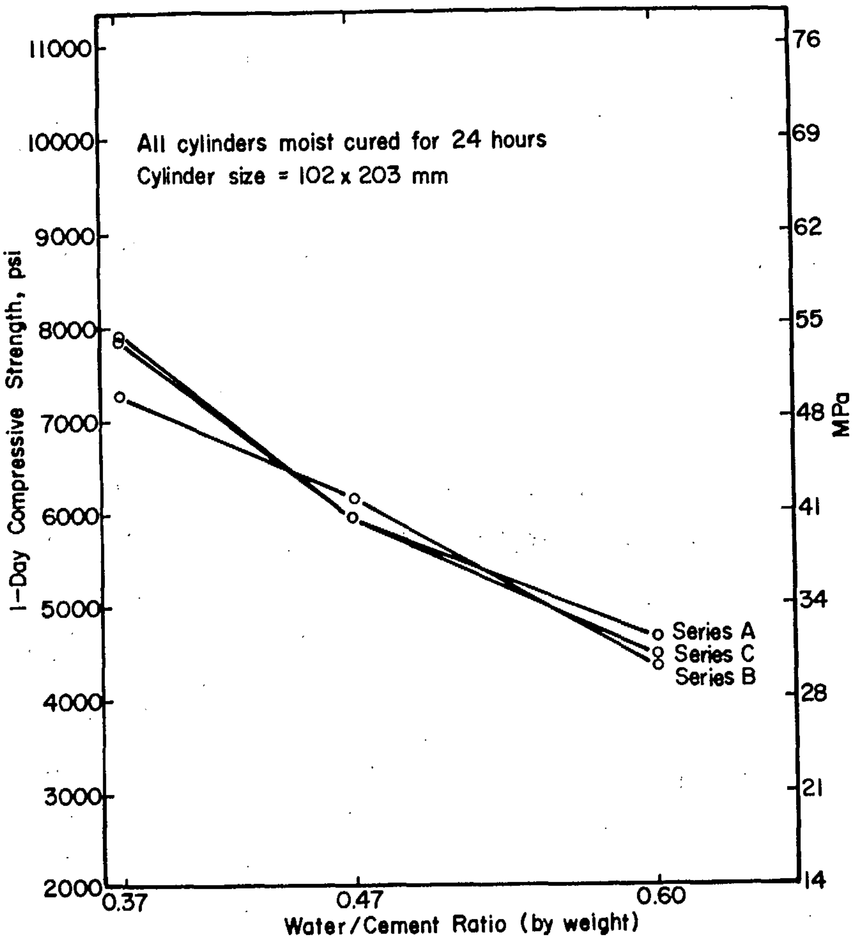


Fig. 1. Relationship between Water/Cement Ratio and Compressive Strength of High Alumina Cement Concrete at One Day

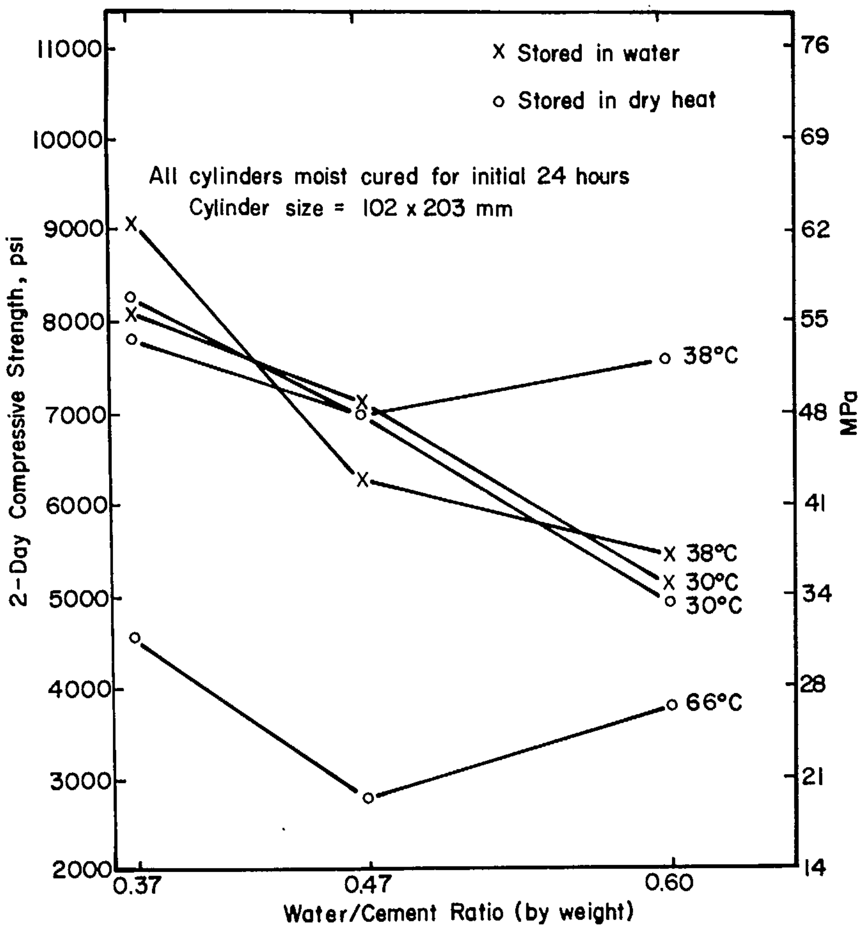


Fig. 2. Relationship between Water/Cement Ratio and Compressive Strength of High Alumina Cement Concrete at 2 days

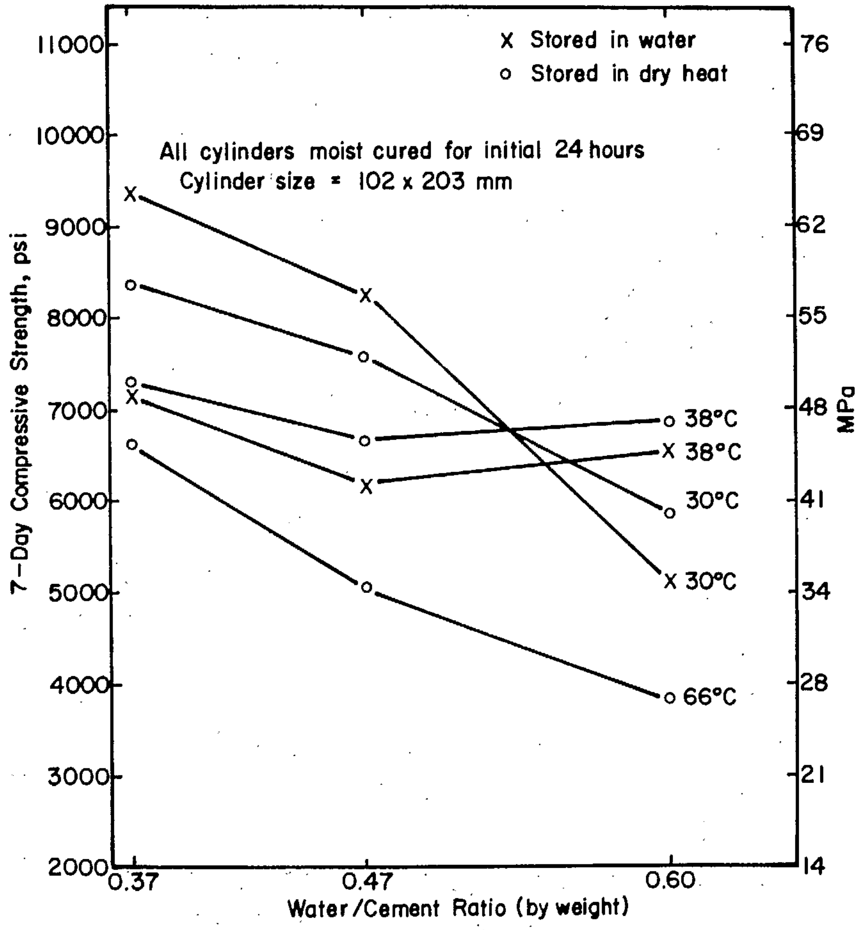


Fig. 3. Relationship between Water/Cement Ratio and Compressive Strength of High Alumina Cement Concrete at 7 days

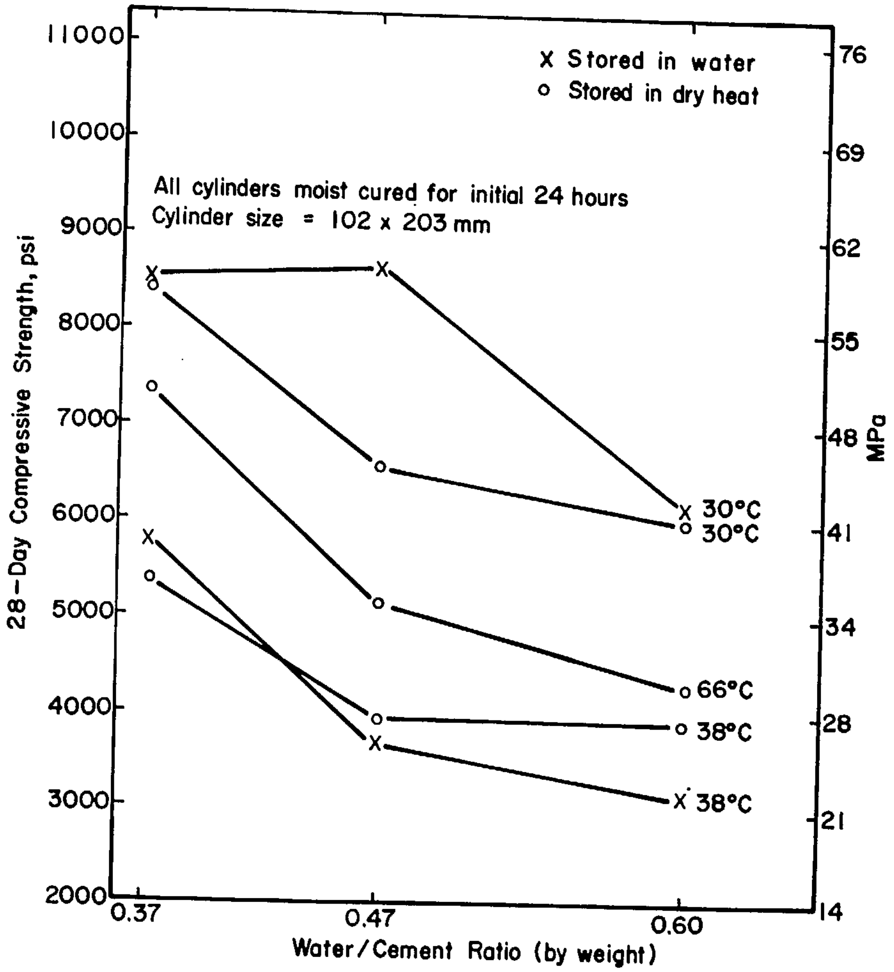


Fig. 4. Relationship between Water/Cement Ratio and Compressive Strength of High Alumina Cement Concrete at 28 Days

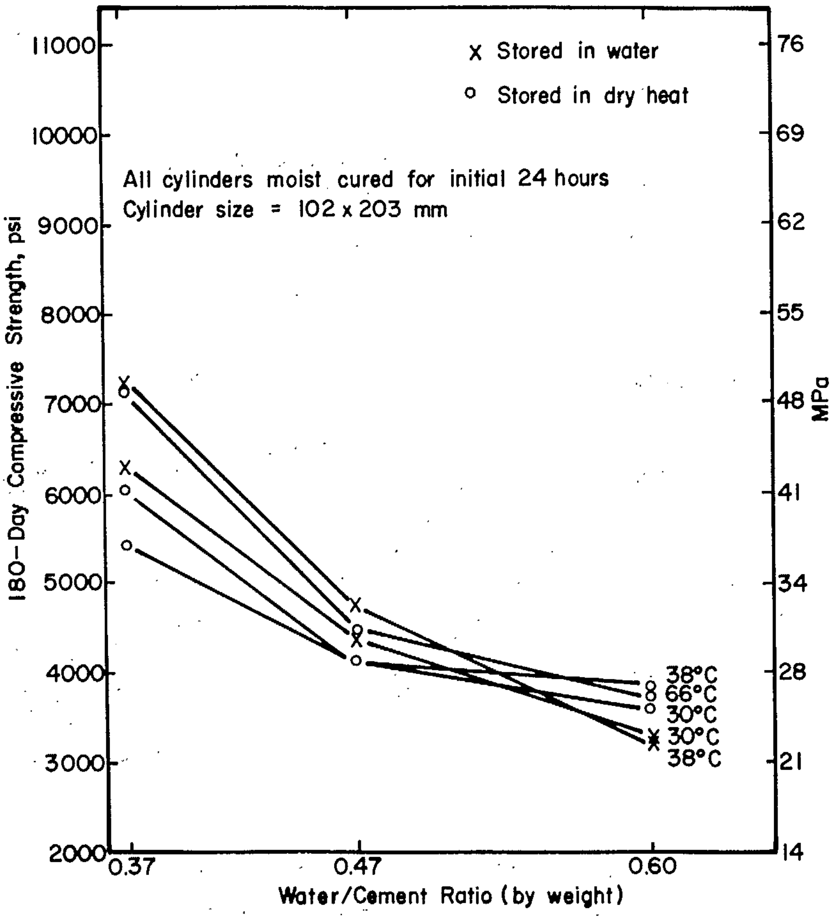


Fig. 5. Relationship between Water/Cement Ratio and Compressive Strength of High Alumina Cement Concrete at 180 Days

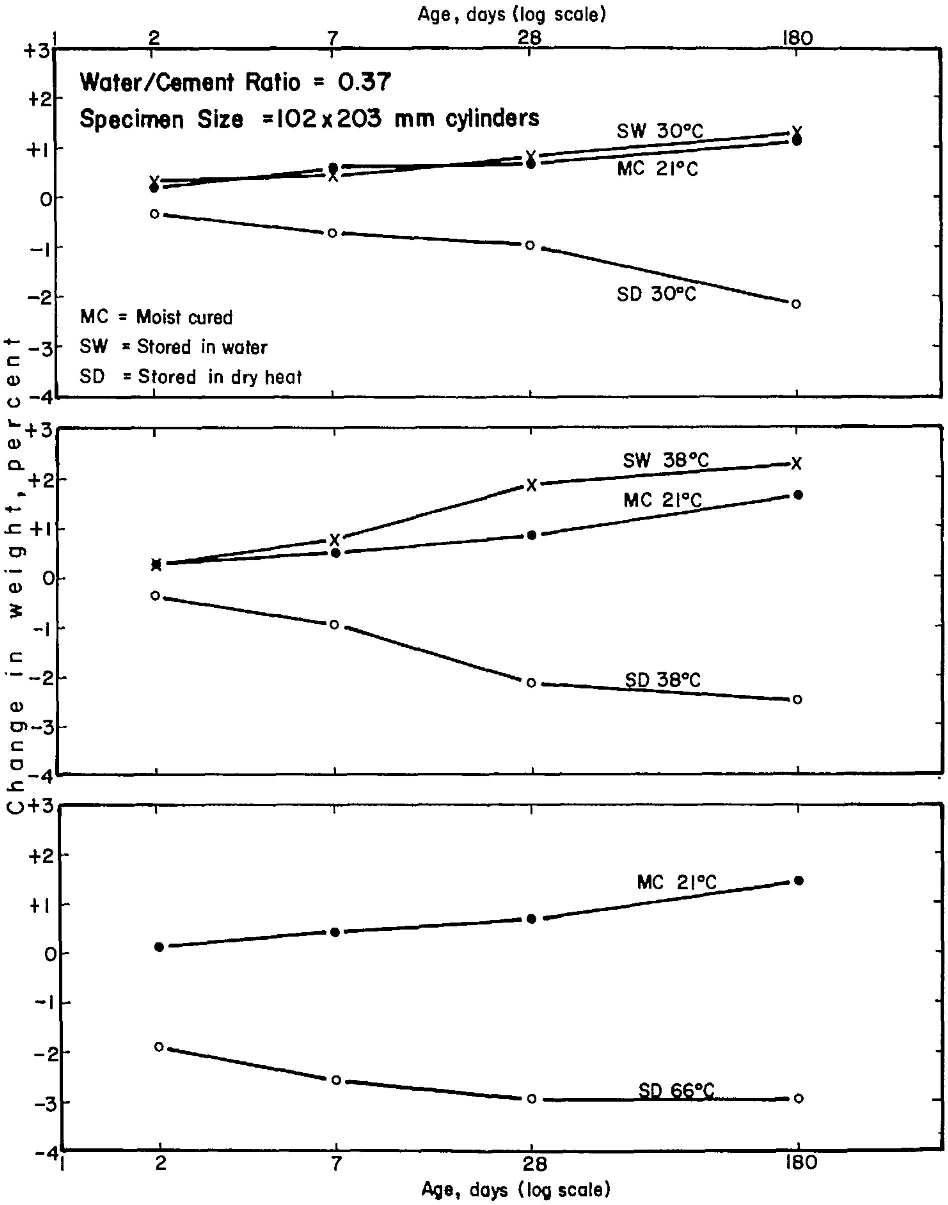


Fig. 6. Relationship between Age and Changes in Weight of Test Cylinders - W/C = 0.37.

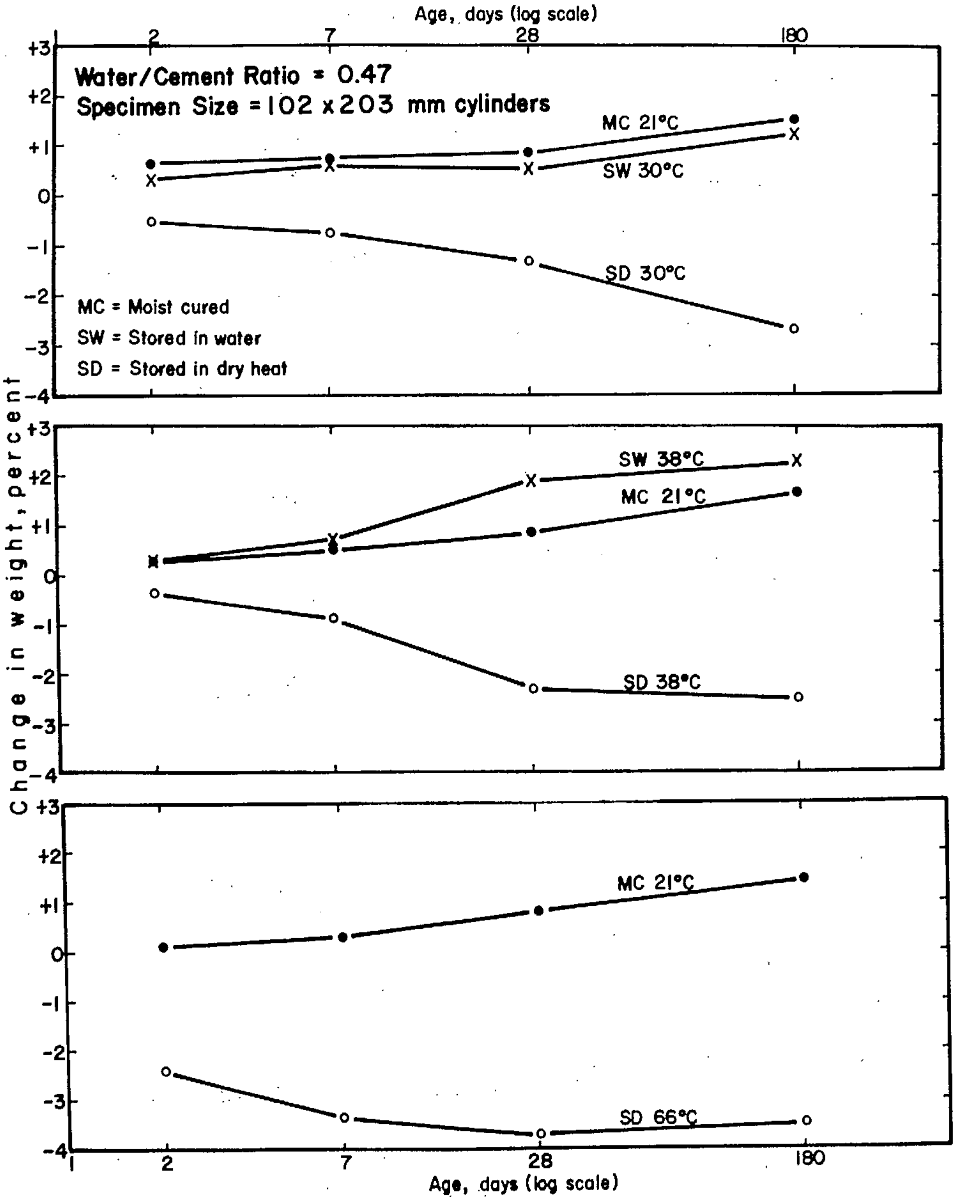


Fig. 7. Relationship between Ages and Changes in Weight of Test Cylinders - W/C = 0.47

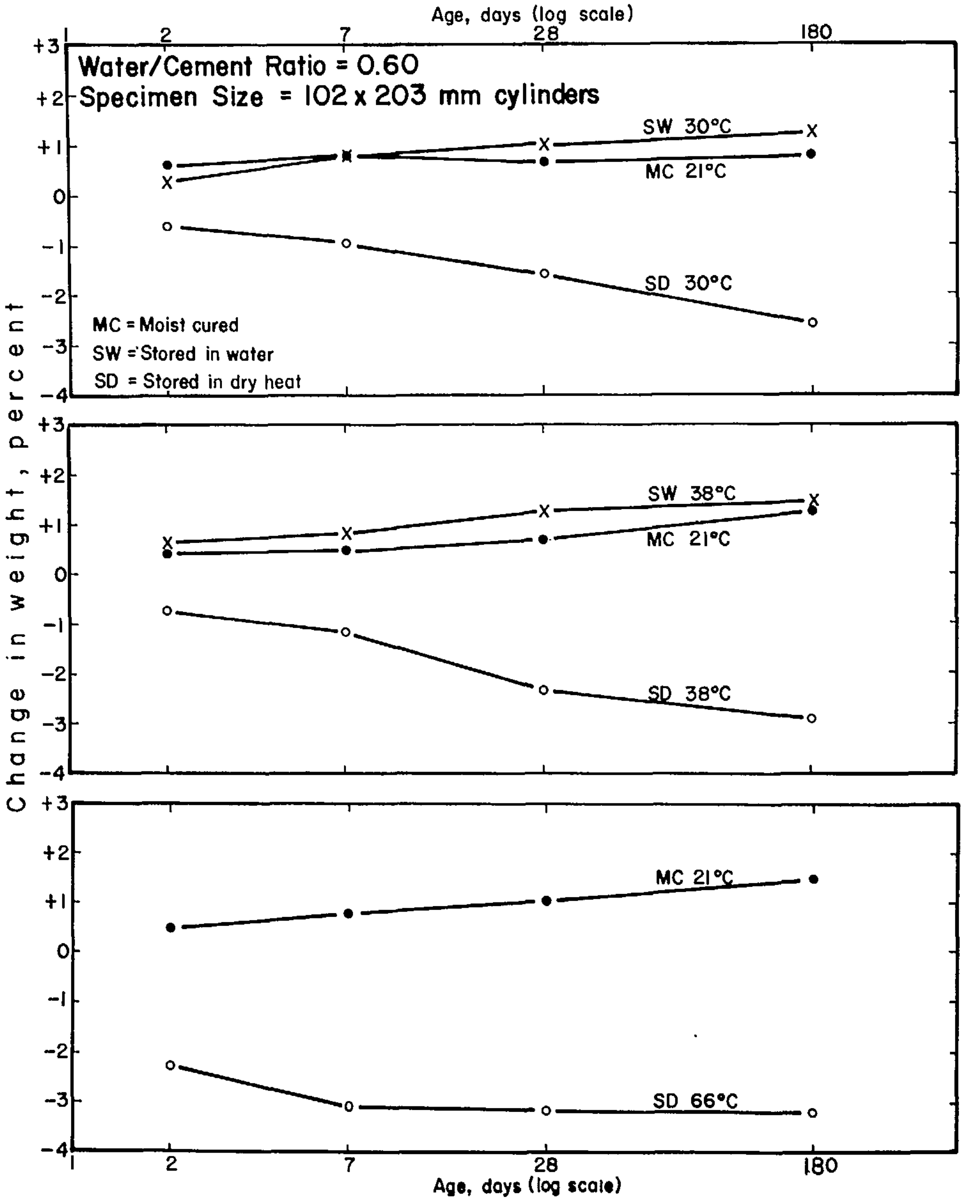


Fig. 8. Relationship between Age and Changes in Weight of Test Cylinders - W/C = 0.60

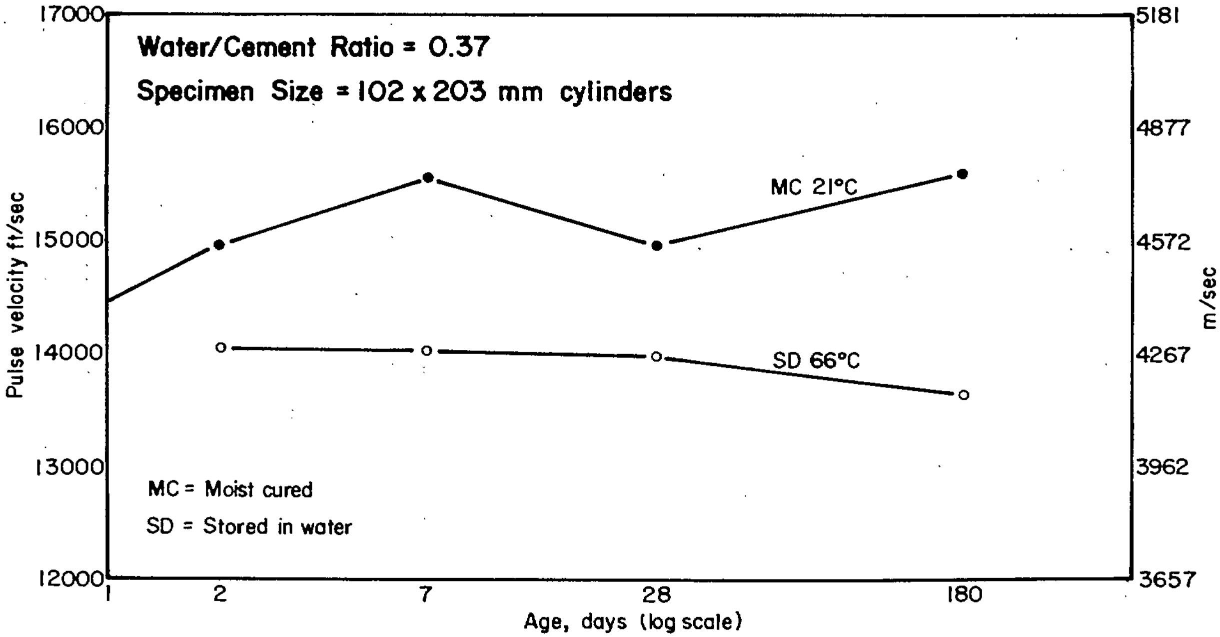


Fig. 9. Relationship between Age and Changes in Pulse Velocity of Test Cylinders - W/C = 0.37

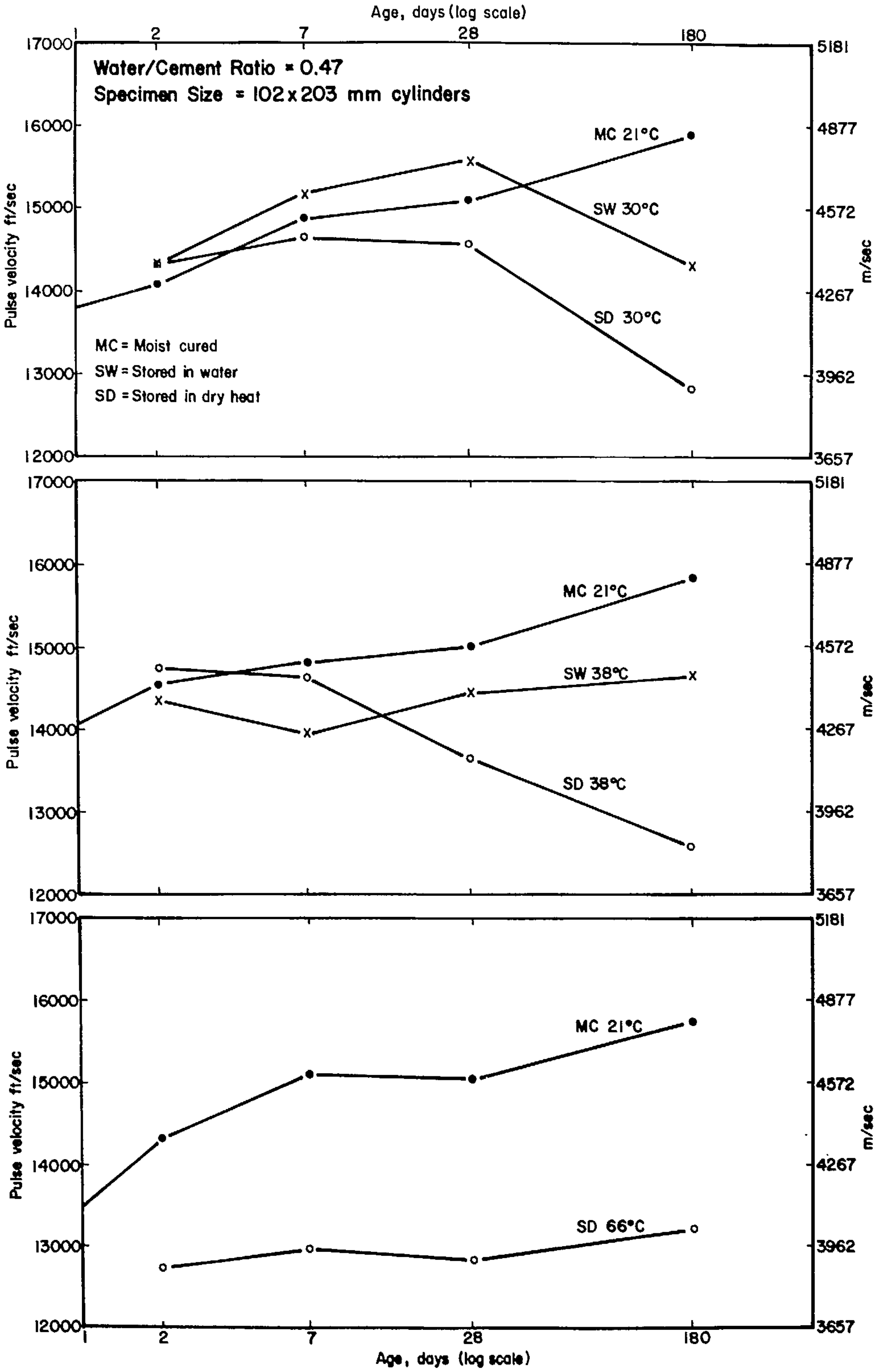


Fig. 10. Relationship between Age and Changes in Pulse Velocity of Test Cylinders - W/C = 0.47.

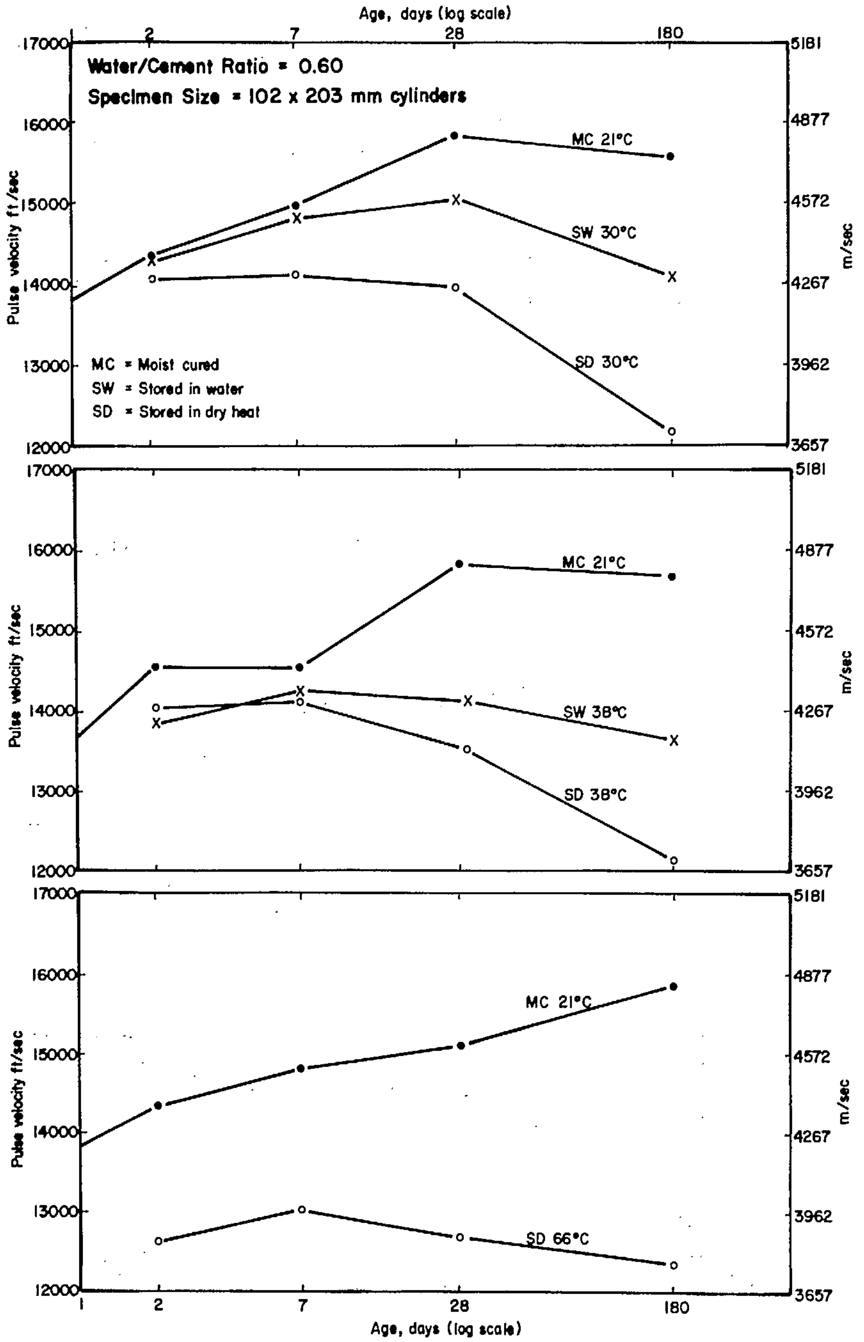


Fig. 11. Relationship between Age and Changes in Pulse Velocity of Test Cylinders - W/C = 0.60.

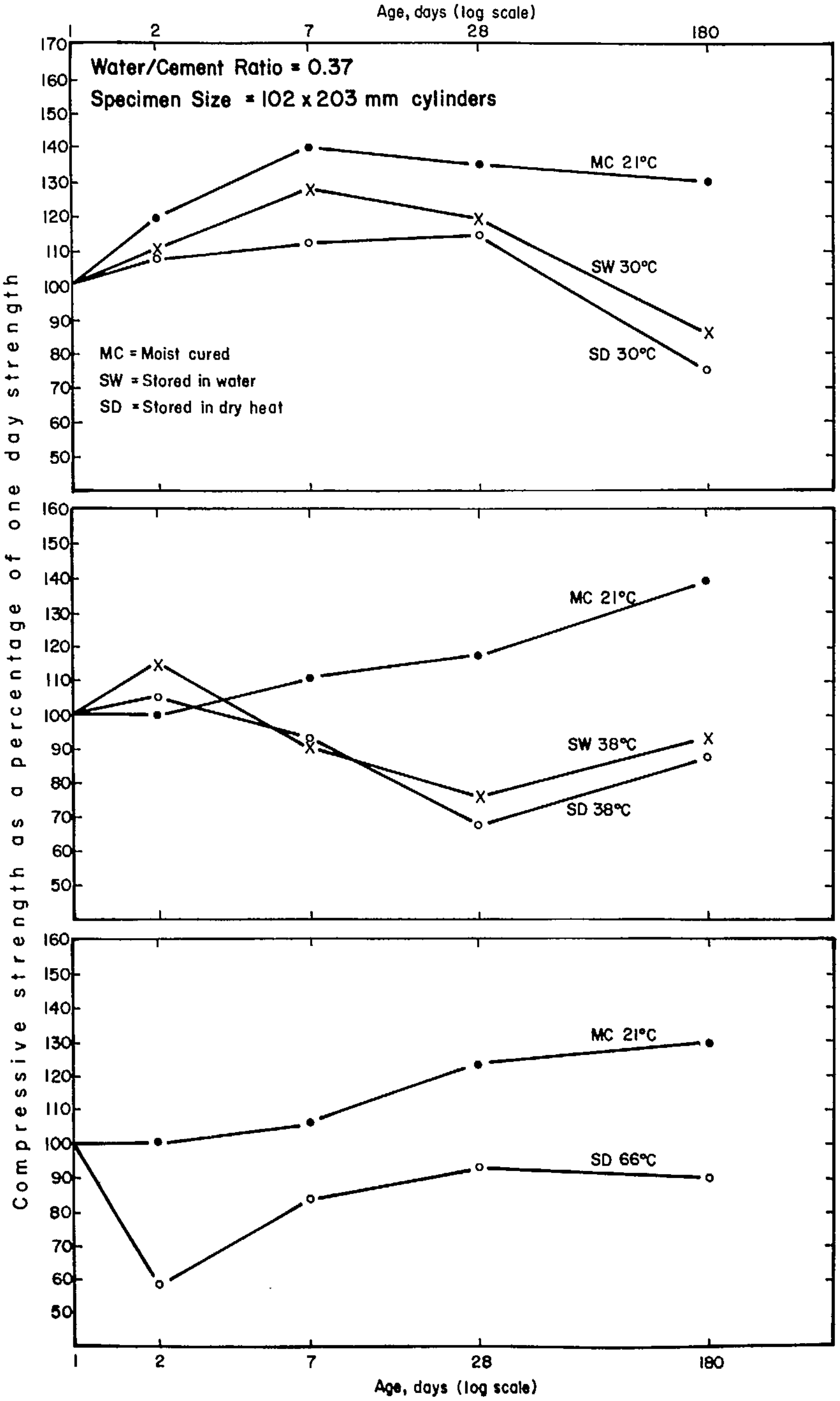


Fig. 12. Relationship between Age and Compressive Strength as a Percentage of One-Day Strength - W/C = 0.37

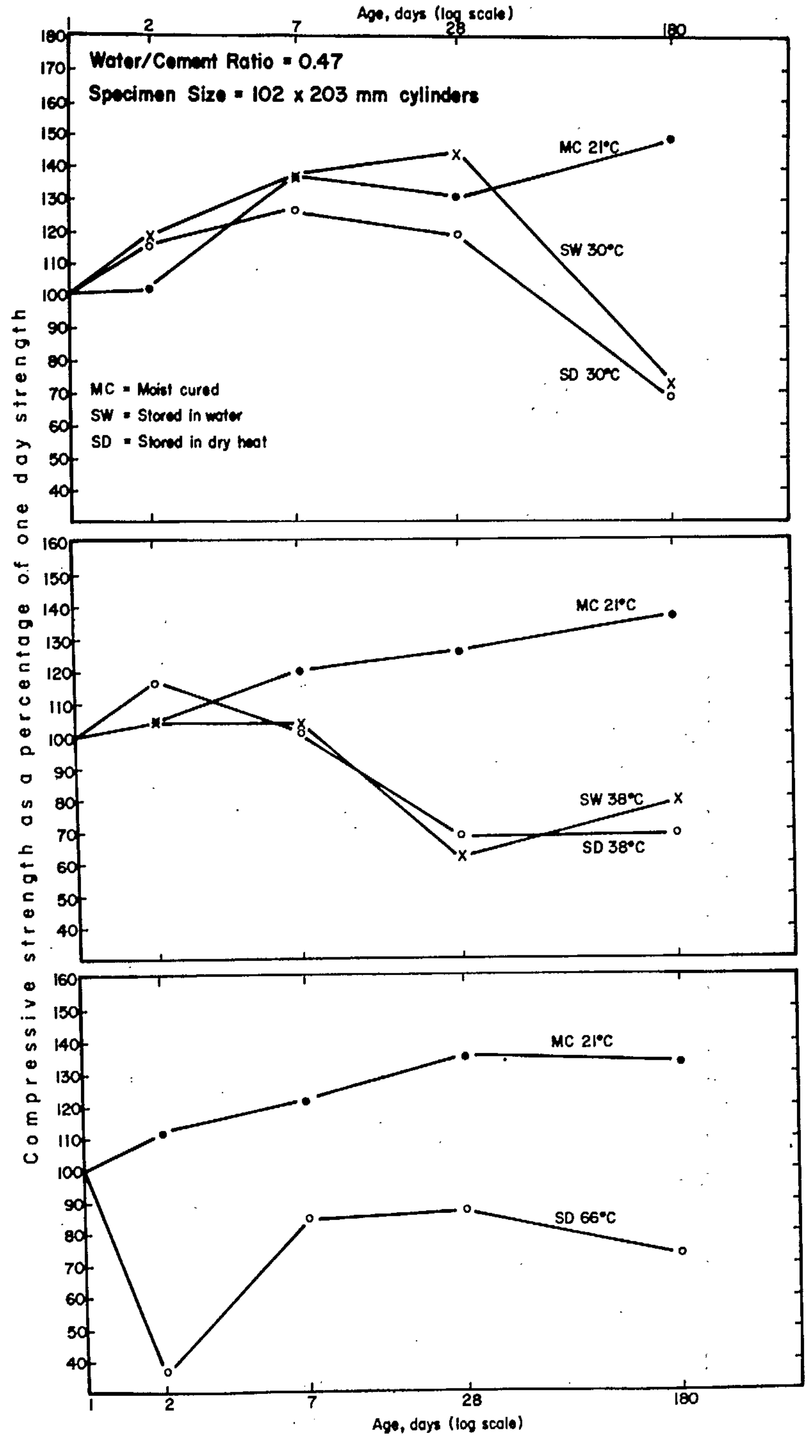


Fig. 13. Relationship between Age and Compressive Strength as a Percentage of One-Day Strength - W/C = 0.47

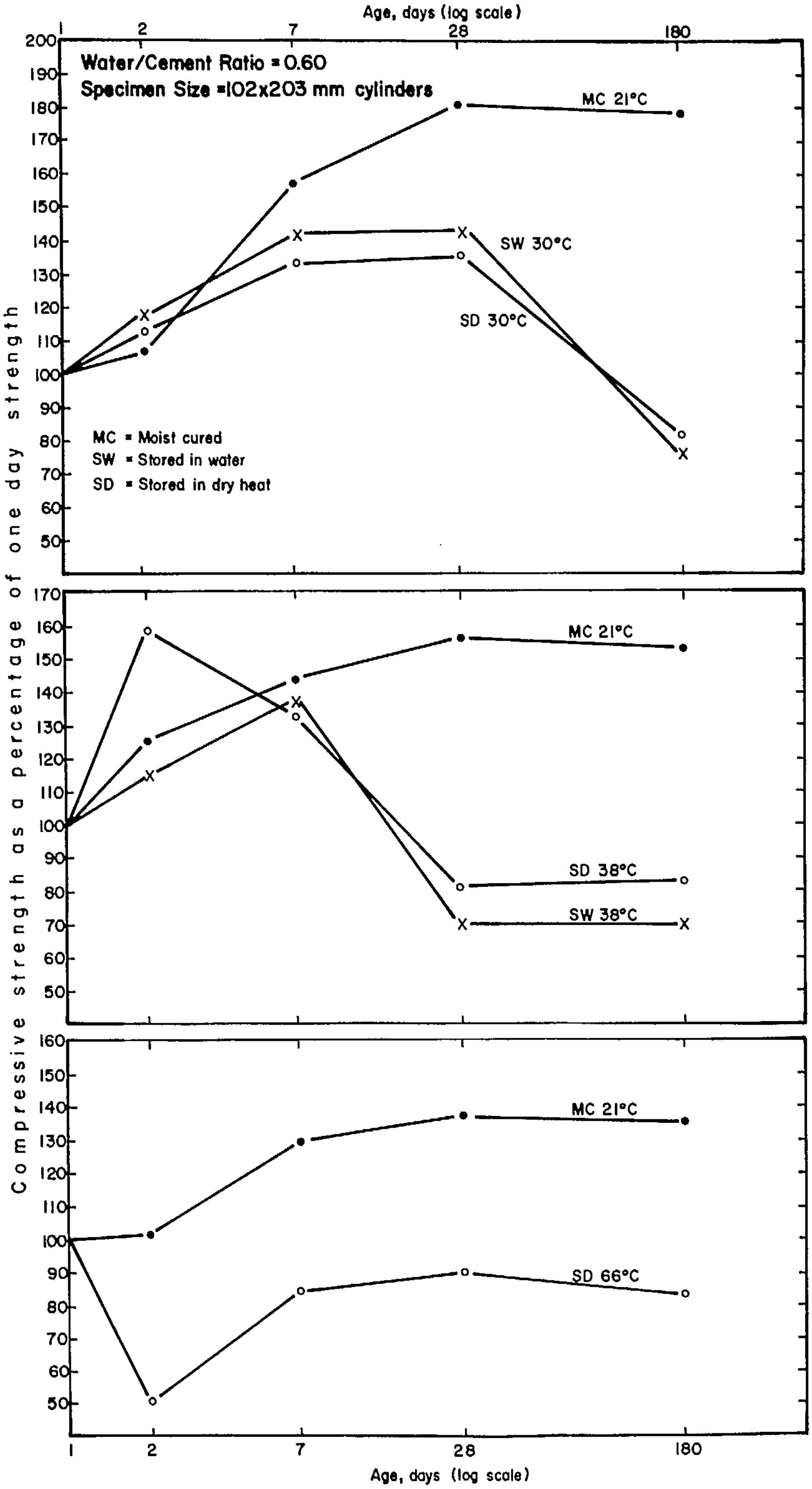


Fig. 14. Relationship between Age and Compressive Strength as a Percentage of One-Day Strength - W/C = 0.60

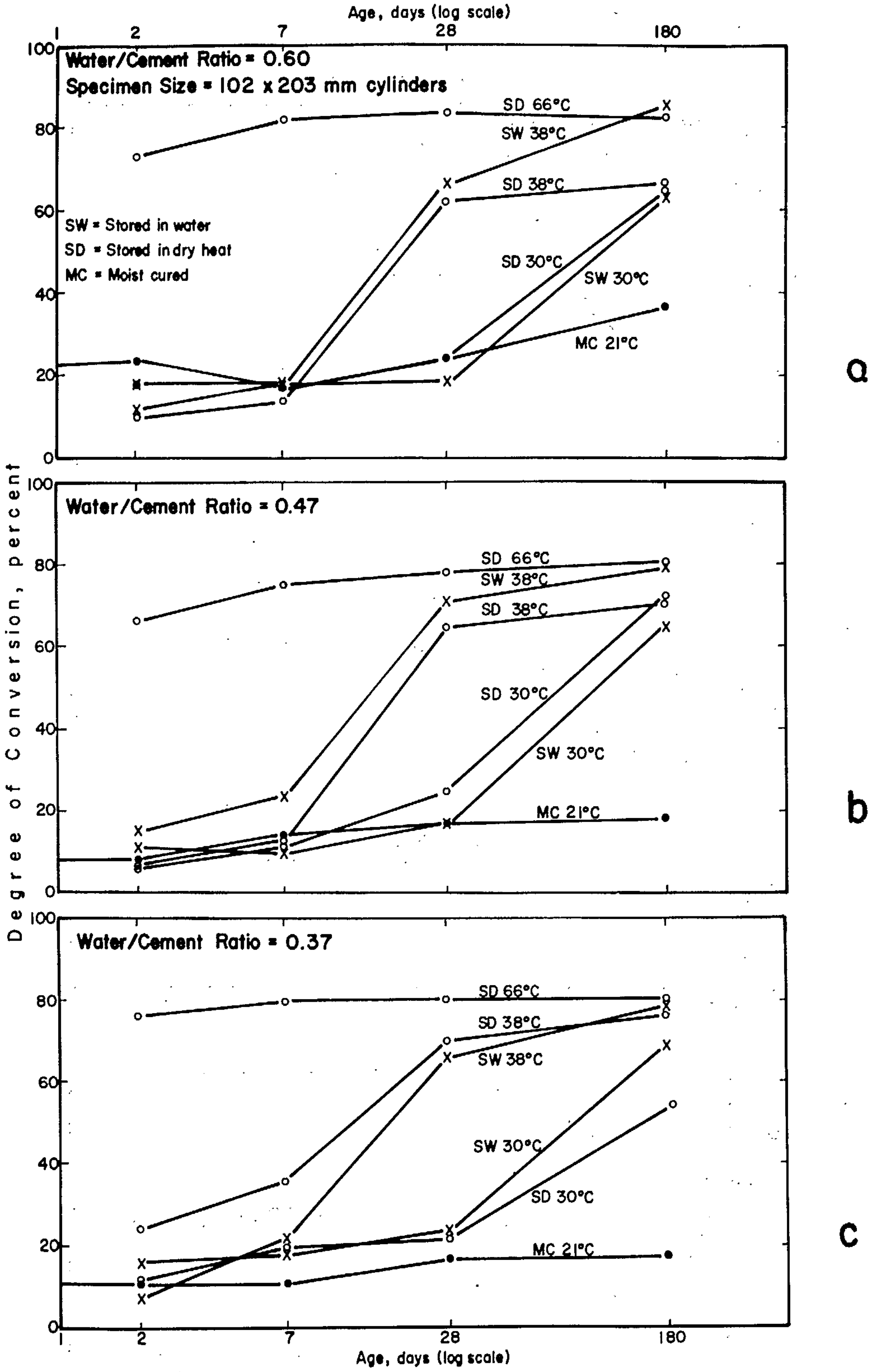


Fig. 15. Relationship between Age and Degree of Conversion of High Alumina Cement Concrete. (a) W/C = 0.60; (b) W/C = 0.47; (c) W/C = 0.37

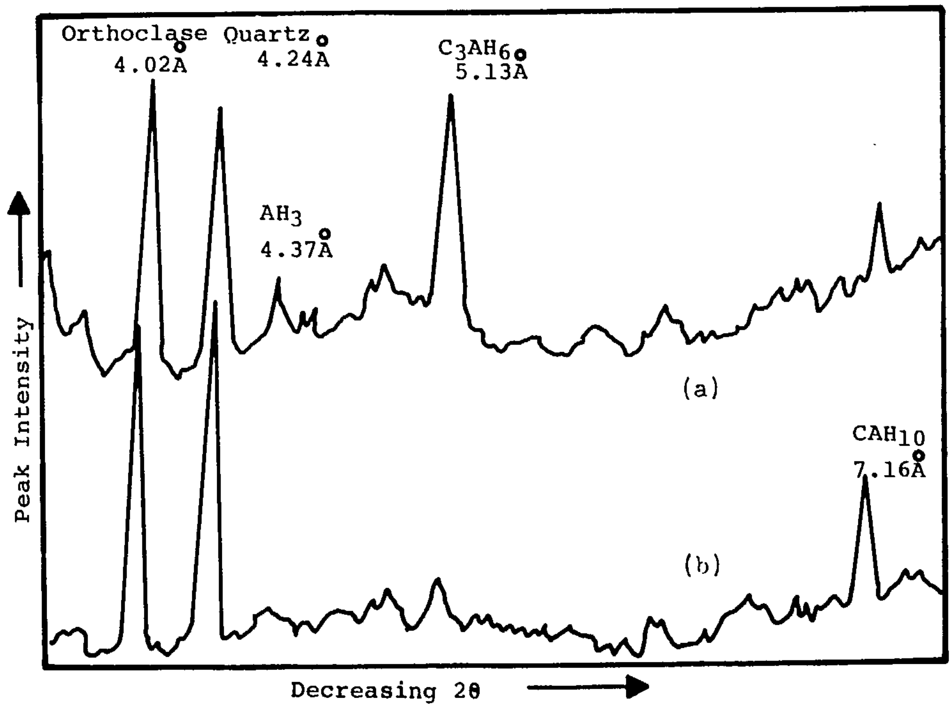


Fig. 16. X-ray Diffraction Patterns of the Unconverted and Converted High Alumina Cement Concrete

(a) Converted Concrete

(b) Unconverted Concrete

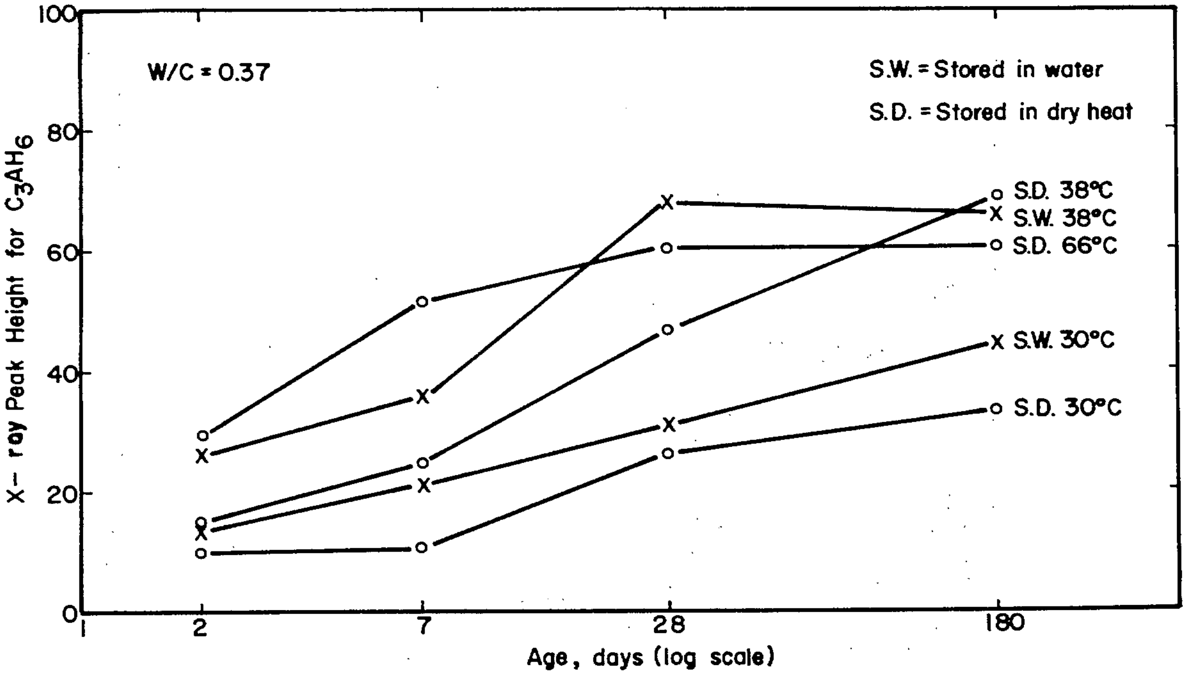


Fig. 17. X-ray Peak Intensity for C_3AH_6 at 5.13Å for High Alumina Cement Concrete After Various Storage Conditions

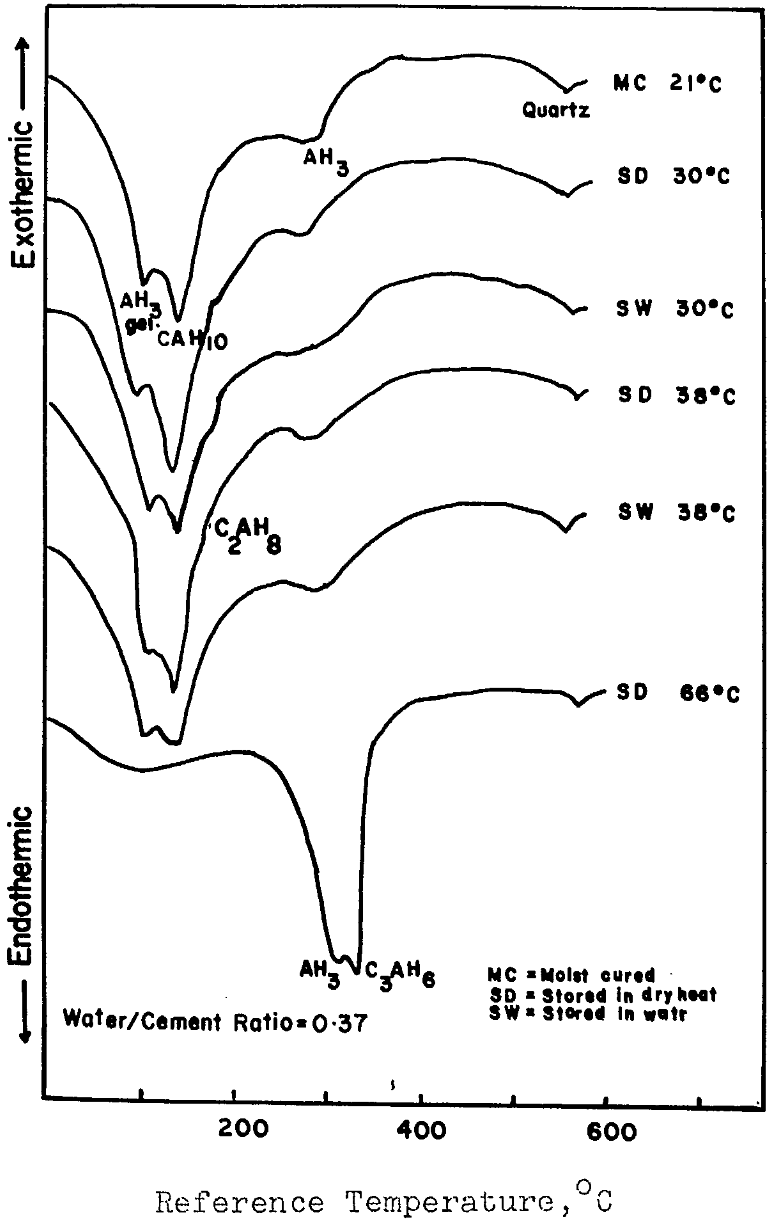


Fig. 18. Thermograms of 2-Day-Old Unconverted and Converted High Alumina Cement Concrete After Various Storage Conditions - W/C = 0.37

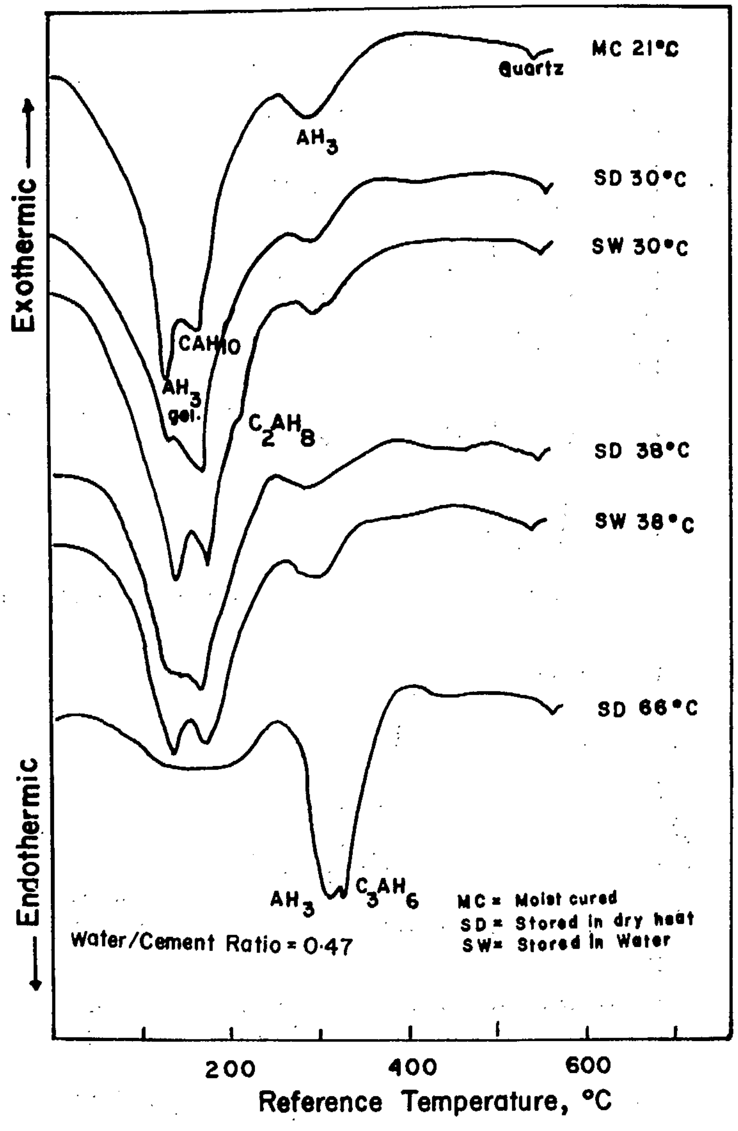


Fig. 19. Thermograms of 2-Day-Old Unconverted and Converted High Alumina Cement Concrete, After Various Storage Conditions - W/C = 0.47

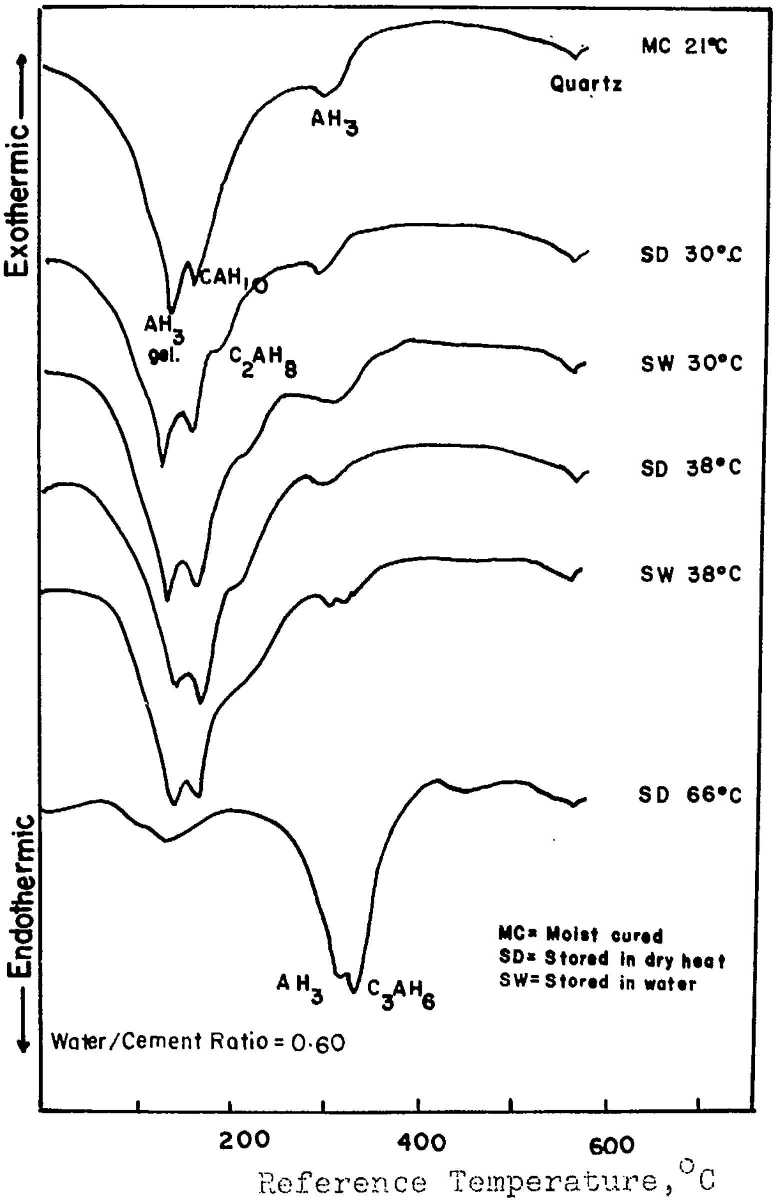
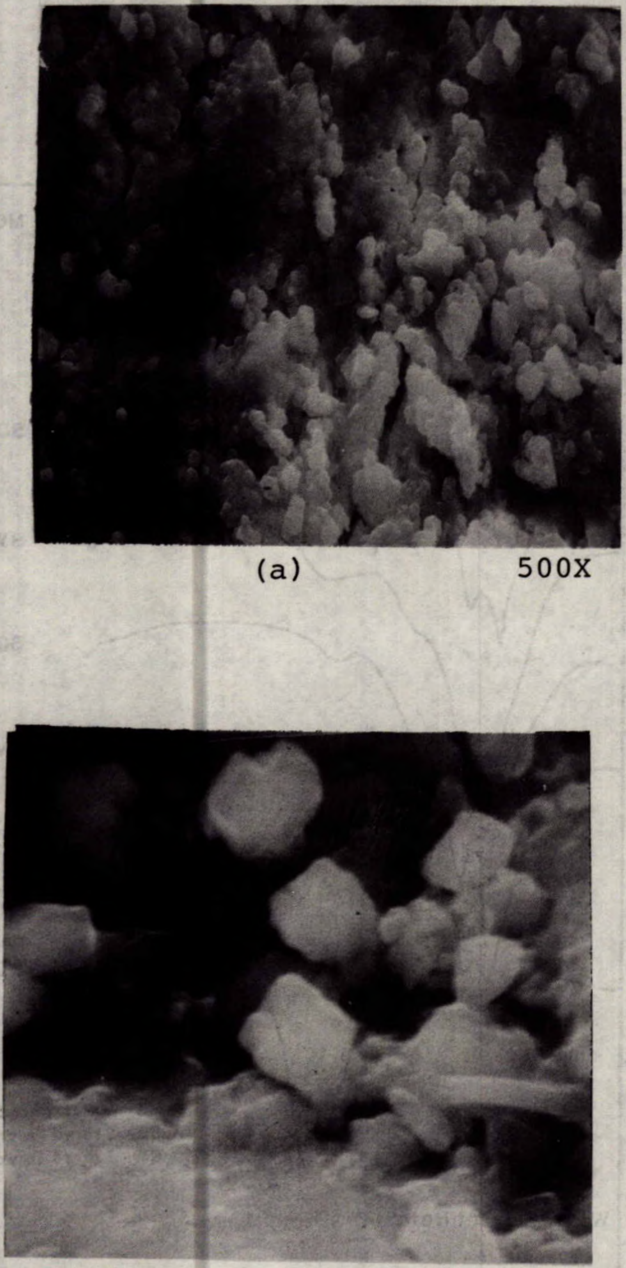


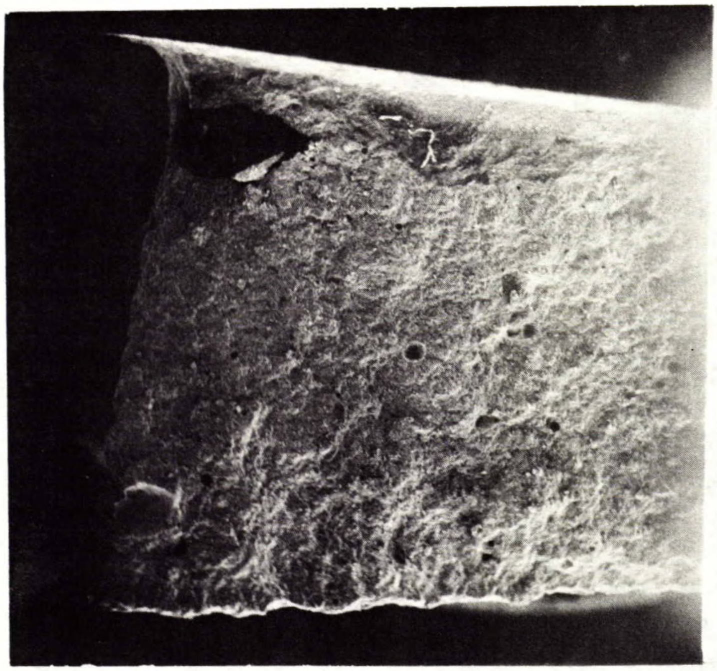
Fig. 20. Thermograms of 2-Day-Old Unconverted and Converted High Alumina Cement Concrete After Various Storage Condition - W/C = 0.60



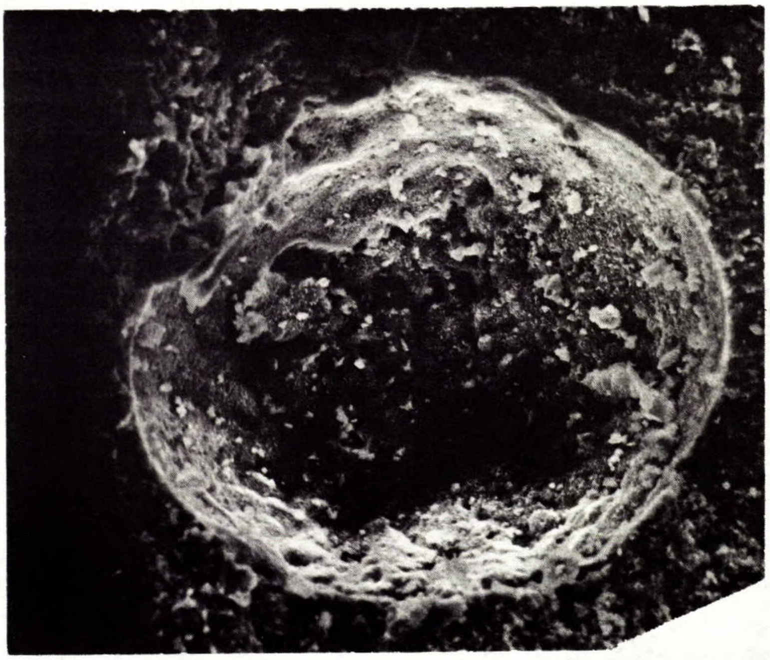
(a) 500X

(b) 700X

Fig. 21. SEM Micrographs of Converted High Alumina Cement Concrete (W/C = 0.37) cured at 66°C Under Dry Heat Conditions for 28 Days



(a) 190X



(b) 450X

Fig. 22. SEM Micrographs of Unconverted High Alumina Cement Paste - W/C = 0.25

(a) 5000X

(b) 9000X

Fig. 23. SEM Micrographs of Unconverted High Alumina Cement Paste (W/C = 0.25) Under Increasing Magnification

CANMET REPORTS

Recent CANMET reports presently available or soon to be released through Printing and Publishing, Supply and Services, Canada (addresses on inside front cover), or from CANMET Publications Office, 555 Booth Street, Ottawa, Ontario, K1A 0G1:

Les récents rapports de CANMET, qui sont présentement disponibles ou qui ce seront bientôt, peuvent être obtenus de la direction de l'Imprimerie et de l'Edition, Approvisionnements et Services, Canada (adresses au verso de la page couverture), ou du Bureau de Vente et distribution de CANMET, 555 rue Booth, Ottawa, Ontario, K1A 0G1:

77-8 Index of commercial flocculants - 1977; H.A. Hamza and J.L. Picard;
Cat. No. M38-13/77-8, ISBN 0-660-01152-2; Price: $1.00 Canada, $1.20 other countries.

77-14 Pit slope manual - Chapter 7 - Perimeter blasting; P. Calder;
Cat. No. M38-14/7-1977, ISBN 0-660-01011-9; Price: $2.25 Canada, $2.70 other countries.

77-20 A pilot plant investigation of thermal hydrocracking of Athabasca bitumen - Effect of recycle of heavy oil on product quality; C.P. Khulbe, B.B. Pruden, J.M. Denis and W.H. Merrill;
Cat. No. M38-13/77-20, ISBN 0-660-0098; Price: $1.50 Canada, $1.80 other countries.

77-21 An apparatus for measurement of thermal diffusivity in air; V.V. Mirkovich;
Cat. No. M38-13/77-21, ISBN 0-662-00746-8; Price: $1.25 Canada, $1.50 other countries.

77-25 Pit slope manual - Supplement 3-1 - Laboratory classification tests; M. Gyenge;
Cat. No. M38-14/3-1977-1, ISBN 0-660-00995-1; Price: $1.25 Canada, $1.50 other countries.

77-26 Pit slope manual - Supplement 3-2 - Laboratory tests for design parameters; M. Gyenge and G. Herget;
Cat. No. M38-14/3-1977-2, ISBN 0-660-00996-X; Price: $1.50 Canada, $1.80 other countries.

77-27 Pit slope manual - Supplement 3-3 - In situ field tests; M. Gyenge and B. Ladanyi;
Cat. No. M38-14/3-1977-3, ISBN 0-660-00997-8; Price: $1.25 Canada, $1.50 other countries.

77-28 Pit slope manual - Supplement 3-4 - Selected soil tests; M. Gyenge, B. Ladanyi;
Cat. No. M38-14/3-1977-4, ISBN 0-660-0098-6; Price: $1.50 Canada, $1.80 other countries.

77-29 Pit slope manual - Supplement 3-5 - Sampling and specimen preparation; M. Gyenge and B. Ladanyi;
Cat. No. M38-14/3-1977-5, ISBN 0-660-00999-4; Price: $1.50 Canada, $1.80 other countries.

77-32 Pilot plant thermal hydrocracking of GCOS (Great Canadian Oil Sands) bitumen. 3. Effect of heavy oil recycle on reactor fouling; C.P. Khulbe, B.B. Pruden, J.M. Denis and W.H. Merrill;
Cat. No. M38-13/77-32, ISBN 0-660-0069-1; Price: $1.00 Canada, $1.20 other countries.

77-34 X-ray fluorescence on-line analysis of lead-zinc ore fractions in mineral slurries; A comparative study between wavelength-dispersive and energy-dispersive techniques; S.K. Kawatra and J.L. Dalton;
Cat. No. M38-13/77-34, ISBN 0-660-01151-4; Price: $1.00 Canada, $1.20 other countries.

77-35 Hydrocracking of Athabasca bitumen: Effect of coal-base catalyst on pilot plant operation; C.P. Khulbe, B.B. Pruden and J.M. Denis;
Cat. No. M38-13/77-35, ISBN 0-660-01081-X; Price: $1.00 Canada, $1.20 other countries.

77-37 Evaluation of a two-stage thermal and catalytic hydrocracking process for Athabasca bitumen; R. Ranganathan, B.B. Pruden, M. Ternan and J.M. Denis;
Cat. No. M38-13/77-37, ISBN 0-660-01157-3; Price: $1.00 Canada, $1.20 other countries.

77-43 A review of some chemical aspects of the formation of coke during thermal hydrocracking of bitumen; K. Belinko and J.M. Denis;
Cat. No. M38-13/77-43, ISBN 0-660-01158-1; Price: $1.00 Canada, $1.20 other countries.