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HIGH-TEMPERATURE BEHAVIOUR OF ALUMINOUS CEMENT CONCRETES CONTAINING DIFFERENT AGGREGATES

N. G. ZOLDNERS, V. M. MALHOTRA
& H. S. WILSON

MINERAL PROCESSING DIVISION

JULY 1963

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by

N. G. Zoldners*, V. M. Malhotra** and H. S. Wilson***

SYNOPSIS

Changes in physical properties of about 400 concrete test specimens, made with aluminous cement and expanded shale, anorthosite, ilmenite and phonolite aggregates, were studied after exposure to temperatures ranging from 100 to 1100°C. It was found that the aluminous cement concrete loses its strength rapidly in the range of temperatures investigated. Expanded shale aggregate concrete showed the greatest stability, retaining about 20 per cent of its original compressive strength after firing at 1000°C, whereas concrete made with phonolite aggregate was the least heat-resistant of the four types investigated.

The fired concrete specimens were tested for strength and examined petrographically. Probable causes of reduction in strength due to exposure to elevated temperatures are discussed briefly.

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*Head, **Scientific Officer, and ***Senior Scientific Officer, Construction Materials Section, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

A condensed version of this report was presented at the 66th Annual Meeting of the American Society for Testing and Materials, Atlantic City, N. J., on June 24, 1963.

Direction des mines

Rapport de recherches R 109

COMPORTEMENT AUX TEMPÉRATURES ÉLEVÉES DE BÉTONS PRÉPARÉES À L'AIDE DE CIMENT ALUMINEUX ET DE DIVERS AGRÉGATS

par

N.G. Zoldners*, V.M. Malhotra** et H.S. Wilson***

RÉSUMÉ

On a étudié les changements subis par les propriétés physiques d'environ 400 échantillons expérimentaux de béton préparé à l'aide de ciment alumineux et de schiste expansé ainsi que d'agrégats d'anorthosite, d'ilménite et de phonolite, après exposition des échantillons à des températures échelonnées entre 100 et 1,100°C. On a constaté que le béton préparé à l'aide de ciment alumineux perd rapidement sa résistance aux températures susmentionnées. Le béton contenant l'agrégat de schiste expansé est demeuré le plus stable, conservant environ 20 p. 100 de sa résistance primitive à la compression sous une température de 1,000°C, tandis que le béton contenant l'agrégat de phonolite a été celui des quatre bétons étudiés qui s'est le plus mal comporté sous l'action de la chaleur.

Les échantillons de béton soumis à l'action de la chaleur ont fait l'objet d'essais destinés à en déterminer la résistance; ils ont également été examinés pétrographiquement. Les auteurs étudient brièvement les causes probables de la perte de résistance du béton exposé à des températures élevées.

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*Chef, **chargé de recherches et ***chargé de recherches principal, Section des matériaux de construction, Division du traitement des minéraux, Direction des mines, ministère des Mines et des Relevés techniques, Ottawa, Canada.

Une version abrégée du présent rapport a été présentée lors de la 66e Réunion annuelle de l'American Society for Testing and Materials, à Atlantic City (N.J.), le 24 juin 1963.

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INTRODUCTION

Extensive use of concrete for industrial installations exposed to elevated temperatures requires heat-resistant materials. The need for such materials is particularly great in the chemical and metallurgical industries, and for the thermal shieldings of nuclear power plants.

Concretes made with hydraulic cements and conventional aggregates are designed for service in normal environmental conditions. The heat-resistance of these concretes is greatly limited and is governed mainly by the types of cement and aggregate used.

A literature survey revealed that, until recently, little information was available on the effect of dry heat at relatively low temperatures (100 to 300°C) on the properties of concrete made with aluminous cement.

One of the earliest publications in this field was a study by Miller and Faulkner (1927) (1)*, indicating a surprising reduction in strength of aluminous cement concrete after heating.

Recently, relevant studies were published by the Universal Atlas Cement Division of United States Steel Corp., Research Laboratories, Gary, Ind., U.S.A. (2, 3), and by the Lafarge Aluminous Cement Company Limited, London, England (4, 5, 6).

A considerable amount of work has been carried out in Russia during the past five years by the Institute of Scientific Research for Concrete and Reinforced Concrete, at the Academy of Building and Architecture, USSR (7, 8, 9, 10).

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* The numbers in parentheses refer to the list of references appended to this paper.

SCOPE OF RESEARCH

The purpose of this study was to investigate the physical properties of aluminous cement concretes after exposure for one hour to temperatures ranging from 100 to 1000°C. Four aggregates were used having widely different mineralogical and physical properties. These consisted of a manufactured lightweight aggregate, a heat-resistant coarse-grained basic igneous rock, a commercially produced heavy aggregate, and a fine-grained feldspathoidal igneous rock exhibiting some unusual properties.

This study is part of an extensive research project (11, 12, 13) being undertaken by the Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada, to determine the effect of different types of cements and aggregates on concrete exposed to dry heat at elevated temperatures. As part of this programme, the heat-resistant properties were also investigated of some of the rocks being used in this study as aggregate (14, 15). Other thermal properties, such as fusion temperature and thermal elongation, have been studied on a wide variety of rocks by others at the Mines Branch in the course of their investigations.

MATERIALS

Cement

A well known commercially available brand of aluminous, or calcium aluminate, cement was used in this investigation.

The chemical composition of this type of cement is entirely different from that of portland cement. Table 1 gives the chemical composition of the cement used and compares it with typical aluminous cements from other countries, and with normal portland cement Type I.

The principal cementitious constituent in commercial aluminous cements is monocalcium aluminate ($CaO.Al_2O_3$) (3), whereas in portland cements the most important minerals are tricalcium and dicalcium silicates.

Standard specifications for aluminous cements have been drawn up in several European countries. However, there is no uniformity in the chemical characteristics required by these specifications. There is no Standard Specifications for this type of cement in the United States.

Cement used in this work was of a dark grey colour due to the higher iron content, and was coarser ground than Type I portland cement.

TABLE 1

Chemical Analyses of Aluminous and Portland Cements

Chemical Constituents (oxides)	Alum. Cement Used*	Typical Aluminous Cements From Different Countries**			Type I Portland Cement***
		England	Germany	U. S. A.	
	%	%	%	%	%
SiO_2	4.50	4-5	5-8	8-9	19-23
Al_2O_3	39.74	48-40	48-51	40-41	5-8
Fe_2O_3	9.98	8-10	0.1	5-6	2-4
CaO	32.52	36-39	39-42	36-37	62-66
MgO	0.12	1.0	1.0	1.0	1-4
SO_3	0.13	0.1	0.5	0.2	1-2.5
FeO	5.40	5-7	1.0	5-6	
TiO_2	2.03	2.0	1.5	2.0	

*Mines Branch Internal Report MS-AC 62-293.
**Ref. 6, p. 41.
***Ref. R. F. Blanks and H. L. Kennedy, "The Technology of Cement and Concrete", Vol. 1, p. 28.

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Aggregates

Graded fine and coarse fractions were prepared from each of the four types of aggregates used. These are described below:

(1) Expanded shale was manufactured in a lightweight aggregate plant in Ontario. The aggregate was produced in a rotary kiln from a grey Carlsbad shale contaminated slightly by irregularly spaced bands of impure limestone. Coarse material was received as produced, but fines were crushed. Grading was done by screening.

(2) Anorthosite was obtained from a building stone quarry in Quebec. The material was received as crushed rock with a maximum size of about 6 in. The blue-grey rock was coarse-grained (1 cm) and consisted principally of plagioclase crystals and altered olivine. Material was crushed and screened into coarse and fine aggregate fractions.

(3) Ilmenite rock was supplied by a commercial producer of heavy aggregate from a quarry in Quebec. The crushed rock was received in two sizes, minus 1 in. plus 4 mesh, and minus 4 mesh material. It consisted predominantly of ilmenite, $FeTiO_3$, which is black in colour and has a metallic lustre.

(4) An igneous rock, traded as "trap rock", was received from a commercial producer in Quebec. It was supplied as coarse aggregate in graded sizes; fine aggregate had to be obtained by crushing and screening. This fine-grained grey rock was identified as phonolite (tinguaite), which is composed essentially of alkali feldspar, nepheline, natrolite (alteration product of nepheline), and soda pyroxene (19).

A detailed petrographic study was made on each of the four types of aggregates used. The results of these studies are summarized in Table 2.

TABLE 2

General Petrographic Features of Aggregates*

Aggregate	Petrography
Expanded Shale	Medium grey, externally rusty, low density, bloated fragments showing primary shale banding; highly vesicular, fused appearance.
Anorthosite	Medium- to coarse-grained (1-10 mm), blue-grey rock, consisting of plagioclase $Ab_{50}An_{50}$ (95%) and scattered crystals of uralitized olivine (5%). Relict crystals of olivine are surrounded by an inner lining of enstatite and an outer coat of chloritized hornblende.
Ilmenite Rock	Medium- to coarse-grained (2-10 mm), consisting of crystals of black ilmenite (85%); granules of plagioclase $Ab_{50}An_{50}$ (10%) and spinel (1%); books and layers of biotite (1%) and green mica or chlorite (2%); and grains of pyrite (1%). The green mica surrounds and fills cracks in the plagioclase.
Phonolite (Tinguaite)	Fine-grained (1-3 mm), light-grey rock, occasional dark bands. The approximate composition is: K feldspar (30%); nepheline (25%); natrolite (20%); albite (15%); and acmite (10%). The rock has a microscopic trachytic texture.

* After J. A. Soles.

Some physical properties of the four aggregates and the proportions of coarse to fine fractions in the concrete mixes are shown in Table 4.

The gradings of the coarse and fine aggregate used for the various materials are shown in Table 3.

TABLE 3

Grading of Aggregates

Coarse Aggregates					Fine Aggregates				
Sieve Size	Per Cent Passing				Sieve Size	Per Cent Passing			
	Expanded Shale	Anorthosite	Ilmenite	Phonolite		Expanded Shale	Anorthosite	Ilmenite	Phonolite
1 in.	-	100.0	100.0	-	No. 4	100.0	100.0	100.0	100.0
3/4 in.	100.0	95.0	95.0	100.0	No. 8	85.0	92.0	90.0	90.0
1/2 in.	65.0	65.0	65.0	56.0	No. 16	70.0	74.4	67.5	67.5
3/8 in.	30.0	35.0	35.0	37.5	No. 30	45.0	52.2	42.5	42.5
No. 4	0.0	0.0	0.0	0.0	No. 50	30.0	31.4	20.0	20.0
					No.100	20.0	10.8	6.0	6.0

TABLE 4

Physical Properties of Aggregates

Properties	Exp. Shale		Anorthosite		Ilmenite		Phonolite	
	C.A.*	F.A.**	C.A.	F.A.	C.A.	F.A.	C.A.	F.A.
Specific gravity, bulk, SSD†	1.17	1.83	2.77	2.71	4.65	4.67	2.54	2.55
Absorption, per cent by wt.	10.60	-	0.40	0.50	0.30	0.50	0.40	0.50
Mix proportions, per cent (by weight)	40	60	57	43	60	40	52	48

* C.A. = coarse aggregate; ** F.A. = fine aggregate; †SSD = saturated, surface-dry.

CONCRETE MIXES

In order to make the needed number of test specimens, a minimum of six concrete mixes was required for each type of aggregates. A total of 24 mixes was prepared between January and November, 1962. The mixing and moulding of test specimens were done in the mornings between 9:00 to 11:00 o'clock.

Design of Mixes

The same basic mix design was used in all the mixes. It was calculated by the absolute volume method in accordance with the following conditions:

(a) The cement content was kept constant at 480 lb per cu yd of concrete for all the text mixes.

(b) The water-cement ratio (W/C) and the ratio of coarse to fine aggregate were kept constant for each type of concrete.

(c) Air-entraining agent (Darex) was used for the expanded shale mixes. All other mixes were non-air-entrained.

Apart from the air-entraining agent, no other admixtures such as plasticizers or densifiers were used for any of the concretes, because little is known about the effect of additives on aluminous cement concretes (20).

Preparation of Mixes

Each mix was prepared as a 2 cu ft batch, using a counter-current concrete mixer. The total mixing time for each batch was 6 min.

The room-dry coarse and fine aggregate was soaked in water 24 hr prior to its use.

The actual mix proportions used in these test mixes are given in Table 5.

TABLE 5

Concrete Mix Data

Type of Concrete Aggregate	Average Mix Proportions, per cu yd					
	Cement, lb	SSD Aggregates		Water, lb	Admixture, oz	W/C* ratio
		Coarse, lb	Fine, lb			
Expanded Shale	480	704	1041	346	Darex-6	0.718
Anorthosite	482	1828	1346	342	nil	0.710
Ilmenite	471	3323	2224	333	nil	0.706
Phonolite	479	1550	1445	347	nil	0.725

* W/C = Water-cement ratio, by weight.

Characteristics of Fresh Concrete

The concrete mixes were dark in colour, due to the darker shade of the aluminous cement. The considerable harshness of the mixes was significant, because of the relatively coarse grind of the cement (16). Ilmenite and anorthosite concrete mixes were slightly undersanded and their plasticity was rather low.

The expanded shale mixes showed a tendency to lose slump rapidly, although the aggregates were soaked for 24 hr.

Some of the measured properties of the fresh concrete, namely the slump, air content and unit weight, were determined using ASTM standard testing methods*.

The test results are summarized in Table 6.

TABLE 6

Properties of Fresh Concrete

Type of Concrete Aggregate	W/C ratio	Slump, in.	Air, %	Unit Weight, lb/cu ft
Expanded Shale	0.718	1¾	5.0[a]	94.3
Anorthosite	0.710	1½	3.1	148.0
Ilmenite	0.706	1	0.7	234.9
Phonolite	0.725	1	1.2	141.5

a — Air-entraining agent used.

Test Specimens

Eighteen test specimens, consisting of nine 4 x 8 in. cylinders and nine 3½ x 4 x 16 in. beams, were cast from each mix. Three cylinders and three beams from each mix were used as reference specimens. Six cylinders and six beams were kept for two firing exposures, using three specimens of each kind

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* ASTM Standard Methods of Tests for Slump of Portland Cement Concrete (C 143-58), Air Content of Freshly Mixed Concrete by the Pressure and Volumetric Methods (C 231-60 and C 173-58), and Weight Per Cubic Foot (C 138-44).

for every firing. To provide test specimens for ten temperature exposures, five mixes were required.

In addition to the mixes for fire test specimens, one more mix was made for each type of aggregate to provide two 3 x 13 x 17½ in. slabs for thermal conductivity studies.

Table 7 gives a summary of various test specimens prepared from each type of concrete.

TABLE 7

Summary of Test Specimens

Type of Concrete Aggregate	Number of Batches	Number of Specimens For Each Type of Concrete			Total No. of Specimens
		4x8 in. cylinders	3½x4x16 in. beams	3x13x17½ in. slabs	
Expanded Shale	5	45	45	-	90
	1	-	-	2	2
Anorthosite	5	45	45	-	90
	1	-	-	2	2
Ilmenite	5	45	45	-	90
	1	-	-	2	2
Phonolite	5	45	45	-	90
	1	-	-	2	2
	24	180	180	8	368

Moulding and Curing of Test Specimens

The test cylinders were cast in aluminum and the beams in heavy brass moulds with 3/8 in. side plates and 1/2 in. end plates. Stainless steel reference plugs were cast in each end of the beam specimens for length measurements.

Test specimens were moulded by placing concrete in the moulds in two layers and vibrating them on a vibrating table for 10 and 20 sec for each layer. These vibrating times had to be doubled for the expanded shale mixes, because of the poor workability of that concrete. When all test specimens were cast, each mould was topped with a glass plate and covered by water-saturated burlap. The moulded specimens were left in the casting room for 24 hr, during which period the ambient room temperature varied from 17 to 27°C. The temperature was recorded four times: three during the first six hours, and one during the next morning. The temperature ranges recorded for all mixes are shown in Table 8.

TABLE 8

Temperature in the Casting Room

Type of Concrete Aggregate	Date of Mixing	Temperature Range in Casting Room for the First 24 Hr, deg Cent.
Anorthosite	Jan. 11-30,1962	17 to 28
Phonolite	Apr. 25-May 4,1962	20 to 27
Expanded Shale	Sept.11-19, 1962	20 to 24
Ilmenite	Nov. 13-21, 1962	18 to 22

At the end of the initial curing period, the test specimens were removed from the moulds and transferred immediately to the moist curing room at a temperature of 23±2C and 95 per cent relative humidity.

After 7 days, the specimens were removed to a storage room, the ambient temperature of which was 24±°C and the relative humidity 65±5%. Test specimens remained in the dry storage until being fire tested.

PROPERTIES OF HARDENED CONCRETE

The density of the concrete in the saturated, surface-dry condition after 7 days of initial moist curing, and of the room-dry concrete just prior to firing, was determined for each type of concrete on the reference test cylinders. The results are shown in Table 9.

TABLE 9

Concrete Density and Cylinder Compressive Strength

Type of Concrete Aggregate	7 day, SSD	Cylinders in Room-Dry Condition, Before Firing				
	Density*, lb/ cu ft	Age, days	Moisture, %	Density*, lb/cu ft	Strength**, psi	C.V.† %
Expanded Shale	95.0	120	4.2	86.0	2110	4.5
Anorthosite	150.9	180	2.6	145.3	2720	6.9
Ilmenite	236.3	95	1.7	232.3	5960	6.7
Phonolite	142.8	118	3.0	136.9	2680	11.7

* Mean of 5 x 9 = 45 cylinders.
** Mean of 5 x 3 = 15 cylinders.
† Coefficient of Variation.

The "between batch" coefficients of variation (21) for different types of concrete, as calculated for the compressive strengths of the cylinders before firing, are also given in Table 9. These are 4.5, 6.9 and 6.7 per cent for expanded shale, anorthosite and ilmenite concrete mixes, respectively, reflecting excellent to good (22) uniformity among the mixes. The high value of 11.7 per cent for the coefficient for phonolite concrete mixes was due to the low compressive strength of one of the mixes, which was 730 psi lower than the average strength of the other phonolite mixes.

HIGH-TEMPERATURE EXPOSURE

Before test firings were commenced, the 7-day moist-cured specimens were air-dried at the ambient room temperature. The drying period varied from 95 days for ilmenite to 180 days for anorthosite concrete.

During this period, the moisture content of the concretes from SSD condition was appreciably reduced and ranged from 1.7 per cent for ilmenite concrete to 4.2 per cent for expanded shale concrete.

The preconditioning of fire-test specimens by air-drying was necessary to avoid spalling damage of concrete, which may be caused by pressure of the steam driven out during firing tests. Test specimens designated for fire tests were marked with a fire-resistant green chrome paint. All firings were conducted in the furnace described below.

Description of Furnace

A gas-fired, down-draft furnace, with internal dimensions of 20 in. wide, 42 in. deep and 24 in. high, was used to fire the concrete test specimens. It was heated by six burners, three on each side. Heating was controlled by changing the size of the burner tips, by regulating the flow of gas and of natural draft to each burner, and by varying an induced draft, using a damper in the exhaust flue. This created a reducing atmosphere in the furnace.

Two thermocouples, connected to a recording pyrometer, were used to record temperature. One was situated vertically in the centre of the heating chamber of the furnace, above the test specimens, and the other horizontally, immediately below the specimens. A vertical section of the furnace is shown in Figure 1.

Arrangement of Specimens in Furnace

Three cylinders and three beam test specimens were fired at one time. The cylinders were laid on their sides at the back of the heating chamber and the beams were set on their $3\frac{1}{2}$ x 16 in. faces in front of the cylinders. The arrangement is shown in Figure 2.

Preliminary Firing

A preliminary firing with dummy test specimens was made to determine the heating rate of the furnace up to 1000°C. It was found to be close to the standard heating rate specified

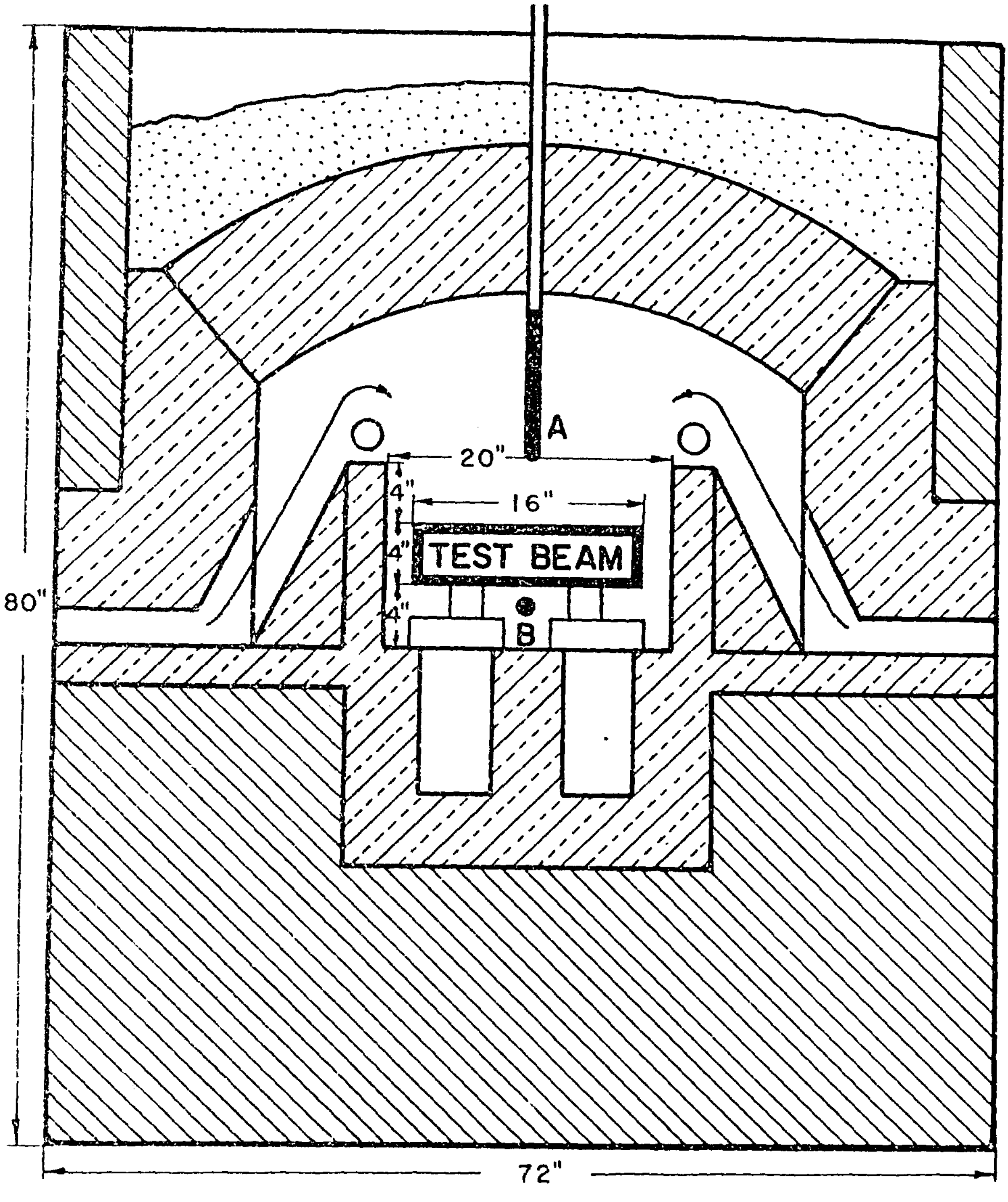

Figure 1. Down-Draft Gas-Fired Furnace. (Vertical cross section)

A. Top thermocouple (vertical)
B. Bottom thermocouple (horizontal)

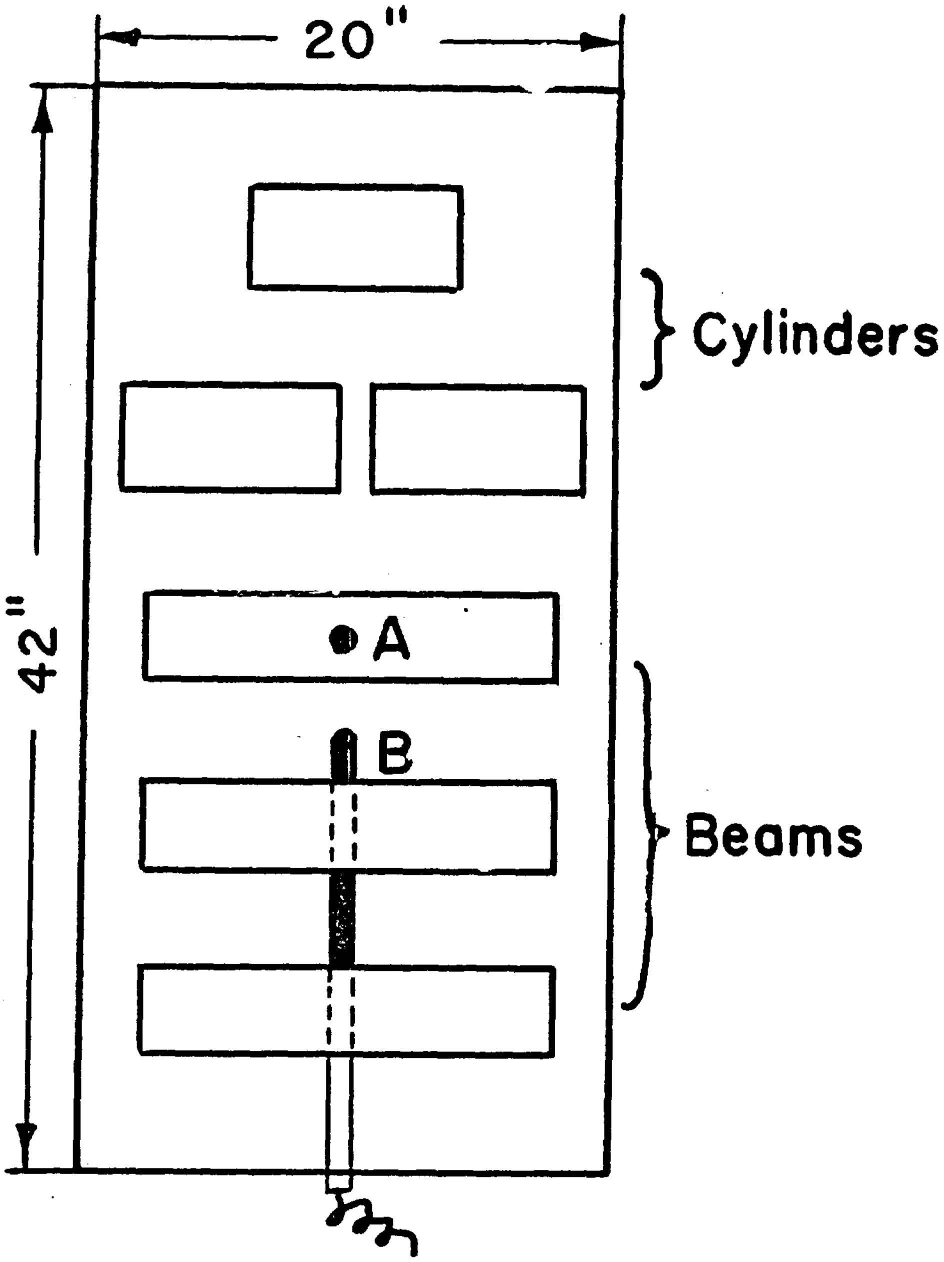

Figure 2 - Arrangement of Test Specimens in Furnace. (Plan)

A. Top thermocouple (vertical)
B. Bottom thermocouple (horizontal)

by ASTM designation E119-61*. The recorded heating curves obtained in the preliminary firing, and the standard time-temperature curves, are shown in Figure 3.

Firing Procedure

The test specimens from each type of concrete were fired at temperatures in 100-degree increments up to 1000°C. Slight deviations from this procedure were made where necessary, either by omitting a certain firing temperature, or by making additional firings at intermediate temperatures. For example, no firing was carried out at 100 and 900°C on phonolite concrete, whereas on anorthosite concrete, additional firings were made at 150 and 1100°C.

In each firing, the specimens were heated to the desired temperature and held at that temperature for one hour. Of the two thermocouples, the bottom one showed temperatures 100 to 250 degrees lower than those shown by the top one. The nominal temperature of the firing environment was assumed to be that shown by the bottom thermocouple. The top thermocouple measured the temperature of the combustion gases before they reached the test specimens. It decreased gradually during the heat-soaking period. Figure 4 shows typical temperature records.

After firing, the test specimens were left in the closed furnace to cool slowly. When they had cooled overnight to about 100°C, they were placed in a desiccator to bring them to ambient room temperature in about 4 hours, and held there until being tested.

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* Standard Methods of Fire Tests of Building Construction and Materials (E119-61), 1961 Book of ASTM Standards, Part 5, p. 1135.

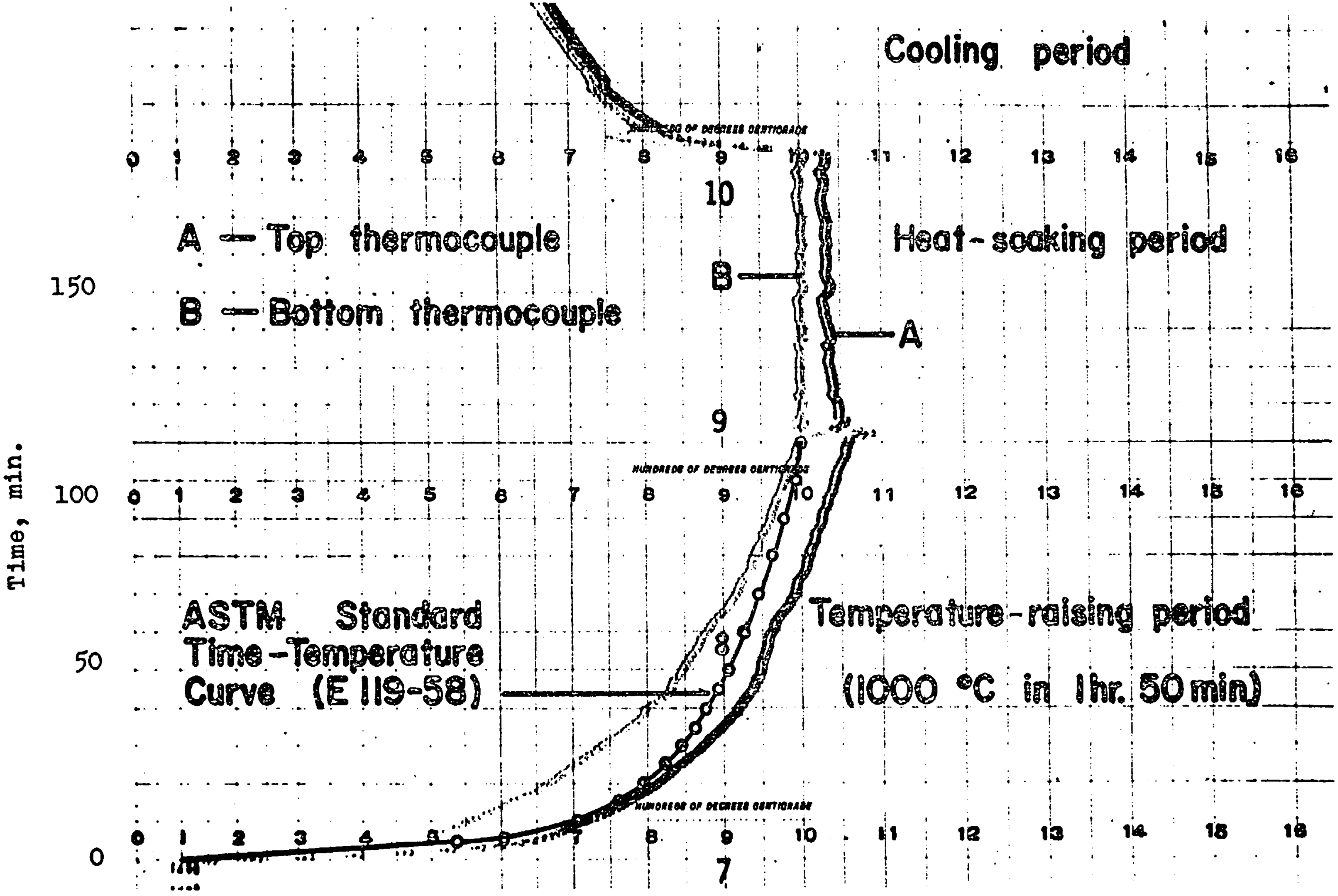

Figure 3. Preliminary Firing - Temperature Record.

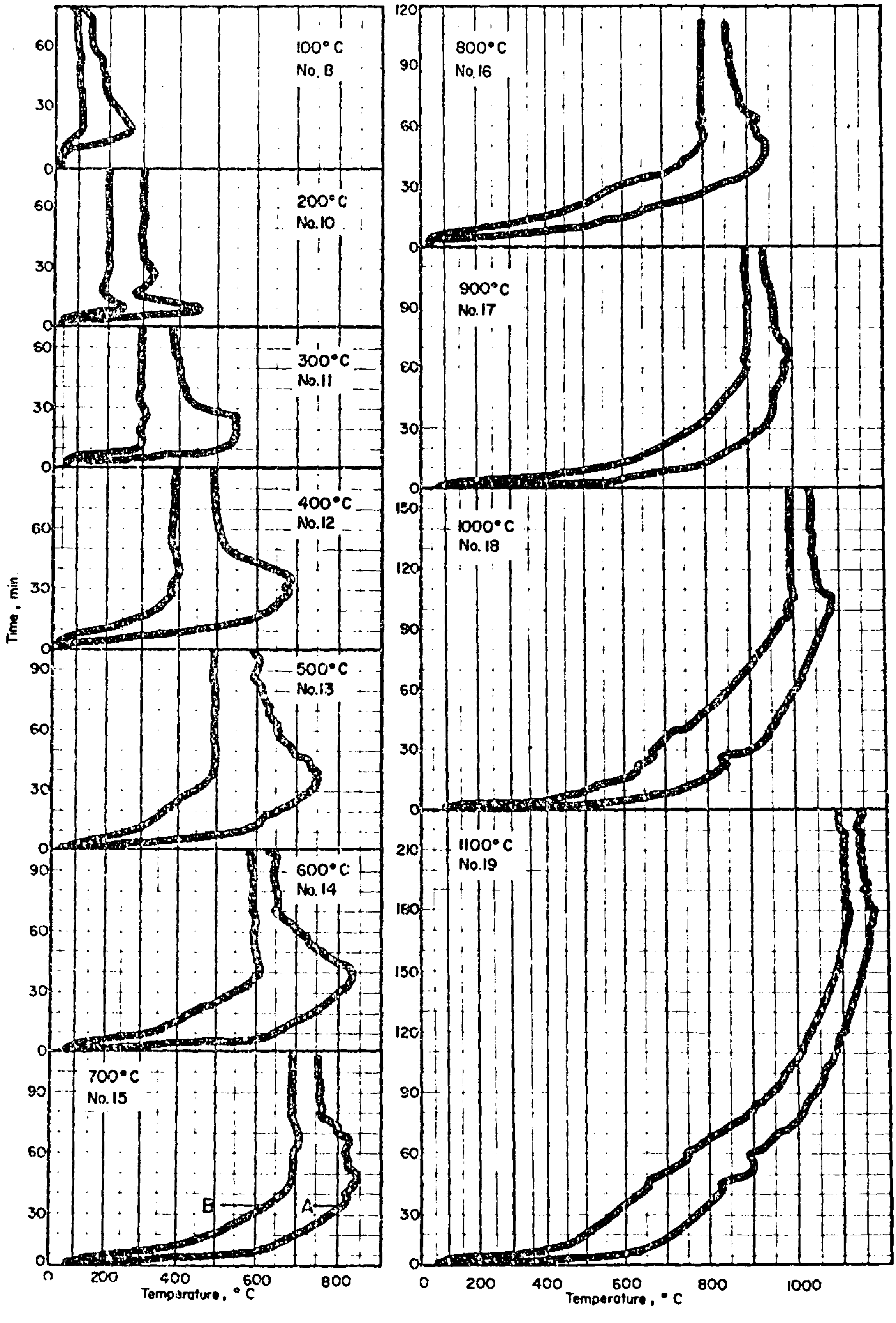

Figure 4. Typical Furnace Temperature Record (anorthosite concrete firing).

A. Top thermocouple
B. Bottom thermocouple

Special Firing For Expanded Shale Concrete

Before the expanded shale test specimens were fired at 1000°C, small test pieces, approximately 1 x 2 x 3½ in., were cut from an unfired reference beam and fired in a natural-gas-fired, up-draft furnace with a hearth area of 4 x 8 in., in which they could be observed during firing.

The test pieces were heated to furnace temperatures of 1000, 1070 and 1100°C at an average rate of about 14 deg/min, held at the maximum temperature for 1 hr, and allowed to cool to about 600-700°C before being removed from the furnace.

These firings indicated that at a temperature of about 1070°C some underbloated shale particles started bloating, resulting in pop-outs and dilation of concrete.

Special Firing For Phonolite Concrete

Firing of the phonolite concrete specimens at a temperature of 1000°C had indicated the tendency of the aggregate and cement to fuse together. In order to determine the rate at which fusion progresses, one special test firing was made. A test piece, 1½ x 1¾ x 3½ in., was cut from an unfired reference beam, placed on a 1 in. refractory slab and fired in the up-draft furnace described above. The furnace was heated at an average rate of about 10 deg/min and the development of fusion was observed by opening the furnace door at intervals between 1000 and 1250°C. At 1100°C, small blisters were observed on the test piece, and at 1225°C it started to deform. At 1250°C, fusion had progressed to the extent that the test piece flowed over the sides of the slab and onto the hearth of the kiln (see Figure 10).

PHYSICAL TESTS AND METHODS

Physical Tests

To evaluate the effect of high temperatures on concrete, the following tests were carried out on the fired test specimens:

1. Weight change determinations
2. Length change determinations
3. Compression tests on cylinders
4. Flexural tests on beams
5. Ultrasonic pulse velocity determinations
6. Thermal conductivity tests on slabs
7. Petrographic examinations of the heated-and-broken test specimens

It is to be noted that the test specimens were subjected to weight, length and pulse velocity measurements before and after firing.

Methods

Test specimens were weighed with an accuracy of ± 0.001 lb.

The lengths of the beams were measured, using a 16 in. comparator designed and built at the Mines Branch. It is equipped with an Ames dial gauge reading to 0.0001 in.

Concrete cylinders were tested for compressive strength according to ASTM Standard Method C 39-61*. For all compression tests, an Amsler testing machine of 600,000 lb capacity,

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* Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39-61), 1961 Book of ASTM Standards, Part 4, p. 721.

with a pendulum dynamometer, was used.

Concrete beams were tested for flexural strength according to ASTM Standard Method C 78-59*. The beams were tested in a manually-operated, lever-type Tinius Olsen testing machine, of which the dial in the 10,000 lb range is graduated to 1 lb.

Ultrasonic pulse velocity was determined by a UCT electronic concrete tester**. This instrument operates at a 100 kc frequency with 0.1 microsecond sensitivity.

Thermal conductivity was determined on 3 x 13 x 17½ in. concrete slabs in accordance with ASTM Standard Method C 202-47***.

The petrographic examination of the fired and broken test specimens was carried out by the Division's petrologist, using chemical analysis where necessary.

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* Standard Method of Test for Flexural Strength of Concrete (using simple beam with Third Point Loading) (C 78-59), 1961 Book of ASTM Standards, Part 4, p. 734.

** Manufactured by A. E. Cawkell, Electronic Engineers, 6-7, Victory Arcade, The Broadway, Southall, Middx., England.

*** Standard Method of Test for Thermal Conductivity of Castable Refractories (C 417-60), 1961 Book of ASTM Standards, Part 5, p. 397.

TEST RESULTS

The results of physical tests made on the four types of concrete are compiled in Tables 10 to 13. These tables show the results of weight, length and pulse velocity measurements before and after firing. Also shown are compressive and flexural strengths of fired test specimens in comparison with strengths of companion reference test specimens.

Thermal conductivity values for the four types of concretes are shown in Table 14. For each test slab, the determination of thermal conductivity was made at four temperatures, with the mean temperature ranging from about 140 to 860°C. Due to the excessive thermal deformation and buckling of the test slab, no tests could be made on ilmenite concrete at 800°C.

Physical features of fired and broken concrete test specimens are shown in Table 15. Included also are the results of stereomicroscopic studies of the ruptured surfaces, i.e. aggregate mortar bond and amount and nature of ruptured aggregate particles.

Photographs in Figures 5 to 10 illustrate the damage sustained by different concrete test specimens after firing at various temperatures.

TABLE 10

Summary of Test Results - Expanded Shale Concrete

Exposure Temperature, deg Cent	Weight Measurements, lb				Beam Length,(1) Dial Readings, in.		Compressive Strength, psi				Flexural Strength of Beams, psi		Pulse Velocity, fps	
	Cylinders		Beams				Cylinders		Beam Ends					
	Before	After	Before	After	Before	After	Refer.(2)	Fired	Refer.(2)	Fired	Refer.(2)	Fired	Before	After
100	5.083	5.042	11.088	11.010	0.0096	0.0105	2020	1560	2550	1850	390	315	11,500	11,100
200	5.073	4.825	10.939	10.489	0.0210	0.0203	2110	1340	2650	1720	385	240	11,200	9,980
300	5.089	4.692	10.832	10.152	0.0074	0.0020	2250	1180	2380	1460	340	190	11,100	8,960
400	5.057	4.639	10.973	10.199	0.0076	0.0016	2110	1150	2650	1510	385	150	11,300	8,770
500	5.090	4.668	10.936	10.087	0.0100	0.0011	2020	880	2550	1250	390	115	11,400	7,970
600	4.952	4.477	10.997	9.993	0.0245	-0.0069	2160	960	2460	1670	395	135	11,530	8,670
700	5.055	4.543	11.009	9.910	0.0277	-	2250	980	2380	1230	340	130	11,200	7,050
800	4.954	4.478	10.584	9.630	0.0330	0.0004	2030	820	2440	1190	385	165	10,800	7,100
900	4.966	4.448	10.846	9.779	0.0197	-0.0320	2160	550	2460	990	395	160	11,050	7,620
1000	4.987	4.520	10.846	9.789	0.0358	0.0140	2030	450	2440	550	385	137	11,300	7,290

(1) Beam gauge length = 14.0000 in.
(2) Reference, not fired companion test specimens.

TABLE 11

Summary of Test Results - Anorthosite Concrete

Exposure Temperature, deg Cent	Weight Measurements, lb				Beam Length,(1) Dial Readings, in.		Compressive Strength, psi				Flexural Strength of Beams, psi		Pulse Velocity, fps	
	Cylinders		Beams				Cylinders		Beam Ends					
	Before	After	Before	After	Before	After	Refer.(2)	Fired	Refer.(2)	Fired	Refer.(2)	Fired	Before	After
100	8.397	8.383	18.562	18.527	0.0903	0.0908	2730	2675	3570	3350	510	460	14,440	13,850
150	8.397	8.326	18.455	18.295	0.0800	0.0808	2730	2395	3570	3155	510	425	15,090	13,630
200	8.414	8.228	18.352	18.013	0.0811	0.0794	2660	1990	2640	2810	495	290	14,360	11,770
300	8.325	8.036	18.861	18.338	0.0850	0.0825	2630	1620	2790	2650	530	235	15,150	9,310
400	8.498	8.150	18.494	17.755	0.0989	0.0950	3070	1500	3525	2810	520	190	14,310	7,180
500	8.313	7.905	18.813	17.990	0.0890	0.0884	2630	1325	2790	2090	530	160	14,500	5,510
600	8.421	7.999	18.646	17.758	0.0873	0.0935	2740	1150	2775	1820	485	140	14,310	4,750
700	8.320	7.876	18.570	17.654	0.0948	0.1054	2740	1120	2775	1830	485	125	14,660	4,620
800	8.329	7.869	18.369	17.418	0.1019	0.1248	2660	830	2640	1340	495	105	14,810	3,750
900	8.416	7.960	18.486	17.514	0.1098	0.1360	2510	560	2695	1145	545	95	14,590	3,160
1000	8.434	7.964	18.491	17.511	0.0484	0.1060	2510	450	2695	645	545	85	14,750	2,540
1100	8.475	8.018	18.423	17.405	0.0829	0.1724	3070	990	3525	1210	520	110	14,370	4,140

(1) Beam gauge length = 14.0000 in.
(2) Reference, not fired companion test specimens.

TABLE 12

Summary of Test Results - Ilmenite Concrete

Exposure Temperature, deg Cent	Weight Measurements, lb				Beam Length,(1) Dial Readings, in.		Compressive Strength, psi				Flexural Strength of Beams, psi		Pulse Velocity, fps	
	Cylinders		Beams				Cylinders		Beam Ends					
	Before	After	Before	After	Before	After	Refer.(2)	Fired	Refer.(2)	Fired	Refer.(2)	Fired	Before	After
100	13.591	13.534	29.329	29.294	0.0226	0.0256	6170	2330	7000	5085	756	538	16,150	14,250
200	13.600	13.335	29.616	29.122	0.0463	0.0476	6170	2010	7000	2850	756	355	16,400	12,300
300	13.504	13.160	30.037	29.382	0.0265	0.0271	5900	1720	7580	3210	695	285	15,600	11,500
400	13.541	13.091	30.029	29.175	0.0167	0.0137	5900	1360	7580	2340	695	180	15,820	7,490
500	13.508	13.012	29.935	28.913	0.0130	0.0073	5390	1300	7940	2130	780	145	15,720	6,820
600	13.604	13.109	29.883	28.806	0.0200	0.0168	5390	1360	7940	2370	780	145	15,900	5,360
700	13.510	12.969	29.724	28.597	0.0262	0.0262	5870	1320	7210	1950	700	90	16,400	4,620
800	-	-	-	-	-	-	-	-	-	-	-	-	-	-
900	13.697	13.183	29.926	28.777	0.0239	0.1072	6470	532	6940	1450	836	70	16,500	*
1000	13.711	13.179	29.609	28.402	0.0282	0.1804	6470	290	6940	790	836	40	16,200	*

(1) Beam gauge length = 14.0000 in.
(2) Reference, not fired companion test specimens.
* Too much damaged to be tested.

Table 13

Summary of Test Results - Phonolite Concrete

Exposure Temperature, deg Cent	Weight Measurements, lb				Beam Length,(1) Dial Readings, in.		Compressive Strength, psi				Flexural Strength of Beams, psi		Pulse Velocity, fps	
	Cylinders		Beams				Cylinders		Beam Ends					
	Before	After	Before	After	Before	After	Refer.(2)	Fired	Refer.(2)	Fired	Refer.(2)	Fired	Before	After
200	7.937	7.725	17.669	17.094	0.0775	0.0775	2765	1900	5060	2920	510	325	16,350	13,090
300	7.755	7.406	17.793	17.023	0.0657	0.0650	2140	970	3890	2290	545	185	15,510	8,175
400	7.942	7.439	17.789	16.649	0.0577	>0.1423	2820	220	5015	*	538	55	15,600	*
500	7.967	7.396	17.896	16.550	0.0335	>0.1665	2820	520	5015	*	538	60	15,875	*
600	7.909	7.332	17.901	16.458	0.0816	0.1161	2740	945	5520	1430	680	80	15,505	3,270
700	7.774	7.185	17.946	16.529	0.0674	0.1023	2140	640	3890	1345	545	90	15,500	3,550
800	7.820	7.177	17.697	16.239	0.0603	0.1260	2765	510	5060	1220	510	80	15,750	3,480
1000	7.980	7.293	18.064	16.396	0.0748	>0.1252	2950	1030	6000	1155	670	110	16,345	3,990

(1) Beam gauge length = 14.0000 in.
(2) Reference, not fired companion test specimens.
* Too much damaged to be tested.

TABLE 14

Thermal Conductivity of Concrete

Type of Concrete Aggregate	Room-Dry Concrete Density, lb/cu ft	Thermal Conductivity		
		Mean Temperature		Coefficient "k", Btu x in. per hr x ft^2 x deg Fahr.
		deg Fahr	deg Cent	
Expanded Shale	84.96	290	143	4.29
		570	299	3.28
		1051	566	3.28
		1484	807	3.42
Anorthosite	143.25	298	148	7.79
		603	317	6.86
		1132	611	6.01
		1577	858	6.14
Ilmenite	231.01	330	165	9.91
		654	345	8.26
		1195	646	7.84
		1470	800	-
Phonolite	134.90	288	142	9.60
		437	225	8.51
		662	350	6.07
		1549	843	5.04

TABLE 15

Physical Features of Broken Heat-Exposed Concrete Cylinders and Beams*

Type of Concrete	Temperatures, deg Cent	Colour, Appearance	Aggregate-Mortar Bond	Broken Aggregate	Conclusions
Expanded Shale	Unheated	Medium dark grey	Good, strong bond	20-30% on beams	- - -
	200 to 300	Medium grey, whitened in core	Bond appears strong.	15-20% on beams 5% on cylinders	Bond weakened by heating.
	400 to 700	Outer 3/4 in. rim brownish grey; map cracking, few larger cracks.	From strong to becoming firm, mortar slightly powdery.	10% or less on beams; nil on cylinders	Map cracking - weakening of bond, suggests shrinkage of paste.
	800 to 1000	Light pinkish grey to rusty tan; core rusty pink; more cracking.	Bond weak to very weak; mortar powdery and friable.	5% or less on beams	Colour indicates oxidation of iron. Aggregate appears to be unaffected.
Anorthosite	Unheated	Dull grey	Good, strong bond	30-40% on beams	- - -
	100 to 300	Dull grey, pinkish in core	Bond appears strong.	35 to 20% on "	- - -
	400 to 700	Greyish pink, darker border; disintegrates along edges at 700°.	Strong to fairly strong bond	20 to 10% on "	Slight oxidation of ferrous impurities; mortar becoming weak.
	800 to 1000	Pinkish to yellowish brown; fine map cracking at 1000°.	Weak bond, mortar appears friable.	10 to 5% on "	Mortar weak, aggregate particles appear sound.
	1100	Brownish rim, pinkish core	Bond strong; mortar cemented.	15-20% on beams	Mortar sintered, "ceramic bond" started.
Ilmenite	Unheated	Medium dark grey	Good, strong bond	20-25% on beams	- - -
	100 to 300	Medium grey, whitened rim; no visible cracks.	Bond good; aggregate clean on broken cylinders.	10% on beams; nil on cylinders	Whitening suggests chemical deterioration of mortar.
	400 to 700	Medium grey to rusty pink; few map cracks.	Bond fairly strong; mortar slightly flaky to powdery.	5% on beams, mostly small aggregate.	Bond weakening; flaking suggests deterioration of mortar.
	800 to 1000	Pinkish buff, rusty rim; few map cracks.	Bond appears firm to weak; mortar friable to flaky.	5 to 2%, fracture along cleavages.	Concrete probably weaker than at 700°; mortar very weak and crumbling.
Phonolite	Unheated	Dull grey	Good strong bond	30-50% on beams	- - -
	200	Muddy grey; darker rim.	Bond appears strong.	15-20% on beams	No significant change.
	300 to 600	Light brownish to pinkish grey; extensive cracking above 400°.	Bond weak to very weak; at 500° largely disintegrated.	5 to 0% on "	Phonolite unstable at 400-600°. Later reaction with air causes decomposition.
	700 to 800	Pinkish grey interior, yellowish brown rim; abundant cracks.	Bond poor; flaky mortar; exterior more cohesive.	0 to 5%; at 800° aggregate appears sound.	Mortar weakened; higher temperatures slightly improve resistance to air attack.
	1000	Rusty brown; large cracks; boils, pop-outs at surface.	Bond appears strong; mortar cemented; ringing sound.	20 to 30%; most particles ruptured.	Some aggregate particles melted; incipient fusion strengthen mortar bond.

* After J. A. Soles.

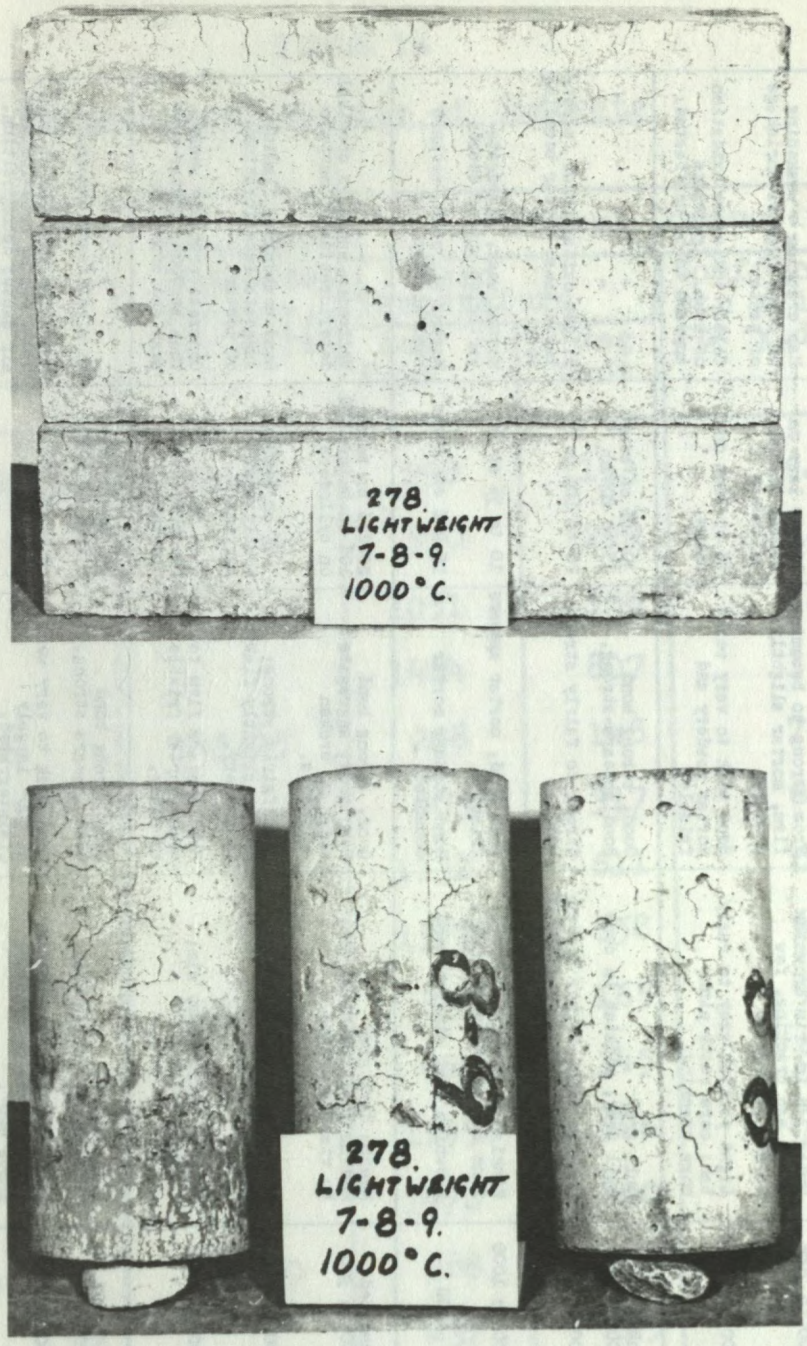
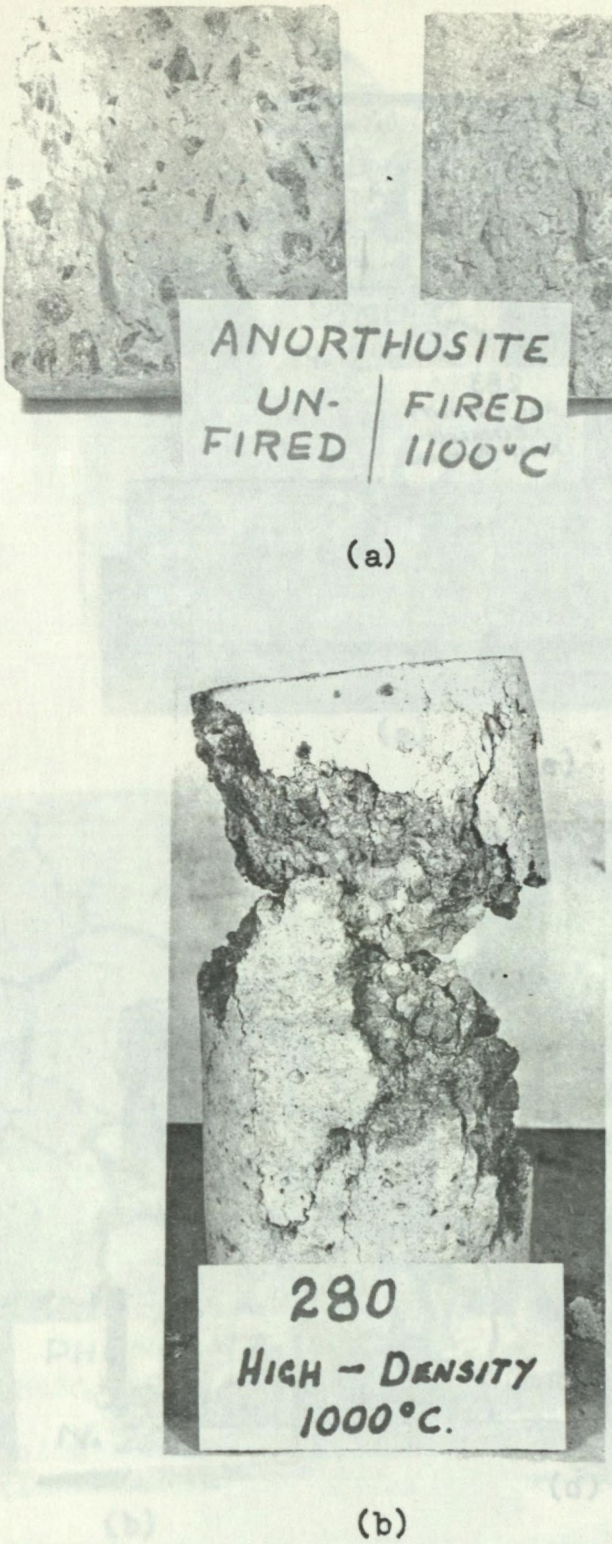


Figure 5. Expanded Shale Concrete Test Specimens Fired at 1000°C.

(Note extensive surface cracking.)



(a)

(b)

Figure 6. Unfired and Fired Test Specimens After Testing.

(a) Ruptured surfaces of anorthosite concrete beams.
(b) Ilmenite concrete cylinder shows very poor aggregate-mortar bond. Mortar crumbled on handling.

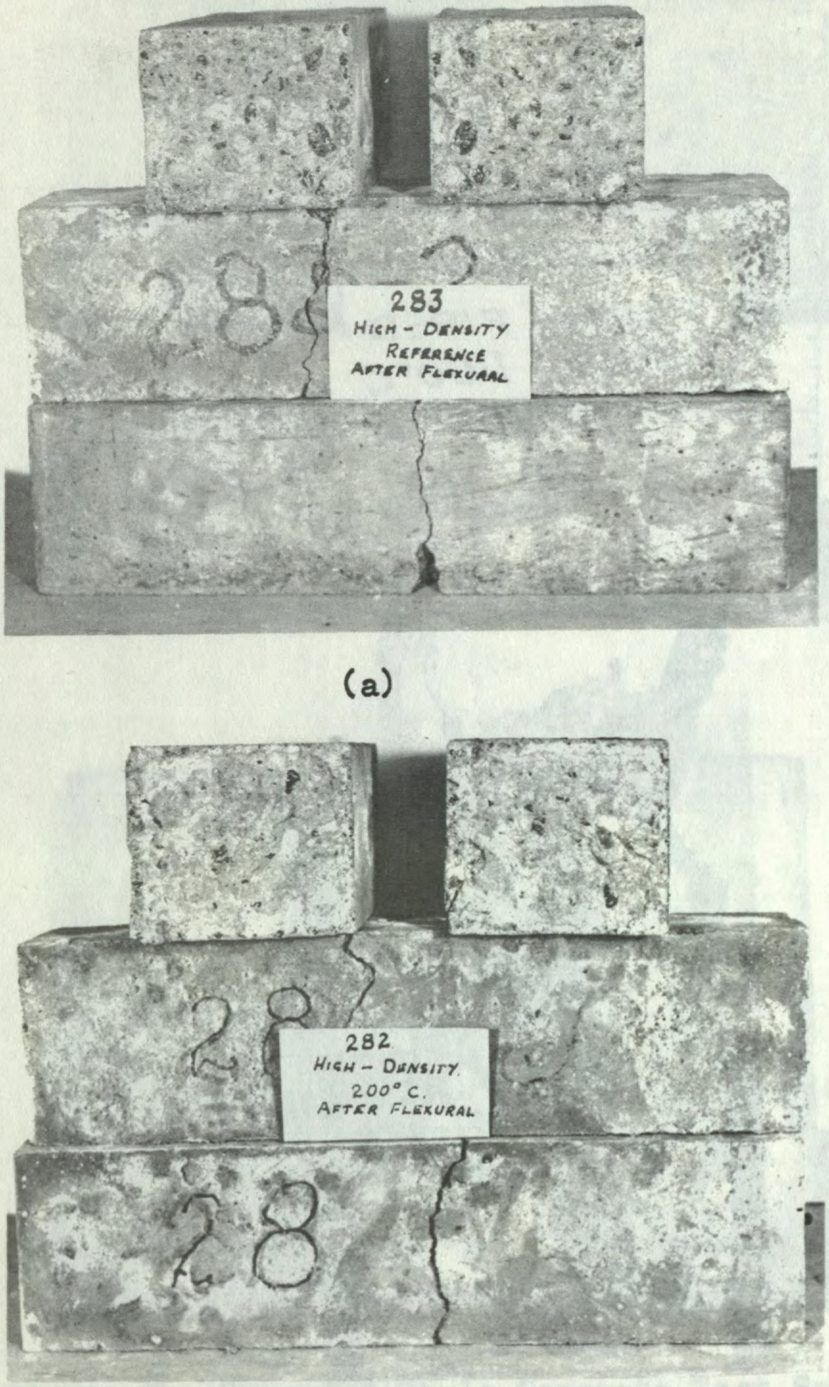
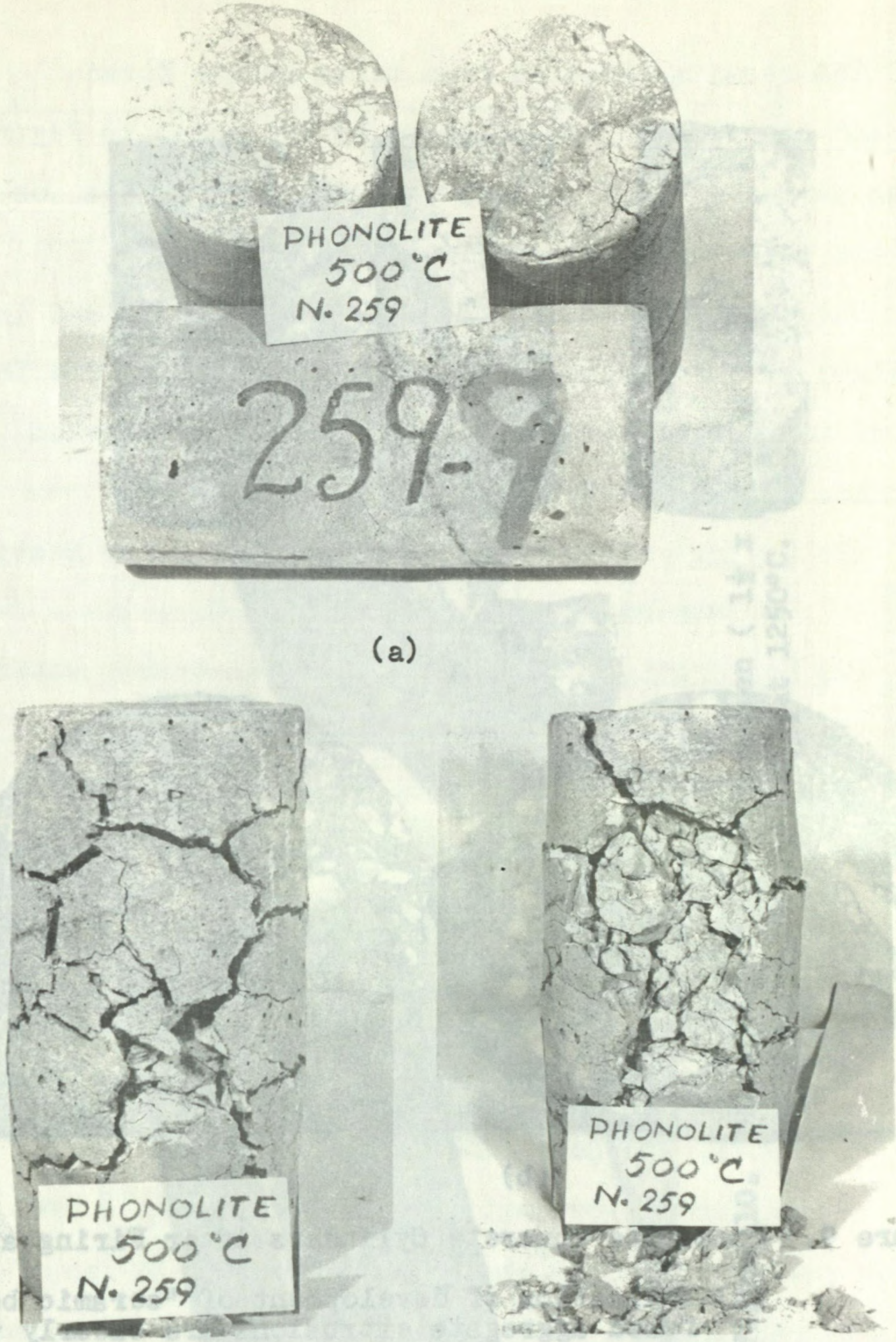


Figure 7. Unfired and Fired Ilmenite Concrete Beams After Testing.

(a) Approx. 25% of aggregate on ruptured surface broken

(b) Approx. 10% of aggregate on ruptured surface broken



(a)

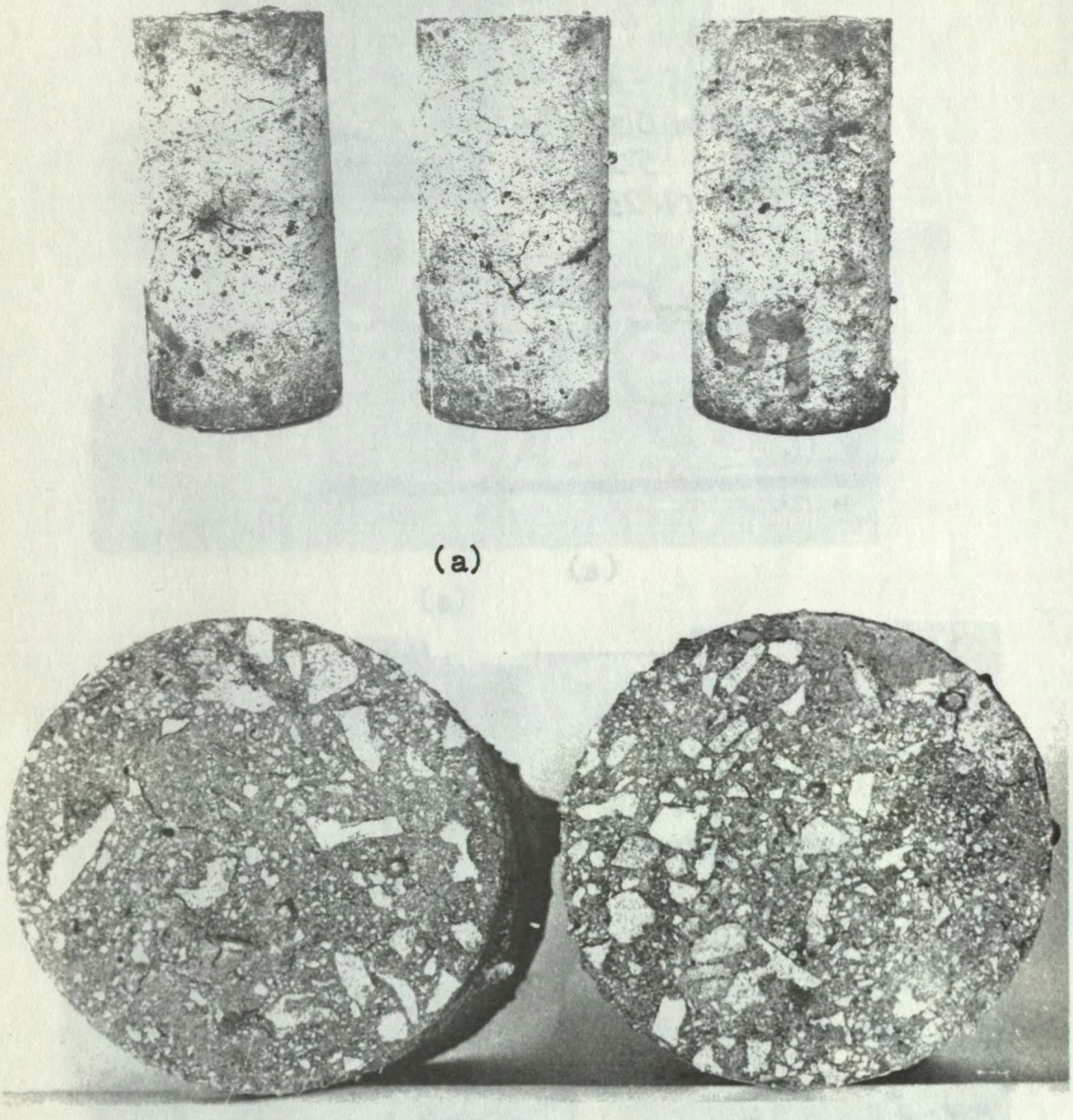
(b) (c)

Figure 8. Phonolite Concrete Cylinders After Firing at 500° C.

(a) Immediately after removal from furnace
(Note aggregate exposed on cylinder ends.)

(b) After 7 days of air storage, at room temperature

(c) Same cylinder after 15 days' air storage



(a)

(b)

Figure 9. Phonolite Concrete Cylinders After Firing at 1000°C.

(a) Indication of development of "ceramic bond"; fused aggregate extrusions are clearly visible.

(b) Ends of same cylinders. (Note glossy surfaces of exposed phonolite.)

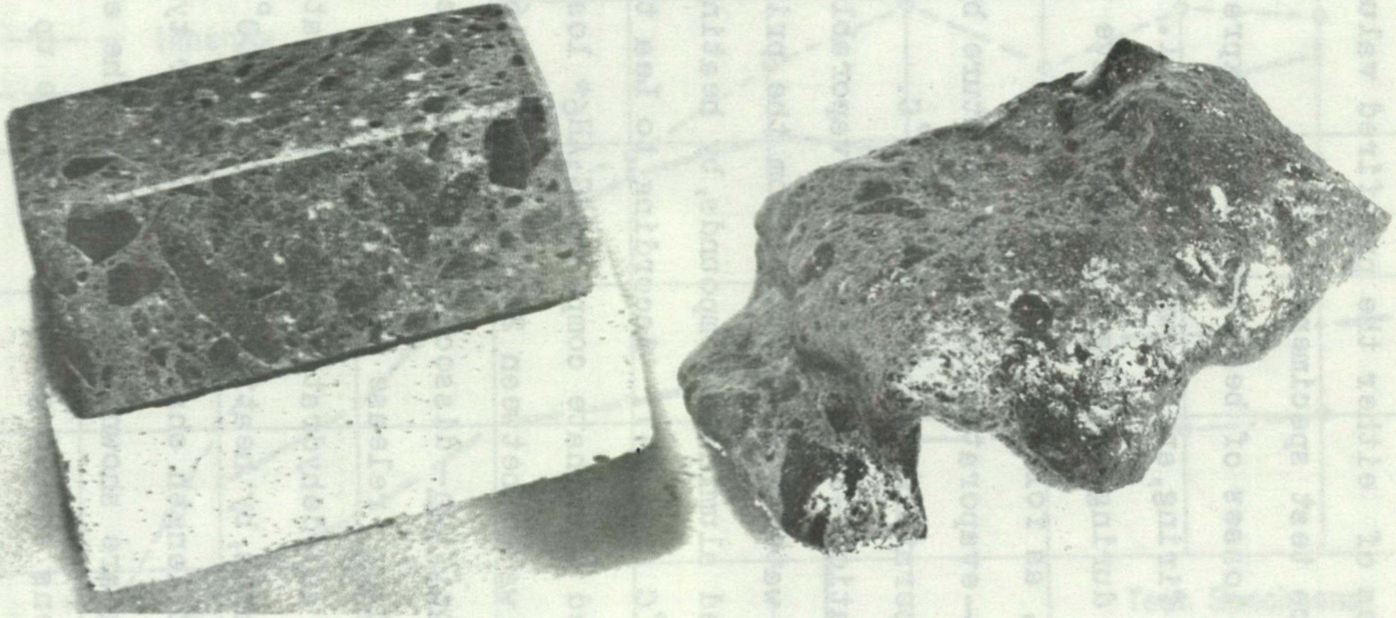


Figure 10. Phonolite Concrete Specimen (1½ x 1¾ x 3½ in.) Before and After Firing at 1250°C.

DISCUSSION OF RESULTS

The results obtained from tests on the fired specimens and compiled in Tables 10 to 13 are shown in Figures 11 to 15 as percentages of either the pre-fired values or the values of the reference test specimens.

The weight losses of beam specimens, expressed in per cent of weight before firing, are shown in Figure 11. The loss of weight of concrete during heating appears to have taken place in three stages, as follows:

(1) Drying—evaporation of free moisture by heating to temperatures from 100 to 200°C.

(2) Dehydration—loss of the non-evaporable water, or the water of hydration from the principal hydrated aluminate compounds, by heating from 200 to 300°C (23, 24). According to Lea the cubic hydrated aluminate compound C_3AH_6* loses most of its water between 225 and 275°C (16).

(3) Decomposition—dissociation of hydrous aggregate following release of the chemically bonded water, and dehydration of other hydrated aluminates, by heating from 300 to 800°C (24).

The permanent length changes for the four types of concretes after firing are shown in Figure 12. The expanded shale concrete specimens showed gradual shrinkage up to 500°C; beyond this temperature, the shrinkage increased rapidly to a

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* For abbreviations, see footnote p. 46.

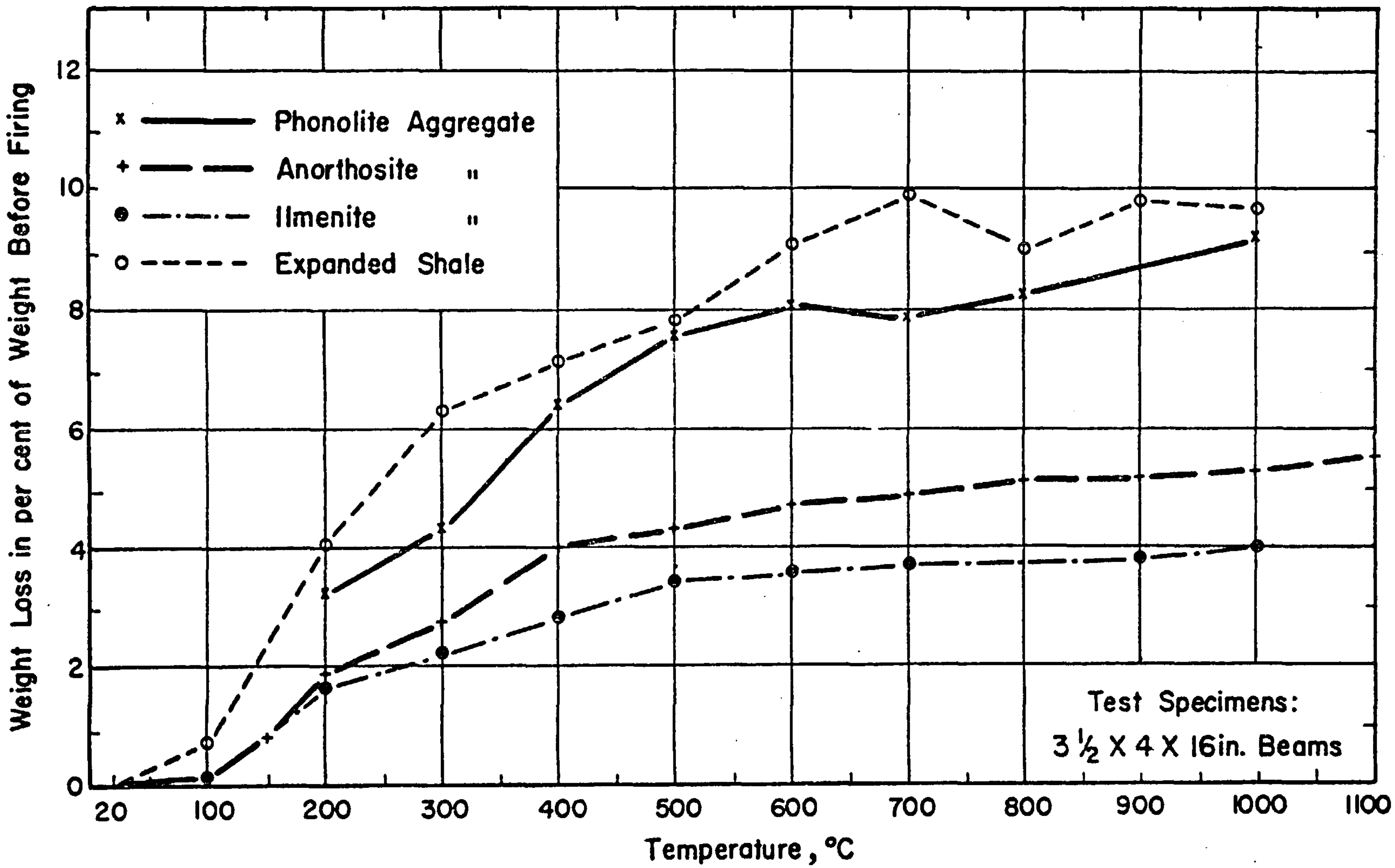


Figure 11. Weight Loss.

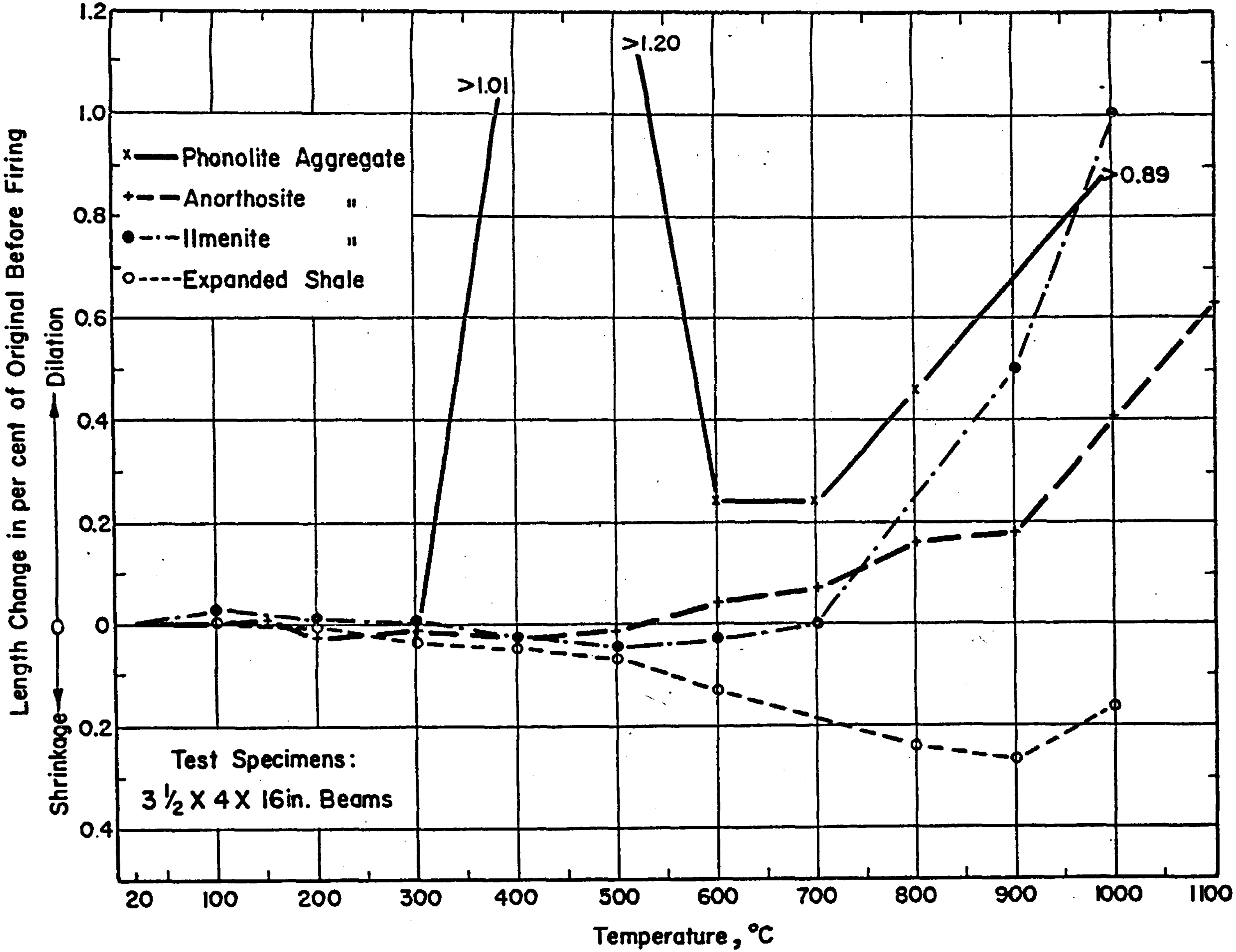


Figure 12. Length Change.

maximum of 0.25 per cent at 900°C. When heated to 1000°C a slight dilation of the fired test specimen was recorded, which probably could be attributed to the development of gases in underbloated shale particles.

All concretes showed a slight dilation followed by contraction, after being exposed to temperatures ranging from 100 to 500°C. A special case was phonolite concrete, which suffered a sudden large expansion between 300 and 600°C. After firing at 500°C its permanent expansion was greater than 1.2 per cent; the fired test specimens were cracked extensively, indicating a decrease in aggregate-cement bond strength (see Figure 8, a). When left in air for several days, the cracks gradually opened until the concrete disintegrated and specimens crumbled (see Figure 8, b and c). The decomposed, powdery particles of aggregate suggested a rehydration or slaking of the calcined rock under the attack of atmospheric moisture. This was substantiated by the slight weight increase of test specimens.

The expansion characteristics of phonolite concrete indicate that 300°C is a critical temperature for the phonolite (tinguaite), the temperature of dissociation of which, as determined by the thermogravimetric method, is from 300 to 450°C (13).

All concretes, except that made with expanded shale, showed marked expansion after being exposed to temperatures over 700°C. Of the three, ilmenite concrete showed the greatest permanent expansion, which was over 1.0 per cent after being fired at a temperature of 1000°C.

It is known from published data (6, 25) that neat, hydrated aluminous cement paste produces permanent shrinkage after exposure to high temperatures; when fired at 1000°C its shrinkage is about 2.0 per cent. Therefore, assuming no expanding phase is formed by cement-aggregate reaction, the resultant expansion of concrete exposed to temperatures over 700°C is probably caused by the expansion characteristics of the mineral constituents of the particular aggregate used.

Compressive strength changes for the four types of concretes after firing at different temperatures are shown in Figure 13.

Concretes made with expanded shale and anorthosite aggregates lost about half of their strength after exposure to 400°C. The compressive strengths reached a minimum of 25.8 and 17.9 per cent, respectively, after firing at 1000°C. A slight regain in strength occurred for anorthosite concrete at 1100°C.

The compressive strength of phonolite concrete specimens reached a minimum of 7.8 per cent at 400°C. Between 400 and 1000°C its strength recovered to a value of 34.9 per cent at 1000°C.

Ilmenite concrete lost more than 60 per cent of its strength after exposure to 100°C. This is comparable with

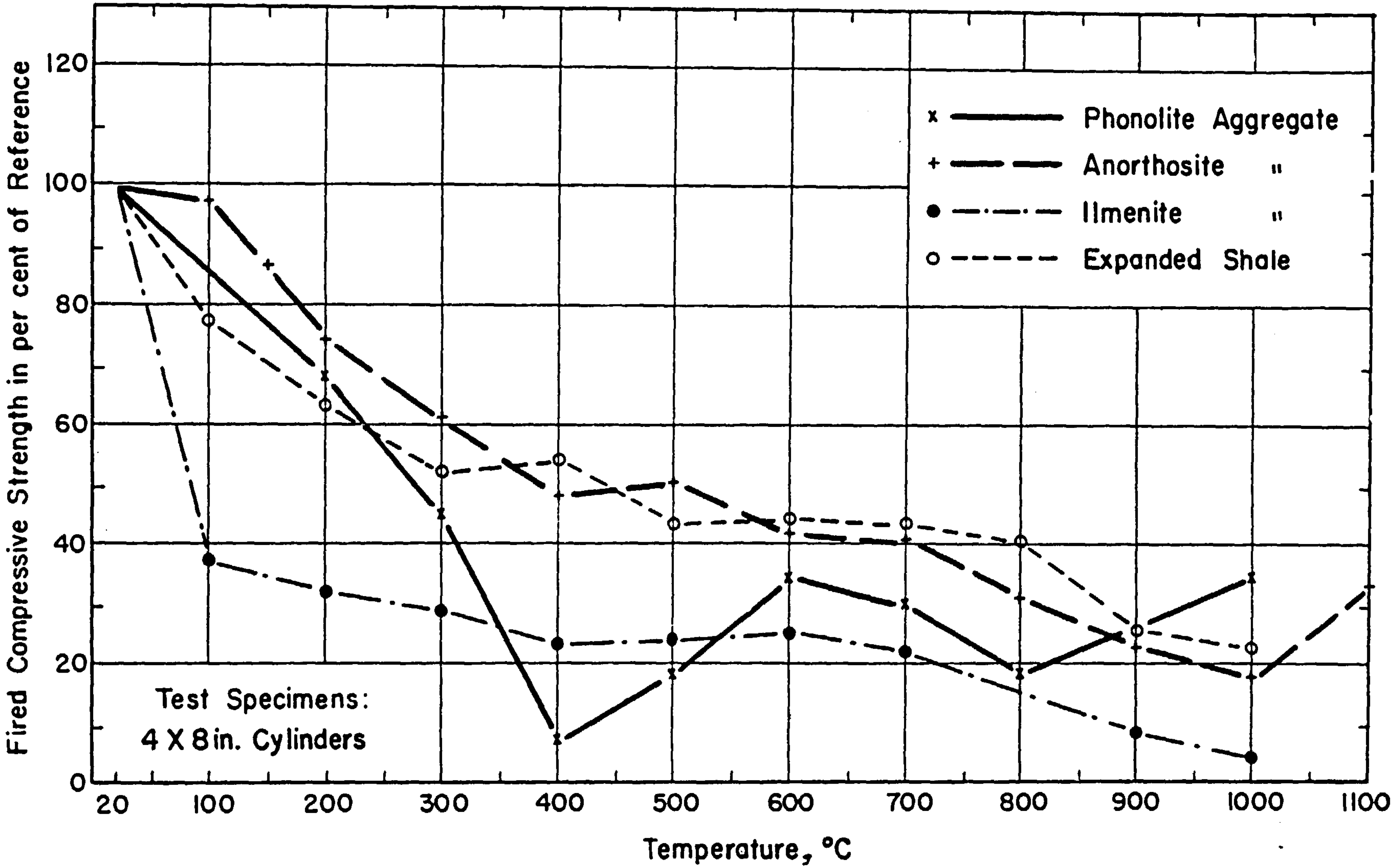


Figure 13. Compressive Strength.

relative strength losses reported by Miller and Faulkner (1), and by Arshinov (9), on aluminous cement concretes made with different aggregates. After firing at 1000°C, the residual strength of ilmenite concrete was only 4.5 per cent. This could be due to a complete breakdown of the bond between the cement paste and aggregate (see Figure 6, b).

The gain in strength at 1000 and 1100°C for phonolite and anorthosite concretes, respectively, is probably due to the development of a ceramic bond. This was substantiated by visual inspection of fired test specimens and microscopic examination of the broken sections of concrete (see Table 15 and Figure 9).

It is interesting to note that the fired strengths of concretes made with anorthosite, ilmenite and phonolite aggregates after heating at 200°C are about the same, ranging from 1900 psi (phonolite) to 2010 psi (ilmenite). The cold strengths of these concretes were 2765 and 6170 psi, respectively. This levelling of the residual strength of concrete after heat exposure at low temperatures is largely governed by the strength of the fired cement bond, and is independent of the concrete cold strength (26). At higher temperatures, however, aluminous cement can begin to react with some aggregate, first in the solid state (e.g. with expanded shale aggregate) and then when aggregate-cement fusion occurs. A ceramic bond is formed, which increases the concrete strength after cooling (5, 16). Such a bond was formed at about 1000°C with phonolite aggregate.

Changes in flexural strength of beams of the four types of concretes after firing are shown in Figure 14. The flexural strengths dropped rapidly upon exposure to temperatures up to 500°C. At this temperature the fired strength ranged from about 10 per cent of the original for phonolite concrete to about 30 per cent for expanded shale and anorthosite concretes. Between 500 and 1000°C, flexural strengths for anorthosite and ilmenite concretes decreased gradually, reaching a minimum of about 5 per cent for ilmenite concrete at 1000°C. The expanded shale concrete beams, on the contrary, showed a strength recovery, reaching about 35 per cent at 1000°C. The anorthosite concrete showed a slight regain in flexural strength at 1100°C, as it had in compressive strength.

The ultrasonic pulse velocities of the four types of concretes fired at different temperatures are shown in Figure 15.

The pulse velocities of expanded shale concrete specimens showed a gradual drop with increased temperature, reaching a minimum value of 64.5 per cent at 1000°C.

The pulse velocities of the other concretes dropped to less than 40 per cent with firing temperatures increasing up to 600°C. Between 600 and 1000°C temperature the pulse velocities of anorthosite concrete specimens decreased continuously, reaching a minimum of 28.8 per cent at 1000°C; the phonolite concrete specimens showed a slight recovery of pulse velocity

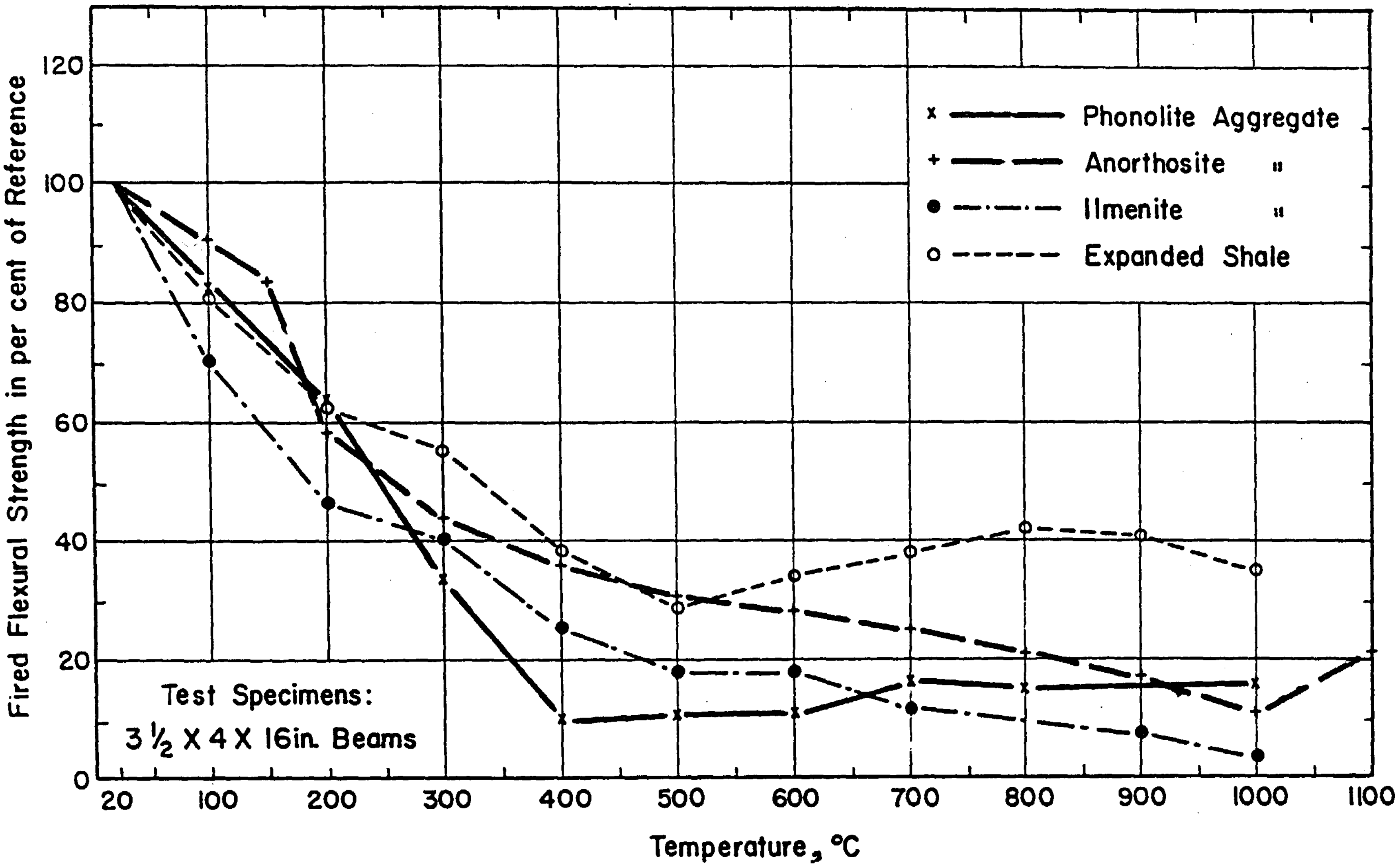
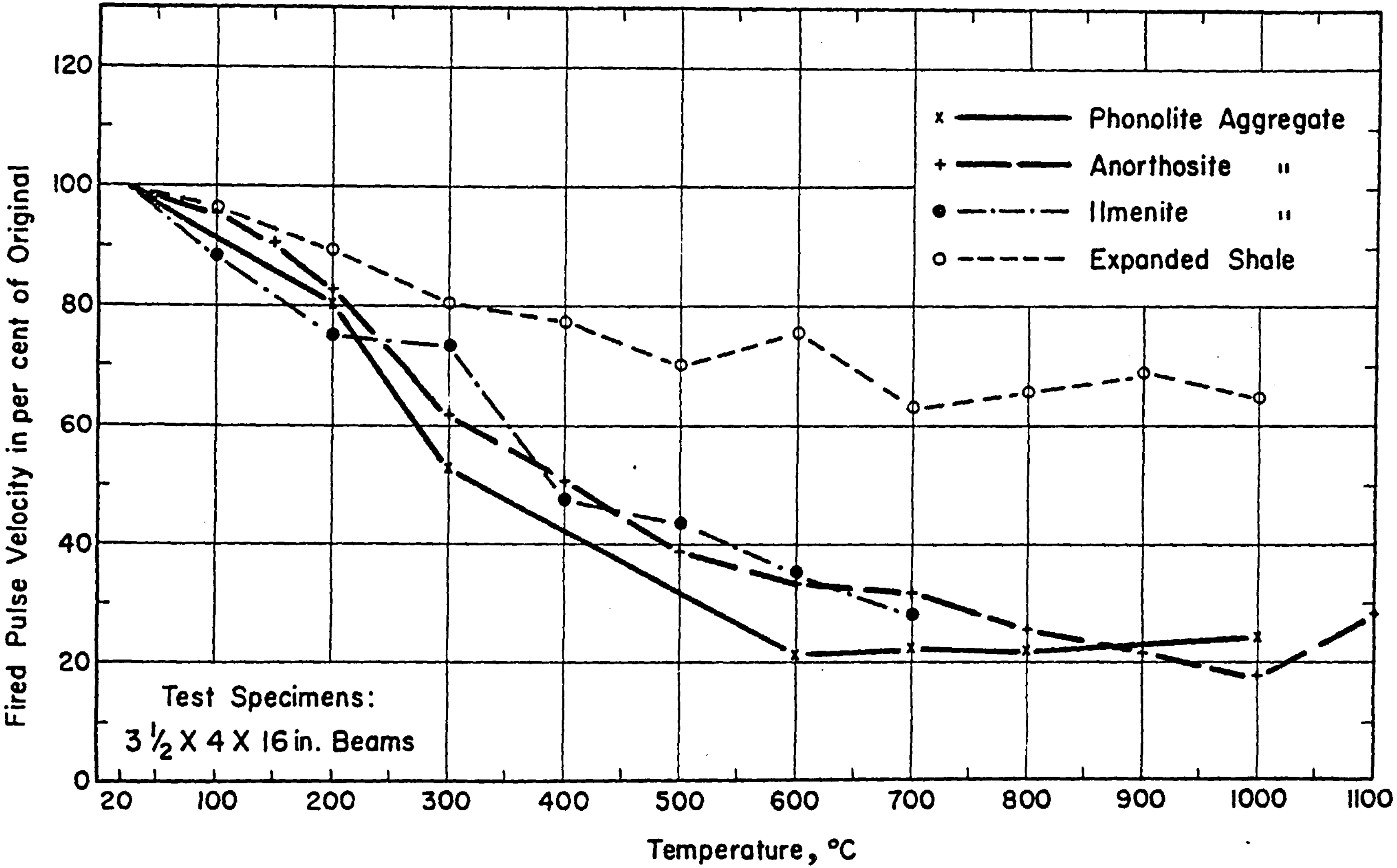


Figure 14. Flexural Strength.



Test Specimens:
3 ½ X 4 X 16 in. Beams

Figure 15. Ultrasonic Pulse Velocity.

values, reaching 24.4 per cent at 1000°C. This is probably due to the development of a ceramic bond which causes closure of microcracks developed in the interior of the concrete specimens.

It is believed that the reduction in compressive and flexural strengths of aluminous cement concretes at lower ranges of elevated temperatures is related primarily to changes in the principal compound composition of the cement, although the type of aggregate also affects the strength of fired concrete greatly.

According to Schneider (25), the major decrease in the strengths of aluminous cement between 110 and 300 to 500°C is due to the dehydration of its principal compounds C_3AH_6, AH_3 and CAH_{10}* and the formation of their dehydration products. The changes in strength noted at temperatures from 300 to 500°C cannot be related to the formation or the presence of any one compound. Above 700°C the loss in strength was possibly due to grain growth and the formation of new compounds.

Wells and Carlson (27) suggested that the loss in strength at elevated temperatures was due to crystallization of AH_3 from alumina gel.

No measurements were made to determine Young's modulus or the dynamic modulus of elasticity of the fired test specimens. It has been suggested (7, 24, 25) that changes in the strength of fired concrete are accompanied by similar,

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* The abbreviations used for the conventional formulae are: $C_3AH_6 = 3\ CaO.Al_2O_3.6H_2O$ (cubic compound); $AH_3 = Al_2O_3.3H_2O$ (alumina hydrate gel, gibbsite); $CAH_{10} = CaO.Al_2O_3.10H_2O$ (pseudohexagonal compound).

although not always proportional, changes in the elastic moduli.

The values compiled in Table 14 show the effect of different aggregates on the thermal conductivity of concrete. Of the four types of concretes tested, ilmenite had the highest thermal conductivity, closely followed by phonolite and anorthosite concretes.

The "k" values for expanded shale concrete agree closely with the graph established by Hansen and Livovich (28) for the relationship between thermal conductivity (at a mean temperature of 1000°F) and fired unit weight of insulating concretes made with aluminous cement.

The "k" factor decreases with decreasing free moisture content in concrete.. Ruh and Renkey showed (29) that thermal conductivity decreases still further at temperatures ranging from 200 to 800°C, when the chemically combined water is driven off.

In this study the decreasing "k" values accompanying increasing mean temperatures indicate gradual weakening of the aggregate-mortar bond and a general deterioration of fired concrete. An increase in thermal conductivity in certain cases at the higher temperatures probably indicates the development of ceramic bond (29).

CONCLUSIONS

It is concluded, from this study, that aluminous cement concretes on exposure to dry heat deteriorate rapidly. Extended deterioration of concrete is dependent to a large degree on the type of the aggregate.

Concretes made with expanded shale, anorthosite, ilmenite or phonolite aggregate lose more than 50 per cent of their strength after exposure to 400°C. At temperatures between 400 and 1000°C the loss in strength continues, but at a relatively slower rate.

Expanded shale concrete showed the greatest stability, whereas phonolite concrete was the least heat-resistant of the four types investigated.

The reduction in strength of the four concretes after exposure to temperatures up to 300°C was caused, to a large degree, by a reduced fired mortar strength, which, it is believed, is primarily due to changes in the principal compound composition of the cement. Between 300 and 1000°C the deterioration is governed mainly by the physical properties and mineral constituents of the aggregate.

The results of this study should be considered when designing aluminous cement concrete structures exposed to dry heat, and investigation of the concrete under the anticipated exposure conditions is recommended.

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