

NATIONAL BUREAU OF STANDARDS REPORT

5601

QUARTERLY REPORT

ON

EVALUATION OF REFRACTORY QUALITIES OF
CONCRETES FOR JET AIRCRAFT WARM-UP, POWER CHECK,
MAINTENANCE APRONS, AND RUNWAYS

by

W. L. Pendergast, E. C. Tuma, L. E. Mong
and E. Trattner



U. S. DEPARTMENT OF COMMERCE
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NBS PROJECT

0903-20-4428

October 1, 1957

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Refractories Section
Mineral Products Division

Sponsored by

Department of the Navy
Bureau of Yards and Docks

Reference: NT4-59/NY 420 008-1

NBS File No. 9.3/1134-C

Approved:

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Chief, Refractories Section

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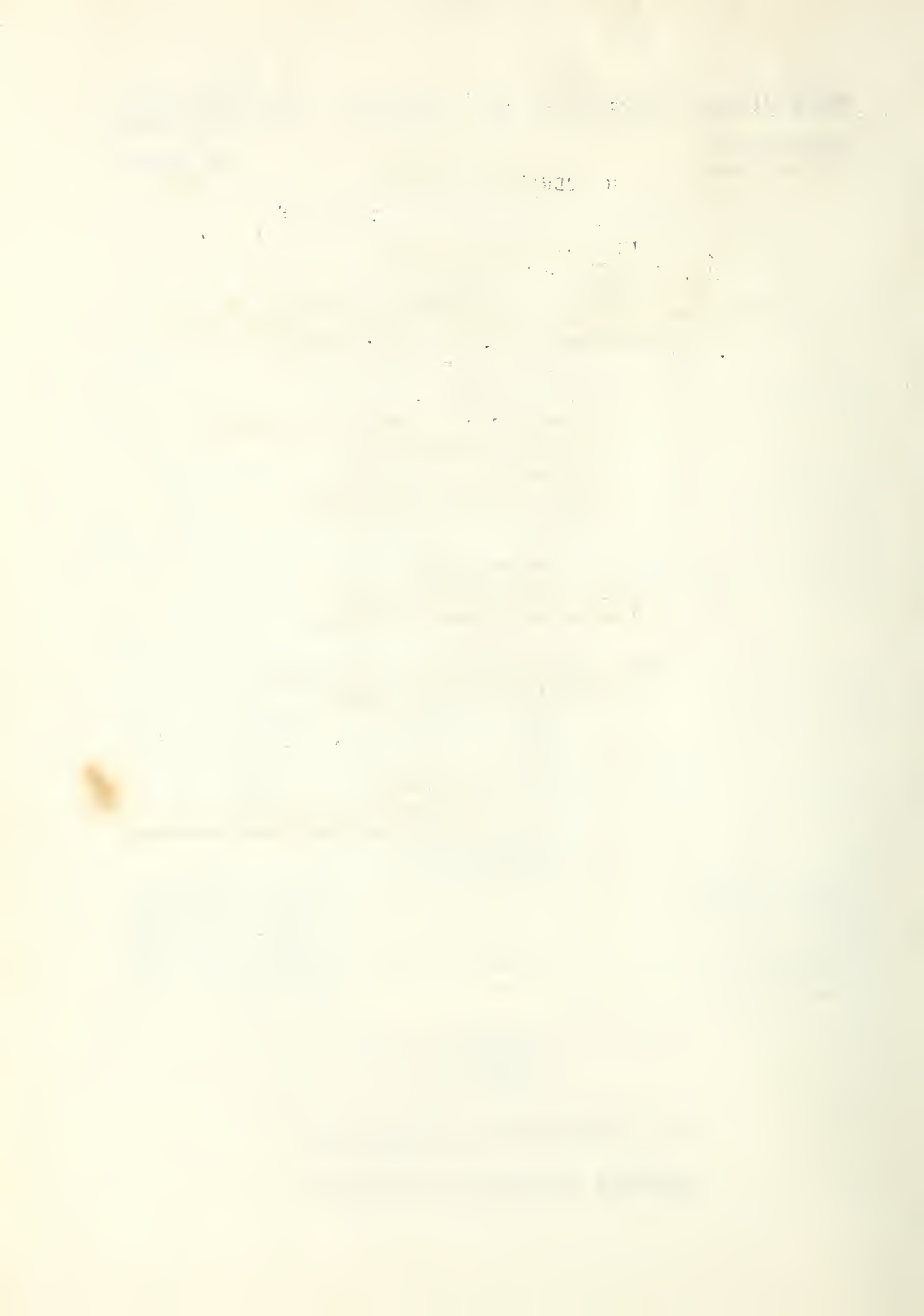
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1. INTRODUCTION

This phase of the project includes the determination of the cause or causes of failure that occur in concrete aprons and runways exposed to jet exhaust gases. A combustion chamber that delivers hot gases at velocities and temperatures approximating those of field conditions is being used. The approach includes instrumentation of the concrete test panels to determine the heat gradients and stresses set up during flame impingement at several locations on the test area and at varying depths below the surface.

2. ACTIVITIES

2.1 Pressure Developed in Concrete
During Heating

2.1.1 Bomb Tests

Pressure-temperature curves were drawn from data taken in eight tests in which the stainless steel bomb and mercury filling was used. The materials, treatments, and data showing the characteristics of the curves are given in Table I.

The evacuation drying (to constant weight) removed nearly all of the capillary water and left "chemically combined" water. Materials tested without this treatment contained appreciable amounts of capillary water, and the concretes had comparatively large amounts. The bonding of the water to the

material, whether chemical or capillary, would be expected to cause deviations of the pressure-temperature curves from the steam curve. The capillary bonding would be expected to vary according to the fineness of the capillaries, and it has been shown, N.B.S. Report No. 5353, p. 3, that brick aggregate with interstitial water does not appreciably alter the pressure-temperature curve from the steam curve.

The extent of these bonding effects are indicated in Table I. The temperature for initial pressure (100°C for the steam curve) is elevated for all samples having capillary water to values ranging from 105° to 170° . Where aggregate was present the values were nearly the same. It appeared that Portland cement developed the finest capillary texture and Alcoa cement the coarsest, possibly because the Alcoa was aged only 7 days and had not had adequate time to develop its fine crystal structure. The offsets from the Steam Curve at the higher pressures showed the persistence of the capillary effects.

Samples, containing chemical combined water only, gave rather high temperatures for initial pressure and large offsets from the Steam Curve. For Portland cement this chemical bonding was similar to the capillary bonding. For the high alumina cements, however, the temperature required to separate chemical combined water or "threshold temperature" rose to 212 and 222°C . Also, for the high aluminas, there was a

Table I. Data from Pressure-Temperature Curves for Neat Cements and Concretes.

Material ^{1/}	W/C Ratio	Percent Water	Fog Room Curing Days	Evacuation Drying		Temperature-Pressure Curves									
				Time	Loss	H e a t i n g					C o o l i n g				
						Temp. for Initial Pressure	Temp. offset at pressures of ^{2/}				Rapid Pressure Rise	Crosses Steam Curves	Temp. offset at pressures of ^{2/}		
							200 psi	400 psi	850 psi	200 psi			400 psi		
				Hrs.	%	°C	°C	°C	°C	°C	°C	°C	°C	°C	°C
P	.275	27.5	12	none	-	170	+22	+17	+5	none	165	- 1	nil		
P	.275	27.5	80	191	2.25	168	+23	+20	+5	272 to 274	185	+ 1	+ 1		
PB	.65	15.2	13	none	-	147	+ 8	+ 7	+7	none	170	- 5	- 5		
L	.275	27.5	16	none	-	150	+10	+ 7	0	none	155	- 5	- 7		
L	.275	27.5	16	164	(.4	212	+25	+15	+5	210 to 225	150	- 7	- 5		
A	.275	27.5	7	none	-	105	+ 7	+ 8	0	none	280	-15	-10		
A	.275	27.5	7	213	1.8	222	+35	+12	0	222 to 242	180	- 3	- 3		
AB	.65	15.2	21	none	-	140	+ 7	+ 7	+11	none	182	+ 5	+ 7		

^{1/} P = neat Portland cement, L = neat Lumnite cement, A = neat Alcoa cement, PB = Portland cement - brick aggregate concrete, AB = Alcoa cement - bric aggregate concrete.

^{2/} Difference in temperature from Steam Curve at pressures indicated.

succeeding temperature range where the pressure rose very rapidly indicating the decomposition of hydrates. The corresponding range in rapid pressure rise was found to be much higher for the Portland cement indicating a second decomposition. These changes for chemical water were masked in the tests where capillary water was present.

Since water was liberated from the material, the cooling curves were not greatly different from the steam curves, a convergence of the curves in the range from 150 to 185°C may possibly indicate some recombination of water with the material to form hydrates having water contents much lower than in the original cured cement.

As pointed out previously, sufficiently high pressures to fracture concretes are developed at temperatures well within the operating temperatures of pavements exposed to jet aircraft. While thorough drying by evacuation raises the temperature of this evolution of water, the rise is not sufficient to avoid explosive spalling, especially for open-throttle operation.

The data given in Table I is useful in comparing the pressure development of different cementing materials, but does not consider other factors, such as the permeability of the concrete, in its performance in heat-resisting applications.

2.1.2. X-ray Examinations

X-ray examinations of neat Portland cement, after three heating cycles (280°C) in the bomb test, were made. The

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patterns were similar for each treatment, and showed peaks corresponding to those reported for Tobermorite, $4-5 \text{ CaO} \cdot 5\text{SiO}_2 \cdot 5\text{H}_2\text{O}$, (Reference given in literature reviewed) dicalcium silicate, $2\text{CaO} \cdot \text{SiO}_2$, and calcite, Ca CO_3 . The absence of other peaks made positive identification of these minerals uncertain.

Additional samples of Portland, Alcoa, and Lumnite cements, after heating to approximate constant weight at progressively higher temperatures, are being collected for X-ray examination.

2.2 Water in Concrete During Curing and Drying

The dependency of humidity on the concentration of water in cured concrete has been utilized to estimate the transfer of water during drying. In this study, specimens described in Figure 2 of N.B.S. Report 4869, October 1956, have been prepared. The ceramic hygrometers originally ordered for this study were unstable, and the manufacturer decided to replace them with miniature hygrometers of the Dunmore type which had recently become available. Three quarters of the order has been filled.

Although these hygrometers were carefully calibrated by the manufacturer, some tests were made in this laboratory to detect the calibration drift. Known relative humidities were obtained by means of saturated salt solutions^{1/} and these

^{1/} Letter Circular LC1026, U. S. Department of Commerce, National Bureau of Standards, April, 1957.

humidities were used in checking the hygrometers. A comparison of the results with the manufacturer's data indicated differences ranging as high as two percent, with the exception of one hygrometer which was obviously defective. Inasmuch as different secondary standards were employed in the measurement of relative humidities in the two laboratories, the uncertainties involved by the use of these methods could account for such differences. The correlation of secondary standards with the primary standard lacks precision, and humidity measurements by means of secondary standards may be considered as art rather than a science.^{1/} In spite of the possible inaccuracies, the Dunmore type hygrometers have advantages of rapid response and reliable electric indicators, both valuable in measuring relative humidities which are progressively changing.

Hygrometers have been inserted in one set of tiles, as shown in Figure 2 of N.B.S. Report 4869, October 1956. These tiles have one three-by-three inch face exposed to drying at 40 percent relative humidity and 77°F. The humidities within the tiles and the change in weight of the tiles are being recorded.

2.3 Relation of Properties of Neat Cements, Aggregates, and Resulting Concretes

2.3.1 Weight Changes During Curing and Drying

Neat cement cubes (1 x 1 x 1 inches) were prepared from each of three cements, Portland, Lumnite, and Alcoa using the

same W/C ratio (0.275). These cubes were cured for 28 days in the fog-room and stored at 50 percent relative humidity at 73°F. Their weights were determined at seven-day intervals and the percent change in weight, based on one day weight, are shown in Figure 1. These curves indicate larger, but comparative, changes than those for concrete shown in Figure 1, N.B.S. Report 4767, July 1956. The larger ratio of exposed area to volume accounts for the more rapid rate changes. It should be noted that after 28 days drying the water content of the cubes ranges from 25 to 30.5 percent. These values are much higher than the values for concretes designed using these cements, which range from 6.5 to 8.5 percent. and very much higher than the aggregate used, which range from 0.3 to 1.7 percent. The Alcoa cement absorbed appreciably more water during curing than was absorbed by either the Lumnite or Portland, but its loss during drying was quite similar to that for Lumnite.

2.3.2 Resistance of Neat Cement Cubes to Jet Impingement

The cubes were retained, in contact with each other, in a holder, and arranged as shown in Figure 2 during the test, which was a five-minute exposure to gases at 1200°F at velocities of 1200 ft/sec.^{2/} Dummy cubes were used to frame the nine test cubes. This arrangement of the cubes was made to obtain an equal average temperature for the cubes of each

^{2/}

Temperatures and instrumentation of panels during test are shown in Figures 2 and 3, N.B.S. Report 4767, July 1956.

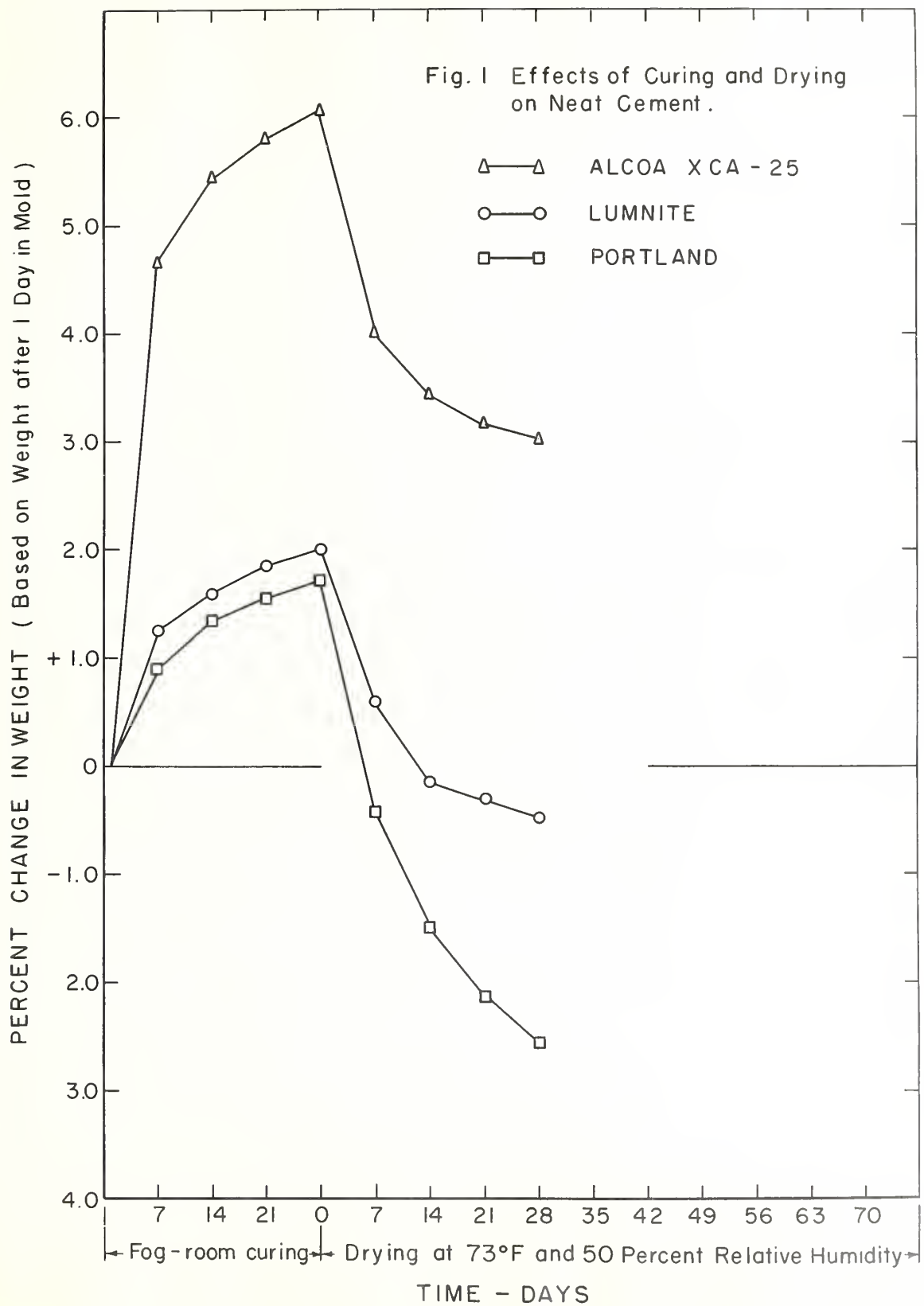
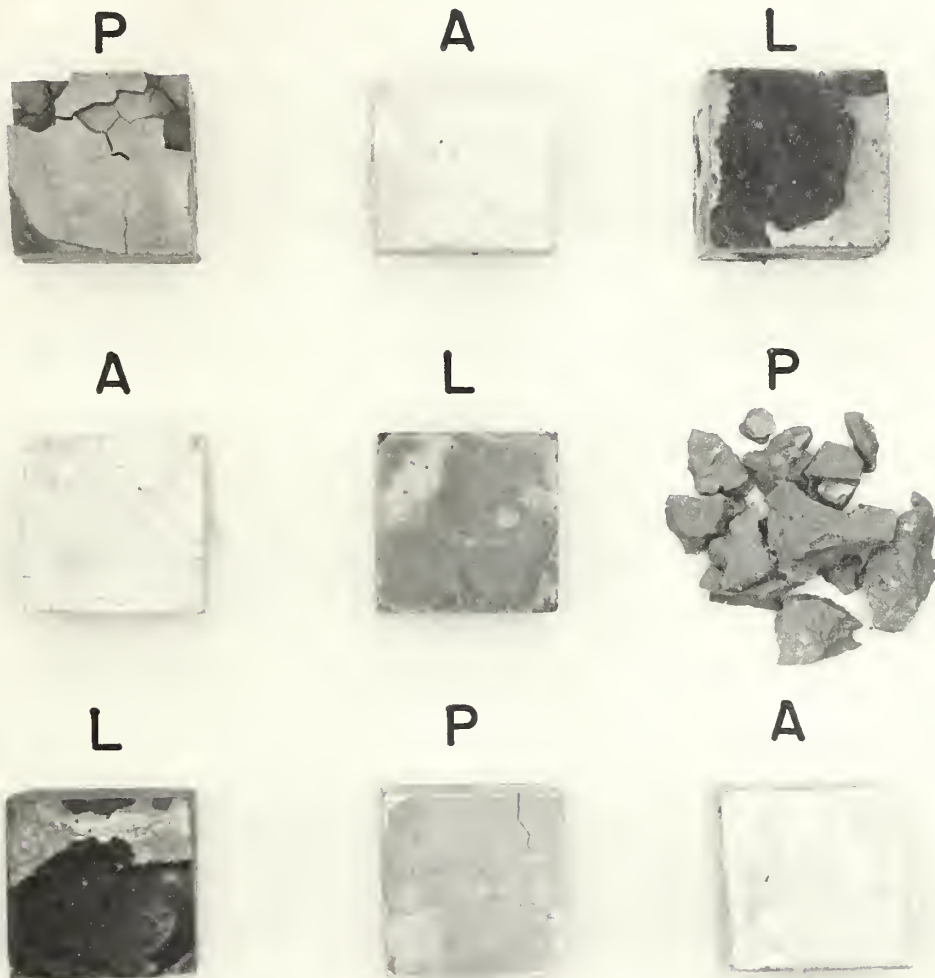


FIG.2 EFFECT OF JET IMPINGMENT TEST ON
NEAT CEMENT.

A = ALCOA XCA-25, L = LUMNITE, P = PORTLAND



composition. The temperature determined adjacent to the cubes and at their exposed surface ranged from 1320°F near the center to 1150°F at the outer edge.

All cement cubes failed in this first test (Figure 2). Although the Lumnite appears to be slightly superior to the others. One portland cube shattered completely. In general, the resistance of these cubes to jet-impingement was much less than that shown by the aggregate material alone, N.B.S. Report 4869, p. 3, October 1956. The concretes, of comparable age, designed using these cements and aggregates, differed in their resistance to the jet impingement ranging from severe failure to no appreciable failure.

2.4 Tests of Conventional Sand and Gravel Concrete Panels with a Protective Topping

Two (18 x 18 x 6 inch) panels were subjected to the jet-impingement test during the period covered by this report. These panels were fabricated in two layers. The backing layer was four and one-half inches thick and fabricated with the conventional sand-gravel-portland cement concrete and cured. The exposed layer or topping was one and one-half inches thick. The topping for one panel was designed with Lumnite cement and diabase aggregate. In the second panel Alcoa cement was substituted for Lumnite. The topping was cured for 14 days in the fog-room and stored for 14 days at 50 percent relative humidity and 73°F .

The impingement test caused no observable loss to the panel containing Lumnite cement. In contrast, the panel containing Alcoa cement showed considerable loss. The loss was greater than that shown by any other panel containing diabase aggregate.

The difference in the performance of the two panels cannot be explained on the basis of the water content at the time of test or by fabrication differences. A factor to which the difference might be attributed is permeability since the other properties were quite similar.

2.5 Thermal Conductivity

In the previous report, it was pointed out that high thermal conductivity of concretes designed with the high-alumina cement, Alcoa, might account for the better results obtained in the jet-impingement test compared with those containing portland cement. The thermal conductivity of a concrete designed with Alcoa cement has been determined^{3/} and found to be 6.55 Btu/hr.ft^3 (116°F/inch). This value is slightly less than that for other concretes of similar density as shown in Figure 3, N.B.S. Report No. 1817, June 1952.

^{3/} Thermal conductivity was determined by the Building Technology Division, Heat Section of the National Bureau of Standards.

2.6 A Study of the Mechanism by which Water Vapor Travels Through Concrete

A constant temperature chamber has been outfitted to hold the water-vapor-transfer apparatus. Improvement of the apparatus has continued. The plastic containers, used at first to hold a reservoir of an appropriate water-salt solution proved inadequate; the containers could not be properly sealed. The plastic containers were replaced by 125 ml ground stopper gas washing flasks. Air, under slight pressure, from the diaphragm pumps was bubbled through the water-salt solution at the bottom of the flask, and then circulated through the cylindrical chambers. This arrangement, however, did not prove quite satisfactory. The sodium chloride, used in one of the flasks, had a tendency to crystallize at the end of the bubbling tube and cause it to be stopped up. Hereafter, the air over the water-salt solution will be circulated without bubbling it through.

2.7 Miscellaneous

Three hundred pounds of emery was submitted by the Walter Maguire Company, Inc. for use as an aggregate in this project. A sieve analysis of the material indicated that the sizing of the emery, all passing a 3/8 inch sieve, would make it impracticable to design concrete for other than surfacing or topping. This material will be used as an aggregate in topping concrete test panels.

A review of the following articles was made during the quarter.

Crystal Chemistry of Hydrous Calcium Silicates; II
Characterization of Interlayer Water, George L. Kalousek
and Rustom Roy. J. Amer. Ceram. Soc., Vol. 40, No. 7, July
1957.

Examination of Tobermorite, $4-5\text{CaO} \cdot 5\text{SiO}_2 \cdot 5\text{H}_2\text{O}$, and
Xonotlite, $5\text{CaO} \cdot 5\text{SiO}_2 \cdot \text{H}_2\text{O}$, by infra-red absorption revealed
striking structural similarities and differences in the two
phases. In the 8 to 15μ region the absorption was the same
for both: the difference arose from the manner in which water
and hydroxyl ions were bonded. Tobermorite exhibited strong
absorption at 6.2μ , a band which is generally associated with
interlayer water, and at 2.9μ , a band generally attributed
to bonded (OH). The mineral Xonotlite did not show these two
bands but contained the band at 2.75μ generally associated
with free (OH). Synthetic Xonotlite prepared at 300°C was
essentially the same as the mineral, but samples prepared at
progressively lower temperatures exhibited the 2.9μ in
increasing intensity. The fibrous form of Tobermorite showed
a band at 6.5 to 7.0μ which increased in intensity with
increasing amounts of CaO in the solid. This band was also
found in the 14 a.u. 1.0 CaO:SiO₂ hydrate, but not in the
Xonotlite. The great volume stability of Xonotlite during
drying and wetting is readily explained on the basis of the

present results. Shrinkage of Tobermorite during drying at temperatures up to 650°C may be due to the removal of both interlayer water and bonded (OH). The changes in absorption during drying at room temperature were too small to permit drawing any conclusion. Similarities and differences between Tobermorite and certain clays are discussed.

The System $\text{CaO-Al}_2\text{O}_3\text{-H}_2\text{O}$, A. J. Majumdar and Rustum Roy, J. Amer. Ceram. Soc., Vol. 39, No. 12, December 1956.

Phase equilibria have been determined in the system $\text{CaO-Al}_2\text{O}_3\text{-H}_2\text{O}$ in the temperature range 100° to 1000°C under water pressures up to 3000 atmospheres. Only three hydrated phases are formed stably in the system: Ca(OH)_2 , $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 6\text{H}_2\text{O}$, and $4\text{CaO}\cdot 3\text{Al}_2\text{O}_3\cdot 3\text{H}_2\text{O}$. Pressure-temperature curves delineating the equilibrium decomposition of each of the phases have been determined and some thermochemical data have been deduced therefrom. It has been established that both the compounds $\text{CaO}\cdot\text{Al}_2\text{O}_3$ and $3\text{CaO}\cdot\text{Al}_2\text{O}_3$ have a minimum temperature of stability which is above 1000°C . The relevance of the new data to some aspects of cement chemistry is discussed.

The Chemistry of Cement and Concrete. F. M. Lea, C. H. Desch, Second Edition revised 1956. Edward Arnold Publishers, London.

Chapters IV, IX, X, XI and XVI pertaining to the composition of cements and hydrated products were reviewed.

U. S. DEPARTMENT OF COMMERCE

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NATIONAL BUREAU OF STANDARDS

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