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# NATIONAL BUREAU OF STANDARDS REPORT

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5736

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



U. S. DEPARTMENT OF COMMERCE  
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NBS PROJECT

NBS REPORT

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Refractories Section  
Mineral Products Division

Sponsored by

Department of the Navy  
Bureau of Yards and Docks

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Approved:  
Dr. Samuel Zerfoss  
Chief, Refractories Section

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REPORT  
ON  
THE  
RESULTS  
OF  
THE  
EXPERIMENTS  
ON  
THE  
EFFECT  
OF  
TEMPERATURE  
ON  
THE  
SOLUBILITY  
OF  
SOLIDS  
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BY  
J. H. P. L. VAN DER  
SANDT

PH.D. THESIS  
SUBMITTED TO THE  
FACULTY OF  
SCIENCE  
IN  
PARTIAL  
FULFILLMENT  
OF  
THE  
REQUIREMENTS  
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THE  
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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

During the period covered by this report five additional pressure-temperature curves were drawn from data taken in five tests in which a stainless steel bomb and mercury filling was used. All of the new data was obtained on concretes. The results of these tests that appear in Table 1 corroborate the findings previously reported.

The effect of the presence of the aggregate was to limit the temperature for initial pressure to values of 140 to 168°C which is only slightly higher than that for brick aggregate alone (133°C). The removal of capillary water by





Table 1. Data from Pressure-Temperature Curves for Neat Cements and Concretes.<sup>1/</sup>

Material <sup>2/</sup>	W/C Ratio	Water Content <sup>3/</sup>	Fog Room Curing Days	Evacuation		Temperature-Pressure Curves						Cooling			
				Drying		Temp. for Initial Pressure °C	Heating			Maximum Temp. °C	Pressure psi	Rapid Pressure Rise °C	Crosses Steam Curve °C	Temp. Offset at Pressures of <sup>4/</sup>	
				Time	Loss %		Temp. Offset at Pressures of <sup>4/</sup>							200 psi °C	400 psi °C
							200 psi °C	400 psi °C	850 psi °C						
F	0.275	27.5	12	none		170	+22	+17	+5	280	870	none	165	-1	nil
P	do	do	80	191	2.25	168	+23	+20	+5	274	740	272-274	185	+1	+1
P-B	0.65	15.2	13	none		147	+8	+7	+7	295	1100	none	170	-5	-5
P-B	do	do	28	144	1.55	168	+30	+25	2/ 5/	288	648	none	no	+30	+11
L	0.275	27.5	16	none		150	+10	+7	0	278	900	none	155	-5	-7
L	do	do	16	164	0.40	212	+25	+15	+5	280	900	210-225	150	-7	-5
L-B	0.65	15.2	28	none		150	+10	+5	0	293	1175	212	no	nil	-3
L-B	do	do	28	144	1.57	150	+15	+10	0	280	940	none	250	+5	0
A	0.275	27.5	7	none		105	+7	+8	0	290	1090	none	280	-15	-10
A	do	do	7	213	1.8	222	+35	+12	0	290	975	222-242	180	-3	-3
A-B	0.65	15.2	21	none		140	+7	+7	+11	285	770	none	182	+5	+7
P-D	0.65	15.2	28	none		150	+12	+7	+2	305	1410	300	450	nil	-2
P-D	do	do	56	144	2.26	145	+11	+7	-5	280	945	none	no	-5	-3
B		18.8	12	none		133	none	none	none	297	1230	none	275	-5	-5

<sup>1/</sup> Some of this data appeared in Table 1 of N.B.S. Report 5601.

<sup>2/</sup> P = neat Portland cement, L = neat Lumnite, A = neat Alcoa XCA-25

B = crushed building brick aggregate, saturated.

PB = concrete designed with Portland cement and crushed building brick aggregate.

LB = concrete designed with Lumnite cement and crushed building brick aggregate.

AB = concrete designed with Alcoa cement and crushed building brick aggregate.

PD = concrete designed with Portland cement and diabase aggregate.

<sup>3/</sup> Based on dry weight of batch.

<sup>4/</sup> Difference in temperature from Steam Curve at pressures indicated.

<sup>5/</sup> Maximum pressure 650 psi.





room temperature evacuation did not effect the temperature of initial pressure possibly due to a development of open pores. This performance is in contrast to the behavior of the Lumnite and Alcoa neat cements for which specimens without capillary water had an appreciably higher temperature of initial pressure.

The characteristics of all the pressure-temperature curves were discussed in detail in N.B.S. Report 5601.

It should be noted that all of the concretes studied had temperatures of initial pressure within the range of 140 to 168°C regardless of their condition of dryness. Pavement concretes of the same design can be expected to develop pressures in the same temperature range.

Since in all hydraulic cements, or concretes containing such cements, the ever present damaging water, capillary and/or combined, is present and some means must be developed for the egress of such water.

#### 2.1.2 X-ray Examination

Samples of Portland, Alcoa, and Lumnite cements were taken from the bomb charges for X-ray examinations.<sup>1/</sup> The samples were taken after hydration, after evacuation at room temperature, after the heating cycle of the bomb test, and

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<sup>1/</sup> X-ray examination made by S. Schneider, Refractories Section.



after heating to constant weight at progressively higher temperatures. The purpose of this study was to gain more information concerning the hydrates present in the cements after the several treatments and to determine the maximum temperature for complete hydration of these comparatively stable compounds containing only a small percentage of water. The crystalline materials identified by X-ray are given in Table II. The amorphous compounds are not included.

The alumina present in the six samples may have been added as excess alumina during the process of manufacture. The hydration of the cement was not completed, during the 14-day curing period, as indicated by the presence of anhydrous cementing materials, CA and  $CA_2$  but these compounds were apparently reduced to non-detectable amounts during the bomb test. The hydrates  $C_2AH_8$ ,  $CAH_{10}$ , and  $AH_3$  were present in the cured cement but were not detected following the bomb test. The hydrated compounds present following the bomb test were AH and  $C_4A_3H_3$ <sup>2/</sup> which persisted even after heating at 600°C in air atmosphere. The temperature will be increased to determine the maximum necessary for the stability of these compounds. The heat treatment such as that occurring

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<sup>2/</sup> This compound is the same as reported by Johnson and Thorvaldson, "Hydration of the Aluminates of CA: V Hydrothermal Decomposition Products of  $C_3A$  at 350°C." Can. J. Research 21B, 236-46 (1943).



Table II. Compounds, Identified by X-ray in Alcoa XCA Cement-<sup>1/</sup>.

Treatment	A <sup>2/</sup>	CA <sup>2/</sup>	CA <sup>2/</sup>	C <sub>2</sub> AH <sub>8</sub>	CAH <sub>10</sub>	C <sub>3</sub> AH <sub>6</sub>	AH <sub>3</sub>	AH	C <sub>4</sub> A <sub>3</sub> H <sub>3</sub>	Z
Bomb #13 Before Bomb Test - 14 days Curing	X	X	X	X	X		X			
Bomb #13 Plus 7 Days Evacuation	X	X	X		X		X?			
Bomb #12 After Bomb Test Max-Temp. 280°C	X							X	X	
Bomb #12 After Bomb Test +200°C	X		X			X		X	X	
Bomb #12 After Bomb Test +300°C	X					X <sup>3/</sup>		X	X	
Bomb #12 After Bomb Test +600°C	X							X <sup>3/</sup>	X <sup>3/</sup>	X <sup>4/</sup>

<sup>1/</sup> A = Al<sub>2</sub>O<sub>3</sub> ( ), C = CaO, H = H<sub>2</sub>O.

<sup>2/</sup> Anhydrous cement.

<sup>3/</sup> Shift of X-ray reflections probably indicate dehydration of compound.

<sup>4/</sup> Unidentified compound: Similar to C<sub>4</sub>A<sub>3</sub>H<sub>3</sub> - Strong Line d = 3.1628.



in the bomb test or jet impingement test, where water is retained, produces a different set of hydrated compounds in the cured cement.

## 2.2 Water in Concrete During Curing and Drying

The study of the correlation of the concentration of water to the humidity within cured concrete is being continued. Specimens described in Figure 2 of N.B.S. Report 4869 were fabricated, humidity, and weight readings are being taken while drying at 35 percent relative humidity at 77°F.

The hygrometers furnished were miniature Dunmore type and had a calibration accuracy of  $\pm$  two percent. Four sets of hygrometers are necessary to indicate the humidity throughout the range of 40 to 100%.

Large errors were noted when hygrometers were substituted in order to change the range, and the first set did not indicate 100 percent when inserted in the tile, upon removal from the fog room. These large errors seem to be corrected by sealing the assembly joints with Glyptol cement. These joints were exposed to the flow of the drying air.

Another source of error was the unreliable seal between the concrete tile and the plastic, moisture proof, jacket. This seal has been improved, in preparing a new set of tile now under study, by cementing the plastic sheet to the tile





with, Pliobond. The sealing is important when determining the moisture gradient within the tile at different depths.

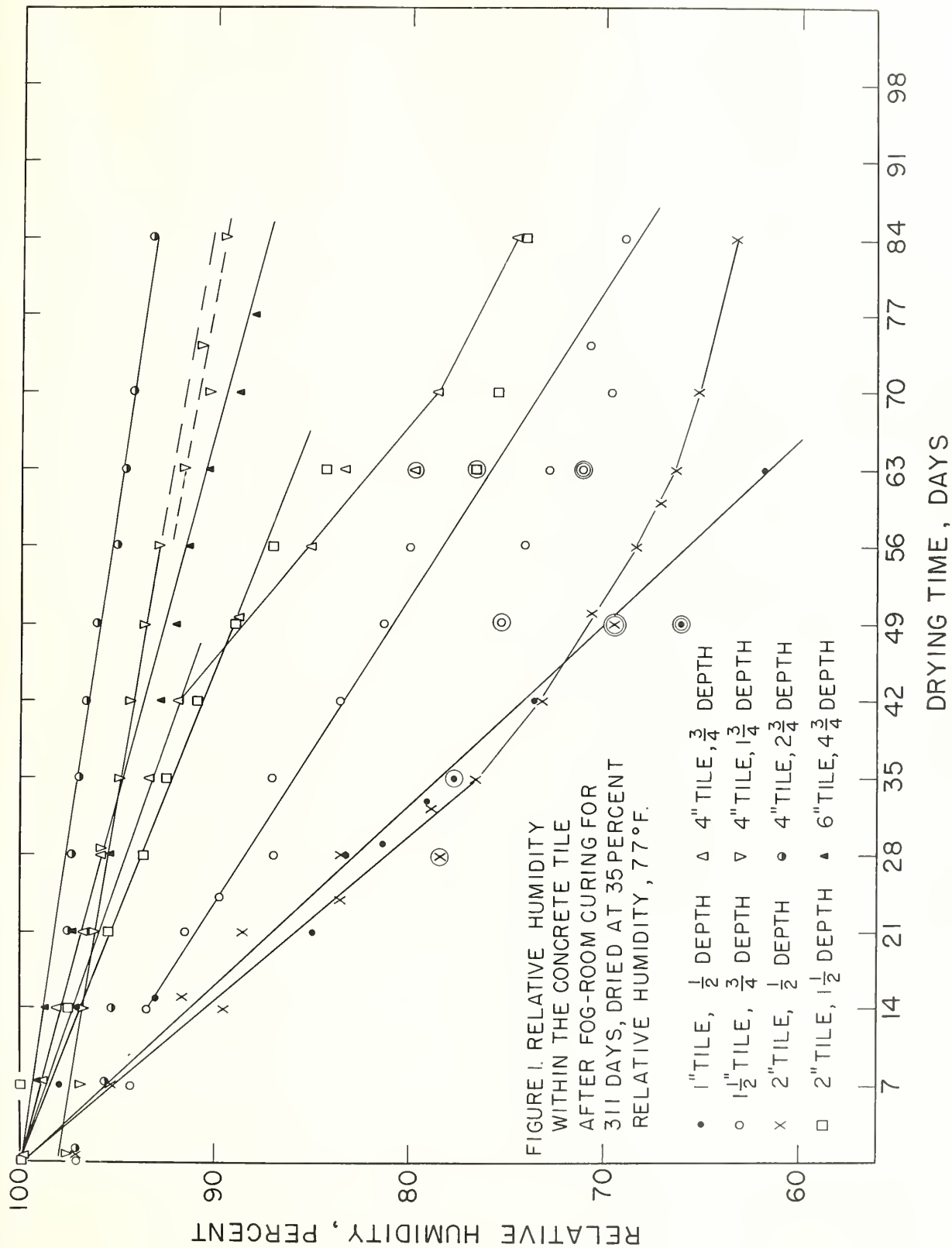
The relative humidity at a given position in the concrete test tile and the time of drying was plotted in Figure 1. For the higher humidities their relations were linear but the error in the measurements made this relation uncertain at the lower humidities. The main source of error was the substitution of hygrometers. The first reading of the replacement hygrometer is indicated on the graph, Figure 1, by single or double circles around the symbol (data point) for the second and third humidity ranges, respectively.

Although some of the data for these replacements were within the tolerance, the offset greatly effected the slope of the line. It is believed that this error was due not only to an exchange of air in the tile cavity but also to leakage in the assembly joint.

A comparison of the humidities after 63 days of drying indicated (1) relative humidities from 61.8 to 66.5 at the one-half inch depth in the one, two, and six inch tiles; and (2) a range of 76.7 to 82.0 at three-quarter inch depth in the one and one-half and four inch tile.

The increase in humidity with depth was also indicated from the data for the two and four inch tile but the data for the six inch tile, not included in Figure 1, was contradictory.







In general low humidities were found near the surface of the tile and increased with depth. However, the humidity near the wet face (opposite the exposed face) of the six inch tile was unexpectedly less than that of a similar location in the four inch tile. Such discrepancies were assumed to result from the uncontrollable variation in fabrication of the tile.

As pointed out in N.B.S. Report 4767 the water exchanged in a specimen depends on the composition of the concrete, the depth of the specimen, and the drying treatment. The rate of drying of an individual tile can be expressed as a constant; calculated from weight loss curve according to equation (2) of this report (N.B.S. 4767). Drying constants ( $a_d$ ) were calculated for tile (Figure 1) in the present study and plotted with the rate of humidity change (for the period of 35 to 42 days) in Figure 2.

For this graph the rate of humidity changes were those at mid-depth of the one and one and one-half inch tile and interpolated values at mid-depths for the two and four inch tile. The graph indicates considerable correlation in the rates of humidity change with water loss. This correlation will probably become somewhat different as the tile dries.





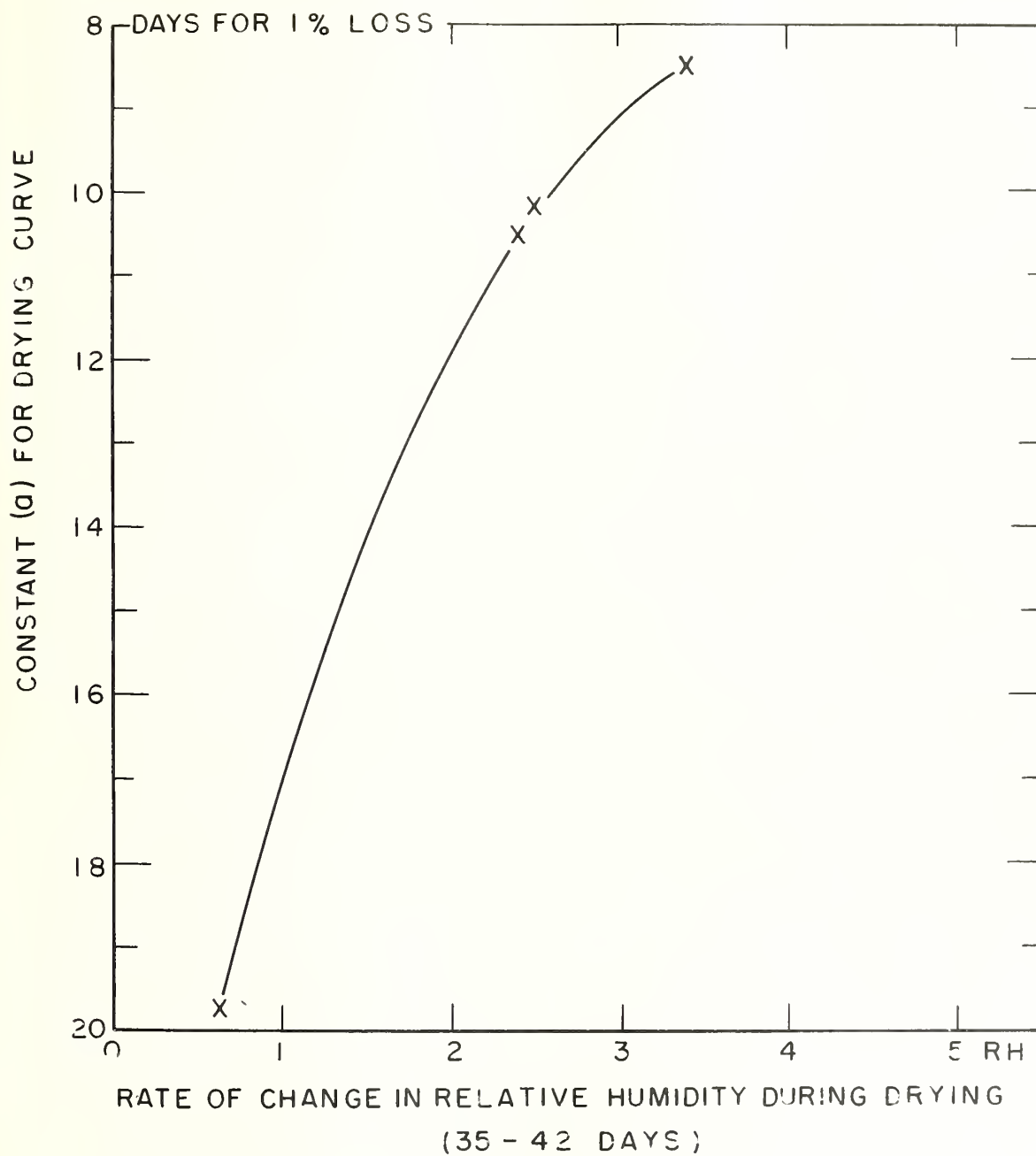


FIGURE 2. RELATION OF CHANGES IN RELATIVE HUMIDITY TO WATER LOSS.



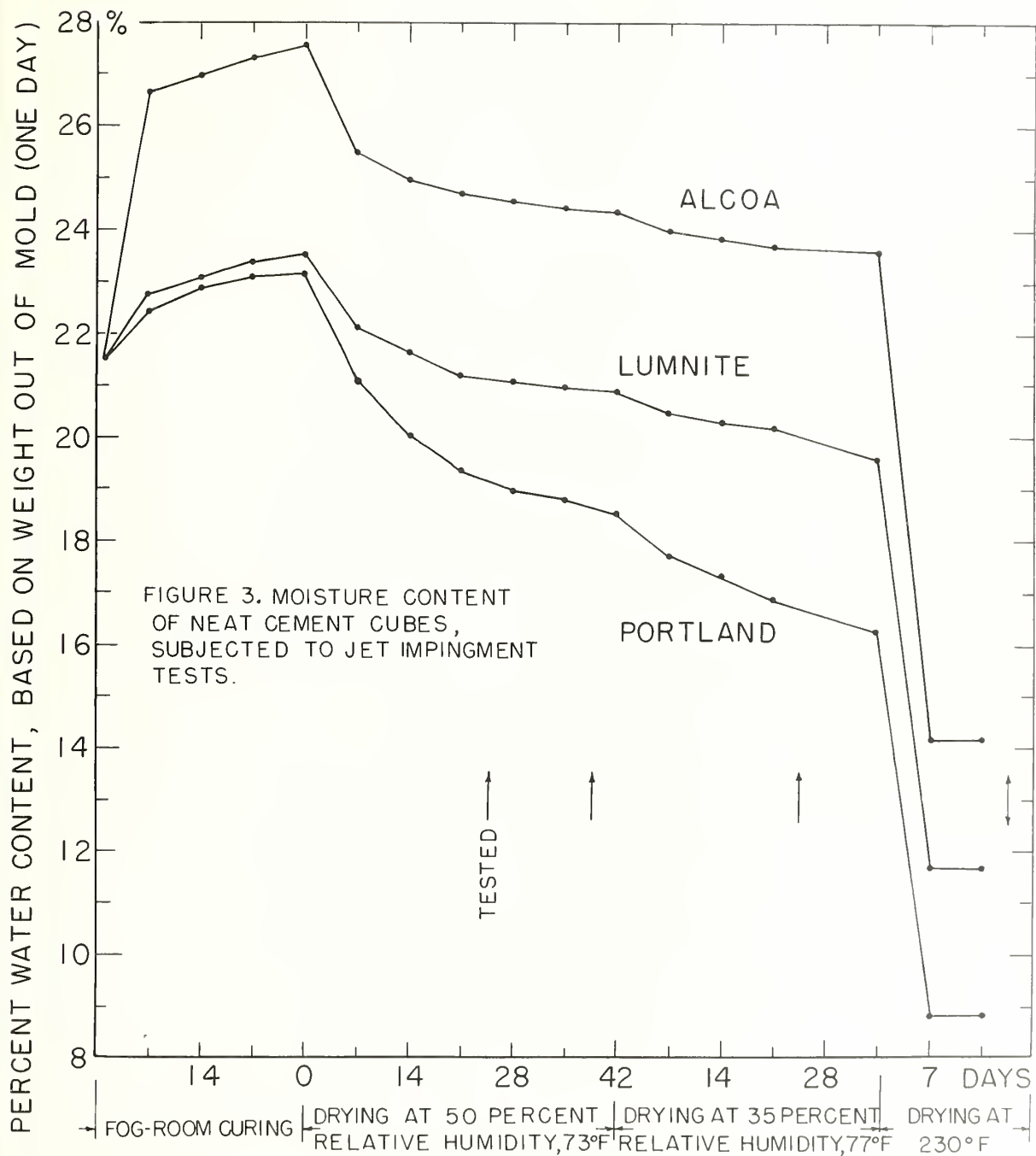
### 2.3 Resistance of Neat Cement Cubes to Jet Impingement Test

A set of 12, one inch cubes, were fabricated using neat Alcoa, Lumnite, and Portland cement. Twenty-seven and one-half percent mixing water was used. After a 28 day fog-room curing the cubes were subjected to drying treatments and jet impingement tests as shown in Figure 3. As previously reported all cement cubes fractured in the first test. The second set of cubes, after additional drying showed no improvement in the jet impingement test although there was a small reduction in water content. The third set indicated some improvement in their resistance to the test with a correspondingly large reduction in water content caused by the drying at 35% relative humidity at 77°F. The drying treatment at 230°F removed all the capillary water in seven days and the last set of specimens tested after 14 days at 230°F showed the best resistance to this test. The damage to this set was restricted to the structurally weak corners and edges.

### 2.4 Jet Impingement Tests on Topping Materials

Eighteen by eighteen by four inch panels were fabricated from conventional portland cement - sand and gravel concrete. These panels were finished with a two inch topping (total thickness six inches). Both topping mixes were designed with Lumnite cement, one contained diabase aggregate the other emery aggregate.







The design of the toppings was as follows:

	Diabase Topping	Emery Topping
Cement content, sack/yd <sup>3</sup>	7.5	14.7
Ratio of cement to coarse to fine, by weight	1:2.12:1.54 <sup>1/</sup>	1:0:2 <sup>2/</sup>
Water cement ratio	0.41	0.32
Gallons per cubic yard	44.9	52.3
Workability	very good	slight excess water

<sup>1/</sup> Maximum size on 3/4" screen.

<sup>2/</sup> All through 3/8" screen.

Both panels were cured 14 days in fog-room and dried 14 days at 50% relative humidity and 73°F.

These panels were subjected to the jet impingement test. The panel made with Emery topping, Figure 4(L-E) shows a large loss and the imbedded thermocouples are visible. No damage was apparent for the Diabase topping.





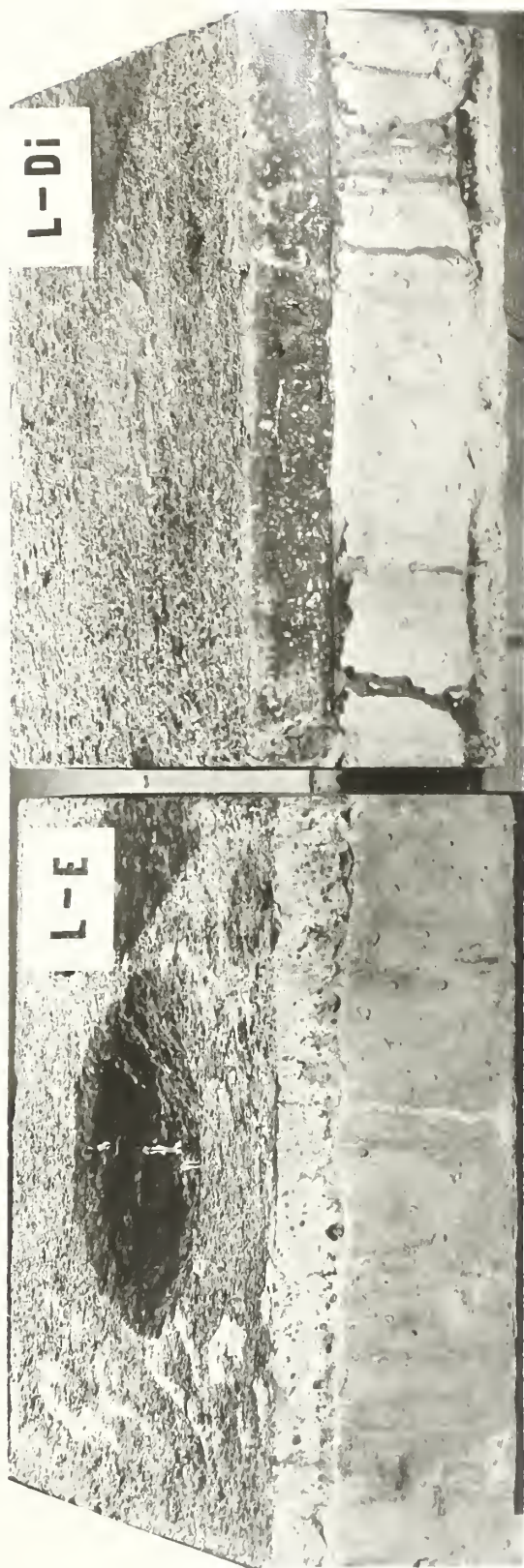


Figure 4. Panels Subjected to the Impingement Test Made With Emery Topping (L-E) and Diabase Topping (L-Di).



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