DEPARTMENT OF MATERIALS AND CONSTRUCTION

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A TECHNIQUE FOR THE DETERMINATION OF A THERMAL CHARACTERISTIC OF STONE

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SYNOPSIS

A technique has been developed for the direct determination of the thermal diffusivity of rocks. The particular application has been to crushed stone coarse aggregates used in Indiana concrete pavement construction.

Values of thermal diffusivity are given for two aggregates with good field performance and for two with poor field performance when used in concrete pavements. Values for both wet and dry stone are presented.

Increases in the value of thermal diffusivity of 20 to 60 percent were found for less than five percent saturation of the voids of the stone.

Studies at the Joint Highway Research Project of Purdue University (*10*)[2] have shown that the field performance of concrete pavements is frequently a function of the coarse aggregate used in their construction. These field studies were correlated with laboratory experiments which included freezing and thawing and thermal shock tests. The result was the separation of some aggregates into two classes—those with significantly good and those with significantly poor field performance of the concrete pavement (*8*).

The purpose of this study was to develop a method for the determination of the thermal diffusivity of rocks, to evaluate this diffusivity for typical coarse aggregates, and to determine the effect of absorbed water on this thermal property. It was hoped that an insight into durability factors might be gained by the study.

Thermal diffusivity, α, is defined mathematically as $\alpha = \frac{k}{\rho C_p}$ where

k = Thermal conductivity, Btu.-ft. per hr.-deg. F.-sq. ft.

ρ = Density, lb. per cu. ft.

C_p = Heat capacity, Btu. per lb.-deg. F.

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[2] Italicized figures in parentheses refer to the list of references at the end of the paper.

Thermal diffusivity is a property of the material and may be thought of as a measure of the rate of change of temperature of a substance when a thermal differential is applied to it.

Clark (*2*) and Clark and Birch (*3*) made measurements of the thermal conductivity of rocks and gave temperature, composition, compression, and wetting as variables. Niven (*7*) and Thomson (*9*) reported experimental methods and data on the conductivity and diffusivity of stone and concrete. With the exception of Thomson the authors referred to were interested primarily in the geological applications of the data obtained. High temperatures and extreme conditions of stress were covered. Niven (*7*) reported results which indicate that the thermal conductivity of stone decreases with increasing temperature and decreasing magnesium carbonate content. Clark (*2*) presented data indicating an increase of from 14 to 30 percent in the thermal conductivity of limestone by soaking the stone in water for 24 hours.

The data available in the literature fail to specify many important physical properties. Thomson (*9*) gave the specific gravity of the stone tested but did not state values for void space or chemical analyses of the stone, which are of great importance in the comparison of data. A fossiliferous limestone with a specific gravity of 2.74 had a diffusivity equal to 0.0137 sq. ft. per hr. while limestone with a specific gravity of 2.60 had a diffusivity of 0.0317 sq. ft. per hr., a difference of 130 percent. A corresponding difference in thermal conductivity (0.396 and 0.924 Btu-ft. per hr.-deg. F.-sq. ft.) was given.

Two limestones and two dolomitic limestones, one of each with good and one with poor field performance were chosen for the tests. Table 1 gives the physical properties of these rocks.

The approach decided upon was the direct determination of thermal diffusivity by the use of transient phenomena measurements. The method chosen used spheres of the rocks with a thermocouple mounted at the center and another at the surface. The sphere was given a thermal shock with steam and the temperature differential between the center and surface of the sphere was measured and plotted as a function of time elapsed after the thermal shock. The thermal diffusivity was then calculated from these data.

TABLE 1
PHYSICAL CHARACTERISTICS OF THE ROCKS TESTED (4)

	Good Field Performance		Poor Field Performance	
Source No.[a]	67-2S	1-1S	47-2S	9-1S
True Specific Gravity	2.77	2.87	2.77	2.87
Absorption, percent,				
24 Hr. Immersion	0.64	1.28	2.78	4.78
Vacuum Saturation	0.70	2.03	5.65	6.87
Saturation, percent,				
24-Hr. Immersion	45	54	72	65
Vacuum Saturation	48	87	93	97
Total Pore Volume[b]	0.041	0.066	0.108	0.240
Volume of Pores Larger Than 0.005 mm.[b]	0.024	0.044	0.026	0.143
Volume of Pores Smaller Than 0.005 mm.[b]	0.017	0.022	0.082	0.097

[a] Joint Highway Research Project code numbers.
[b] Expressed as a ratio of volume of pores to volume of solids.

Each stone was tested dry and after vacuum saturation to determine the effect of absorbed water on the diffusivity.

EXPERIMENTAL

The preparation of a sphere from the rough sample was done in four steps. A rough cube 2 in. on a side was broken from the sample. The cube was then chipped to a rough polyhedron. Next the piece was ground to a rough sphere of approximately 1½-in. diameter on an abrasive wheel, care being taken to keep the sphere cooled with water and to grind very lightly. In the last step this blank was reduced in the special rotary grinding fixture shown in Figure 1. The final grinding was done in a drill press or lathe, the initial work being done dry and the final steps with a slurry of rock powder and water. A grinding speed of 375 rpm. was used and three or four spheres were completed before it became necessary to refinish the soft steel fixture. The final result was a sphere of rock which varied from true by only a few thousandths of an inch.

The holes in the finished spheres were drilled perpendicular to the bedding planes by using the drill fixture shown in Figure 1 and positioning the sphere under the drill using the finger holes provided.

The thermocouple wire used met three requirements—small diameter to insure small heat capacity and quick response to temperature change, high thermoelectric power in the temperature range of 190 to 230 deg. F., and good reproducibility. General Electric Copper-Copnic thermocouple wire, number 36 A.W.G. (0.005 in. diameter) was found satisfactory. The wires, in series with 100 to 150 ohms, were electrically welded using an oil-covered mercury contact across 115 volts.

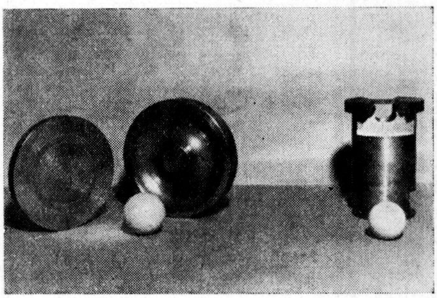

Figure 1. Grinding Fixture and Drill Fixture

Since the beads formed were approximately three times the wire diameter, those couples which were to be mounted in the centers of the spheres were annealed and reduced to wire diameter by grinding with a fine grade of emery paper glued to glass. The couples were re-annealed after being ground and were then examined under a low-power microscope for location of the couple interface and for possible faults.

The calibration consisted of comparing couple e.m.f.'s using a hypsometer. Matched pairs of couples were set aside for use as "surface" and "center" thermocouples for the spheres.

The thermocouples were then threaded full length through 0.025-in.-o.d. by 0.008-in.-i.d. by 24-mm., two-hole magnesia insulators. This type of insulation was found to be necessary because insulating lacquers and varnishes which were tried failed to wet the wire properly and gathered in drops on the leads, causing short circuiting due to incomplete coating.

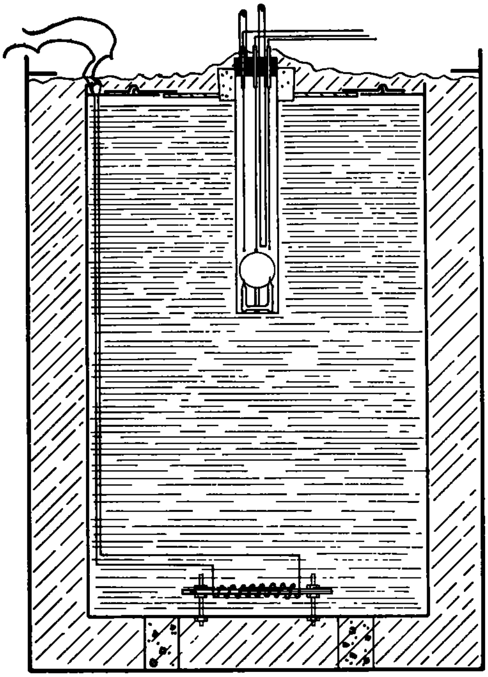

Figure 2. Test Apparatus with Sample in Place

The "center" thermocouples were fitted with small porcelain insulators over the magnesia insulator. The porcelain insulator acted as an adapter to mount the thermocouple in the $\frac{1}{16}$-in. diameter hole drilled to the center of the sphere. A cold-solder type of cement was found to be satisfactory for mounting the couples and for acting as a bond between insulators.

The test apparatus is shown in Figure 2. The constant temperature bath consisted of a 45-gal. drum as a container for a smaller drum of transformer oil surrounded by rock wool insulation. The heater element was 20 ohms of resistance wire wound on a transite board. Power input was regulated by a variac operating on 115-volt input.

The test container was a section of 50-mm. pyrex tubing $10\frac{1}{2}$ in. long and fitted with a rubber stopper containing steam inlet and outlet lines and three ports for the thermocouples. A cork stopper was used as a collar, fitting a hole cut in the Transite top of the constant temperature bath. The sphere rested on a glass tripod in the test vessel. Spines of glass were drawn on the tips of the erect legs, resulting in practically point contact and effectively isolating the sphere from contact with the container. Minimum size of the container was found to be important because larger containers proved to be too difficult to wash clear of air when the steam was switched into the test container.

The steam-line arrangement was such that the steam was allowed to exhaust to atmosphere until time for a run. The steam was then switched to the container inlet. The inclusion of a simple trap with a baffle to separate water from the wet steam was found to be necessary.

A Leeds and Northrup Portable Precision Potentiometer was used for the e.m.f. measurements.

The technique was designed to give the closest possible approach to the mathematical solution of conduction in a sphere (*1,5*). The specific mathematical solution of Fourier's conduction equation used in these experiments was that of a sphere initially at a constant temperature whose surface is changed, at time zero, to a new temperature and held there. The temperature difference between surface and center and the corresponding time determine the diffusivity according to the integrated equation of the form given by Jakob (*6*), Ingersoll (*5*), and Carslaw (*1*).

In this work the surface thermocouple and the center thermocouple were set up to read the temperature difference. A third couple was mounted in the vessel, and referred to a cold junction at the ice point, as a method of measuring the initial temperature and the temperature of the steam, fixing the initial temperature difference as well as indicating the temperature at which the determination was made.

For the "dry" tests the spheres were dried

24 hr. at 105 to 110 deg. C. in an oven, removed, and mounted in the test container. The container was placed in the constant temperature bath, the connections made, top insulation added, the steam inlet and outlet corked, and the sample allowed to come to equilibrium with the bath. When thermal equilibrium was reached across the sphere a run could be made. Steam was allowed to run through the exhaust line for fifteen minutes or more before being switched to the test vessel. When the steam was admitted to the test vessel a stop watch was started.

Since the temperature at the center of the sphere did not change rapidly, good results were obtained with the null-point instrument by having the operator at the potentiometer call "read" to an assistant when a balance was reached. The corresponding time was read by the assistant and the potentiometer reading was recorded. The time-temperature differential curve was thus built up by a series of points. The maximum temperature difference at time zero was obtained by reading the temperature in the vessel prior to the run by means of the reference couple and determining the temperature of the steam flowing through the container after the run.

The technique followed in testing the spheres in the wet condition was similar. The vacuum-saturated sphere was put in the test vessel, placed in the bath, and insulation added. It was, however, found to be necessary to leave the steam lines open to allow a certain amount of water to evaporate from the sphere before equilibrium could be reached. This resulted in the low values for the degree of saturation which was attained by the rocks. During this period of evaporation a constant temperature difference between bath and sphere was noted. When the degree of saturation dropped to less than ten per cent, evaporation had either ceased or had become very low, and a run could be started.

Three assumptions were made in these tests, i.e., that there was no penetration of the pores of the stone by the condensing steam film, that the surface of the sphere assumed the temperature of the steam rapidly and remained there, and that the film coefficient of

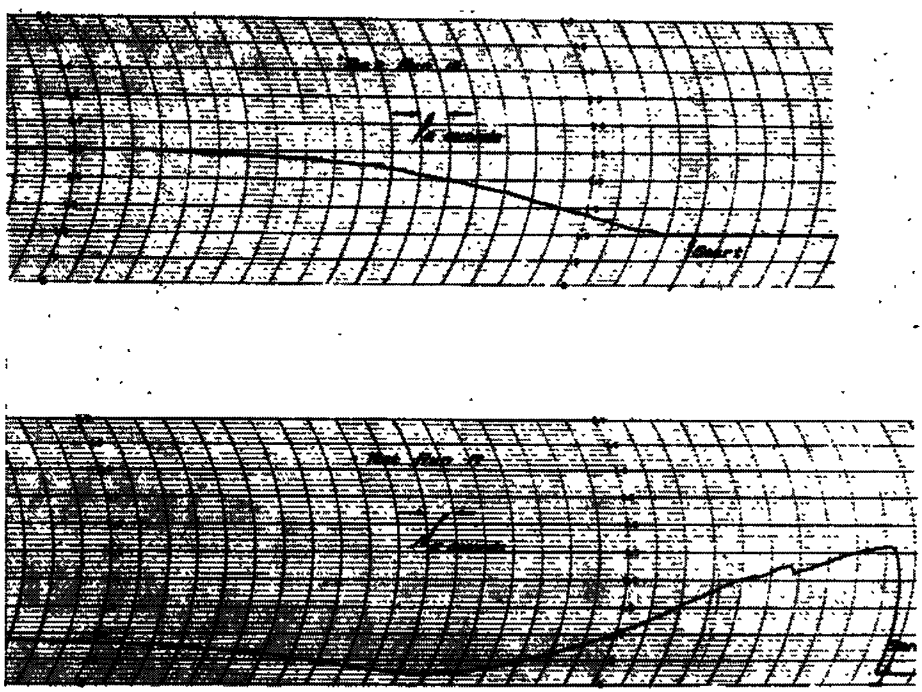

Figure 3. Recorder Strips for Test Runs Using Amplifier and Recorder

the condensing steam was very large relative to the thermal conductivity of the stone in spite of the air initially present in the container. The first two assumptions were checked experimentally and found valid, and the third was based on values obtained from the literature (*6*).

Figure 3 reproduces two records obtained by feeding the thermocouple e.m.f. to an amplifier and recording the amplifier output on an Esterline-Angus high-speed strip recorder. The charts read from right to left and the horizontal divisions are five seconds. The upper curve represents the temperature at the center of the sphere. The steam was admitted at the point marked "start". Some six seconds later the temperature at the center began rising and proceeded upward to an equilibrium value. The lower curve represents the temperature differential between center and surface of the sphere. The differential rose sharply to a maximum and then declined to an equilibrium value. (The little jump in the curve was due to a slug of water getting through the trap and into the test vessel. The trap was subsequently redesigned to prevent this occurrence.) These curves are not part of the data but are included merely to illustrate the thermal changes which took place in the sphere.

Since the runs averaged 1½ to 2 min. and, on the measurement runs, the signal for zero time was given by the observer manipulating the steam rather than by the observer at the recorder, an error of approximately one to one and one-half percent is believed to have been introduced by the assumption made concerning zero time.

In all runs the maximum initial temperature difference was adjusted to approximately five deg. F. in order to operate over a range wherein the thermal diffusivity could be regarded as essentially constant.

DATA AND RESULTS

Tables 2 and 3 present part of the data for dry and wet runs, respectively. Only eight runs out of a total of thirty-five are tabulated, since these tables are merely illustrative of the data obtained.

The solution of the basic differential equation for non-steady-state conduction of heat used in the calculations (see Jakob (6)) is of the form:

$$\frac{\theta}{\theta_0} = 2[e^{-(\pi^2 \alpha\tau)/R^2} - e^{-(4\pi^2\alpha\tau)/R^2} + e^{-(9\pi^2\alpha\tau)/R^2} - \ldots$$

Where θ = Temperature difference between center and surface at time τ
θ_0 = Temperature difference at time $\tau = 0$
α = Thermal diffusivity. sq. ft. per hr.
τ = Time, hr.
R = Radius of sphere, ft.

Since the series converges rapidly, an approximation is made by retaining only the first term in the brackets:

$$\frac{\theta}{\theta_0} = 2[e^{-(\pi^2\alpha\tau)/R^2}]$$

or

$$\alpha = \frac{\ln \dfrac{\theta}{2\theta_0}}{-\dfrac{\pi^2\tau}{R^2}}$$

Since only the ratio of the temperature differences is involved in the calculations and the calibration of copper-constantan thermocouples is substantially linear over relatively large temperature ranges, the ratio of the millivolt readings may be substituted for $\frac{\theta}{\theta_0}$.

The results are presented in Table 4. These final diffusivity values are averages of four runs each. It must be remembered that these are diffusivity values at steam temperature. They are not, however, expected to differ greatly from the diffusivity at lower temperatures since the temperature coefficient of thermal conductivity is small.

As a check on the method the following results are of interest: for sphere 9-1S-2, test run 19, using the amplifier and recorder, gave α = 0.053; test run 20, using potentiometer and stop watch, gave α = 0.0545, a difference of 2.8 percent.

The outstanding result of the experiments is the large change in the thermal diffusivity with a relatively small degree of saturation. The increases in diffusivity range from 20 to 59 percent for saturations of less than five percent. Whether the diffusivity increase would be proportionately large for higher saturations is an important question that cannot be answered at this time. The increase is logically thought to be due to a replacement of air, having a very low thermal conductivity, by water in the pores of the stone, and the greater such replacement the greater

TABLE 2
TEST RUNS OF DRY STONE[a]

Time, sec.	Millivolts		
		Test Run 27	
19	0.160	Sphere: 67-2S-1	
30	0.137	Diameter: 1.520 ± 0.001 in.	
45	0.107		
58	0.080	Final Reading (Steam)	4.234mv
72	0.054	Initial Reading	4.084
88	0.037		
103	0.025	Initial Difference	0.150mv
119	0.013		
		Test Run 24	
5.6	0.190	Sphere: 1-1S-1	
44.0	0.102	Diameter: 1.379 ± 0.002 in.	
54.0	0.058		
67.4	0.037	Final Reading (Steam)	4.270mv
78.4	0.026	Initial Reading	4.082
94.5	0.015		
109.0	0.008	Initial Difference	0.188mv
		Test Run 3	
19	0.215	Sphere: 9-1S-2	
73	0.195	Diameter: 1.409 ± 0.001 in.	
80	0.175		
94	0.150	Final Reading (Steam)	4.253mv
105	0.125	Initial Reading	3.828
114	0.107		
126	0.083	Initial Difference	0.425mv
139	0.060		
		Test Run 10	
14	0.122	Sphere: 47-2S-1	
25	0.108	Diameter: 1.654 ± 0.003 in.	
37	0.095		
47	0.080	Final Reading (Steam)	4.253mv
57	0.067	Initial Reading	4.134
71	0.050		
80	0.040	Initial Difference	0.119mv
89	0.030		
102	0.022		
113	0.015		

[a] In these tabulations and those of Table 3 the first points are doubtful because of the sudden initial change in e.m.f.; the last points are doubtful because of the difficulty of measurement of small e.m.f.'s. However, these points aid materially in indicating the trend of the curve.

the expected diffusivity. The often-contradictory and conflicting data existing in the literature for porous materials show the need for careful specification of moisture conditions existing in the voids.

Comparison of the values for the thermal diffusivity of the wet rocks indicates interesting correlation with absorption data and with volume of pores smaller than 0.005 mm. (Table 1). The thermal diffusivity increases in the order 67-2S, 1-1S, 47-2S, 9-1S and the maximum percent absorption increases in the same order. Furthermore, the volume of pores smaller than 0.005 mm. shows a corresponding increase. The correlation is not exact, but the trends are evident. It may, therefore, develop that the micropore structure of the rock is

TABLE 3
TEST RUNS OF WET STONE

Time, sec.	Millivolts		
		Test Run 32	
12	0.205	Sphere: 67-2S-2	
20	0.197	Diameter: 1.664 ± 0.001 in.	
31	0.177		
41	0.155	Final Reading (Steam)	4.247mv
52	0.130	Initial Reading	4.033
63	0.110		
78	0.083	Initial Difference	0.214mv
89	0.067		
101	0.055		
114	0.042		
		Test Run 25	
17	0.140	Sphere: 1-1S-1	
29	0.092	Diameter: 1.379 ± 0.002 in.	
38	0.065		
51	0.028	Final Reading (Steam)	4.255mv
60	0.017	Initial Reading	4.079
70	0.007		
		Initial Difference	0.176mv
		Test Run 30	
17	0.125	Sphere: 9-1S-2	
25.5	0.085	Diameter: 1.409 ± 0.001 in.	
35	0.048		
43.5	0.022	Final Reading (Steam)	4.251mv
51.5	0.000	Initial Reading	4.097
		Initial Difference	0.154mv
		Test Run 35	
15	0.156	Sphere 47-2S-1	
27	0.120	Diameter: 1.654 ± 0.003 in.	
38	0.098		
48	0.078	Final Reading (Steam)	4.268mv
61	0.051	Initial Reading	4.074
80	0.044		
94	0.036	Initial Difference	0.194mv
107	0.025		

responsible for pavement blowup susceptibility as well as for lack of resistance to freezing and thawing.

Dolomites are indicated to have higher diffusivities than limestones. This tendency is shown also by the data of Niven (*7*) and Clark and Birch (*3*).

The results of these tests alone are not sufficient to demonstrate the magnitude of the stresses set up in concrete by the difference in rate of temperature change between mortar and aggregate. The thermal stresses actually arise from the differences in the coefficients of thermal expansion at a rock-mortar interface. The thermal diffusivity values merely determine the relative temperatures. Thomson (*9*) gives 0.0126 sq. ft. per hr. as the values for α of dry, hardened, portland cement paste. It may readily be seen that the temperature of a piece of rock whose α is equal to 0.066 sq. ft. per hr. will change with sufficient rapidity under non-steady-state heating to produce stresses over and above those expected by assuming isothermal planes passing through the slabs of concrete.

TABLE 4
TABULATION OF RESULTS

Source No.	Thermal Diffusivity of Dry Rock[a]	Deviation from Mean[c]	Thermal Diffusivity of Wet Rock[a]	Deviation from Mean[c]	Saturation	Increase in Diffusivity due to Wetting
Good Field Performance						
	α	%	α	%	%	%
67-2S	0.035	2	0.044	15	3.5	25
1-1S[b]	0.041	2	0.056	5	4.5	38
Poor Field Performance						
47-2S	0.037	4	0.059	10	3.7	59
9-1S[b]	0.055	8	0.066	3	4.6	20

[a] Average of four runs, in sq. ft. per hr. and at 210 deg. F.
[b] Dolomitic limestone
[c] These values represent the maximum deviation of any one diffusivity value from the mean of its group.

The single greatest source of error inherent in this work is temperature measurement. A relatively small absolute error in measurement of the temperature differences could produce a large percentage error.

The limitations of the work are the inability to attain higher degrees of saturation, more commensurate with those found in the field, and the inability to obtain a statistically reliable indication of the accuracy of the method due to the limited number of runs.

SUMMARY

1. A method has been developed for the direct determination of the thermal diffusivity of rocks. This method is capable of precision if relatively large-sized spheres are used and if accurate temperature measurements are made.

2. The change in thermal diffusivity of rocks with moisture content is very large—on the order of 20 to 60 percent for less than five percent saturation of the voids.

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