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Thermal Shock Studies of Ceramic Materials

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Mechanics Division

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A test method has been developed and used at the U.S. Naval Research Laboratory for comparing the thermal shock characteristics of several ceramic materials. The method is simple and inexpensive making use of a circular disk of ceramic material containing a central hole. Thermal stresses are imposed by heating the inside of the central hole with a methane-oxygen torch. Thermocouples, placed at different distances from the central hole, give one information on temperature versus time for various torch flame temperatures. Additional information may be obtained on crack velocities in each material by painting concentric silver strips on the disk surfaces. By connecting each strip to an oscilloscope, one may follow the path of the fracture as the crack progresses across the plate.

Comparative thermal shock data were obtained for two types of alumina, a crystalline glass, and a borosilicate glass. Information obtained through these studies will be used in an attempt to correlate fracture toughness with various thermal shock parameters.

INTRODUCTION

Presently, there is great interest in ceramics and refractory metals, especially since the advent of man's exploration of outer space where there are a number of very critical problems involving high temperature and thermal shock. Thermal shock, as used here, means the rapid buildup of stress in a body due to high gradient thermal changes. The buildup however, is not rapid enough for shock waves to exist. To be sure, thermal shock is not a new problem. It has probably been a problem as long as ceramics have existed. While there is certainly some thermal shock information available, ceramicists have traditionally been more concerned with other problems and have been primarily satisfied with some don'ts where thermal shock is involved. For example, don't pour boiling water into a cold drinking glass.

TEST PROCEDURE

Materials

The materials were chosen in an arbitrary manner with availability and relatively nonhazardous nature (1) having a large part in the choice. The materials chosen were as follows:

1. Alumina A
2. Alumina B
3. Borosilicate glass
4. Crystalline glass

Specimens

It was decided that four types of specimens would be used; a 6-inch-diameter circular disk, notched and unnotched, and a 4-inch-diameter circular disk, notched and unnotched, all containing a central 5/8-inch-diameter hole and shaped as shown in Fig. 1. The shape was chosen for its simplicity, ease of handling, ease of mounting, and availability.

Apparatus

The test apparatus (2) was assembled as shown in Fig. 2. A torch was placed 4.6 centimeters above the specimen with a control thermocouple placed directly on the other side of the hole 15.0 centimeters from the torch. The control thermocouple was made from platinum-platinum (90%) rhodium (10%) and the torch was a natural gas-oxygen torch with a OX-5 tip.

Testing

Arbitrary heat numbers 1, 2, 3, 4, and 5 were assigned to different settings of the controls on the torch and the oxygen pressure (methane pressure was assumed not to vary with use). The settings of the controls were such that a sufficient

NRL Problem F01-05; Project RRMA 33-083/6521/R007-10-01. This is an interim report; work on this problem is continuing. Manuscript submitted October 15, 1963.

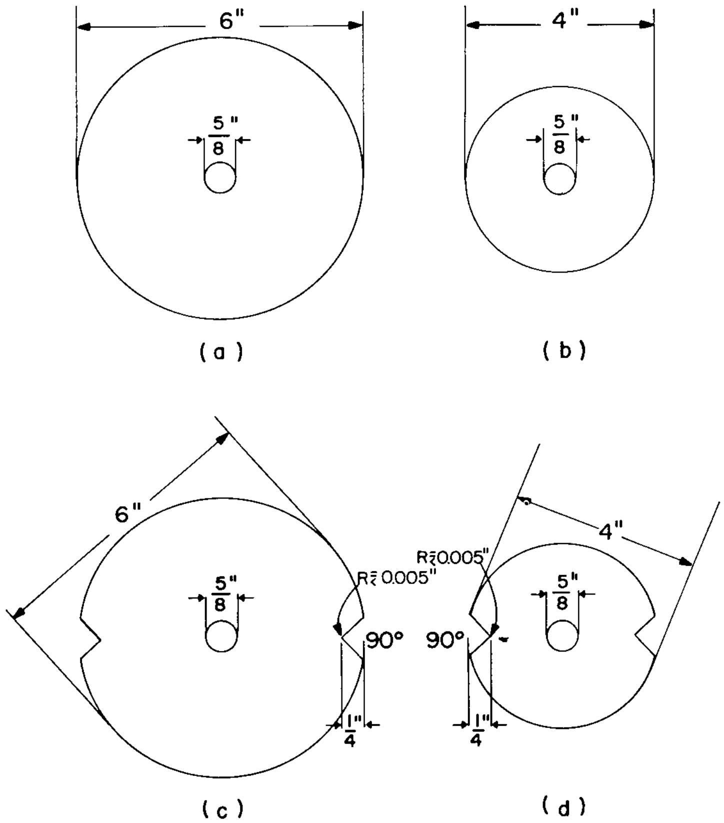

Fig. 1 - The four shapes of specimens. All specimens are approximately 1/4-inch thick.

degree of linearity existed to allow one to extrapolate to the nearest tenth of a heat number.

Specimens shaped as shown in Fig. 1a were prepared from alumina A, borosilicate glass, and crystalline glass (one of each material) with three thermocouples placed at 0.06 centimeter, 0.37 centimeter, and 1.65 centimeters from the edge of the center hole.

When the tests were run, temperature vs time was recorded for each thermocouple. The readings of the three thermocouples that were positioned on the specimen were extrapolated back to the edge of the center hole. These values of temperature vs time were then compared with the temperature of the control thermocouple and the heat numbers. This established the relationship between heat number, control-thermocouple temperature, and temperature of the edge of the central hole for any particular length of time.

This process was then continued for all five arbitrary heats. Now, for a given setting of the gas and oxygen controls, or more reliably for a given temperature of the control thermocouple, the thermal condition of the inside edge of the center hole can be determined for all similarly shaped specimens of the same material. Some typical temperatures for various time periods are shown in Tables 1-3 for the alumina A, the borosilicate glass, and the crystalline glass respectively.

The procedure for obtaining heat numbers vs time curves consisted of mounting the specimen properly, setting the gas and oxygen controls as desired, lighting the torch, and simultaneously starting a stop watch. When the specimen fractured due to stresses induced by heating the inside portion of the specimen, the watch was stopped. The exact heat number was derived by using the voltage recorded from the control thermocouple.

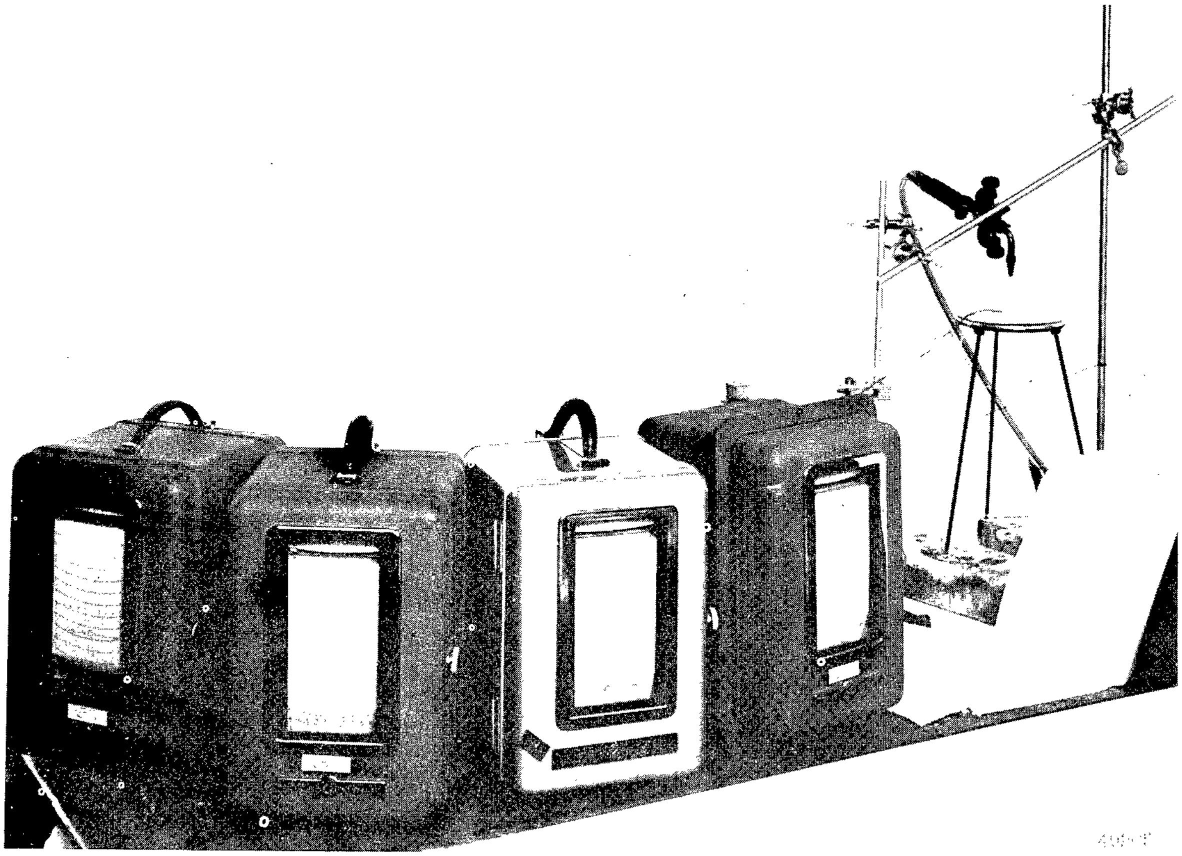

Fig. 2 - The apparatus used for testing

TABLE 1
Some Typical Temperature for Various Time Periods and Heat Numbers using Alumina A

Heat No.	Temperature at Indicated Times (°C)					
	13 sec	25 sec	38 sec	50 sec	100 sec	150 sec
1	205	240	275	290	300	310
2	390	465	535	565	590	605
3	575	690	790	835	870	900
4	760	915	1050	1110	1150	1190
5	945	1140	1310	1380	1435	1485

TABLE 2
Some Typical Temperatures for Various Time Periods and Heat Numbers using Borosilicate Glass

Heat No.	Temperature at Indicated Times (°C)					
	13 sec	25 sec	38 sec	50 sec	100 sec	150 sec
1	180	200	225	240	260	265
2	345	380	435	465	515	530
3	505	560	640	685	760	790
4	665	790	845	910	1010	1040
5	830	920	1055	1130	1255	1295

TABLE 3
Some Typical Temperatures for Various Time Periods and Heat Numbers using Crystalline Glass

Heat No.	Temperature at Indicated Times (°C)					
	13 sec	25 sec	38 sec	50 sec	100 sec	150 sec
1	145	165	170	175	180	190
2	270	310	320	330	340	360
3	400	450	470	485	505	535
4	525	595	620	640	660	705
5	650	740	770	795	820	880

The tests for fracture mode and crack velocity were carried out in much the same manner with the exception of the use of the instrumentation shown in Fig. 3.

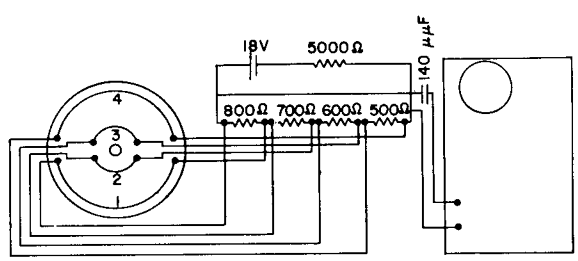

Fig. 3 - Instrumentation and silver painting of a 6-inch-diameter specimen for determining fracture mode and crack velocity

DISCUSSION

Mode of Fracture and Crack Velocity

The 6-inch-diameter specimens of both alumina A and alumina B were used to determine the mode of fracture and the approximate crack velocity. Several different methods of silver painting and instrumenting the specimens were tried. Of these methods, the most successful (3) is shown in Fig. 3. As previously mentioned, thermal stresses were induced in the specimens by heating the inside of the central hole with a methane-oxygen torch. When the specimen fractured, a picture was taken of the sweep across the oscilloscope showing the voltage change and when it took place. From this picture, the direction of crack propagation was determined and the average velocity of the crack was determined between any two circuits on the specimens. However· it was assumed in both these cases (the second more critically than the first) that the circuit broke just as the crack passed beneath it.

Considering the case of the alumina A specimen, the evidence seemed to support this necessary assumption. The fractured specimens and the corresponding oscillogram are shown in Fig. 4. The results of the data obtained from the oscillogram and the records obtained from the thermal shock tests are shown in Table 4.

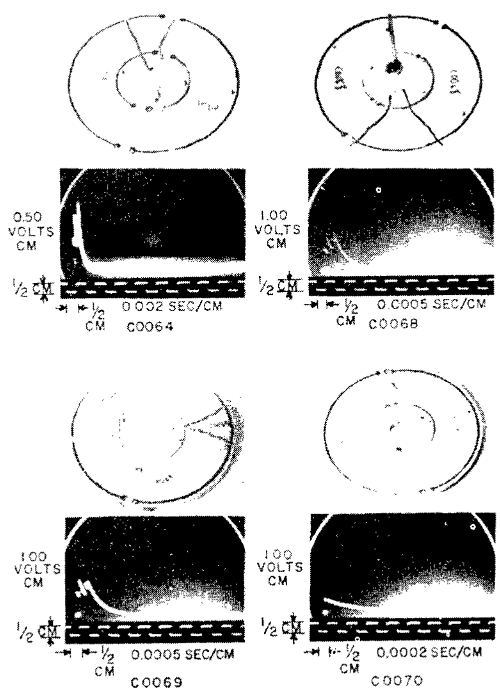

Fig. 4 - Fractured alumina A specimens with corresponding oscillogram

For the case of alumina B, the fractured specimens are very similar to those of alumina A, yet the oscillogram showed erratic breaking orders. This indicated that the breaking of the conducting strips at the proper time could not be depended upon. Nevertheless some information may be obtained regarding fracture velocity if it is assumed that the earliest that an outside strip broke was when the crack was beneath it or at the latest when the crack reached the central hole in the specimen. Also, that the outside strip on the other side broke when the specimen broke into pieces. All of the information obtained on alumina B specimens is given in Table 5 and Fig. 5. Branching of the crack occurs in most of these tests, indicating the attainment of a limiting upper velocity of crack propagation. This velocity is somewhat less than the theoretical limit of $2/\pi$ times the transverse sound velocity.

Notch Sensitivity

As might be expected all of the materials in both sizes were highly sensitive to notching. Since it was obvious that the effective diameter of a specimen was decreased when it was notched, one might think that this was really the reason for the apparent notch sensitivity. Such was not the case. The 4-inch unnotched specimen of any one of the materials showed better resistance to thermal shock fracture than the same material in the 6-inch size with a 1/4-inch notch on each side.

Neither crystalline glass nor the borosilicate glass would fracture due to thermal shock with any heat number tried using unnotched specimens of either size. However, it should be noted

TABLE 4
Direction and Velocity of Crack Propagation for 6-Inch Alumina A Specimens

Specimen No.	Heat No.	Breaking Time (sec)	Maximum Temperature Inside Hole (°C)	Breaking Order	Crack Velocity (ft/sec)		
					Initial Segment	Middle Segment	Final Segment
C0064	3.8	133.6	1100	4, 3, 2, 1	400*	800*	1000*
C0068	3.6	121.9	1050	1, 2, 3, 4	500	1440	1560
C0069	3.6	211.7	1100	1, 2, 3, 4	800	1460	1560
C0070	3.7	182.1	1100	4, -, -, -	< 400	—	—

*±100.

TABLE 5
Direction and Velocity of Crack Propagation for
6-Inch Alumina B Specimens

Specimen No.	Heat No.	Breaking Time (sec)	Breaking Order	Crack Velocity (ft/sec)
C0245	2.6	67.0	4, 3, 2, 1	3310, 2170, 2260, 1490-3110
C0246	2.8	65.0	1, 2, 4, 3	3570, 2990-6010
C0247	2.6	136.0	4, 3, 1, 2	2710, 3270-6860
C0248	2.7	54.5	1, 2, 4, 3	2710, 3580-7640
C0249	2.5	41.7	1, 3, 2, 4	3740-8040

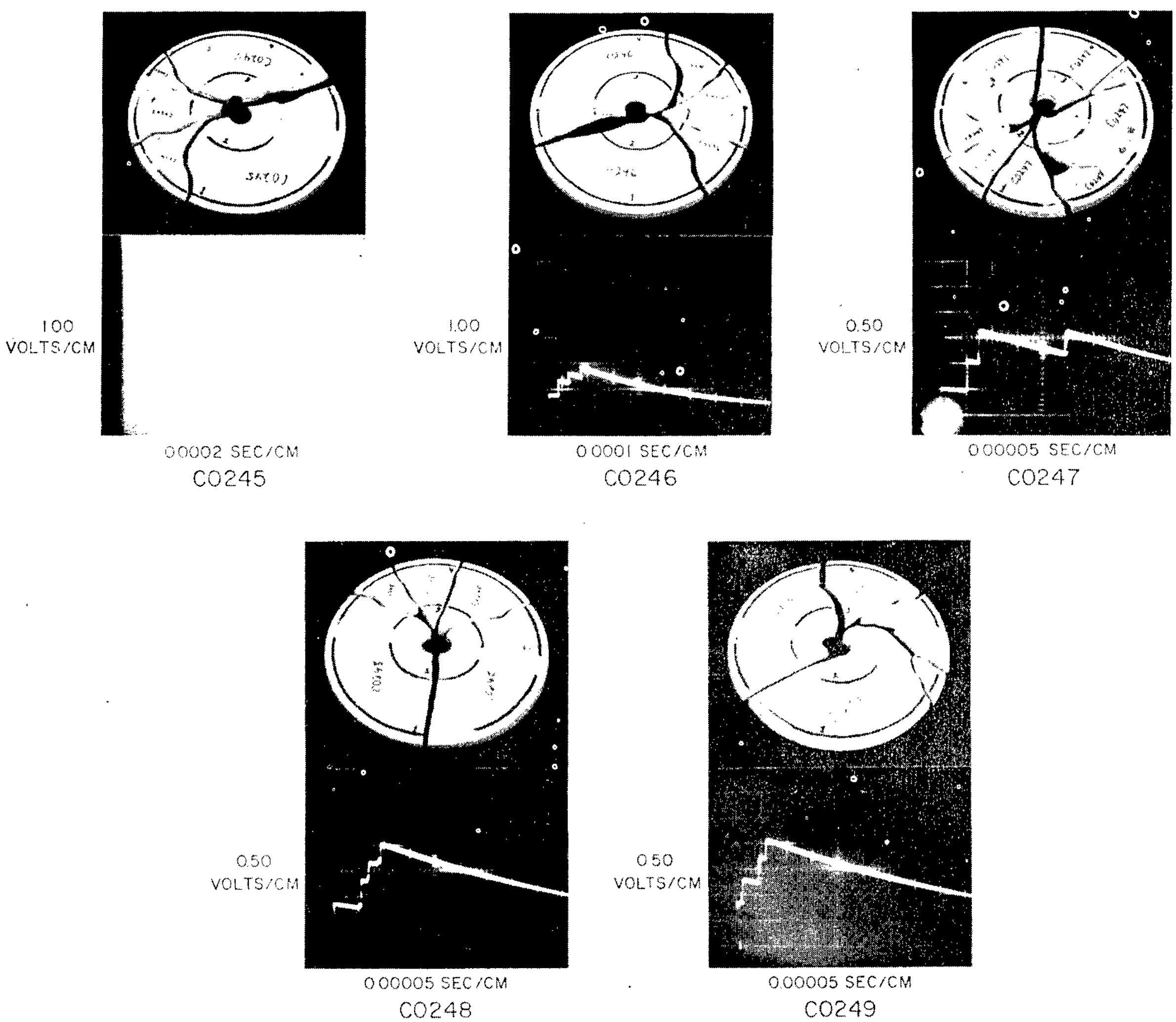

Fig. 5 - Fractured alumina B specimens with corresponding oscillogram

that the extensive melting in the hole and immediate area with the borosilicate glass specimens pointed up another problem that must be considered where materials are to be used at high temperatures (4-6).

For the case of alumina A and alumina B, the notch sensitivity of each material in both sizes is shown in Figs. 6-9. In Fig. 6 it is seen that the time for fracture due to thermal shock differed quite appreciably depending on whether the 6-inch specimens of alumina A were notched. At heat number 4 there possibly was a difference of as much as 100 seconds while at heat number 2 the difference seemed to be about 300 seconds.

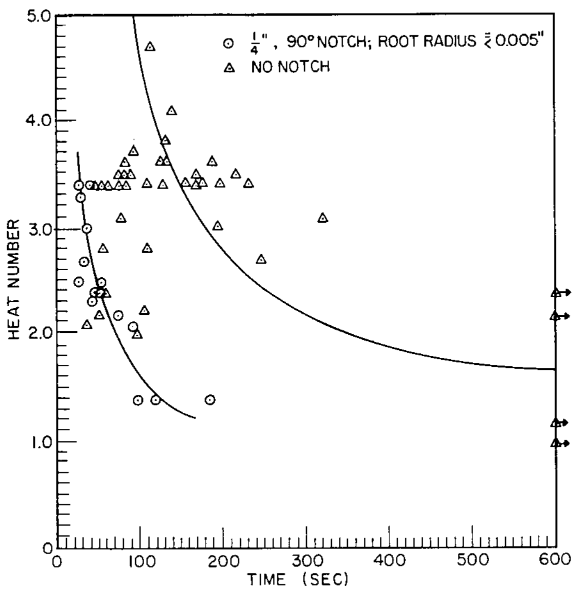

Fig. 6 - Notch sensitivity of 6-inch alumina A specimens

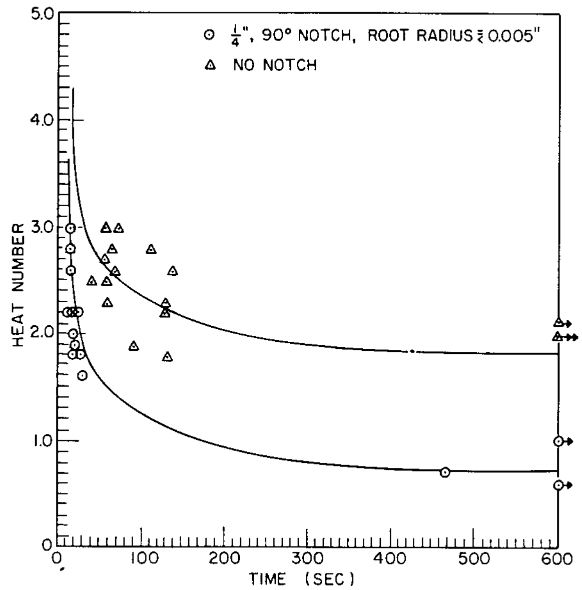

Fig. 8 - Notch sensitivity of 6-inch alumina B specimens

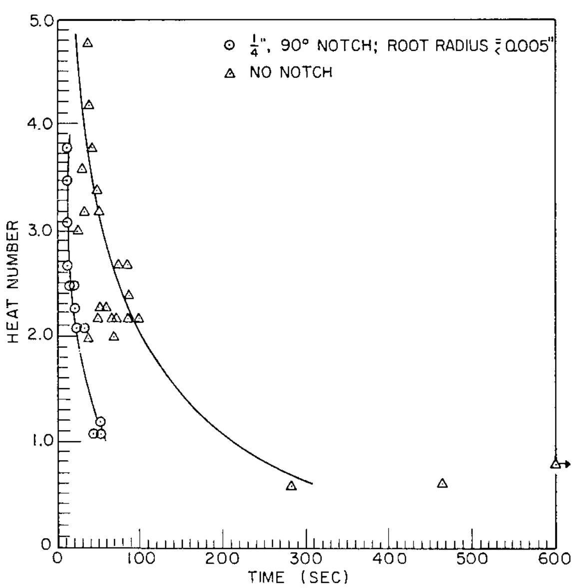

Fig. 7 - Notch sensitivity of 4-inch alumina A specimens

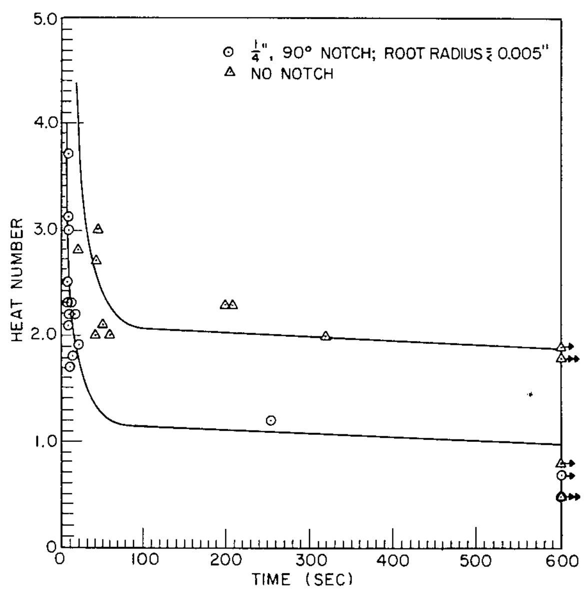

Fig. 9 - Notch sensitivity of 4-inch alumina B specimens

For the 4-inch alumina A specimen, the difference was much less. In Fig. 7, for example, at heat number 4 the difference seemed to be less than 25 seconds, while even at heat number 2 the difference was only about 75 seconds. Considering Fig. 8, it appeared that in the upper range there was not a great deal of notch sensitivity exhibited for the 6-inch specimens of alumina B; however, it appeared that only with great difficulty could an unnotched specimen be fractured by thermal shock at a heat number as low as 2 while notched specimens would fracture with relative ease as low as heat number 1. There was little difference worth noting in the inspection of the notch sensitivity data for 4-inch alumina B specimens in Fig. 9 as opposed to the data shown in Fig. 8.

For the crystalline glass and the borosilicate glass specimens the plots of heat number vs breaking time for notched specimens of both sizes are shown in Figs. 10-13. It should be pointed out here that since the unnotched specimens could not be fractured at all, then the crystalline glass and the borosilicate glass must be considered more notch sensitive than the two aluminas even though the notched thermal shock properties of these two materials might actually exceed those of the two types of alumina in some cases.

Effect of Specimen Dimensions

All materials which fractured in these tests, for both notched and unnotched specimens, differed in time to fracture vs heat number for

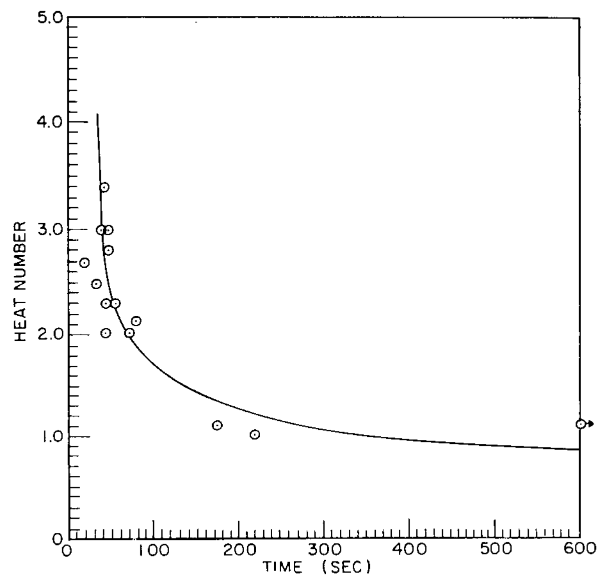

Fig. 11 - Heat number vs breaking time for 4-inch-diameter notched crystalline glass specimens

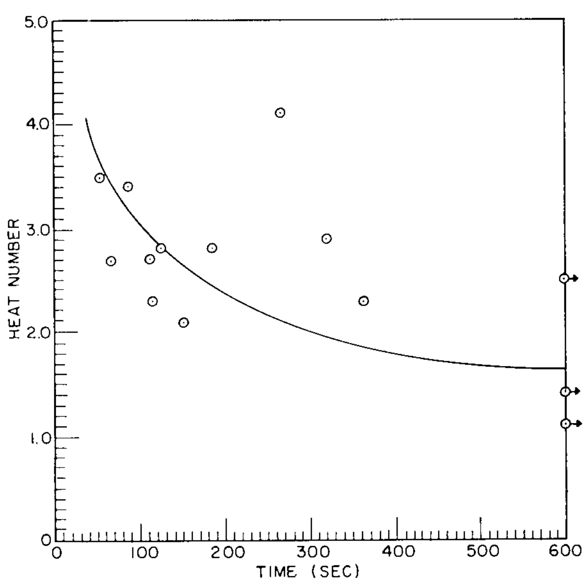

Fig. 10 - Heat number vs breaking time for 6-inch-diameter notched crystalline glass specimens

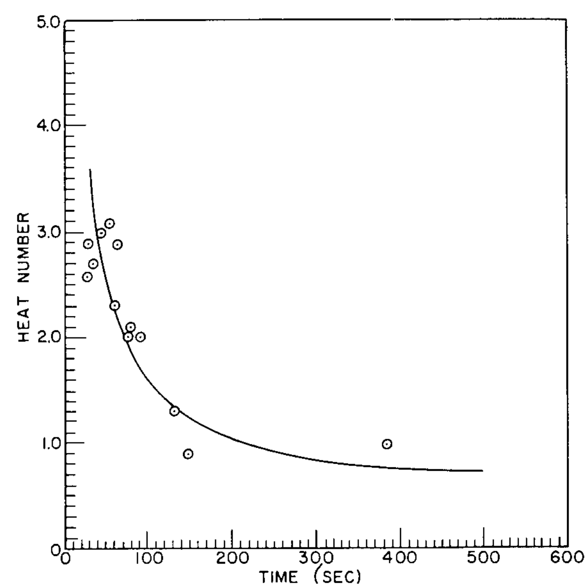

Fig. 12 - Heat number vs breaking time for 6-inch-diameter notched borosilicate glass specimens

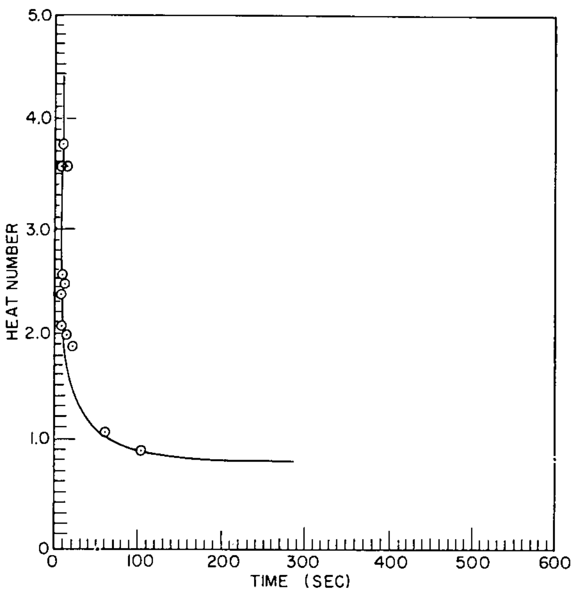

Fig. 13 - Heat number vs breaking time for 4-inch-diameter notched borosilicate glass specimens

the two specimen sizes. The two types of glass did not fracture in the unnotched condition. These differences are shown in Figs. 14-19. In Fig. 14 it is seen that this difference for unnotched alumina A was rather pronounced with a difference between the 4-inch and 6-inch specimens of nearly 100 seconds at heat number 4. The 6-inch specimens of alumina A can seldom be broken at heat numbers as low as 2 while the 4-inch specimens can often be broken at heat numbers even lower than 1. In Fig. 15 it is seen that for notched specimens of alumina A the effect of specimen dimensions was not as pronounced as in the unnotched specimens shown in Fig. 14. However, it was still significant, especially at the lower heat numbers. For alumina B, both notched and unnotched shown in Figs. 16 and 17, only a very slight difference existed between 4-inch and 6-inch specimens. It was difficult to tell if the crossovers really did exist as shown or if in fact there was no difference at heat numbers around 2 for unnotched specimens and heat numbers around 1 for notched specimens. Figure 18 shows that the effect of specimen dimensions for the notched crystalline glass was small at heat numbers above 4 but was of much greater significance at the lower heat numbers. The effect of specimen dimensions on the borosilicate glass shown in Fig. 19 was not very significant although certainly present. The greatest difference for the breaking times was 150 seconds.

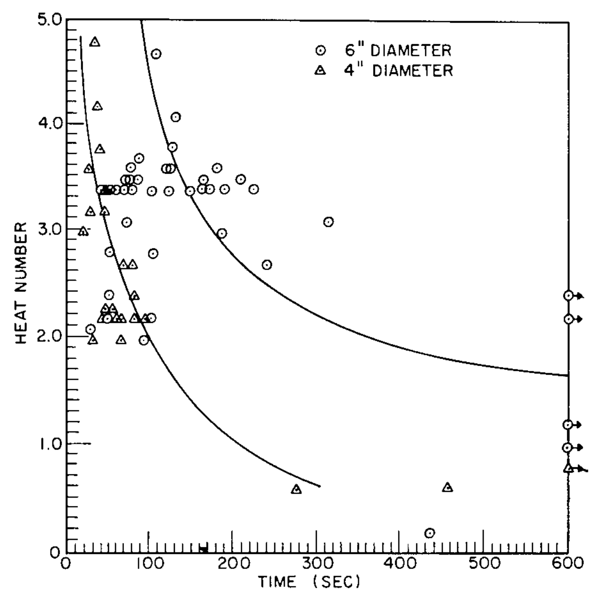

Fig. 14 - Effect of specimen dimensions for unnotched alumina A specimens

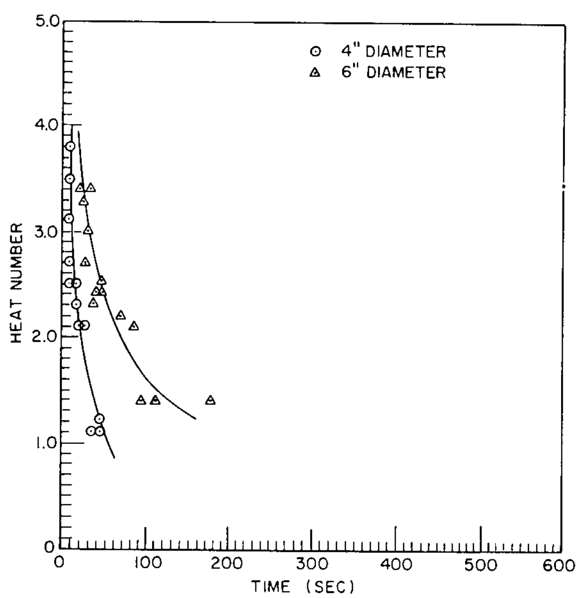

Fig. 15 - Effect of specimen dimensions for notched alumina A specimens

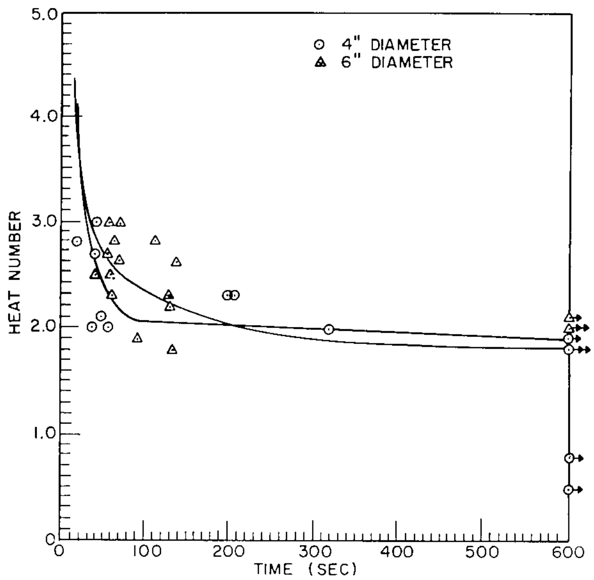

Fig. 16 - Effect of specimen dimensions for unnotched alumina B specimens

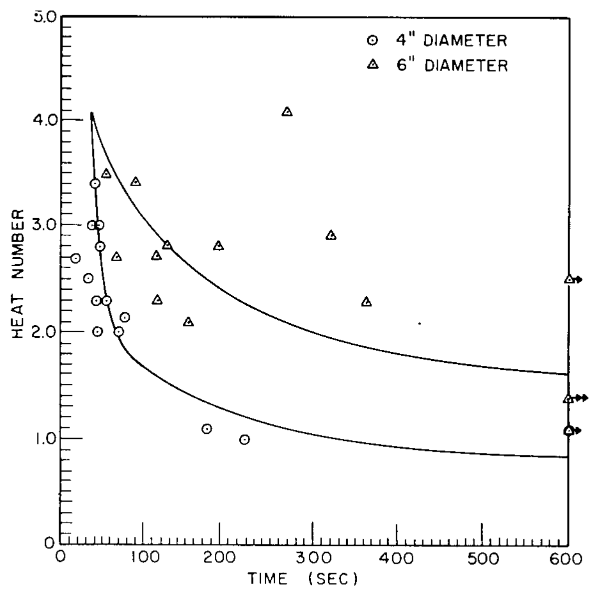

Fig. 18 - Effect of specimen dimensions for notched crystalline glass specimens

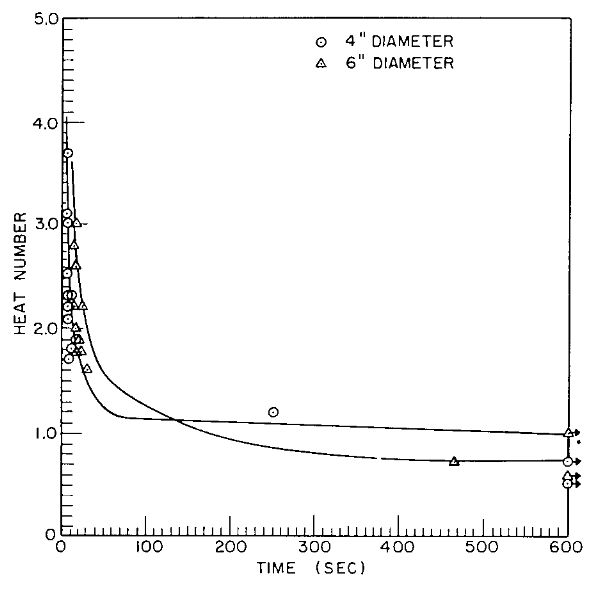

Fig. 17 - Effect of specimen dimensions for notched alumina B specimens

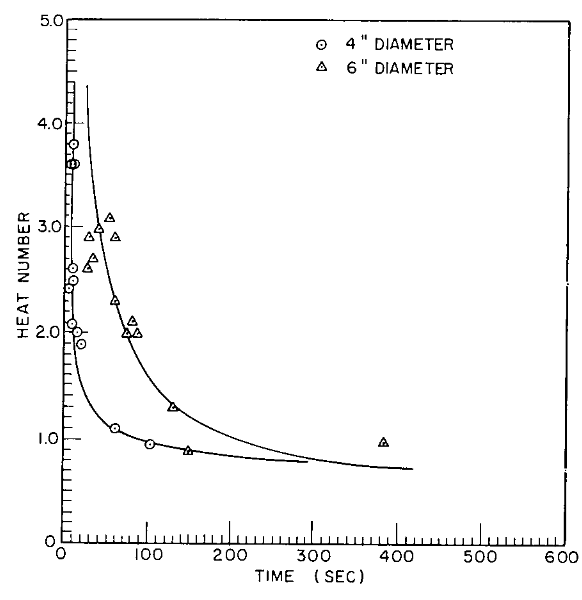

Fig. 19 - Effect of specimen dimensions for notched borosilicate glass specimens

Relative Resistance to Thermal Shock

As expected, each material showed a difference in resistance to thermal shock. No data were obtained for the borosilicate glass or the crystalline glass unnotched specimens by these methods. All of the data obtained is shown in Figs. 20-23. Figure 20 shows that for unnotched 6-inch specimens, alumina A is much better in its resistance to thermal shock than alumina B at heat numbers from

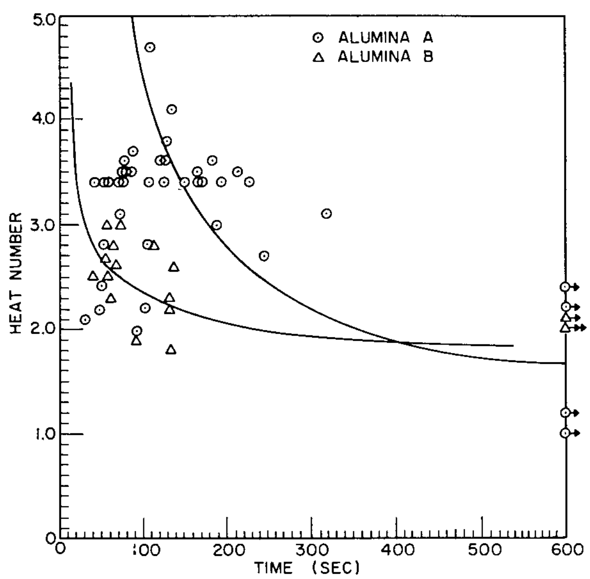

Fig. 20 - Comparison of the resistance of 6-inch-diameter, unnotched specimens of alumina A and alumina B to thermal shock

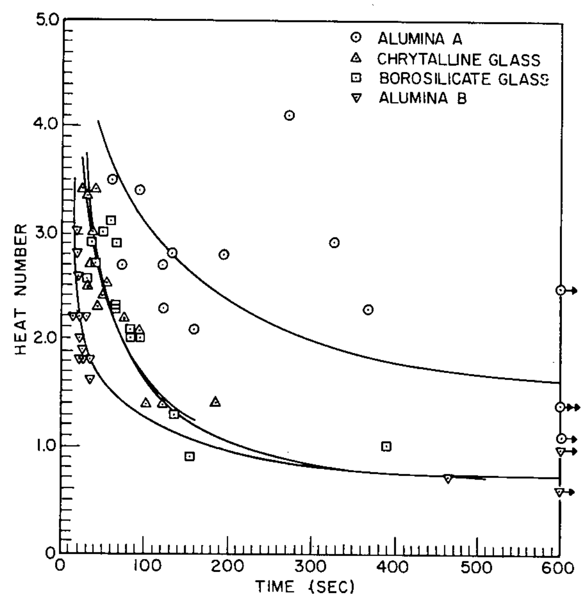

Fig. 22 - Comparison of the resistance of 6-inch-diameter, notched specimens of alumina A, alumina B, crystalline glass, and borosilicate glass to thermal shock

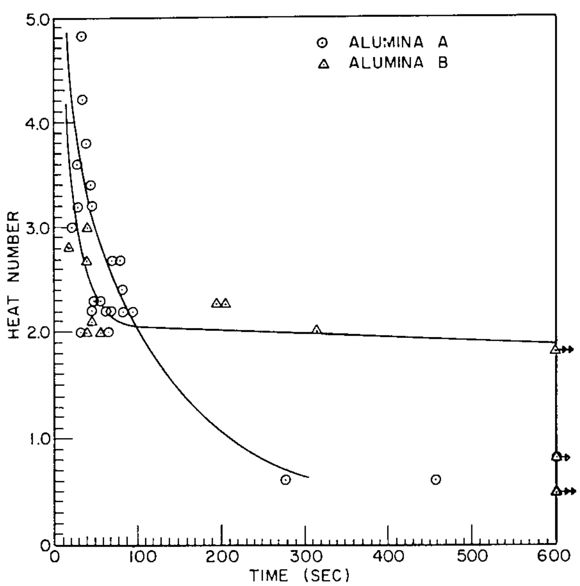

Fig. 21 - Comparison of the resistance of 4-inch-diameter unnotched specimens of alumina A and alumina B to thermal shock

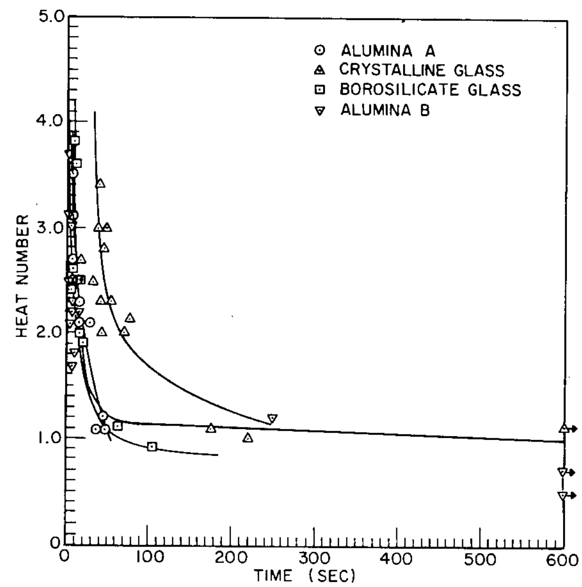

Fig. 23 - Comparison of the resistance of 4-inch-diameter, notched specimens of alumina A, alumina B, crystalline glass, and borosilicate glass to thermal shock

2 to 4. At heat numbers above 4 the difference began to decrease, and below heat number 2 there was little difference in the time required for thermal shock fracture. For the unnotched 4-inch specimens of the same two materials shown in Fig. 21, there was little difference between the two with alumina A somewhat better above heat number 2; however, a crossover was observed below heat number 2. Alumina B does not fracture due to thermal shock below heat number 2 while alumina A fractured as low as heat number 0.5. The relative ability of notched 6-inch specimens of all four materials to withstand thermal shock is shown in Fig. 22. This figure shows that alumina A did much better than any of the other materials with alumina B the poorest; the borosilicate glass and the crystalline glass are about the same with data points spaced about one-third of the distance from the alumina A curve to the alumina B curve. Figure 23 shows the curves for notched 4-inch specimens of all four materials. It is seen that here the crystalline glass was somewhat better than the other three materials, although for this particular type and size of specimen there was little difference between the four materials.

CONCLUSIONS

1. A simple methane-oxygen torch may be used to compare thermal shock characteristics of ceramic materials. Work is reported on here for two types of alumina, a crystalline glass, and a borosilicate glass comparing time to fracture as a function of torch flame temperature.

2. Fracture due to thermal shock in the specimen starts from some notch or flaw on the outside edge of the specimen; the crack runs to the central hole, and then on across, often branching before reaching the outside edge on the other side of the specimen. As cracking progresses the specimen opens up shifting the neutral axis (between tension and compression regions) allowing the fracture to propagate through regions that initially were in compression. Release of strain energy with crack propagation goes into further opening up of the specimen supplying the energy for creation of new fracture surfaces and for popping apart of the specimen parts.

3. As the crack velocity reached a limiting value, branching occurred for most of the tests reported on here. This is in accordance with past experience; however, this limiting velocity was somewhat less than the theoretical limit of approximately $2/\pi$ times the velocity of propagation of transverse waves in the material.

4. Notching does more than simply reduce the effective diameter of the specimen; it points up the notch sensitivity of these materials.

5. Resistance to thermal shock in unnotched specimens does not imply resistance to notch sensitivity for these materials.

6. The claim by Manson (7) that the effects of thermal shock should be checked only "under conditions that simulate as closely as possible the shock severity of the intended application" is substantiated. For specific applications the particular unit under consideration should be thermal shock tested in full scale wherever possible.

REFERENCES

1. Darwin, G. E., and Buddrey, J. H., "Beryllium," New York: Academic Press, Inc., pp. 368-376, 1960
2. McKinney, K. R., and Smith, H. L., "Nonmetallic Materials for Naval Weapons Systems," Report of NRL Progress, pp. 35-36, Sept. 1961
3. McKinney, K. R., and Smith, H. L., "Fracture Study on Short Thick-Wall Cylinders Subjected to Thermal Stresses," Report of NRL Progress, pp. 22-24, July 1962
4. Strauss, E. L., *Am. Ceram. Soc. Bull.* **42**:444 (1963)
5. Contract NOrd 19135(FBM), Washington, to Raytheon Company, Bedford, Massachusetts, pp. 62-124
6. Contract NOrd 18619(FBM), Washington, to Stanford Research Institute, Menlo Park, California, pp. 2-12
7. Manson, S. S., Machine *Design* **30** (June 26):99 (1958)