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NOTICE

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FOREWORD

This report was prepared by the University of Illinois in the Department of Ceramic Engineering, under USAF Contract No. AF 33(616)-87. The contract was initiated under Research and Development Order No. 615-17(D-B), "Ceramic Materials", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Lt R. J. Brinkman acting as project engineer.

ABSTRACT

A combination thermal cycling and compressive loading apparatus was constructed for investigating the effects of pre-compression on the thermal shock resistance of a pure oxide ceramic body.

It was found that selective compressive loads applied in the direction of the long axis of a rectangular bar composed of alpha alumina effectively increased its thermal shock resistance. This was indicated by a progressive increase in the average modulus of rupture of each of several groups of bars; each group being uniformly thermal shocked at a compressive load of 1000, 3500 and 7000 psi, respectively. It was also found that about one half of the total improvement was obtained with the initial load of 1000 psi.

The sonic technique was used as a method of detecting flaws in thermal shocked but unloaded specimens. When specimens were thermal shocked in compression, however, the sonic method, as used in this investigation, was unable to indicate the presence of flaws.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. E. SORTE
Colonel, USAF
Chief, Materials Laboratory
Directorate of Research

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I. INTRODUCTION

1. Statement of the Problem

The usefulness of ceramic components composed of pure refractory oxides in aircraft power plants is limited notwithstanding their many excellent properties. Although these materials possess adequate strength and chemical stability at high temperatures, their resistance to sudden changes in temperature (thermal shock) is quite low. When failures occur, it is usually because the "thermal stresses", induced by unequal expansion, exceed the tensile strength of the body. Brittle materials usually fail in tension when subjected to thermal shock, therefore, loading a specimen of this material in compression during all cycles of temperature change may conceivably delay failure and subsequently improve its thermal shock resistance.

The problem in this investigation was to determine if a compressive load of known magnitude applied to a test shape of pure oxide ceramic could improve its thermal shock resistance. Specifically, this involved the design and instrumentation of equipment by means of which thermal cycling of unloaded and loaded test pieces could be accomplished. A method of detecting, if possible, the presence of critical flaws, in the test piece, at the instant of their occurrance was also desired.

2. Scope of the Investigation

The investigation dealt principally with the effect of mechanically applied compressive loads on the thermal shock resistance of a pure oxide ceramic body. It was anticipated, however, that should the results of the study prove encouraging the methods used in it could be applied to an evaluation of various other ceramic and cermet materials. In a broader sense, the principle of compressive loading might be utilized in due time in engineering design to eliminate or at least to effectively minimize failures due to thermal shock and/or impact.

In this study only α alumina was used. It was chosen as the most suitable material for the following reasons:

1. It is inexpensive.
2. It has no polymorphous inversions.
3. It is stable at high temperatures.
4. It has no pyro-plastic tendencies up to 2000°F.
5. It requires no specialized processing equipment.

II. EXPERIMENTAL PROCEDURES

1. Specimen Preparation

a. Raw materials

As described in the scope of this investigation relatively pure aluminum oxide in finely divided form was selected for the test material. The specimen shape adopted as being most useful and reproducible was a rectangular bar nominally 1 in x 1 in x 6 in. in dimensions. As the first step in obtaining such test specimens suitable quantities of Alcoa A-10 alumina (325 mesh) were wet milled in a steel ball mill to a particle size of more than 90 per cent smaller than 10 microns. After suitable hydrochloric acid leaching, washing, drying and blending the material so obtained was mixed with 8 per cent Ceremul R and pelletized by rubbing through a 14 mesh screen.

By the procedure described, a material quite suitable for forming by dry press methods was obtained but it was laborious and time consuming. From results obtained later, it was found that A-10 alumina used as received would produce specimens differing very little in quality and it was therefore used in all formal experimentation.

b. Forming and firing

Five per cent of Ceremul R (in the case of A-10 material) was added to the alumina and blended in a Simpson mixer. The charge was then granulated by rubbing it through a 14 mesh screen. The test pieces were dry pressed at 2700 psi, which was the highest pressure obtainable, in a Model K08 Dennison press. The amount of material used in each specimen was weighed to insure uniformity.

In developing the technique of specimen forming a heavy case hardened mold box was designed and fabricated for use in a large 200 ton "Hydro Power" press. A number of bars were made in this mold at pressures up to 10,000 psi but due to such problems as limited production, and difficulty in setting the heavy bars in the furnace so as to obtain uniform firing and freedom from warping it was decided to carry on the work with the more rapidly produced and uniform bars pressed at 2,700 psi.

The binder was burned out of the specimens by holding them at a temperature of 300°F for 12 hours in a Globar furnace after which the temperature was raised to 2400°F and maintained 8 hours in order to develop sufficient strength for subsequent handling. Final sintering at 3000°F was performed in a gas fired Remmey furnace. It was found that by setting the bars upon alumina bubbles in a vertical position in the furnace, dimensionally uniform specimens, free from warping and cracking were obtained.

Due to the capacity of the furnace and the individual vertical setting required, not more than fifty bars could be fired in any given lot.

2. Methods for Detecting Flaws

A suitable means of flaw detection was recognized as being of primary importance. This was due to the fact that the early detection of conditions sure to cause eventual failure such as small fissures created by a gradual weakening of the bonds between crystals was the important matter. If the method were sensitive enough to detect the instant of failure, so much the better. Various methods were considered. Among them were the use of fluorescent oils, microscopic replicas, radiographic methods to detect internal fracture voids, sonic and ultra sonic techniques.

Since a general study of such methods could not be accomplished within the structure of the research contract, it was decided to particularly develop the use of the sonic technique. There were several reasons for this, a) the technique gave some promise of being sensitive enough to detect small internal flaws, b) the equipment was available and c) considerable knowledge of its use had been acquired. Cross-breaking, or modulus of rupture tests were adopted as a means for verifying the existence of any serious internal flaws in the specimen.

3. Effect of Thermal Shock on Unloaded Bars

For evaluating the sonic technique as a method of flaw detection fifty test bars were prepared using the methods described in item one (1) of this Section. After firing, the fundamental resonant frequency of flexural vibration (F) was determined for each bar. The values obtained are given in Table I. The average value ($\bar{F}$) of the frequency was 4558 cps with a standard deviation (σ) of 108 cps. The coefficient of variation (v) of 2.3 per cent, determined by statistical analysis of the data, was noted to be just within the reported accuracy of the testing apparatus.

As the next step, twenty five bars were taken from the total and subjected to five moderately severe thermal shock cycles. The resonant frequency of each bar was again determined and compared to that obtained for it in the unshocked condition. The average frequency for all of the unshocked bars, 4558 cps, dropped to 3666 cps for the thermal shocked bars. The differences in frequency were considered to be significant and were interpreted as indicating that structural alterations had been incurred as a result of thermal shocking.

All bars in the test series were then subjected to cross-breaking in a Dillon Tester which is equipped with a dynamometer head. The average modulus of rupture for the unshocked bars was 3557 psi. For all thermal shocked bars with residual strength enough to measure the average value dropped to 245 psi. These data are summarized in Table I.

4. Effect of Thermal Shock on Bars Loaded in Compression

a. Description of apparatus

The apparatus, shown in Figure 1, consists of a combination thermal shock and compressive loading device. The sonic testing unit, with a test bar in place, is shown in the foreground. Two opposed Carver Laboratory Press heads (No. 11713-166) are mounted horizontally, rigid, and level on the bed of a 50 in tool room lathe. They are fixed in position by two steel columns as shown. A Blackhawk 20 ton "Porto-Power" hydraulic ram (No. RC-251) is mounted horizontally on one head. Carver swivel bearing plates are attached to the ram piston and to the opposite head. They swivel on large hardened steel balls. Their purpose is to compensate for any nonparallelism occurring in the ends of the specimens. Pressure is applied by means of a Blackhawk "Porto-Power" hydraulic pump (No. P-85) capable of developing a line pressure of 10,000 psi. A Greer hydro-pneumatic accumulator (No. 30A-½) with a capacity of one quart connected to the hydraulic system in parallel with the ram serves to maintain constant pressure as the specimen expands and contracts during the thermal shock cycle.

Two diametrically opposed gas-air precision burners of the type used in sealing off operations in glass working are used to apply heat to the test bars. Their tips are placed with axes normal to the long axis of the specimen and midway from its ends so that the heating zone is confined to a narrow region in the center of the bar. With this arrangement it is possible to produce a clean fracture in the center of the test bar normal to its axis.

Provision for cycling was accomplished by supporting the burners on a raising and lowering mechanism constructed of Castaloy "Flexframe" units. The burners could thereby be lifted away from the specimen at specified intervals, thus allowing it to cool in still air, and then returned to their original position. By this method it was not necessary to disturb the flame setting which was maintained constant throughout an entire experiment.

Shown in the picture at the upper left is an apparatus installed for the purpose of picking up and transmitting sonic vibrations from a test bar loaded in compression and undergoing thermal shock cycling. The mode of vibration of a prism loaded axially was difficult to predict. The position of the excitor was therefore considered to be of importance in an attempt to induce the prism to vibrate in a prescribed manner. In this case, the excitor is positioned beneath the specimen, the sonic pulses being transferred to the specimen with the aid of a nickel rod which is heat resistant, in contact with the bottom face of the prism. This arrangement, it was hoped, would induce the prism to vibrate in a relatively simple mode, the plane of vibration being vertical and containing the prism axis. If the prism could be induced to vibrate under load, then the sonic pulses originating therefrom would be transferred to a crystal pickup by means of a nickel rod in contact with the top of the specimen. The two rods are in the vertical position normal to the axis of the prism and equidistant from its ends where the maximum deflection may be expected to occur. This region is also the heating zone. Since crack orientation relative to the plane of vibration was thought to be important in the success of this method, it was believed that by applying heat by mutually opposed precision burners perpendicular to the prism axis a better control might be achieved. It was hoped that the apparatus would signal some change in frequency at the instant a crack developed, but such was not the case. Although it was designed to, and did, pick up vibrations from heated but unloaded ceramic bars that were investigated in a previous research problem it could not be depended on to give reliable signals at any stage in this study. It is possible that some adjustment, modification or repair of the drive, pickup or receiving mechanism would have served to clear up the trouble. It did not, however, appear to be desirable to undertake this in view of the limited amount of contractual time remaining.

b. Testing in compression

For evaluating the effect of pre-compression on the thermal shock resistance of pure oxide ceramics, forty rectangular bars of A-10 grade alumina were prepared and their fundamental resonant frequencies obtained. Thermal shocking under axial compressive load was performed by first centering the specimen between the bearing plates of the loading apparatus with disks of 1/32 in asbestos sheet inserted between the swivel bearing plates and the ends of the bar to aid in equalizing and distributing the load. The hydraulic pump was then operated until the desired pressure was indicated on a pressure gage. At this point, a pressure valve between pump and accumulator was closed so that the ram and accumulator operated as a closed system. Heat was then applied to the bar by lowering the lighted and adjusted burners into position. After one minute the burners were raised and the bar allowed to cool for one minute. After one complete test, of five such cycles, the temperature of the specimen in the narrow region of the hot zone was found to be about 1100°F. After the tests were completed the bars were allowed to cool for ten minutes while still in compression since it was found that early release would cause them to fracture, usually with an audible report.

The forty available specimens were tested in groups of eight each so that the best possible average values could be used. The first group received no thermal shock treatment. The second group was subjected to thermal shock but not to compressive loading. In order to make sure that the bars of this group received the uniform thermal shock treatment they were, in turn, supported on stainless steel rods in the loading apparatus in the exact position occupied by loaded bars. The remaining three groups were thermal shocked five cycles in the manner described at loads of 1000, 3500, and 7000 psi, respectively.

c. Evaluation, methods and results

As previously noted, the initial plan in this investigation was to use sonic techniques in detecting the degree of thermal shock failure and if possible the instant of failure. Since the apparatus designed to transmit sonic vibrations during the progress of the test would not function, the thermal shock cycling was carried on independently of it through the four stages of loading described above.

The bars, after loading and cycling, were again referred to the sonic test conducted at room temperature. All such tests were followed by cross-breaking or modulus of rupture tests. Table II shows the resonant frequency and modulus of rupture values for eight control bars tested in the as fired condition. The frequency values, which were quite uniform averaged 6106 cps. There was more scatter in the modulus of rupture values but their average was 7877 psi.

When similar tests were performed on thermal shocked bars some interesting variations in results were obtained. As shown in Table III, thermal shocking of unloaded bars (specimens 16 and 28) produced large decreases in resonant frequency and cross-breaking strength. These results were expected since they were in keeping with those of previous tests on unloaded bars. However, when a compressive load of 1000 psi was applied during thermal shock cycling the average resonant frequency decreased by very little, from 6143 to 5962 cps. The cross-breaking strength though, increased from a very low nominal value of a few hundred psi to an average of 2806 psi. When the load was increased further, to 3500 and then to 7000 psi, no changes at all in frequency could be detected although modulus of rupture tests showed that faults were present in the specimens. Even so the cross-breaking strength continued to increase with increasing load. It averaged 3257 psi at a load of 3500 psi and 5554 psi at a load of 7000 psi.

These data are summarized in Tables II and III and Figure 2.

III. SUMMARY

In the work on the effect of pre-compressive loading on the thermal shock resistance of pure oxide ceramics described in this report a number of procedures were worked out and followed. Test bars of A-10 alumina were prepared and evaluated. As first steps, the fundamental resonant frequency in flexural vibration and the cross-breaking strength of sound bars were determined. Other bars were subjected to controlled thermal shock cycling at loads of zero, 1000, 3500 and 7000 psi. They were then evaluated by sonic methods and final modulus of rupture (cross-breaking) tests.

It was found that compressive loading progressively increased the resistance to thermal shock as shown by corresponding increases in specimen cross-breaking strength. The increase was not linear (cf Figure 2). The 1000 psi load accounted for about one half of the strength gained at 7000 psi. This trend, if verified and found applicable to other bodies, might be useful to design engineers. Its implication is that selected bodies could be greatly improved in thermal shock resistance by only moderate compressive loading.

The sonic method of flaw detection served quite well in cases where thermal shock cycling under no load was carried on. With application of load, however, the effectiveness of this method first decreased and then disappeared entirely. It was thought that flaws developed under compressive loading may be held so firmly together as to cause the test piece to vibrate as a whole.

IV. DISCUSSION

Data obtained on flaw development in a specimen being alternately heated and cooled under compressive load, by some suitable method would be useful. However, concurrent data on the cross-breaking strength and impact resistance might be of definite significance. Based on the known strength of dense ceramic bodies in compression it is postulated that such loading should greatly increase the resistance of ceramics and cermets to impact induced brittle fracture.

V. CONCLUSIONS

1. Pre-compressive loading, in the direction of the principle axis increases the thermal shock resistance of α alumina bodies.

2. Moderate initial loads produce the greatest improvement. With increasing loads, the improvement continues but at a declining rate.

3. Due to the sharp increases in thermal shock resistance brought about by moderate loading it appears that the principle might be used with good results in the design of selected ceramic

and cermet components.

4. The sonic technique will apparently serve to detect internal flaws in unloaded specimens which have been subjected to thermal shock but it becomes ineffective in cases where the specimens are thermal shocked under compressive load.

TABLE I. - COMPARISON OF RESONANT FREQUENCIES AND MODULI OF RUPTURE OF THERMAL SHOCKED AND UNSHOCKED SPECIMENS OF ALPHA ALUMINA

Spec. number	%(1) por.	Resonant frequency(2) (unshocked)	(shocked)	Frequency difference	Modulus of rupture(3) (unshocked)	(shocked)
144-2	61.3	4475	----	----	3012	----
144-3	60.1	4475	3350	1125	----	*
144-4	60.4	4500	----	----	1963	----
144-5	61.5	4625	3325	1300	----	*
144-6	60.6	4500	----	----	3284	----
144-7	61.7	4375	4125	250	----	478
144-8	61.5	4450	4000	450	----	267
144-10	60.4	4475	----	----	4744	----
144-11	61.5	4500	4500	450	----	197
144-12	60.7	4450	----	----	2500	----
144-13	60.5	4600	4300	300	----	311
144-14	61.1	4600	----	----	2942	----
144-15	60.5	4500	3700	800	----	63
144-16	60.6	4575	----	----	3977	----
144-17	62.0	4600	1550	3050	----	*
144-18	61.4	4600	----	----	3675	----
144-19	61.3	4550	4200	350	----	386
144-20	60.3	4500	----	----	1939	----
144-21	60.5	4625	3975	650	----	314
144-22	61.1	4475	----	----	5289	----
144-24	60.9	4550	----	----	5859	----
144-25	59.5	4650	2300	2350	----	*
144-26	61.0	4625	----	----	3879	----
144-27	59.8	4675	2400	2275	----	*
144-28	58.1	4650	----	----	2955	----
144-29	60.1	4650	3450	1200	----	*
144-30	59.7	4500	----	----	4120	----
144-32	60.3	4500	----	----	3063	----
144-33	60.1	4475	4000	475	----	470
144-34	60.2	4700	----	----	5587	----
144-35	61.0	4700	2850	1850	----	*
144-36	59.8	4625	----	----	4139	----
144-38	60.1	4700	1900	2800	----	11
144-40	60.6	4600	----	----	3684	----
144-41	59.7	4475	3100	1375	----	*
144-42	60.5	4425	----	----	1808	----
144-43	60.1	4450	4300	150	----	446
144-44	59.9	4625	----	----	5229	----
144-45	60.5	4550	4550	100	----	576

* No appreciable strength.
1. Per cent porosity based on dry weight.
2. Cycles per second.
3. Pounds per square inch.

(TABLE I - Cont)

Spec. number	%[1] por.	Resonant frequency[2] (unshocked)	(shocked)	Frequency difference	Modulus of rupture[3] (unshocked)	(shocked)
144-46	60.5	4600	----	----	4251	----
144-47	59.8	4600	4300	300	----	298
144-48	60.2	4600	----	----	4038	----
144-49	60.0	4675	4500	175	----	1219
144-50	59.8	4425	----	----	3005	----
144-51	59.6	4500	4225	375	----	262
144-52	60.3	4675	----	----	2213	----
144-53	60.6	4625	4400	225	----	458
144-54	61.8	4600	----	----	2879	----
144-55	60.4	4600	----	----	2464	----
Average	60.1	4558	3666	----	3557	245
σ	4.5	108	522	----	1121	15

TABLE II. - CONTROL TESTS: RESONANT FREQUENCIES AND MODULI OF RUPTURE OF PRESUMABLY SOUND ALUMINA BARS TESTED IN THE AS PRESSED AND FIRED CONDITION

Spec. number	Resonant frequency (1)	Modulus of rupture (2)
147-4	6050	7900
147-7	6150	7200
147-10	6000	3200
147-15	6150	9204
147-20	6050	8050
147-23	6250	10184
147-24	6200	8424
147-34	6000	8855
Average	6106	7877

1. Cycles per second.
2. Pounds per square inch.

TABLE III. - RESONANT FREQUENCIES AND MODULI OF RUPTURE OF ALUMINA BARS THERMAL SHOCKED UNDER VARIOUS PRE-COMPRESSIVE LOADS

Spec. number	Resonant frequency(1) (unshocked)	(shocked)	Frequency difference	Modulus of rupture (shocked)	Pre-load(2)
147-5	6200	Failed	----	----	None
147-11	6200	"	----	----	"
147-16	6150	2500	3650	477	"
147-21	6300	Failed	----	----	"
147-25	6100	"	----	----	"
147-27	6100	"	----	----	"
147-28	6100	4500	1600	160	"
147-41	6150	Failed	----	----	"
Average	6162				
147-1	6050	5500	550	2415	1000
147-2	6250	6200	50	2669	"
147-12	6250	6100	150	225	"
147-14	6050	6000	50	4424	"
147-18	6200	6100	100	3100	"
147-38	6000	5800	200	4480	"
147-39	6000	5800	200	4266	"
147-40	6350	6200	150	870	"
Average	6143	5962	181	2806	"
147-6	6100	6100	0	3542	3500
147-13	6000	6000	0	3657	"
147-26	6100	6100	0	4000	"
147-29	6200	6200	0	4004	"
147-30	6100	6100	0	3498	"
147-31	6400	6400	0	1632	"
147-35	6000	6000	0	3340	"
147-37	6150	6150	0	2385	"
Average	6131	6131		3257	
147-3	6200	6200	0	4347	7000
147-8	6200	6200	0	4836	"
147-9	6200	5950	250	*	"
147-17	6200	6200	0	4200	"
147-19	6100	6100	0	12320	"
147-22	6000	6000	0	3200	"
147-32	6000	6000	0	4100	"
147-33	6100	6100	0	5880	"
Average	6125	6094		5555	

* Cracked when unloaded too soon after thermal shock cycling.
1. Cycles per second.
2. Pounds per square inch.

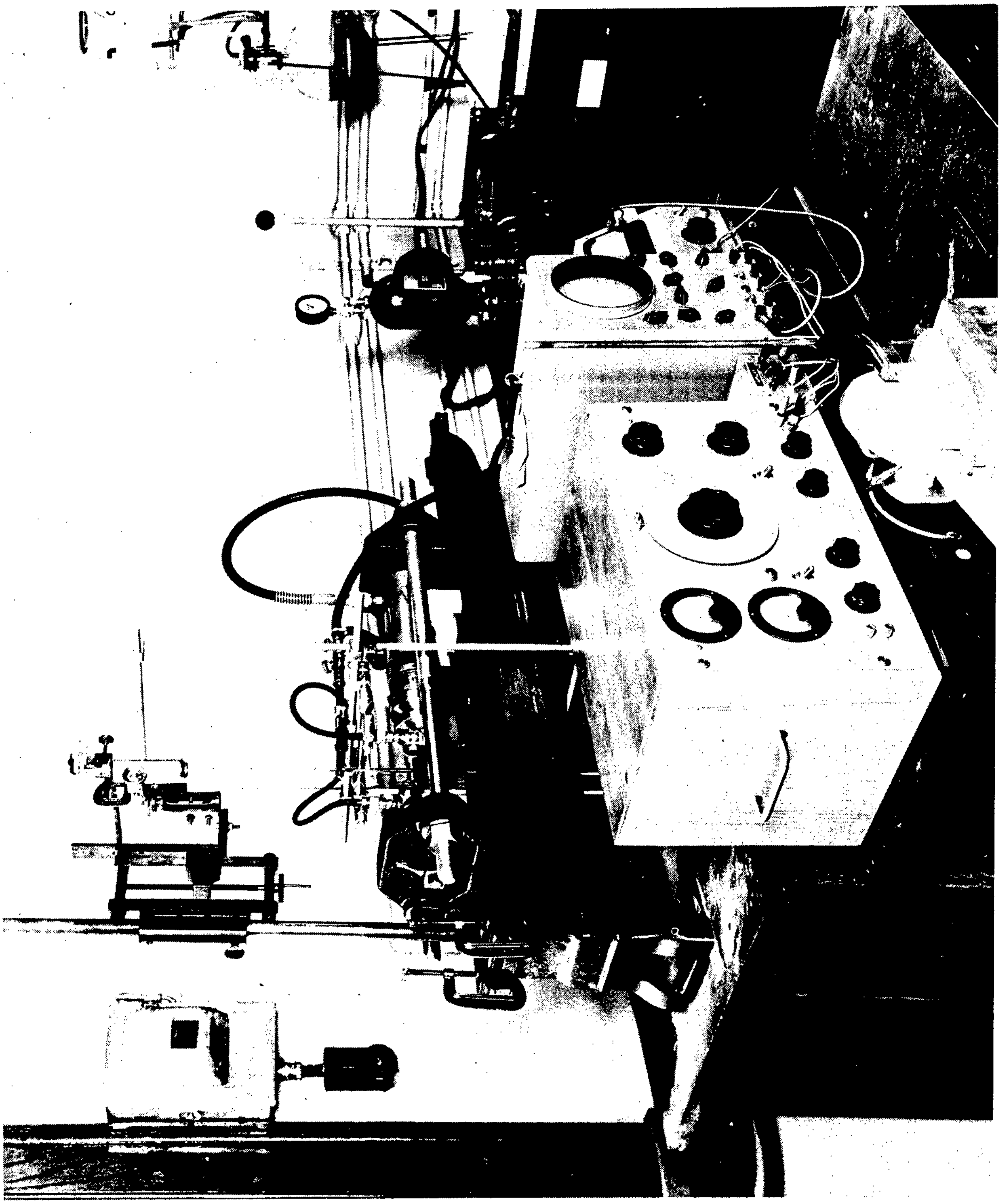

Figure 1. - A combination thermal shock and compressive loading device with sonic testing equipment shown in the foreground.

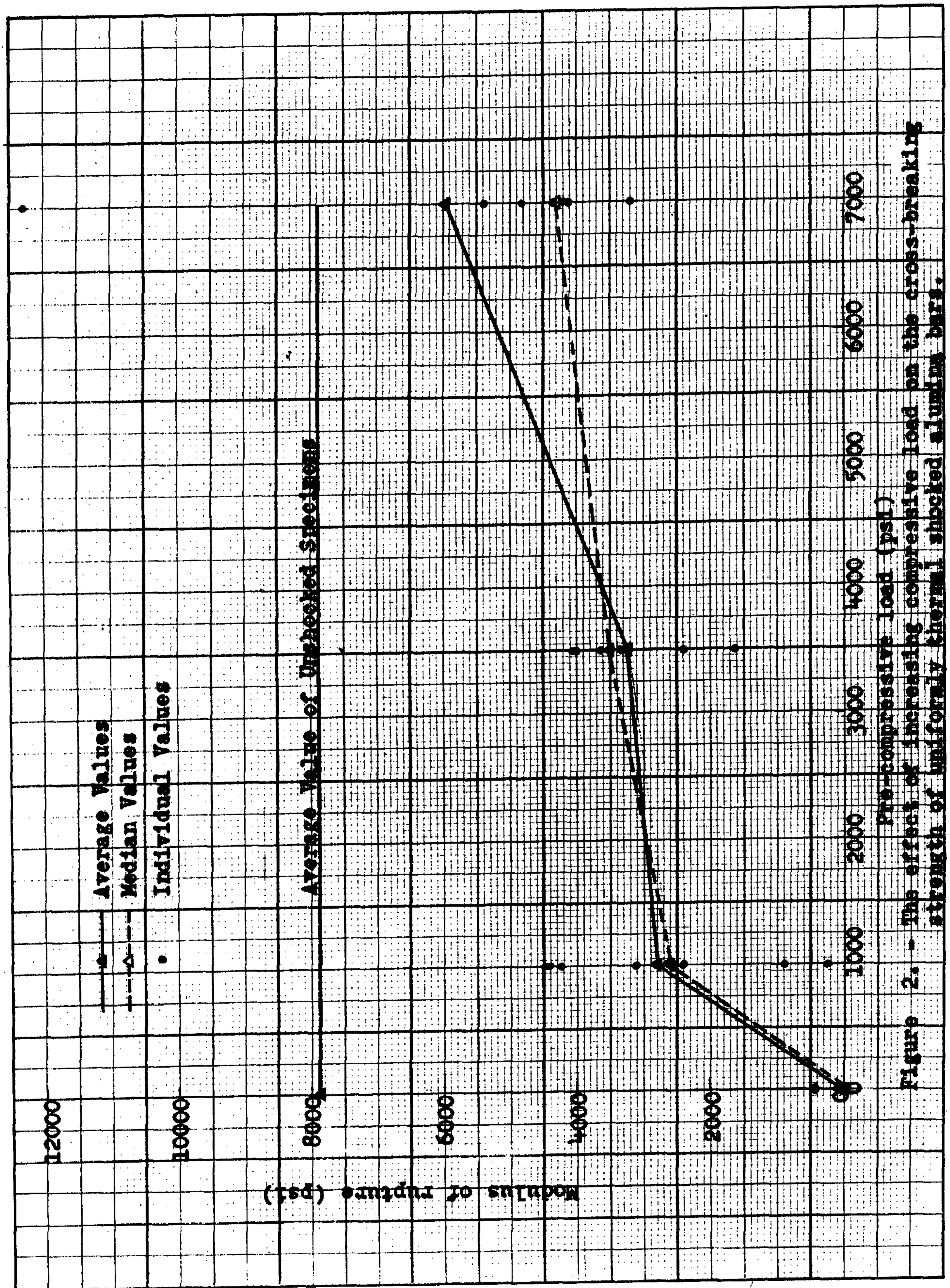

Figure 2. - The effect of increasing compressive load on the cross-breaking strength of uniformly thermally shocked alumina bars.