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RESEARCH MEMORANDUM

PRELIMINARY INVESTIGATION OF ZIRCONIUM BORIDE CERAMALS FOR GAS-TURBINE BLADE APPLICATIONS

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PRELIMINARY INVESTIGATION OF ZIRCONIUM BORIDE CERAMALS FOR GAS-TURBINE BLADE APPLICATIONS

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SUMMARY

Zirconium boride ZrB_2 ceramals were investigated for possible gas-turbine-blade application. Included in the study were thermal shock evaluations of disks, preliminary turbine-blade operation, and observations of oxidation resistance. Thermal shock disks of the following three compositions were studied: (a) 97.5 percent ZrB_2 plus 2.5 percent B by weight; (b) 92.5 percent ZrB_2 plus 7.5 percent B by weight; and (c) 100 percent ZrB_2.

Thermal shock disks were quenched from temperatures of 1800°, 2000°, 2200°, and 2400° F. The life of turbine blades containing 93 percent ZrB_2 plus 7 percent B by weight was determined in gas-turbine tests. The blades were run at approximately 1600° F and 15,000 to 26,000 rpm.

The results of this investigation were as follows:

1. The best thermal shock performance was yielded by the 97.5 percent ZrB_2 plus 2.5 percent B composition. This result compared favorably with those for ceramals of TiC plus Co and of TiC plus Ni.

2. Observations of oxidation made during the thermal shock studies indicated that oxidation was slight for the short times involved up to 2000° F. However, a 97.5 percent ZrB_2 plus 2.5 percent B specimen held for 100 hours at 2000° F was severely oxidized. The oxide scale was ZrO_2.

3. A blade of 93 percent ZrB_2 plus 7 percent B was operated for 35 minutes at 1600° F and 26,000 rpm. A second blade ran for 40 minutes at 1600° F and 15,000 to 26,000 rpm, while a third failed under 1600° F and below 15,000 rpm.

INTRODUCTION

In a continuing effort to develop ceramal materials for aircraft power-plant applications, zirconium boride ZrB_2 ceramals have been considered. Data given in reference 1 indicate that cemented zirconium boride possesses excellent transverse rupture strength at approximately 2400° F and also has good oxidation resistance. These data suggest further investigation of this material for gas-turbine-blade application. 2496

The objectives of this investigation at the NACA Lewis laboratory were threefold: (a) to determine the thermal shock behavior, (b) to determine the turbine blade performance, and (c) to observe the oxidation resistance of ZrB_2 ceramals.

APPARATUS AND PROCEDURE

Experimental materials. - Experimental materials were furnished by the American Electro Metal Corporation.

Ceramal thermal shock disks. - The thermal shock disks were 1/4 inch thick by 2 inches in diameter and were either hot pressed or cold pressed and sintered. Three compositions were investigated (a) 100 percent ZrB_2, (b) 97.5 percent ZrB_2 plus 2.5 percent B, and (c) 92.5 percent ZrB_2 plus 7.5 percent B. Fabricating information (i.e., powder size, temperature, pressure) was not furnished for any of the disks. Some of the thermal shock specimens were made of virgin powders, and others were made of recrushed powder, which is hot-pressed material recrushed and remilled. The history of the disks is summarized in table I. All specimens except numbers 21 to 25 were inspected by radiographic methods to detect internal flaws and by a penetrant-oil method to detect surface flaws and porosity. These tests were performed at this laboratory. Specimens 9 to 20 were surface-ground in an effort to remove surface porosity. After these disks had been ground about 0.03 inch under-sized without elimination of the porosity, grinding was discontinued. Specimens 21 to 25 had been annealed, subjected to approximately 50 thermal shock cycles, and radiographically inspected by the manufacturer, before they were received at this laboratory. The details of the annealing treatment and the thermal shock procedure are not known. The surfaces of these specimens were oxidized after this preliminary testing and for that reason surface inspection could not be made at this laboratory.

Ceramal blades. - The ceramal turbosupercharger-type blade used is illustrated in figure 1(a). This blade shape is similar to that used in the investigations reported in references 2 and 3. The ceramal and the alloy blades had the same airfoil configuration; the ceramal blade root, however, had a 100 percent greater cross section (fig. 1). The roots of the ceramal blade roots were electroplated with 0.008 inch of copper. The blades were made of 93 percent ZrB_2 plus 7 percent B and were hot pressed. These blades were radiographically inspected for internal flaws and penetrant-oil inspected for external flaws. Data for the blade specimens are presented in table I.

Evaluation Procedures

Thermal shock evaluation. - The thermal shock apparatus consisted of a furnace in which a specimen was heated and an air chamber into which the specimen could be quickly moved for quenching. This apparatus is illustrated in figure 2. The air velocity was 265 feet per second (50 lb/min at 70-80° F). The specimen was kept in the furnace for 10 minutes and then withdrawn into the quenching air stream for 5 minutes. The test consisted of 25 quench cycles from 1800° F, 25 quench cycles from 2000° F, 25 quench cycles from 2200° F, and 25 quench cycles from 2400° F for a total of 100 cycles or until specimen failure, whichever occurred first. A more detailed description of the apparatus and procedure is given in reference 2. Qualitative observations of oxidation were made concurrently with the thermal shock evaluation.

Turbine blade evaluation. - The apparatus consisted essentially of a small gas turbine supplied with hot gases from a turbojet combustion chamber (see fig. 3). The inlet-gas temperature was measured by chromel-alumel thermocouples attached to the turbine guide vanes immediately upstream of the blades. A more detailed description of this apparatus is given in reference 4. Each of the ceramal blades was run separately at an estimated temperature of 1600° F. The operating schedule for the first two blades specified rapid acceleration to 15,000 rpm and step-wise speed increases up to 26,000 rpm and then extended operation at this speed. The third blade was brought directly up to 26,000 rpm. At this speed the midspan stress for the ceramal blade was approximately 14,700 pounds per square inch. The midspan stress of Stellite 21 alloy control blades was approximately 20,000 pounds per square inch. A ten-blade alloy control sample was run along with each ceramal blade. The remaining blades in the wheel were metal and were cut to about 3/4 their original length to prevent their failure and yet permit operating speeds to be reached. The wheel was dynamically balanced before operation and thereafter as necessary.

Oxidation evaluation. - In addition to the qualitative oxidation observations made in the thermal shock evaluation, a specimen of 97.5 percent ZrB_2 plus 2.5 percent B (virgin powder), taken from a failed thermal shock disk, was kept for 100 hours at 2000° F in a commercial-type heat-treat furnace. The composition of the oxide scale formed was determined from X-ray diffraction studies by the Debye-Scherrer method.

Density evaluation. - The percent of theoretical density was determined for some of the disks after thermal shock evaluation. The surfaces were carefully ground to remove all scale. The actual density was determined from weighings in water and air. Water absorption of the disks during these weighings was negligible.

The theoretical density was computed with 6.1 (ref. 5) and 2.3 (ref. 6) grams per milliliter used as the densities of ZrB_2 and B, respectively, assuming a mechanical mixture of the two. 2496

RESULTS AND DISCUSSION

Thermal Shock Evaluation

The thermal shock performance of the specimens is presented in table II(a). The best results were obtained with disks 21 to 25, which contained 2.5 percent B plus 97.5 percent ZrB_2. These disks were made in a hot press designed to reduce residual stress. Thermal shock resistance has been improved compared with the other 2.5 percent B plus 97.5 percent ZrB_2 disks but it is not known whether this is attributable to the hot press or to the annealing. Except for disk number 23, which may have been defective in view of its poor performance compared with disks 21, 22, 24, and 25, these disks survived the full 100 thermal shock cycles in addition to the approximately 50 heat shock cycles they had received before being received at this laboratory. It was not known whether recrushed or virgin powders were used for these disks.

Assuming no other major variables have been changed, it would appear from a study of the results for the disks 3 and 13 to 20 that virgin powders yield better results than recrushed powders. Study of the data for disks 4 to 8 and disks 16 to 20 does not suggest any effect of density on thermal shock resistance.

The thermal shock resistance of the 97.5 percent ZrB_2 plus 2.5 percent B ceramal compares favorably with that of (CbTaTi)C, modified TiC plus Co, and TiC plus Ni ceramals reported in reference 3.

Oxidation evaluation. - In general, the oxidation during the thermal shock evaluation was negligible to slight, through 2000° F for specimens 1 to 20. The total time at temperature for the first 50 cycles was, however, only 8.3 hours. At 2400° F the oxidation was moderately severe. The scale was tightly adhering and could not be readily removed by either chipping or vapor blasting. Since specimens 21 to 25 were already oxidized before the NACA thermal shock testing, it was not possible to determine the extent of oxidation for these specimens at the lower temperatures; however, upon completion of

100 cycles (i.e., up through 2400° F and 16.7 hr of testing) oxidation had also become moderately severe.

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The 97.5 percent ZrB_2 plus 2.5 percent B specimen, which was kept at 2000° F for 100 hours, underwent serious oxidation, as illustrated in figure 4. A "Maltese cross" effect associated with oxidation occurring at the metal-scale interface is noted. Also to be noted is the occurrence of a number of zones within the scale. This specimen experienced a weight gain of 9.7 milligrams per square centimeter. Originally it had a cross-sectional area of 0.367 square centimeter and after heating had an unaffected area of 0.148 square centimeter, 60 percent of its original cross-sectional area having become oxidized. The X-ray diffraction study showed the scale to be composed only of ZrO_2.

Density evaluation. - The percents of theoretical densities reported by the fabricator and those determined during the course of this study are reported in table II. It can be noted that the values determined during this investigation are, in all cases, lower than those reported by the fabricator. Although the reason for the discrepancy is not known, the surface porosity revealed by use of a penetrant-oil inspection method on the disks tends to confirm the lower values. Possible reasons for discrepancy are:

(a) The assumption of a chemical reaction in the fabricator's calculations (new compound formation, solution, etc.) rather than a mechanical mixture of the components. This would not account for the discrepancy in specimens 10, 11, and 12, which are 100 percent ZrB_2.

(b) The use of differing densities for the components in the calculations.

(c) The possibility that some physical change during the thermal shock testing has altered the density.

Turbine blade evaluation. - The results of the turbine blade evaluation are presented in table II(b). The first blade failed after a total of 55 minutes at the various conditions listed in the table. The second blade failed during the approach to 15,000 rpm and 1600° F. This blade ran for a total of 10 minutes. Subsequent examination of the failed section indicated that this blade may have been defective. The third blade failed after 40 minutes of operation, 35 minutes of which were at 26,000 rpm and 1600° F, and the other 5 minutes used in approaching this condition. Oxidation was negligible during these runs. No alloy blades failed during these tests.

The ceramal blades had a 100 percent greater cross-sectional area in the root than did the alloy control blades in order to compensate for their brittleness. In addition, the roots of the ceramal blades were copper plated in order to achieve a better load distribution. However, these expedients were insufficient to prevent the failure of the ceramal blades at the neck-roll junction of the roots.

The objective of the blade evaluation was to study the performance of this ceramal at a speed (26,000 rpm) at which the stress at the midspan in the Stellite 21 alloy control blades would be comparable to the stresses at the midspan of the blades in a current jet engine. At 26,000 rpm the stress at the midspan of the control blades was 2496
20,000 pounds per square inch. This value is similar to that encountered at the midspan in the blading of the J33 turbojet engine. Simultaneously it was desired to determine whether or not there was any limiting speed below 26,000 rpm; hence, the first two blades were brought to 15,000 rpm and increased by increments thereafter. The first two runs indicated that the quality of the material varied widely and that operation should be limited to low speeds. Because of the limited number of blades available, and because the variation in quality indicated that the performance in the first two runs was probably not indicative of the potential performance of other blades, it was decided to take the third blade directly to the final operating conditions; the results indicate that it might be possible to operate at this condition for short times.

As stated previously, all the blades failed in the root and consequently the ability to withstand the severe conditions which exist at the midspan of the airfoil cannot be satisfactorily judged. Blade life could probably be prolonged by the use of improved root designs. When the ZrB_2 blades reported herein are compared with the TiC plus Co and TiC plus Ni blades reported in reference 3, it is seen that the ZrB_2 ceramal blades ran for shorter times under similar conditions than did the reference blades. However, in view of the facts that a very small number of ZrB_2 blades were investigated and that there is only limited experience in the fabrication of ZrB_2 blades, these results must be regarded as very preliminary.

SUMMARY OF RESULTS

The results of this investigation may be summarized as follows:

1. The best thermal shock performance was yielded by the 97.5 percent zirconium boride ZrB_2 plus 2.5 percent boron B composition. This composition survived 100 quenching cycles; 25 cycles at each of the following temperatures: 1800°, 2000°, 2200°, and 2400° F.

2. Observations of oxidation during the thermal shock studies indicated that oxidation was, in general, slight for times less than 8.3 hours up through 2000° F. However, a 97.5 percent ZrB_2 plus 2.5 percent B specimen held for 100 hours at 2000° F was severely oxidized. The oxide scale was ZrO_2.

3. A 93 percent ZrB_2 plus 7 percent B blade was operated for 35 minutes at 26,000 rpm and 1600° F. A second blade ran for 40 minutes at speeds of 15,000 to 19,500 rpm at 1600° F. A third blade investigated failed below 15,000 rpm and under 1600° F.

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3. Hoffman, C. A., and Cooper, A. L.: Investigation of Titanium Carbide Base Ceramals Containing Either Nickel or Cobalt for Use as Gas-Turbine Blades. NACA RM E52H05, 1952.

4. Hoffman, Charles A., and Ault, G. Mervin: Application of Statistical Methods to Study of Gas-Turbine Blade Failures. NACA RN 1603, June, 1948.

5. Anon.: Properties of Cemented Zirconium Boride. American Electro Metal Corp. Feb. 15, 1950. (Contract N6-ONR-256)

6. Anon.: Metals Handbook, 1948 Edition. Am. Soc. Metals (Cleveland), 1948.

TABLE I. - SPECIMEN DATA
(disks and blades)

Specimen number	Percent ZrB_2	Percent B	Percent of theoretical density		Results of surface inspection	Results of radiographic inspection	Fabrication method
			Manufacturer's value	NACA value			
1[a]	97.5	2.5	99.0		Surface cracks and porosity	Internal crack or void	Sintered
2[a]	97.5	2.5	98		Surface porosity	Internal crack near circumference	Sintered
3[a]	97.5	2.5	100		Edge damaged	Segregation	Hot pressed
4[a]	92.5	7.5	100		Nicks on surface	Void near edge	Hot pressed
5[a]	92.5	7.5	98		General surface porosity	Segregation	Hot pressed
6[a]	92.5	7.5	94		General surface porosity	Segregation	Hot pressed
7[a]	92.5	7.5	93		General surface porosity	Segregation	Hot pressed
8[a]	92.5	7.5	92		Very porous surface	Satisfactory	Hot pressed
9[a]	92.5	7.5	96.5		General surface porosity	Segregation	Sintered
10[a]	100	0	98	92	General surface porosity	Internal pin holes	Hot pressed
11[a]	100	0	99.0	94	General surface porosity	External pin holes - segregation	Hot pressed
12[a]	100	0	99.8	92.6	General surface porosity	Internal pin holes - segregation	Hot pressed
13[b]	97.5	2.5	100	89	General surface porosity	Segregation	Hot pressed
14[b]	97.5	2.5	99.5	88.5	General surface porosity	Internal pin holes - segregation	Hot pressed
15[b]	97.5	2.5	99	88.5	General surface porosity	Segregation	Hot pressed
16[a]	97.5	2.5	100	88.5	General surface porosity	Segregation	Hot pressed
17[a]	97.5	2.5	99	88.5	General surface porosity	Segregation	Hot pressed
18[a]	97.5	2.5	97	89.5	General surface porosity	Internal pin holes - segregation	Hot pressed
19[a]	97.5	2.5	95	84.0	General surface porosity	Segregation	Hot pressed
20[a]	97.5	2.5	81		General surface porosity	Internal pin holes - segregation	Hot pressed
21	97.5	2.5	98.5[c]				Hot pressed annealed
22	97.5	2.5	98.5[c]				Hot pressed annealed
23	97.5	2.5	98.5[c]				Hot pressed annealed
24	97.5	2.5	98.5[c]				Hot pressed annealed
25	97.5	2.5	98.5[c]				Hot pressed annealed
26[d]	93	7			Satisfactory	Satisfactory	Hot pressed
27[d]	93	7			Satisfactory	Satisfactory	Hot pressed
28[d]	93	7			Satisfactory	Satisfactory	Hot pressed

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[a]Virgin powder.
[b]Recrushed powder.
[c]The percent of theoretical density was at least 98.5 percent as reported by the manufacturer.
[d]Turbine blade.

TABLE II. - SUMMARY OF ZIRCONIUM BORIDE CERAMAL INVESTIGATION

(a) Thermal shock evaluation of disks.

Specimen number	Boron content, percent	Number of cycles at -				Total number cycles	Type of failure
		1800° F	2000° F	2200° F	2400° F		
1	2.5	10				10	Fracture
2	2.5	3				3	Crack
3	2.5	25	25	25		75	Fracture
4	7.5	25	25	8		58	Fracture
5	7.5	7				7	Crack
6	7.5	25	4			29	Crack
7	7.5	25	3			28	Crack
8	7.5	25				25	Fracture
9	7.5	9				9	Fracture
10	0	25	25			50	Crack
11	0	15				15	Crack
12	0	25	25			50	Crack
13	2.5	25	11			36	Crack
14	2.5	25	25	1		51	Crack
15	2.5	25	25			50	Crack
16	2.5	25	25	25	25	100	
17	2.5	25	25	25	3	78	Fracture
18	2.5	25	25	25	21	96	Fracture
19	2.5	25	25	25	3	78	Fracture
20	2.5	25	25	25	25	100	
21	2.5	25	25	25	25	100	
22	2.5	25	25	25	25	100	
23	2.5	3				3	Fracture
24	2.5	25	25	25	25	100	
25	2.5	25	25	25	25	100	

(b) Turbine blade evaluation.

Specimen number	Estimated blade temperature, °F	Speed, rpm	Time, min	Total time, min	Remarks
26	to 1600	to 15,000	15	15	
	1600	15,000	10	25	
	1600	16,750	10	35	
	1600	18,000	10	45	
	1600	19,500	10	55	Blade failure occurred at neck-roll junction
27	to 1600	to 15,000	10	10	Blades failed upon approach to 1600° F and 15,000 rpm. Failure occurred in neck-roll junction
28	to 1600	to 26,000	5	5	
	1600	26,000	35	40	Blade failed at neck-roll junction

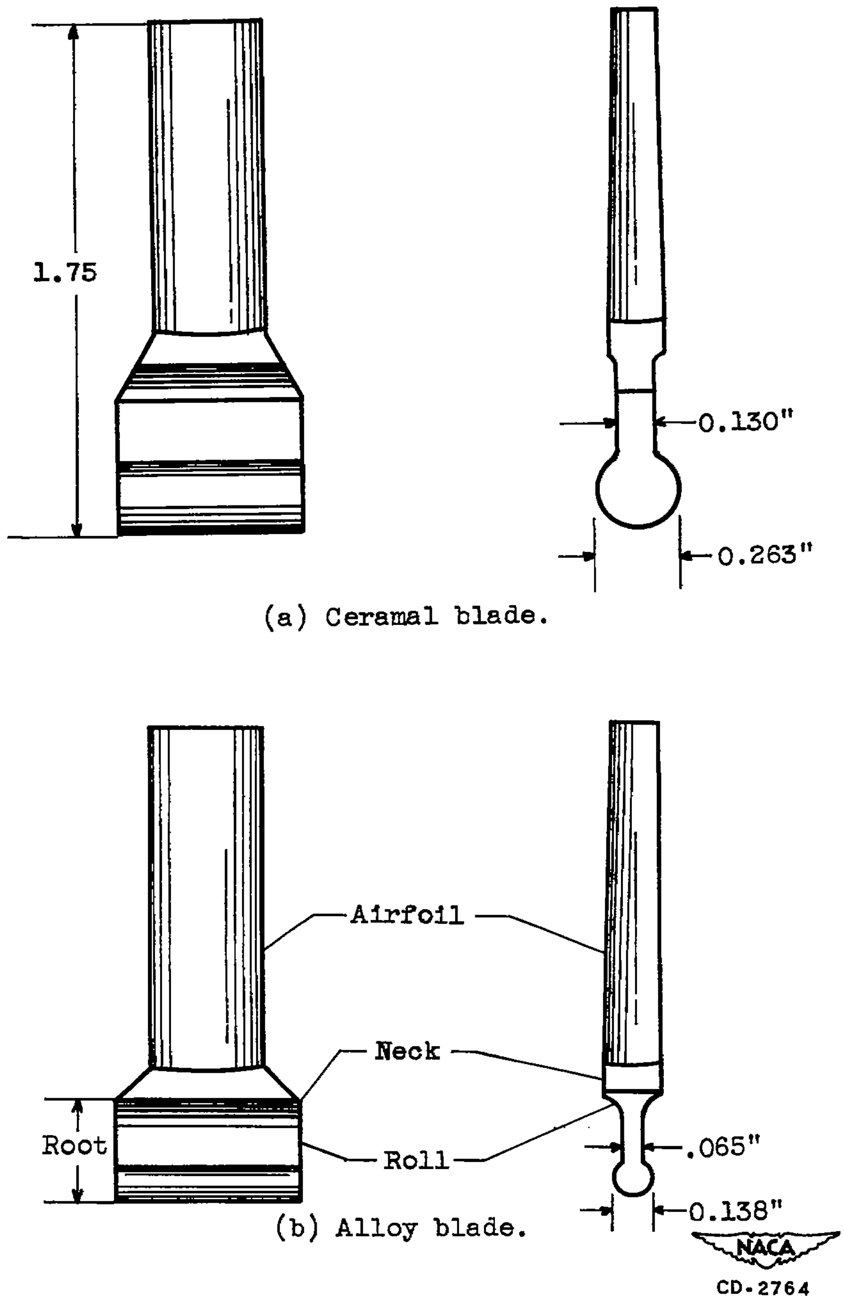

Figure 1. - Types of blade design used in investigation.

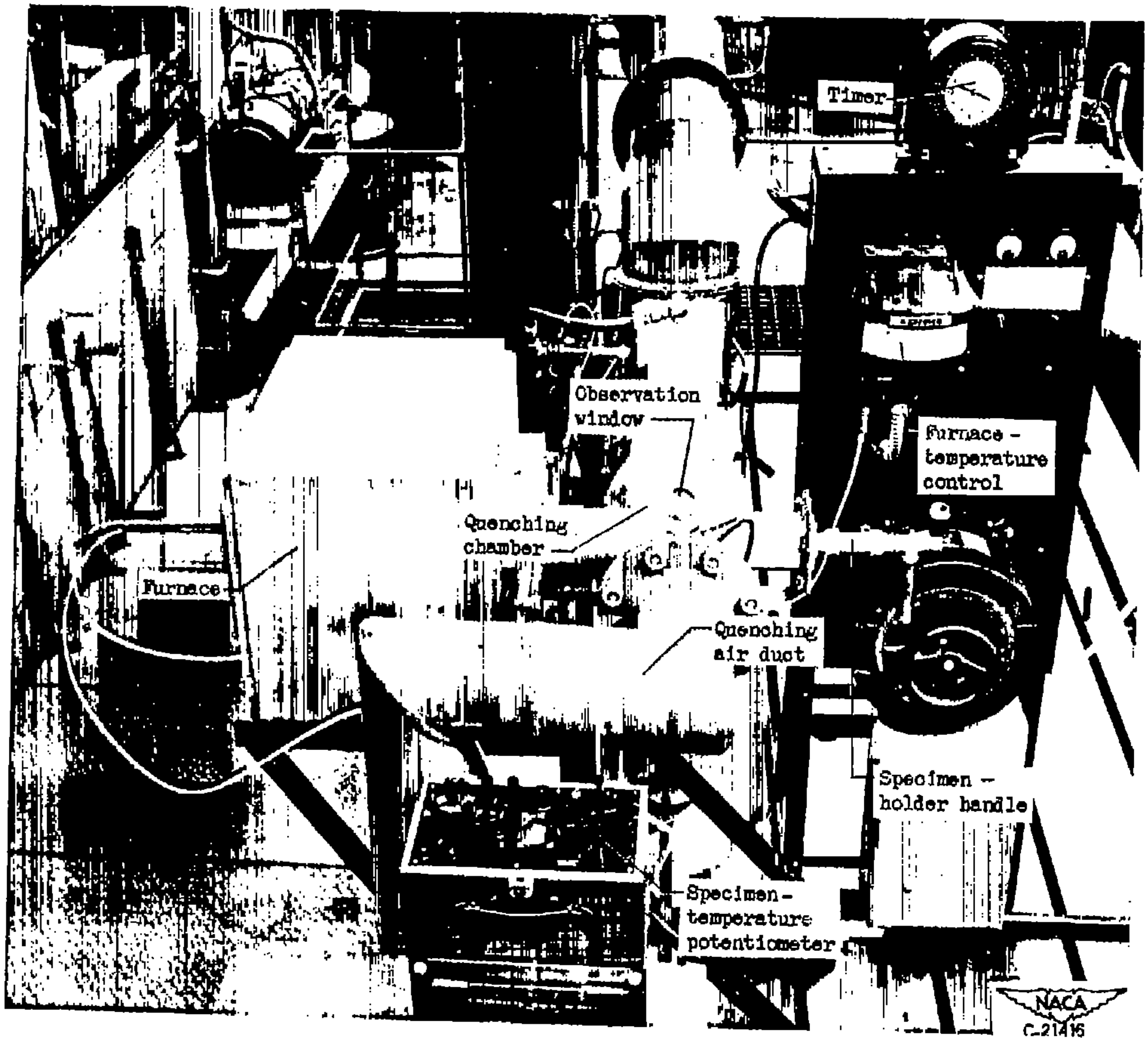

Figure 2. - Thermal-shock apparatus.

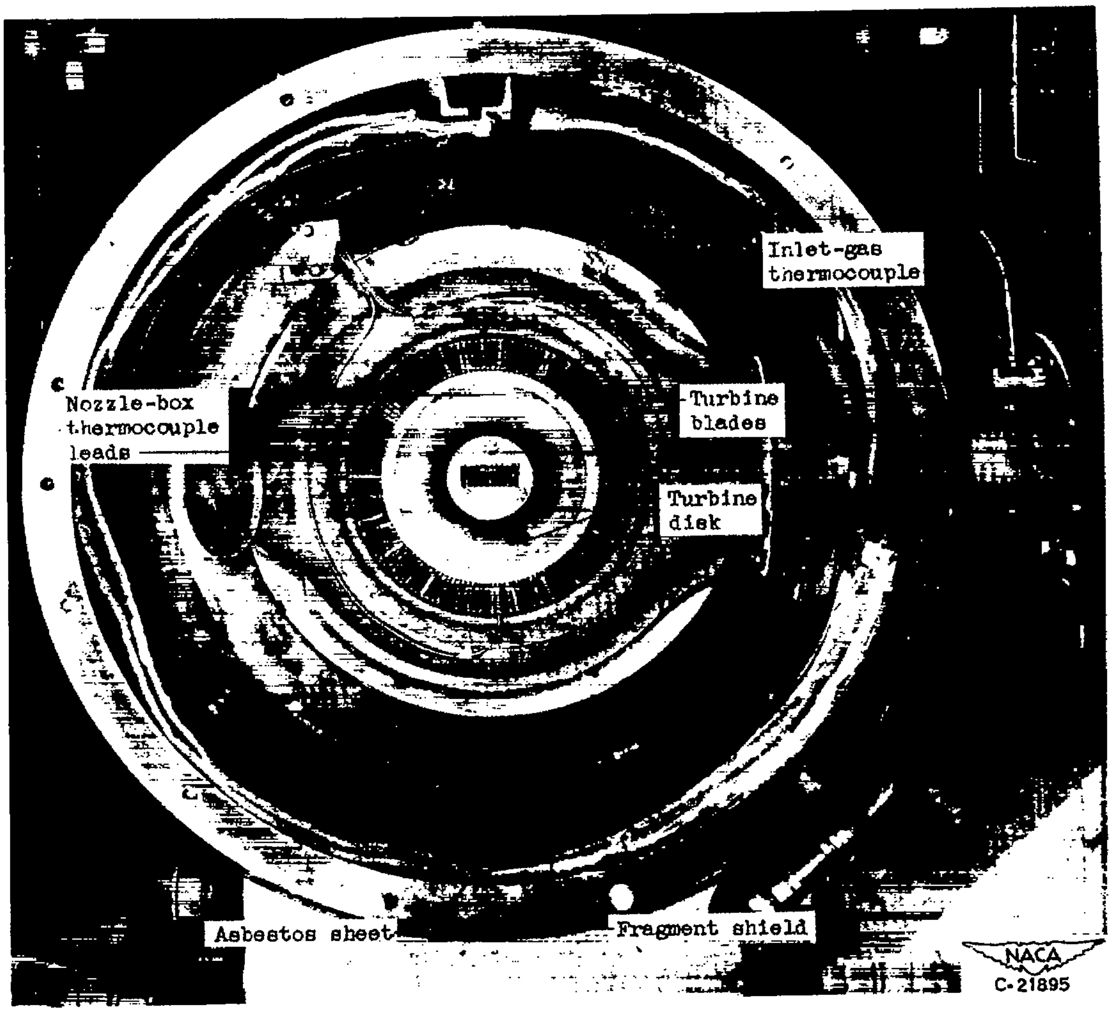

Figure 3. - Blade-evaluation unit.

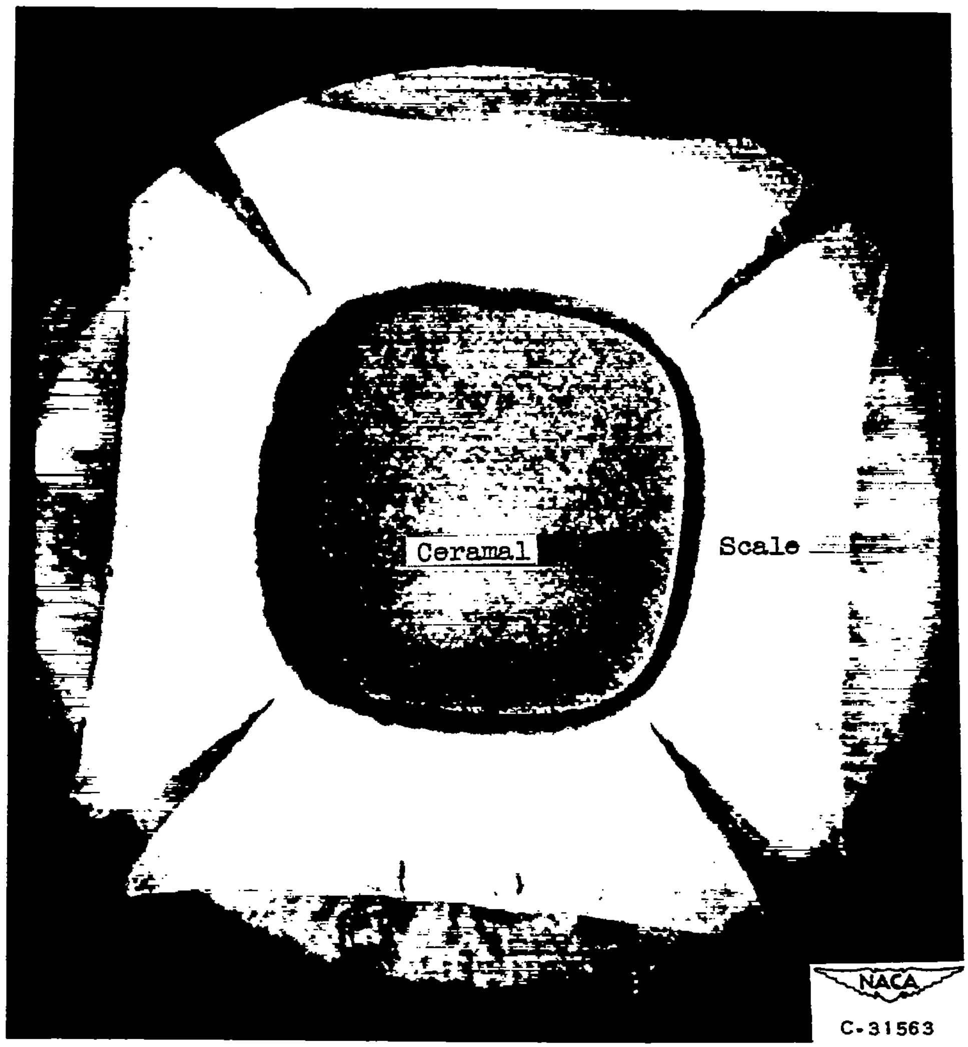

Figure 4. - Ceramal specimen containing 97.5 percent zirconium boride plus 2.5 percent boron (virgin powders) after 100 hours at 2000° F in air atmosphere. X15.

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