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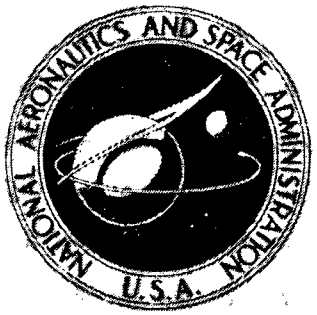

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FABRICATION OF CERMET BEARINGS FOR THE CONTROL SYSTEM OF A HIGH-TEMPERATURE LITHIUM-COOLED NUCLEAR REACTOR

by Howard G. Yacobucci, Richard L. Heestand, and Donald E. Kizer

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16. Abstract

The techniques used to fabricate cermet bearings for the fueled control drums of a liquid-metal-cooled reference-design reactor concept are presented. The bearings were designed for operation in lithium for as long as 5 years at temperatures to 1205° C. Two sets of bearings were fabricated from a hafnium carbide - 8-wt. % molybdenum - 2-wt. % niobium carbide cermet, and two sets were fabricated from a hafnium nitride - 10-wt. % tungsten cermet. Procedures were developed for synthesizing the material in high-purity inert-atmosphere glove boxes to minimize oxygen content in order to enhance corrosion resistance. Techniques were developed for pressing cylindrical billets to conserve materials and to reduce machining requirements. Finishing was accomplished by a combination of diamond grinding, electrodischarge machining, and diamond lapping. Samples were characterized in respect to composition, impurity level, lattice parameter, microstructure and density.

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SUMMARY

This report describes the techniques used to fabricate cermet bearings for the fueled control drums of a liquid-metal-cooled reference-design reactor concept. The bearings were designed for operation in lithium for as long as 5 years at temperatures to 1205° C. Two sets of bearings were fabricated from a hafnium carbide - 8-weight-percent molybdenum - 2-weight-percent niobium carbide cermet, and two sets were fabricated from a hafnium nitride - 10-weight-percent tungsten cermet. These materials were selected on the basis of corrosion resistance and fabricability as determined in a preceding lithium compatability testing program in which a series of carbide and nitride cermets were evaluated. Since purity, in respect to oxygen content, greater than that available in commercial material was required for maximum corrosion resistance, procedures were developed for synthesizing the material in high-purity inert-atmosphere glove boxes. Specimens were then consolidated by vacuum hot pressing. This required developing techniques for pressing cylindrical billets in order to conserve materials and to minimize the amount of grinding required.

Finishing of the bearings was done by diamond grinding reference flat and cylindrical surfaces, electrodischarge machining the radius, and diamond lapping to the required tolerances and finishes.

All starting materials were characterized with respect to composition and impurity levels. Bearings of a specific conposition were fabricated from a single lot of the refractory compound and metal binder phase.

Representative samples from each batch of material used in bearing fabrication and a hot-pressed sample from each batch were characterized in respect to composition, impurity level, lattice parameter, microstructure and density. Dimensions and surface finish were determined for each bearing.

INTRODUCTION

The NASA Lewis Research Center has recently terminated work on a technology program for a compact, fast-spectrum nuclear reactor for space electric power generation. This report covers a part of the work performed under that program. Reference 1 describes the liquid-metal-cooled reactor concept used to identify problems associated with advanced, high-temperature reactors of this type. In the course of this study, several reactivity control methods were considered. These were movable fuel, movable poison, and movable reflector.

The fuel in the movable fuel concept is asymmetrically positioned in six rotatable control drums (fig. 1) which are cooled by flowing lithium. Although this arrangement provides for a large amount of reactivity control, it also requires bearings that can operate in lithium for as long as 5 years at temperatures to 1205^{o} C. The design of these cermet bearings, which are essential to the development of this control system, is discussed in reference 2; this report discusses their fabrication.

The work reported herein was performed by Battelle's Columbus Laboratories (BCL) under subcontract to the General Electric Company - Nuclear Systems Programs (GE-NSP), Cincinnati, Ohio. It was part of an overall program conducted by GE-NSP for NASA Lewis under contract NAS 3-13447. The fabricational techniques presented herein utilize procedures previously developed by BCL under a companion program (ref. 3) in which lithium compatibility test specimens of six cermet compositions were prepared. On the basis of corrosion resistance and fabricability, as determined in the lithium compatibility testing program, two of the six cermet materials were selected for bearing fabrication. These are a hafnium carbide - 8-weight-percent molybdenum - 2-weight-percent niobium carbide (HfC-8Mo-2NbC) cermet and a hafnium nitride - 10-weight-percent tungsten (HfN-10W) cermet.

It was planned to test these bearings in lithium under simulated control drum operating conditions. However, the program was cancelled shortly after the bearings were fabricated.

EXPERIMENTAL PROGRAM

The objective of this study was to fabricate cermet test bearings of a specified composition, with special emphasis on attaining a desired oxygen level of 100 ppm or less, in accordance with compositions defined by GE-NSP. It was required that the material be fully dense, fine grained, and homogeneous for operation in high-temperature lithium. The type selected for fabrication was the control drum thrust bearing (see fig. 1), which is a modified spherical bearing having two components. The stationary member

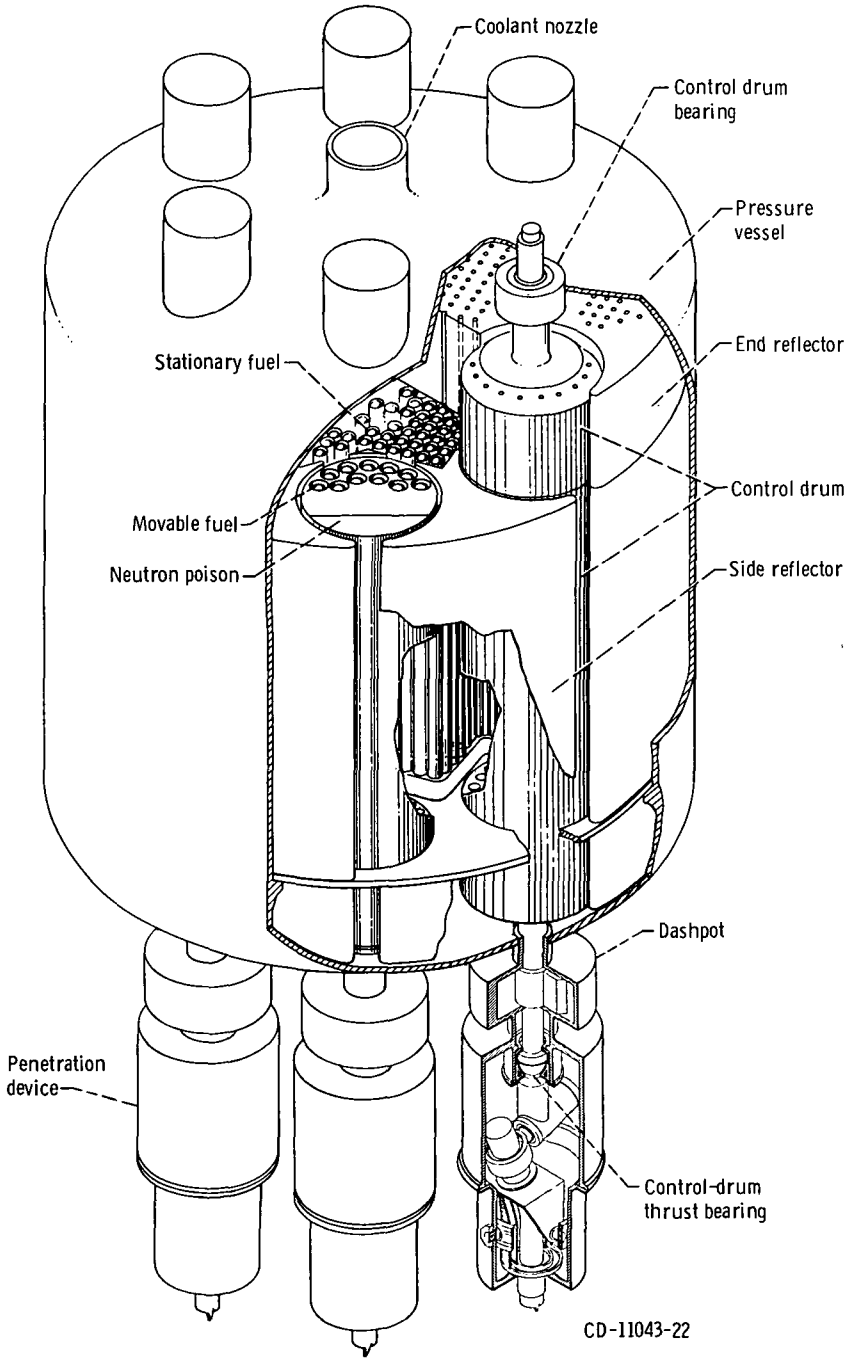

Figure 1. - Compact fast reactor - reference design.

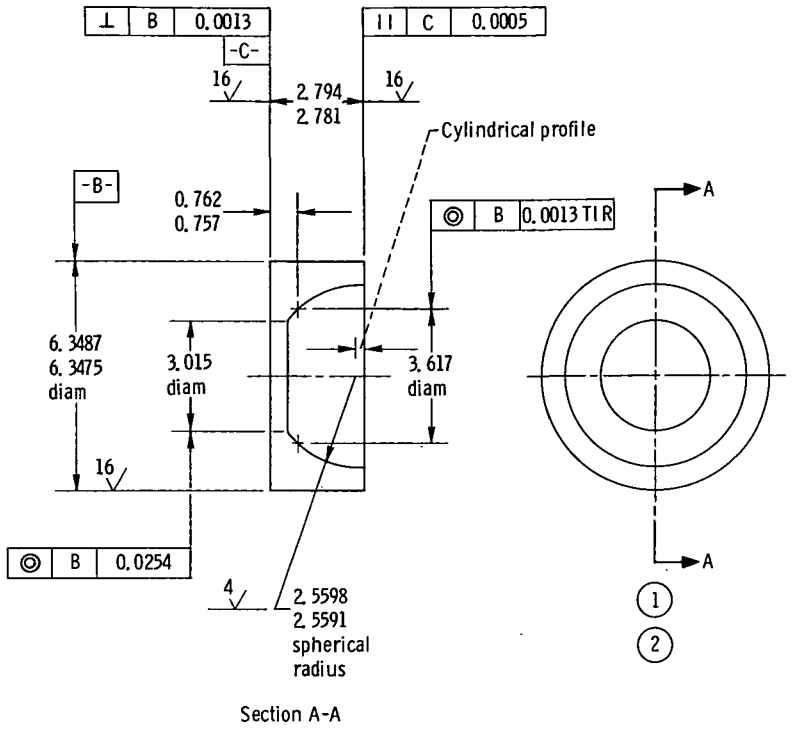

Figure 2. - Control drum spherical bearing 128C8072. Cermet composition: set, 1 HfN-10W; set 2, HfC-8Mo02NbC. (All dimensions are in cm.)

(pt. 128C8072) is shown in figure 2, and the rotating member (pt. 128C8071) is shown in figure 3. Two sets of bearings were made from HfN-10W, and two sets were made from HfC-8Mo-2NbC.

MATERIAL PROCUREMENT

Sources of materials utilized in the program were the same as those for the lithium compatibility test program in order to ensure similarity of composition and characteristics. Approximately 13 kilograms of reactor-grade crystal bar hafnium was procured that had an oxygen content of less than 50 ppm oxygen as certified by vendor analysis. The zirconium content was 3 weight percent.

The amount of hafnium originally required in the subcontract was 7.3 kilograms. However, because of an increase in bearing size it was necessary to procure 13 kilograms. Tungsten powder was purchased that had a Fisher subsieve particle size of 0.78 micrometer, and molybdenum powder of less than 1-micrometer particle size was used from the lithium compatability program. At that time a 2.3-kilogram lot of molybdenum was purchased as a minimum order requirement by the vendor. The oxygen con-

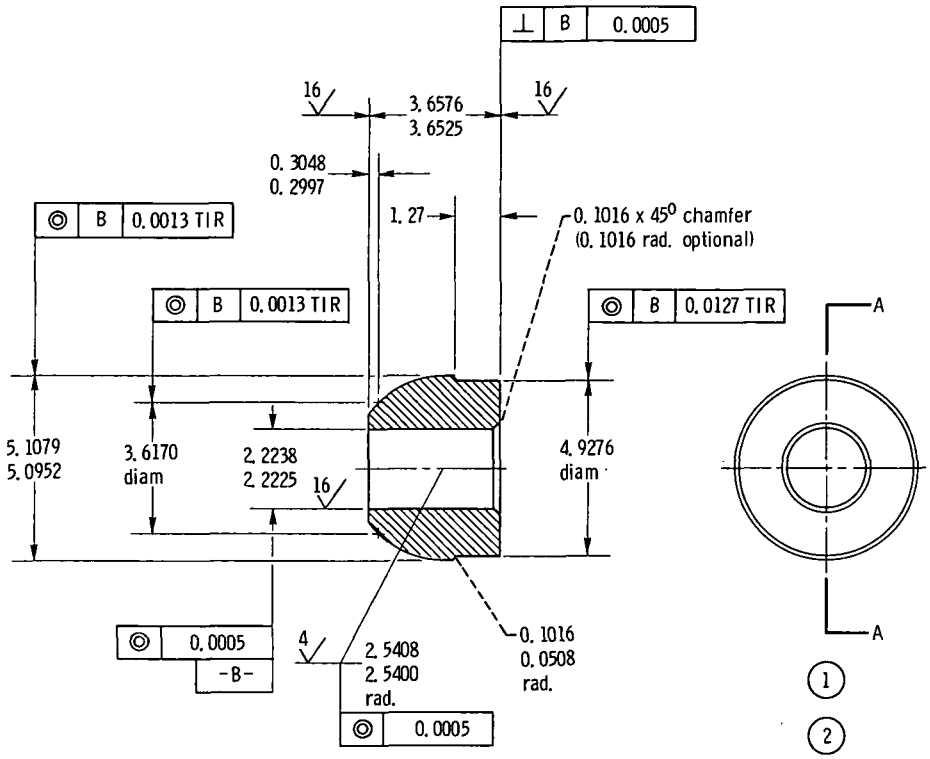

Figure 3. - Control drum spherical journal 129C8071. Cermet composition: set 1, HfN-10W; set 2, HfC-8Mo-2NbC. (All dimensions are in cm.)

tent of the as-received material was unsatisfactory in both cases and was reduced in the tungsten by heat treating in hydrogen for 4 hours at 1100° C. The oxygen content of the molybdenum was reduced by heat treating for 4 hours at 1100° C under a vacuum of 1×10^{-5} torr.

Niobium carbide utilized in the program was from the batch used previously and was also treated in a vacuum of 1×10^{-5} torr for 4 hours at a temperature of 1100° C. Spectrographic-grade graphite powder used in the preparation of HfC was cleaned by heat treating in a vacuum of 1×10^{-5} torr for 6 hours at 1400° C.

Because of the rigid requirements on oxygen content, all starting materials were reanalyzed for oxygen content by either BCL or an independent laboratory. All samples for gas analysis were prepared in a glove box and sealed in ferrovac iron cans with either ferrovac iron or vacuum-melted tin lids. Cans and lids were included as standards for analysis. Each canned specimen was also sealed in an argon-containing bottle for transfer to analysis.

Table I presents the results of analysis conducted on the starting materials. The crystal bar hafnium was analyzed for oxygen content by inert-gas fusion of samples from each piece of crystal bar. Initial samples were prepared in the same manner as ones

TABLE I. - ANALYSIS OF STARTING MATERIALS

Impurity	Source of analysis	Impurity level at less than value indicated[a], ppm												
		O[b]	Si	Fe	Mg	Mn	Al	Mo	Co	Ni	Cr	Ca	C (wt. %)	C (free)
Hf(lot A)[c]	Vendor	50	20	100	10	10	35	5	5	25	20	--	-----	20
	BCL[d]	74	10		15	5	40	10	3	20	30	20	-----	----
Hf(lot B)[c]	Vendor	50	20		10	10	35	5	5	25	20	--	-----	20
	BCL	72	10		15	5	40	10	3	20	30	30	-----	----
W	Vendor	----	6	11	4	1	2	10	1	22	10	1	-----	----
W(as received)	BCL	1500												
W(reduced)	BCL	26												
Mo	Vendor	----	72	40	10	10	20	--	10	22	10	10	-----	----
Mo(as received)	BCL	420												
Mo(reduced)	BCL	100												
NbC	Vendor	260	10	30									11.74	3900
NbC(reduced)	BCL	260											11.20	----

[a]Dashed lines indicate that impurity is below level of detection.

[b]Analyzed by inert-gas fusion.

[c]One lot furnished in two pieces: a sample taken from each piece.

[d]Battelle Columbus Laboratories.

submitted previously, and spectrographic-grade iron cans (23 ppm oxygen) sealed with vacuum-melted tin lids were used. Because of modifications in analytical procedure, however, the presence of the tin indicated high oxygen contents of 98 and 180 ppm in the two batches. Duplicate samples were resubmitted with iron lids used in place of tin and with approximately 1 gram of platinum added for each 0.10 gram of sample to enhance oxygen release. These changes were incorporated in all subsequent inert-gas fusion analyses for gaseous contaminants. The oxygen content was still in excess of that certified by the vendor (<50 ppm) but was not considered excessively high. The contamination levels of all other additives were judged to be sufficiently low to allow the material to be utilized. Although the oxygen level in the highest purity NbC was out of tolerance, previous work indicated that the strong carbothermic reduction during hot pressing of the carbide materials effectively reduces oxygen content.

SYNTHESIS OF HIGH-PURITY MATERIALS

Hafnium Nitride

In the program for the preparation of corrosion test specimens the process selected to synthesize hafnium nitride consisted of reducing the crystal bar hafnium to powder by hydriding (ref. 4), with conversion to the nitride made according to procedures previously developed at BCL (ref. 3). The crystal bar was cleaned by ultrasonic degreasing in absolute ethyl alcohol and was introduced into the vacuum-purged, inert-atmosphere glove box - furnace apparatus shown in figure 4.

The glove box was purged by evacuating it to a minimum pressure of 1×10^{-5} torr and backfilling it with purified argon. Both the moisture level and the oxygen content of the glove box were monitored continuously, and the atmosphere was circulated through a purifier when water-vapor contamination increased to an equivalent dewpoint of -55° C. When the glove box was not in use, all materials were sealed in containers and the atmosphere was recirculated through the purifier continuously. All materials as they entered or were removed from the glove box passed through an isolated, independently evacuated and purged interlock.

The material was placed in tungsten crucibles which were placed in a tungsten mesh furnace attached to the glove box. Batches were approximately 2 kilograms. The door

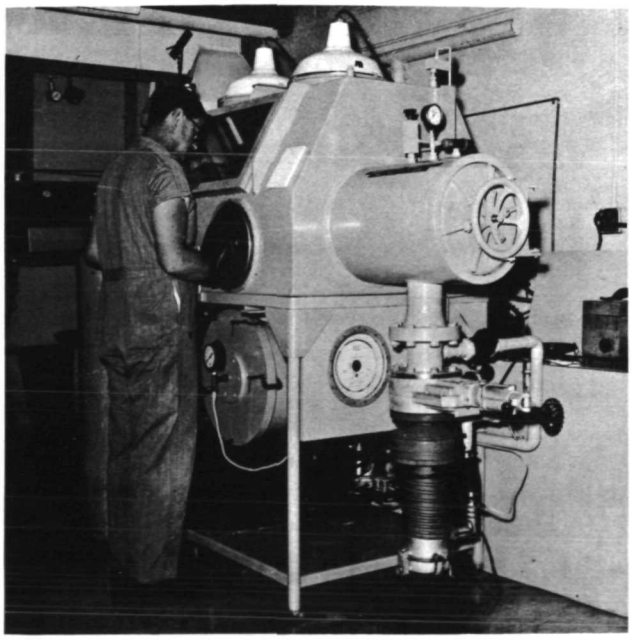

Figure 4. - Combination high-purity inert glove box - vacuum and atmosphere furnace.

TABLE II. - OXYGEN ANALYSES OF HAFNIUM AND HAFNIUM COMPOUNDS

Specimen	Material	Oxygen content, ppm	Sample type
Hf-A	Hf crystal bar	98	Iron can - tin lid
Hf-B	↓	180	Iron can - tin lid
Hf-C		74	Iron can - iron lid - platinum
Hf-D	↓	72	↓
HfH-1A	Hf hydride powder	430	↓
HfH-1B	↓	420	↓
HfH-2A		380	↓
HfH-2B		420	↓
HfH-3A		450	↓
HfH-3B		480	↓
HfH-4A		370	Iron can - copper lid
HfH-4B		44	↓
HfH-5A		120	↓
HfH-5B		220	↓
HfH-6A		310	↓
HfH-6B		140	↓
HfH-7A		20	↓
HfH-7B	↓	40	↓
HfH-8A	HfH stored from previous program	490	↓
HfH-8B	HfH stored from previous program	620	↓
HfH-1BM	HfH-1 ball milled for 18 hr	270	↓
HfN	HfN stored from previous program	32	↓

Spattering of tin precluded obtaining any results in this case. On further discussion with General Electric personnel and the chemical analyst, copper was selected as a potential lid material. Calibration samples and duplicate samples from batches HfH-4 to HfH-7 were submitted for reanalysis. Also samples of hafnium hydride and hafnium nitride stored from the previous program and ball-milled material from batch HfH-1 were submitted. Again results were highly inconsistent.

Further discussions were held with the chemical analyst and General Electric personnel with the conclusion that the high evolution rate of hydrogen on flash melting of the specimens masked any accurate determination of oxygen and that no further attempt would be made to analyze the hydride.

Since hafnium metal powder was to be used for preparation of the nitride and hafnium hydride was to be used directly to prepare the hafnium carbide, only sufficient material was dehydrided for preparation of the nitride. The remaining hafnium hydride was sealed in containers under argon and stored in the glove box pending preparation of the carbide.

prepared as weighed proportions of the required components. Ball milling to achieve mixing of the blends was conducted under argon for a period of 18 hours. The blending for each composition was conducted immediately prior to hot pressing in order that all bearings and characterization specimens of a given composition could be prepared from a single powder batch.

HOT PRESSING OF BILLETS

Billets hot pressed for the bearings and characterization specimens were prepared from single batches of powder of the desired composition. Dies consisted of a reusable ATJ graphite die body with sacrificial ATJ sleeve inserts and reusable AXF graphite punches. Sacrificial ATJ disks were placed between the composition to be pressed and the AXF punches in order to eliminate bonding or damage to the AXF punches. The die cavity was lined with a minimum of one layer of Grafoil followed by one to three 0.0254-millimeter layers of tungsten to minimize contamination of the billet material.

All graphite utilized in the punches and corollary furnace insulation was outgassed

Figure 5. - 68 000-Kilogram-load vacuum hot press.

Die sets were machined during the material synthesis portion of the program for all bearing sizes originally specified. Prior to initiation of hot pressing, however, the smaller bearings[1] were eliminated as a requirement because of design changes and fund limitations.

The pressing of billets for the reference bearings required that hollow cylindrical shapes be fabricated in order to conserve material and minimize grinding and machining costs. This was accomplished by utilizing an annular die with a central graphite rod. To demonstrate feasibility of this technique, with the reference materials, a trial pressing was made with the small dies and the HfN-10W material remaining from the previous program. The critical point in that experiment was the selection of an internal graphite rod that has an expansion coefficient greater than that of HfN-10W so that the cermet was not loaded in tension on cooling. The coefficient of expansion of HfN-10W and HfC-8Mo-2NbC was unknown at that point in the program.

The first pressing was conducted with a center rod of ATJ graphite which has an average expansion coefficient of 6.05×10^{-6} centimeter per centimeter per degree Centigrade throughout the temperature range of 1000° to 2400° C. The pressing was conducted at 2100° C for 4 hours at a load of 6.894×10^{7} N/m^2. Figure 6 shows the fracturing which occurred in the cermet on cooling. A second pressing was made under the same conditions with a central rod of L-56 graphite, which has an average coefficient of expansion of 9.0×10^{-6} cm/cm/$^{\circ}$ C over the temperature range of 1100° to 2500° C. This material performed successfully, as shown in figure 7; however, it was necessary to remove the L-56 graphite rod from the billet by machining. Thus, the coefficient of expansion of HfN-10W must be equal to or less than that of L-56 grade graphite.

Metallographic examination of one side of the specimen gave a metallographic density of 98 percent of theoretical.

Billets of the HfN-10W composition for bearings 128C8072 (fig. 2) and 128C8071 (fig. 3) were pressed successfully by using the annular die configuration. Conditions for pressing are listed in table III.

Experimental pressing of the HfC-8Mo-2NbC composition was also conducted with annular dies used for the small bearings to conserve materials. In the initial pressing of the HfC-Mo-NbC composition in the annular die, cracking occurred on the billet surface, which indicated a reaction with the Grafoil and tungsten diffusion barriers. This experiment was repeated with double layers of Grafoil and tungsten on one end of the billet and double layers of Grafoil and molybdenum on the other end. In the experiment it was found that the molybdenum and Grafoil combination was unsatisfactory because of excessive diffusion. However, double layers of tungsten and Grafoil were effective in

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[1]Originally, it was planned to fabricate all of the cermet bearings found in the control drum system of the reference design reactor concept.

TABLE III. - PRESSING CONDITIONS FOR BILLETS

[Pressure, 68 940 kN/m^2.]

Billet	Material, wt. %	Part	Temperature, °C (a)	Time, hr	Comments
1	HfN-10W	128C8071	2190	3.5	Final ram movement, 0.102 mm/hr.
2	↓	128C8071	2160	3.5	↓
3		128C8072	2150	4	
4		128C8072	2150	4	
5	HfC-8Mo-2NbC	128C8071	2050	2.5	Central rod failed prior to full density.
[b]5	↓	128C8071	↓	4	Final ram movement, 0.102 mm/hr.
6		128C8072		↓	↓
7		128C8072			
8		128C8071			
[c]9	↓	--------	↓		

[a]Optical measurement through sight port, uncorrected.

[b]Repress conditions.

[c]Specimen for characterization.

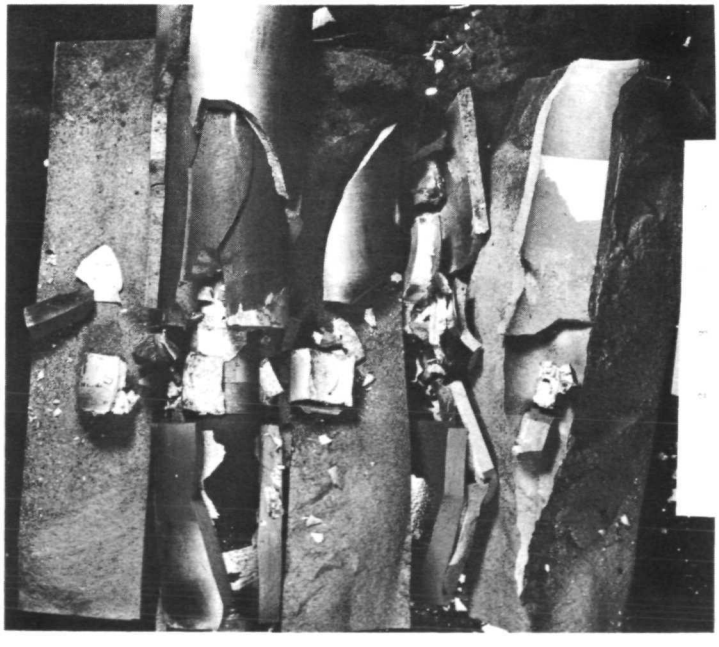


Figure 8. - Graphite die failure.

the repressing of a specimen requires machining of special die components to fit the specimen dimensions.

Since the central graphite mandrel for billets for bearing 128C8072 is larger and comparably less fragile than that required for bearing 128C8071, it was decided to attempt pressing the larger billet. Two successful pressings were accomplished with no unexpected difficulties in removal of the central graphite rod. The pressings were conducted at 2050° C for a period of 4 hours at a pressure of 6.894×10^7 N/m^2. Slight surface cracking was observed on each billet; however, these areas could be removed by grinding. Preliminary measurements of bulk density indicated that the density of each part would be in excess of 95 percent of theoretical.

A second billet for bearing 128C8071 was pressed successfully at 2050° C for 4 hours at a pressure of 6.894×10^7 N/m^2. After preparation of the special die body, the specimen for bearing 128C8071 was successfully repressed at 2050° C for an additional 4 hours at 6.894×10^7 N/m^2. The billet was repressed successfully, and bulk density measurements indicated a density of 93.5 percent of theoretical. This is comparable to the densities obtained on the other HfC-8Mo-2NbC billets, approximately 95 percent of theoretical. The parameters for the hot pressing of all billets for bearing fabrication are presented in table III.

Since insufficient material was present in the hot-pressed HfC-8Mo-2NbC billets for removal of a characterization sample, an additional sample was pressed with the die body and punch for the small bearings. The parameters of time, temperature, and pressure were the same as those used in pressing the bearing billets.

CHARACTERIZATION

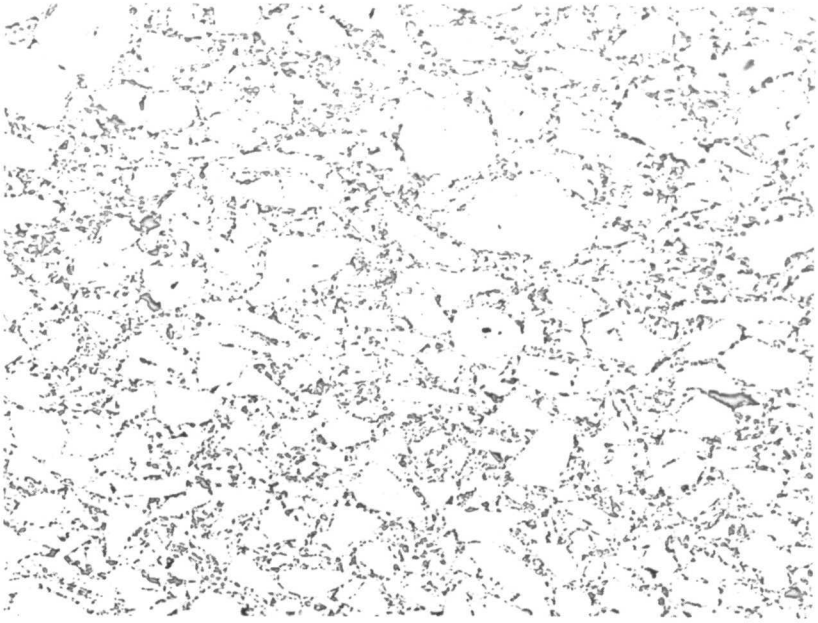
Analysis for the major constituents for each bearing shape is given in table IV, and

TABLE IV. - ANALYSIS FOR MAJOR CONSTITUENTS OF HOT-PRESSED BILLETS

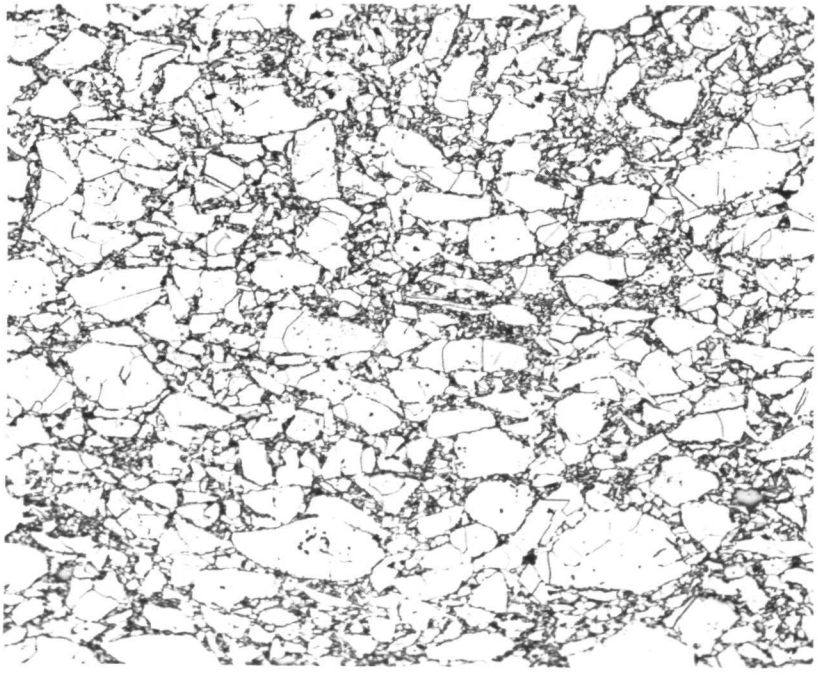
Material, wt. %	Part	Amount of indicated element found in sample[a], wt. %								Major-constituent lattice parameter, anstroms
		Hf	W	Mo	Nb	C	C (free)	N_2	O_2	
HfN-10W	128C8071	83.5	10.1	----	----	----	----	6.27	0.0199	4.526±0.001
HfN-10W	128C8072	83.4	10.1	----	----	----	----	6.25	0.0722	4.526±0.001
HfC-8Mo-2NbC	(b)	82.8	----	8.00	1.82	6.50	0.04	----	0.0430	4.625±0.001

[a]Dashed lines indicate that element is below level of detection.

[b]Sample prepared for characterization.

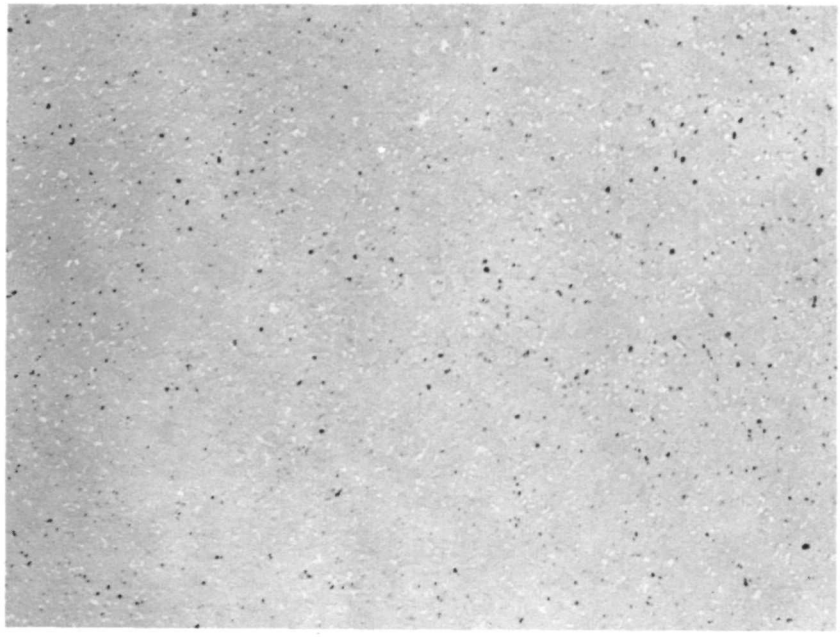


(a) As polished. X100.

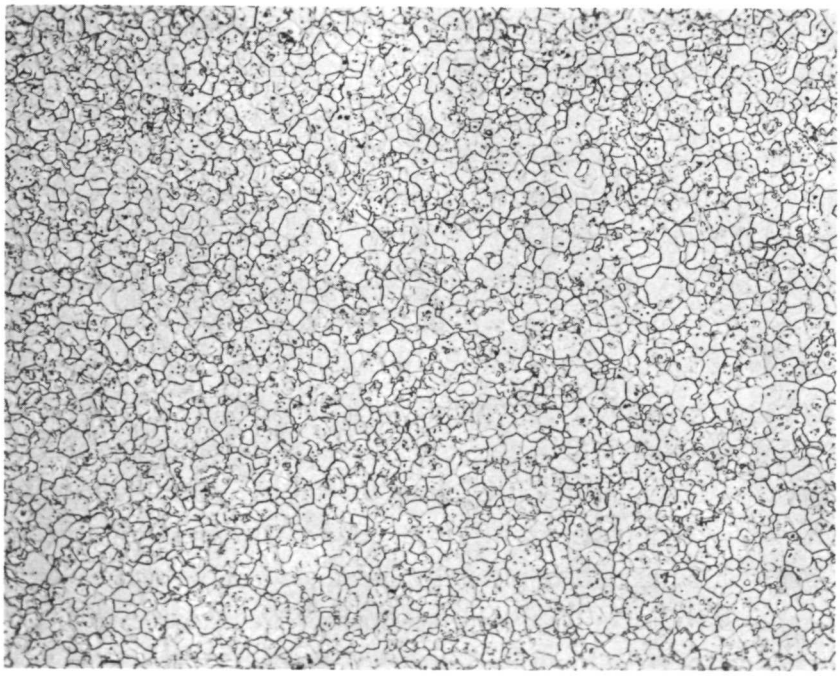


(b) Etched. X100.

Figure 9. - HfN cermet structure for bearing 128C8071.



(a) As polished. X100.



(b) Etched. X100.

Figure 11. - HfC cermet structure of representative sample.

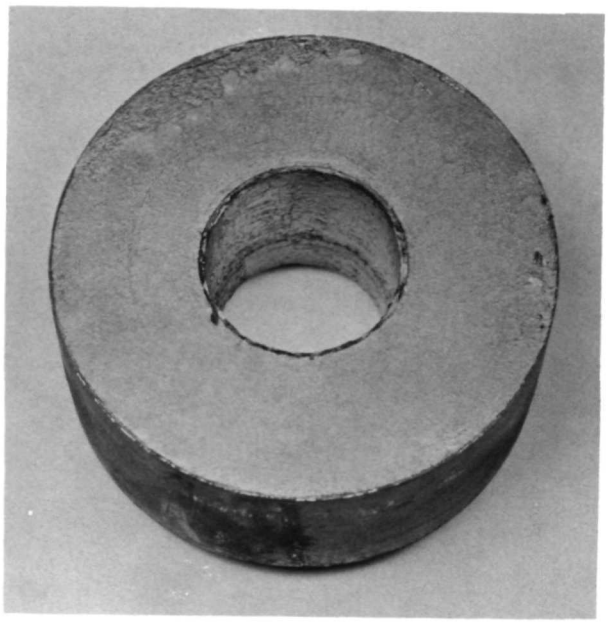


Figure 12. - Hot-pressed billet before grinding.

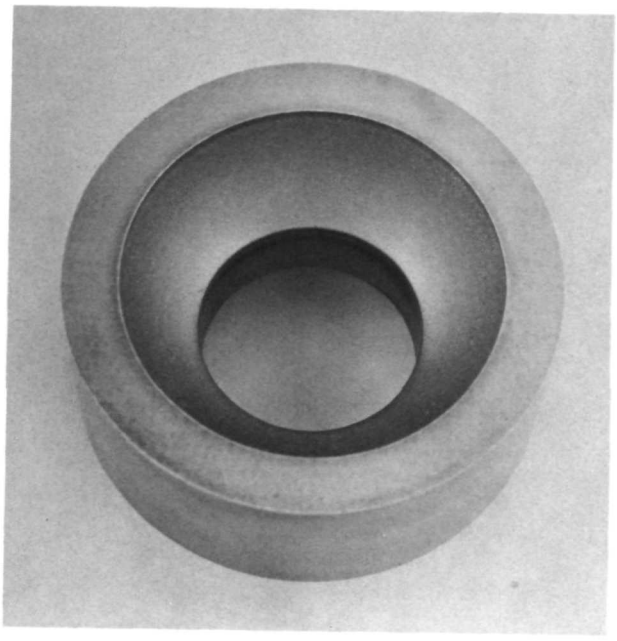


Figure 13. - Bearing as diamond ground and electrodischarge machined.

of 0.254 millimeter per hour was maintained during rough cutting to prevent chipping. However, it was necessary to reduce the cutting speed below 0.0762 millimeter per hour during final cutting. Figure 12 is representative of a billet in the hot-pressed condition. Figure 13 shows a billet which has been diamond ground on the flat and cylindrical surfaces and electrodischarge machined on the radius.

Final diamond lapping for bearing 128C8072 was performed with a special fixture on a lathe with a radius cutter. This same appratus, shown in figure 14, was used for the inspection of the part. For bearing 128C8071 a jig boring machine, shown in figure 15, was used to lap and inspect the radius of the bearing which was not on the centerline axis of the bearing.

All grinding and lapping operations on both materials were extremely time consuming because of the exceptional hardness of the materials.

Results of the inspection on the machined parts are presented in tables VII and VIII. All critical dimensions on the drawings of figures 2 and 3 are listed along with the dimensions determined and the variations of radii. Not all dimensions are within tolerance because of difficulties in working the materials. However, all critical dimensions were ground as close as possible by the methods available and within the funding limitations.

TABLE VII. - DIMENSIONS OF SPHERICAL BEARING 128C8071

Designation	Nominal dimension, cm	Specimen			
		HfN-1	HfN-2	HfC-1	HfC-4
		Dimension, cm			
Length	3.658	--------	--------	--------	--------
	3.653	3.658	3.658	3.655	3.650
Outside diameter	5.1079	5.1059	--------	5.1039	--------
	5.1069	5.1049	5.1059	5.1049	5.1029
Outside diameter	4.9276	4.9276	4.9276	4.9276	4.9276
Inside diameter	2.2238	2.2238	2.2238	2.2238	2.2250
	2.2225	2.2233 (+0.0010, -0.0008)	2.2230 (+0.0005, -0.0003)	2.2230 (+0.0010, -0.0010)	2.2240 (+0.0020, -0.0020)
Radius	2.5408	2.5403	--------	2.5392	--------
	2.5400	2.5397	2.5403	2.5397	2.5387
Concentricity	2.2225	--------	--------	--------	--------
	3.6170	--------	--------	--------	--------
	[a].0013	.0041	.0030	.0051	.0015
Concentricity	2.2225	--------	--------	--------	--------
	5.1079	--------	--------	--------	--------
	[a].0013	.0033	.0015	.0038	.0013
Concentricity	2.223	--------	--------	--------	--------
	4.928	--------	--------	--------	--------
	[a].013	.0005	.0005	.0005	.0005
Flatness	.0005	.0005	.0005	.0005	.0005
Perpendicularity	.0005	.0005	.0005	.0005	.0003
Out of round	.0005	.0010	.0005	.0010	.0020
Length	1.270	1.270	1.270	1.270	1.280
Axial location	1.549	--------	--------	--------	--------
	1.544	1.547	1.552	1.544	1.533

[a]Total indicated runout.

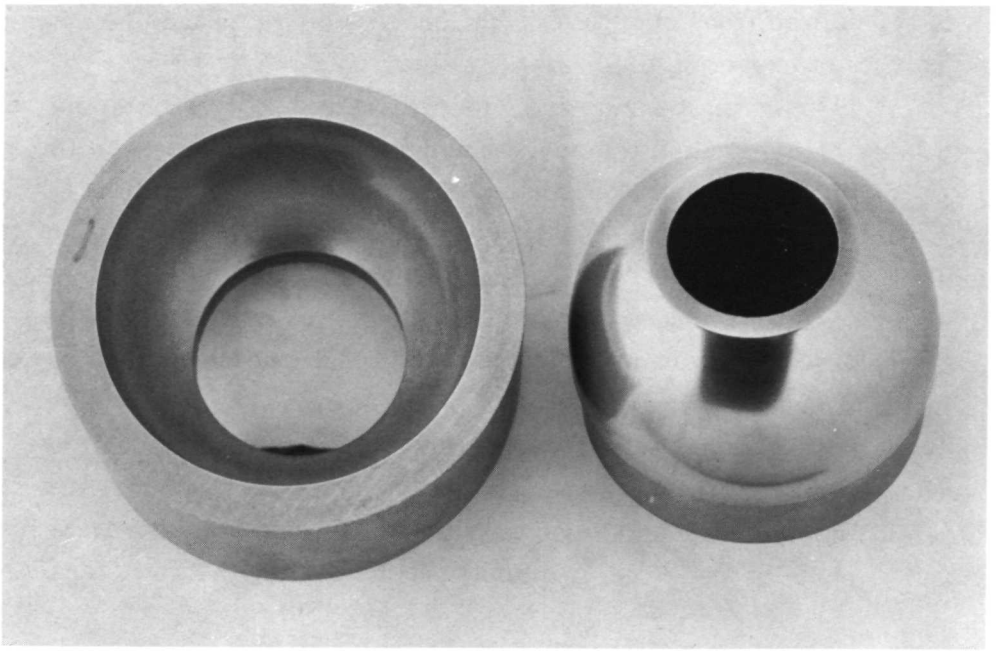


Figure 16. - Typical finished bearings.

CONCLUDING REMARKS

Four bearing components of each of the two required cermet compositions were fabricated, ground to size, and characterized. Procedures used to synthesize the materials were identical to those used in the lithium compatability program conducted previously. Procedures for hot pressing required considerable modification in fabricating billets for the bearings in order to conserve material and avoid excessive grinding during finishing. The pressing of annular billets was successfully accomplished after experimental determination of a technique for selection and removal of a central graphite rod at the pressing temperature. Temperatures and pressures used for hot pressing the parts were in excess of those normally utilized in hot pressing but were required by the refractory nature of the materials.

All handling of the powders was done under vacuum or in high-purity inert atmospheres. The degree of purity maintained in the bearings is significant when it is considered that the materials were handled primarily as powders having a high surface area available for contamination. The synthesis and fabrication procedure required many steps with a minimum of 20 hours at temperatures between 750° and 860° C, 30 hours at 1800° C, and 5 hours above 2000° C.

Analytical difficulties were encountered when attempting to determine the oxygen content of the hydride powders. These analyses could not be conducted because the high

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