RESEARCH MEMORANDUM

RESISTANCE OF VARIOUS MATERIALS TO ATTACK BY MOLTEN BISMUTH-LEAD EUTECTIC AT ELEVATED TEMPERATURES

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NATIONAL ADVISORY COMMITTEE
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RESISTANCE OF VARIOUS MATERIALS TO ATTACK BY MOLTEN BISMUTH-LEAD EUTECTIC AT ELEVATED TEMPERATURES

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SUMMARY

The resistance of 40 materials including alloys, ceramics, ceramals, and pure metals to attack by bismuth-lead eutectic at temperatures between 1500° and 2000° F was investigated. A velocity of 15 feet per second was maintained between the material surface and the bismuth-lead eutectic. Those materials found to be resistant to this attack included 17 of the ceramals and ceramics, graphite, and arc-cast molybdenum. All other materials investigated were appreciably attacked by the eutectic in the form of uniform attack, cavitation, or pitting, as indicated by metallographic analysis. No evidence of intergranular corrosion was observed in any of the materials studied in this investigation. Disintegration rates were estimated in mils per year from linear measurements taken before and after the specimens were subjected to attack by the molten eutectic.

INTRODUCTION

Some static studies of the materials subjected to attack by bismuth-lead molten eutectic are reported in reference 1. The present investigation was conducted to determine the resistance of materials to attack by bismuth-lead molten eutectic in a dynamic system; that is, a system having a relative velocity between the liquid and the material surface.

The attack on materials in a dynamic liquid flow system depends on (a) the velocity of flow, (b) the temperature distribution on various solid parts, (c) the temperature distribution in the liquid, and (d) the geometry of the system, in addition to corrosion, dissolution, and erosion. In a continuous circulating loop (nonisothermal system) the concentration of dissolved material in the liquid increases and decreases cyclically as the liquid passes the hot and cold portions of the system, respectively.

In this investigation, a standard velocity of liquid relative to the surface of the test material was maintained and isothermal test

temperatures of 1500° and 2000° F were held, the concentration of the dissolved material in the liquid may have approached the saturation point. To this extent, the test does not duplicate the condition in continuous nonisothermal flow systems. The tests, however, are believed to afford a preliminary screening of materials for use with the bismuth-lead eutectic.

APPARATUS AND PROCEDURE

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The liquid-metals-attack equipment, shown in figure 1, was used in this investigation. Both flat faces of the specimen ring were covered by a graphite washer allowing only the peripheral surface of the ring to be in direct contact with the molten eutectic bath. The specimen ring with graphite washers was mounted on a graphite shaft which, in turn, was threaded onto a steel shaft. This steel shaft was supported by a water-cooled bearing assembly which was bolter on the water-cooled steel pot cover. The specimen-ring periphery was surrounded by a grid of baffles in the eutectic bath, having a clearance of approximately 0.012 inch at room temperature between the baffles and the specimen periphery. The purpose of the grid of baffles was to render the eutectic bath practically motionless while the specimen was rotated therein, thus causing relative motion between the periphery of the specimen and the eutectic mixture.

The eutectic bath pot was made of graphite, which is known to have a high resistance to attack by the bismuth-lead eutectic. The molten eutectic-bath temperature was maintained by an electric globar furnace and was indicated by a chromel-alumel thermocouple inserted in a porcelain-type protector tube placed within 1/4 inch of the specimen. The furnace temperature, however, was set and controlled at the temperature necessary to obtain the desired eutectic temperature by means of a platinum-platinum and rhodium thermocouple and an automatic temperature-control system. This temperature observation and control system maintained the eutectic-bath-temperature variation within ±5° F at 1500° F as well as 2000° F.

Chemical compositions of the alloys, ceramics, and elements used in this work are given in table I.

The bath consisted of 23 pounds (approximately 62 cu in.) of the lead-bismuth eutectic in order to completely envelope the specimens. Argon at the rate of 10 cubic feet per hour was introduced into the space above the bismuth-lead bath in order to protect it from oxidation.

Test specimens of materials were of two kinds depending upon the availability. Easily available materials were machined as shown in figure 2 according to the following specifications:

Diameter, in.	approximately 2
Hole diameter, in..	0.751
Thickness, in..	0.25
Concentricity, in.	0.0001

The radius of the specimen ring was measured at four equally spaced points, which were numbered on the specimen face. Materials that were difficult to obtain because of the purity and dimensional limitations were machined into segments and inserted into a slotted graphite holder as shown in figure 3. The radial dimension of the segments was 0.325 inch and the thickness, 0.251 inch. Precision measurements were made of the radius of each specimen ring and insert before and after each evaluation period. An electronic height gage and a precision surface plate were used in these measurements providing an accuracy of ±0.0001 inch.

PROCEDURE

The material investigated was cleaned and rinsed in 95-percent ethyl alcohol and then dried. The specimen-ring was placed on the shaft, sandwiched between two graphite washers, that were used to minimize attack on the faces of the specimen ring. The specimen shaft assembly was threaded onto the bearing assembly (fig. 1), and lowered into position in the eutectic bath.

The eutectic bath was heated to 50° F above the operating temperature before use, experience having showed that the eutectic bath temperature decreased 50° F after immersion of a specimen therein. If a material was suspected or known to have poor resistance to thermal cracking, the material specimen ring, assembled on the graphite shaft, was preheated before immersion in the eutectic bath. The preheating process consisted of insertion for 5 minutes in an infrared oven at approximately 450° F and then, after being attached (at preheating temperature) to the drive shaft, suspension for 10 minutes above the eutectic bath surface. The temperature of this surface was near the operating temperature. The specimen graphite-shaft assembly that was attached to the bearing assembly was suspended above the eutectic bath by a collar which was inserted between the bearing assembly and the steel pot cover. Following preheating, the collar was removed, and the bearing assembly was boltered in place as shown in figure 1. The electric motor was then started and gradually brought up to its rated speed by means of a rheostat. Speed of rotation of the specimen was measured with a stroboscope and maintained at approximately 1720 rmp in order to produce a peripheral velocity of the specimen of 15 feet per second.

A material that was unpredictable, with regard to being attacked by an eutectic, was run for a 1-hour period. A material believed to have poor resistance to attack was evaluated for a period of 5 minutes; good resistance, for 24 hours.

The ceramics, ceramals, pure metals, and the refractory alloys were tested at 2000° F. The other alloys showed extremely rapid attack at 2000° F and hence were run at 1500° F.

Upon removal from the eutectic bath, the specimen on the graphite shaft was immersed in a crucible containing powered carbon until cooled to approximately room temperature in order to protect the specimen from oxidation. Particles of the eutectic bath adhering to the specimens, were removed by a combination of heat and mechanical treatment. 2225

The specimen radii before and after test were measured by an electronic height gage and precision surface plate in order to determine the change in dimensions caused by attack. In cases of attack, specimens were carefully machined from the outer periphery toward the center on a precision cylindrical grinder, removing a layer of material 0.0005 inch thick at a pass until the point of the deepest attack had been removed. This point of deepest attack determined the final radius measurement of the specimen.

Microscopic analysis of a cross section of the specimen which included some of the peripheral surface was made in order to determine the mechanism of attack.

The sectioned specimen was mounted, polished, and usually etched so that visible changes in the crystallographic structure could be noted. Each specimen was examined at magnifications of X50 and X1000 for evidence of physical changes.

RESULTS AND DISCUSSION

Three types of attack by the eutectic bismuth-lead bath on the investigated materials are listed in table II. These types are:

(1) Uniform attack: Rate of attack is approximately the same over the entire peripheral surface of the specimen, the surface remaining relatively smooth (see fig. 4).

(2) Caviation: Rate of attack is markedly uneven on the surface of the specimen, the surface becoming rough and sometimes jagged (see fig. 5).

(3) Pitting: Rate of attack appears uniform, the surface appearing smooth and unattacked except in a very few small areas of the specimen; but, upon close examination of the surface, small areas of attack, usually macroscopic in size, are evident (see fig. 6).

The following materials given in table II were found to be totally resistant to attack at a temperature of 2000° F and a period of 24 hours in this investigation: alumina, beryllia, boron carbide, boron carbide - iron, chromium carbide, graphite, K-138, K-138A, K-151A, magnesia, arc cast molybdenum, bolybdenum disilicide, NBS-4811C, fused quartz, silicon carbide - boron carbide, titanium carbide, zircon, zirconia, and zirconium carbide - niobium.

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The remaining materials investigated (table II), underwent appreciable attack, ranging from 1.82×10^2 mils per year for the sintered alloy LT-2, to 4.25×10^7 mils per year for calcium reduced vanadium.

Uniform attack, similar to figure 4, was observed in Hastelloy B, L-605 alloy, LT-2 alloy, sintered molybdenum, nickel, niobium, titanium, vanadium, tungsten, zirconium, 316 alloy, and 347 alloy.

Cavitation as shown in figure 5 occurred in chromium, cobalt, Inconel, and iron.

Pitting similar to figure 6 appeared in the molybdenum tungsten alloy, S-816, tantalum, Vitallium, and zirconium carbide.

No intergranular corrosion was found in this investigation.

The variation in corrosion rates between arc cast and sintered molybdenum is noticable (table II). This variation is corroborated metallographically by comparing the photomicrograph of arc cast molybdenum (fig. 7) with that of sintered molybdenum (fig. 8). The exposed surface of the arc cast molybdenum is relatively smooth and unaffected as compared with the sintered molybdenum. Density measurements, however, indicate no appreciable difference in body porosity. Reduced corrosion resistance, in the sintered specimen might be attributed to occluded gases, which formed invisible phases not present in the arc cast molybdenum.

SUMMARY OF RESULTS

Molten bismuth-lead eutectic alloy at temperatures from 1500° to 2000° F was used as the critical media in attack resistance investigations of 40 different materials having melting points above 2000° F. These materials included pure metals, ceramics, ceramals, and alloys.

Of these materials, alumina, beryllia, boron carbide, boron carbide - iron, chromium carbide, graphite, K-138, K-138A, K-151A, magnesia, arc cast molybdenum, molybdenum disilicide, NBS-4811C, fused quartz, silicon carbide-boron carbide, titanium carbide, zircon, zirconia, and zirconium carbide-niobium were totally resistant to attack for 24 hours at 2000° F. The remaining materials were critically attacked exceeding 500 mils per year. Employment of the ceramics, ceramals, and metals found to be totally resistant to attack in a period of 24 hours appeared promising as container material insofar as resistance to attack by molten bismuth-lead eutectic at 1500° to 2000° F is considered. 2225

Lewis Flight Propulsion Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, June 12, 1951.

REFERENCE

1. Anon.: Liquid-Metals Handbook. Atomic Energy Commission and Bur. Ships, Navy Dept., June 1, 1950. pp. 103, 106, 107.

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TABLE I - MATERIAL COMPOSITION

	Constituents (percent by weight)
	Alloys
Hastelloy B	Ni, 66.4; Mo, 28.0; Fe, 5.5; C, 0.1
Inconel	Ni, 78.7; Cr, 13.5; Fe, 6.8; Si, 0.4; Cu, 0.3; Mn, 0.2
L-605	Fe, 51.0; Cr, 20.0; W, 15.0; Ni, 1.0; Fe, 1.8; Mn, 1.5; Si, 0.5; C, 0.1
LT-2	W, 60.4; Cr, 24.0; Al_2O_2, 12.1; Fe, 0.4; Al, 0.3
Mo-W	Mo, 98; W, 2.0
S-816	Co, 45.0; Cr, 20.0; Ni, 20.0; Nb, 4.0; W, 4.0; Mo, 3.0; Fe, 2.6; Mn, 0.6; C, 0.4; Si, 0.3
Vitallium	Co, 64.0; Cr, 2.8; Mo, 6.0; Mn, 1.0; Si, 0.6; C, 0.2
316	Fe, 67.0; Cr, 17.0; Ni, 12.0; Mo, 1.8; Mn, 1.0; Si, 0.5
347	Fe, 69.0; Cr, 18.0; Ni, 10.5; Mn, 1.0; Nb, 0.8; Si, 0.5
	Ceramics
Al_2O_3	Al_2O_3, 99.5
BeO	BeO, 99.5
B_4C	B, 79.1; combined C, 10.8; free C, 3.9
B_4C-Fe	B_4C, 64; Fe, 36
CrC	Cr, 86.2; C, 12.1; Fe, 0.2
K-138	TiC, 80; Co, 20
K-138A	TiC, 66.3; Co, 18.7; NbC + TaC + TiC, 15
K-151A	TiC, 66.3; Ni, 18.7; NbC + TaC + TiC, 15
MgO	Mg, 60.0; O, 39.6; combined C, 0.1
$MoSi_2$	$MoSi_2$, 99.0; Fe, 0.9; C, 0.1
NBS-4811C	BeO, 82.6; ZrO, 8.3; Al_2O_3, 7.0; CaO, 2.0
Quartz	SiO_2, 99.9
SiC-B4C	SiC, 85; B_4C, 15
TiC	TiC, 96.5; excess C, 1.9; TiO_2 + TiN_4, 1.00; Si, 0.3; Ni, 0.1; W, 0.1; Co, 0.1
Zircon	$ZrSiO_2$, 99.1
ZrC	Zr, 86.2; combined C, 9.4; free C, 4.1
ZrC-Nb	ZrC, 88; Nb, 12
ZrO_2	Zr, 73.40; O, 25.6; combined C, 0.1
	Metals-Element
Chromium	Cr, 98.5
Cobalt	Co, 97.5
Graphite	C, 99.8
Iron	Fe, 99.9
Molybdenum, arc cast	Mo, 98.8
Molybdenum, sintered	Mo, 99.8
Nickel	Mi, 99.9
Niobium	Nb, 99.8
Tantalum	Ta, 99.8
Titanium	Ti, 99.4; C, 0.3; O + N + Fe, 0.4
Vanadium	V, 99.8
Tungsten	W, 99.9
Zirconium	Zr, 99.0

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TABLE II - DISINTEGRATION OF SOLID MATERIALS IN MOLTEN EUTECTIC BISMUTH-LEAD MEDIA

	Material	Type of material	Specimen radius decrease (in.)	Evaluation period (min.)	Temperature (°F)	Estimated disintegration rate, (mil/year)	Type of attack	Results
Alloys	LT-2	Sintered	0.0005	1440	2000	1.825×10^{2}	Uniform	
	Mo-W	Forged, Hot Rolled	.0028	1440	2000	1.022×10^{3}	Pitting	
	Vitallium	Cast	.0040	1440	1500	1.460×10^{3}	Pitting	
	L-605	Hot Rolled	.0188	1440	1500	6.862×10^{3}	Uniform	
	347	Cold Rolled	.0275	1440	1500	1.004×10^{4}	Uniform	
	316	Cold Rolled	.0296	1440	1500	1.080×10^{4}	Uniform	
	S-816	Hot Rolled	.0407	1440	1500	1.486×10^{4}	Pitting	
	Hastelloy B	Cold Rolled	.1371	1440	1500	5.004×10^{4}	Uniform	
	Inconel	Cold Rolled	.1875	60	2000	1.634×10^{6}	Cavitation	Complete Disintegration within 24 hours
Ceramals-Ceramics	Al_2O_3	Hot Pressed	0.0000	1440	2000	0.000	None	
	BeO	Hot Pressed	.0000	1440	2000	.000	None	
	B_4C	Hot Pressed	.0000	1440	2000	.000	None	
	B_4C-Fe	Sintered	.0000	1440	2000	.000	None	
	CrC	Hot Pressed	.0000	1440	2000	.000	None	
	K-138	Sintered	.0000	1440	2000	.000	None	
	K-138A	Sintered	.0000	1440	2000	.000	None	
	K-151A	Sintered	.0000	1440	2000	.000	None	
	MgO	Hot Pressed	.0000	1440	2000	.000	None	
	$MoSi_2$	Hot Pressed, Sintered	.0000	1440	2000	.000	None	
	NBS 4811C		.0000	1440	2000	.000	None	
	Quartz	Fused	.0000	1440	2000	.000	None	
	$SiC-B_4C$	Hot Pressed	.0000	1440	2000	.000	None	
	TiC	Hot Pressed	.0000	1440	2000	.000	None	
	ZrC-Nb	Hot Pressed	.0000	1440	2000	.000	None	
	ZrO_2	Hot Pressed	.0000	1440	2000	.000	None	
	Zircon	Hot Pressed	.0000	1440	2000	.000	None	
	ZrC	Hot Pressed	.0030	1440	2000	1.815×10^{5}	Pitting	
Metals-Elements	Graphite	Extruded	0.0000	1440	2000	0.000	None	
	Mo	Arc cast	.0000	1440	2000	0.000	None	
	W	Sintered	.0015	1440	2000	5.477×10^{2}	Uniform	
	Mo	Sintered	.0053	1440	2000	1.935×10^{3}	Uniform	
	Ta	Sintered	.0055	1440	2000	2.007×10^{3}	Pitting	
	Nb	Sintered	.0030	60	2000	2.628×10^{4}	Uniform	Complete Disintegration within 24 hours
	Cr	Cast	.2378	60	2000	2.066×10^{6}	Cavitation	Complete Disintegration within 24 hours
	Fe	Ingot	.1036[a]	30	2000	1.815×10^{6}	Cavitation	Complete Disintegration within 1 hour
	Co	Cast	.2812	60	2000	2.464×10^{6}	Cavitation	
	Ti		>.3695	9	2000	$>2.156\times10^{7}$	Uniform	
	Ni	Wrought A	.2531	5	2000	2.661×10^{7}	Uniform	Complete Disintegration within 1 hour
	Zr	Iodide	.3425	5	2000	$>3.600\times10^{7}$	Uniform	Complete Disintegration within 5 minutes
	V	Ca reduced	.0488	60	2000	4.275×10^{7}	Uniform	

[a]Approximately

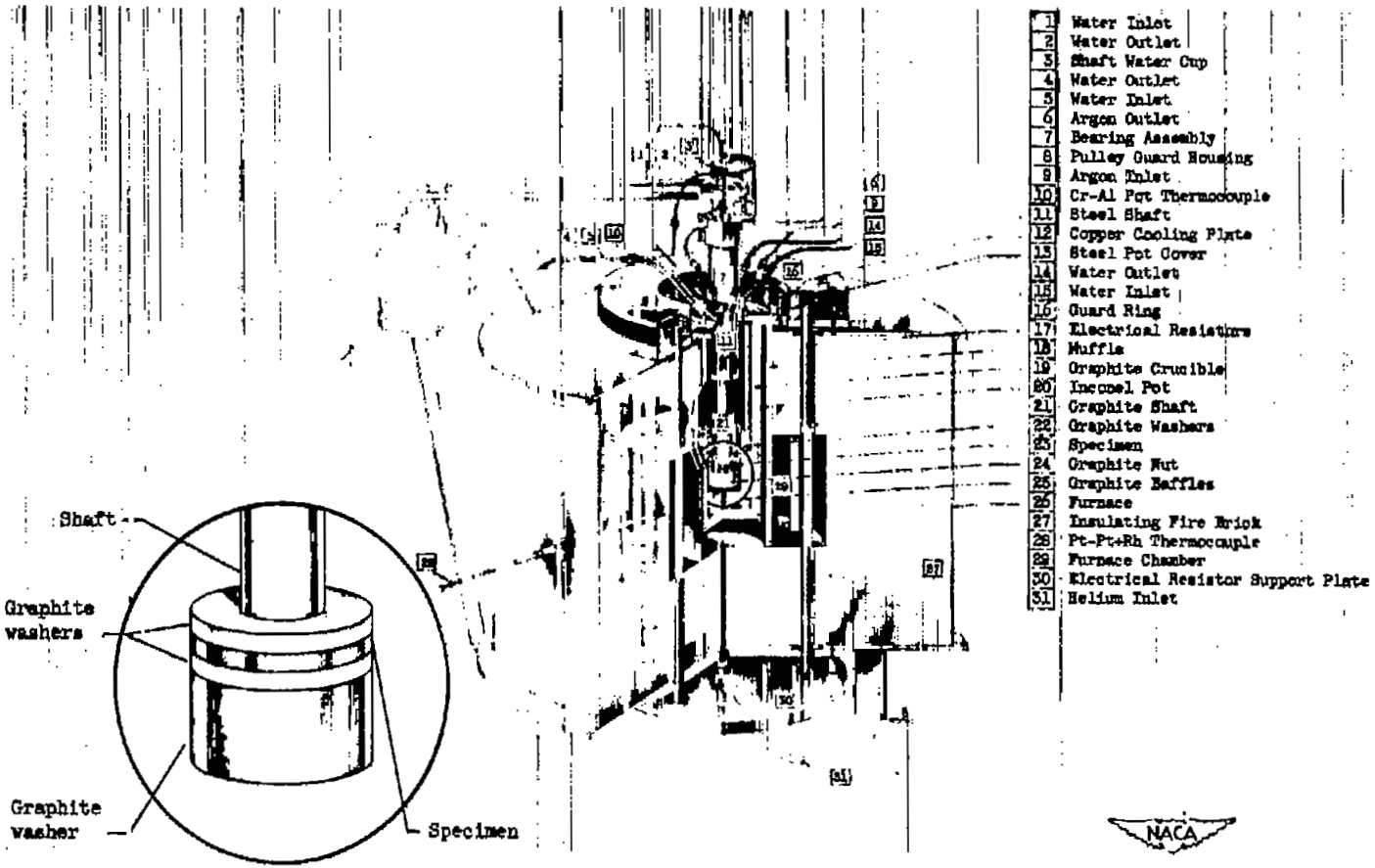

Figure 1. - Liquid-metals-attack evaluation equipment.

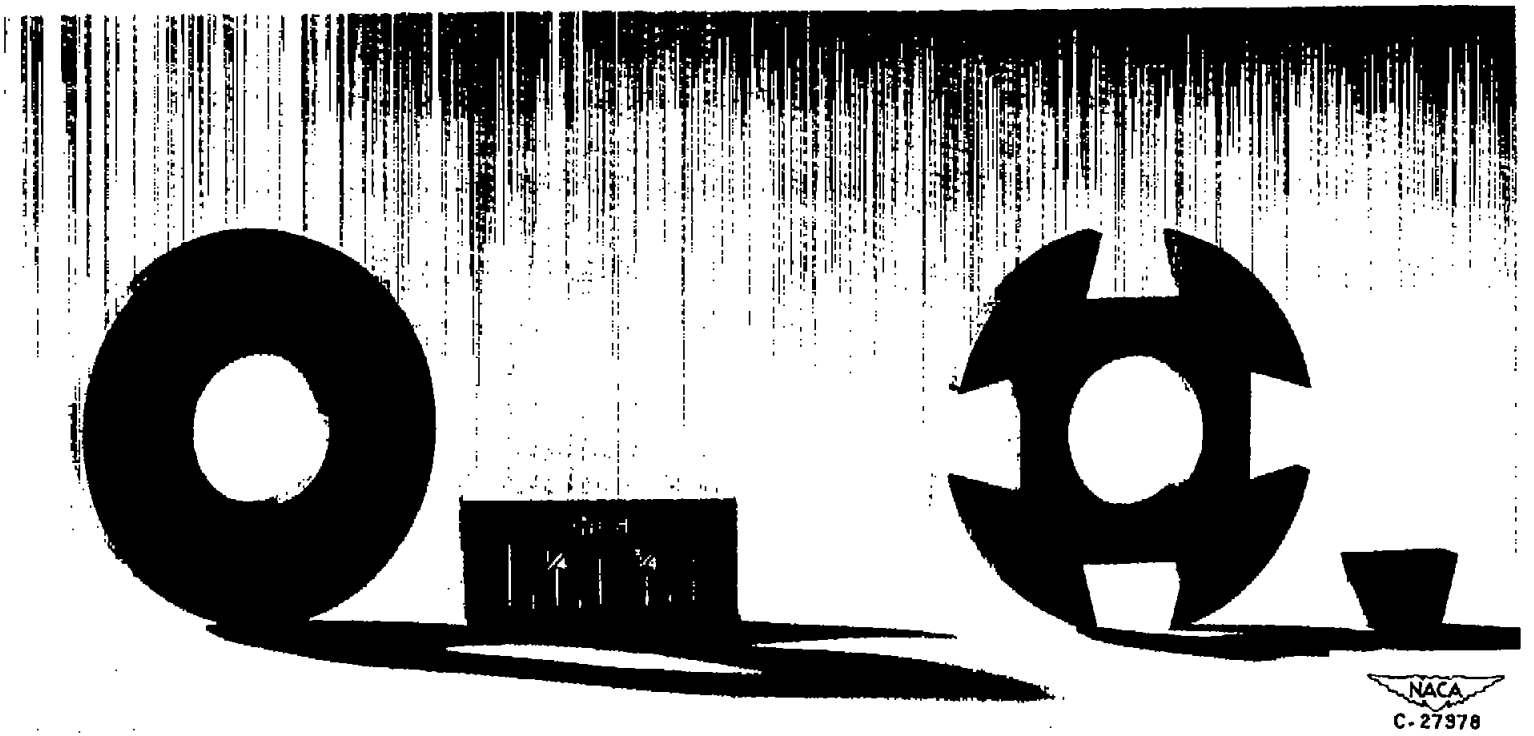

Figure 2. - Specimen ring.

Figure 3. - Slotted graphite holder and insert.

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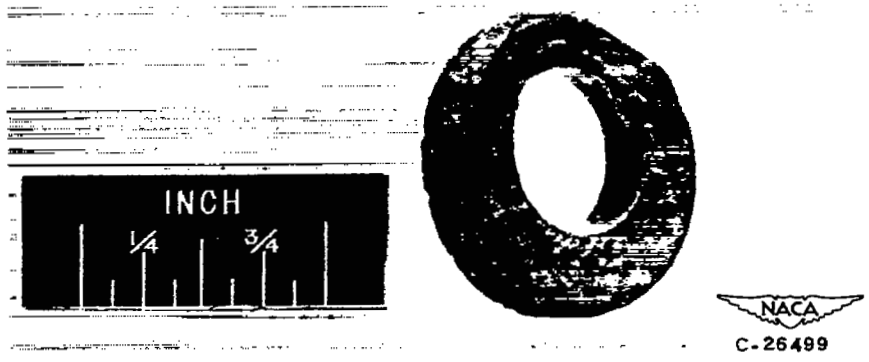

Figure 4. - Uniform attack (titanium, 9 min; 2000° F).

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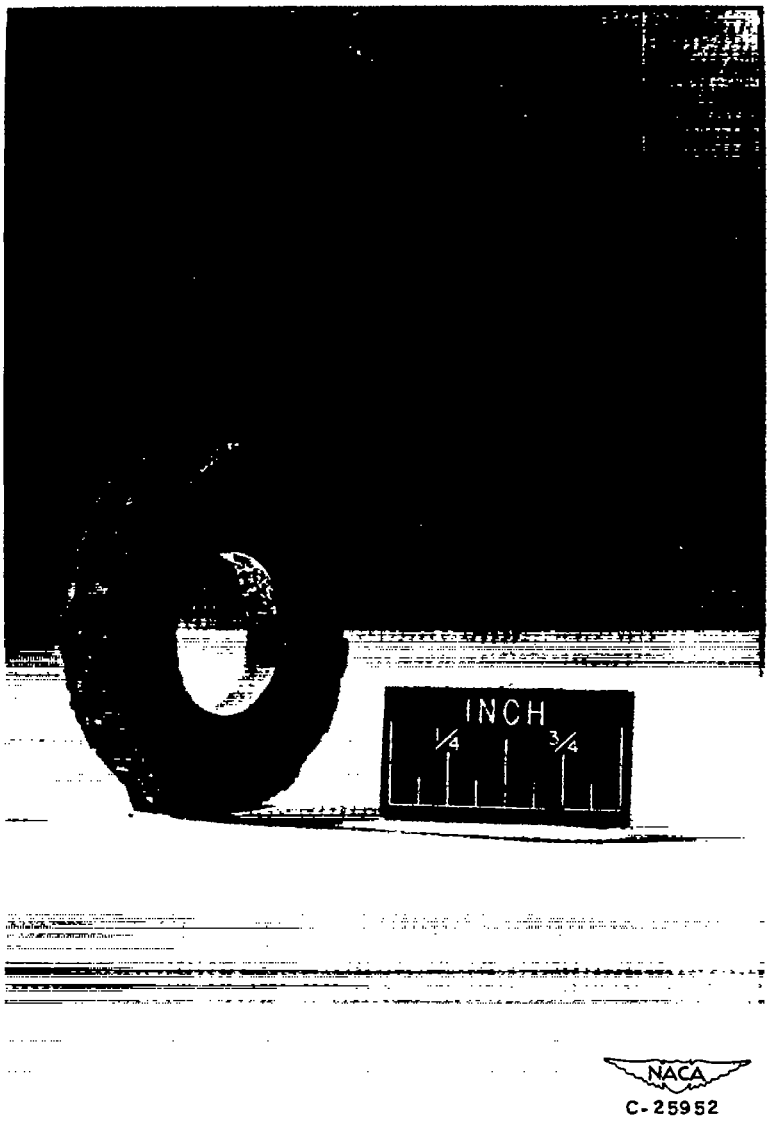

Figure 5. - Cavitation (chromium; 1 hr; 2000° F).

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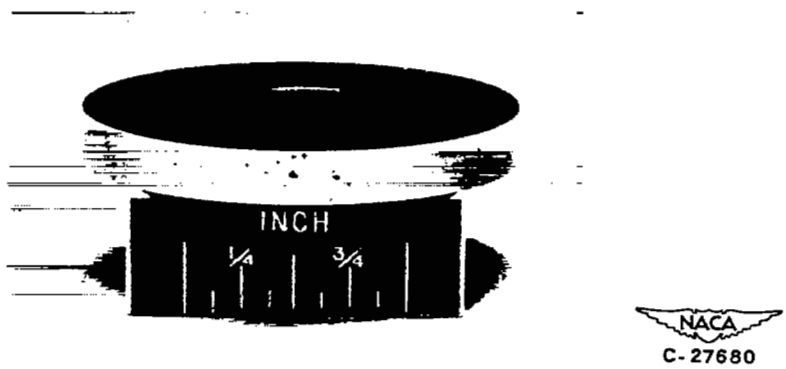

Figure 6. - Pitting attack (vitallium; 24 hr; 1500° F).

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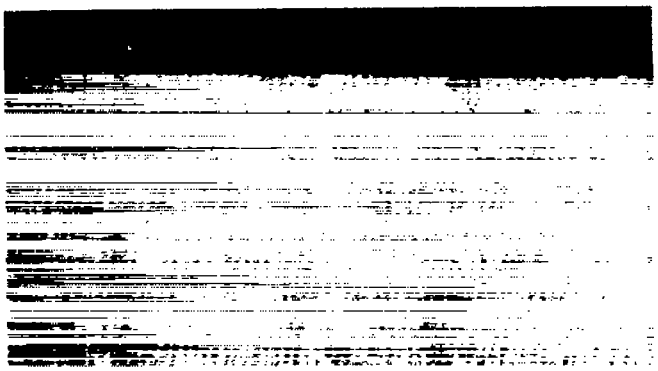

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Figure 7. - Nil effect of bismuth-lead eutectic for 24 hours at 2000° F on surface of arc cast molybdenum. X50; etchant, $KOH-K_3Fe(CN)_6$.

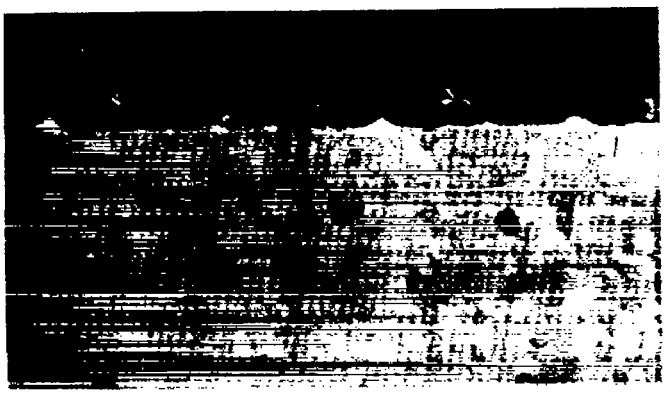

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Figure 8. - Destructive effect of bismuth-lead eutectic for 24 hours at 2000° F on surface of sintered molybdenum. X50; etchant, $KOH-K_3Fe(CN)_6$.