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MATERIALS FOR POTASSIUM LUBRICATED JOURNAL BEARINGS

Quarterly Progress Report No. 11

Quarter Ending January 22, 1966

EDITED BY R. G. FRANK

prepared for
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CONTRACT NAS 3-2534

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION

GENERAL  ELECTRIC

CINCINNATI, OHIO 45215

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Quarterly Progress Report No. 11

Covering the Period

October 22, 1965 to January 22, 1966

Edited by

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CINCINNATI, OHIO 45215

FOREWORD

The work described herein is being performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2534. Its purpose, as outlined in the contract, is (to evaluate materials suitable for potassium lubricated journal bearing and shaft combinations for use in space system turbogenerators) and, ultimately, to recommend those materials most appropriate for such employment.

R. G. Frank, Manager, Physical Metallurgy, Materials and Processes, is administering the program for the General Electric Company. L. B. Engel, Jr., T. F. Lyon, W. H. Hendrixson and B. L. Moor are directing the program investigations. The design for the friction and wear testers was executed by H. H. Ernst and B. L. Moor.

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I. INTRODUCTION

The program reviewed in this eleventh quarterly report, covering activities from October 22, 1965 to January 22, 1966, is performed under the sponsorship of the National Aeronautics and Space Administration. (Its purpose is to evaluate materials suitable for potassium lubricated journal bearing and shaft applications in space system turbogenerators operating over a 400° to 1600°F temperature range.) The critical role of bearings in such systems demands the maximum reliability attainable within today's state-of-the-art. Achieving this reliability requires an interdisciplinary approach employing the best mechanical designs of journal bearings combined with the selection of the optimum materials to serve as the structural members. Satisfying this latter requirement constitutes the aim of this program.

A number of investigators have conducted studies in this field and their contributions have advanced the state-of-the-art considerably (Section VII, Ref. 1). Although their work is significant, there are no common criteria for a comparison of the existing data. Therefore, establishing a unified approach to the development and evaluation of materials for potassium lubricated bearing application is deemed essential. The program involves a comprehensive investigation of material properties adjudged requisite to reliable journal bearing operation in the proposed environment. This includes: 1) corrosion testing of individual materials and potential bearing couples in potassium liquid and vapor, 2) determination of hot hardness, hot compressive strength, modulus of elasticity, thermal expansion and dimensional stability characteristics, 3) wetting tests by potassium and 4) friction and wear measurements of selected bearing couples in high vacuum and in liquid potassium.

In cooperation with the cognizant NASA Technical Manager, 14 candidate materials were selected (Table I) from a compilation of existing data on available materials. The materials reviewed fall into four broad categories:

- Superalloys and refractory alloys with and without surface treatment.
- Commercial metal bonded carbides.
- Refractory compounds such as stable oxides, carbides, borides and nitrides.
- Cermets based on the refractory metals and stable carbides.

Each material is procured from appropriate suppliers to mutually acceptable specifications and subsequently is subject to chemical, physical and metallurgical analyses to document its characteristics before utilization in the program. After the documentation of processes and properties, the candidate materials undergo corrosion, dimensional stability, thermal expansion, compression and hot hardness testing. Considering the bearing material

TABLE I. CANDIDATE BEARING MATERIALS

<u>Material Class</u>	<u>Candidate Material</u>	<u>Nominal Composition</u>
A. Nonrefractory Metals and Alloys	Star J	17%W-32%Cr-2.5%Ni-3%Fe-2.5%C-Bal. Co
B. Refractory Metals and Alloys	Mo-TZM (Arc Cast; Stress-Relieved)	0.5%Ti-0.08%Zr-0.02%C-Bal. Mo
	Tungsten (Arc Cast; Stress-Relieved)	99.96%W (Min.)
C. Fe-Ni-Co Bonded Carbides	Carboloy 907	74%WC-20%TaC-6%Co
	Carboloy 999	97%WC-3%Co
	K601	84.5%W-10%Ta-5.5%C
D. Refractory Compounds - Oxides, Carbides, Borides	Lucalox	99.8%Al ₂ O ₃ (Min.)-0.1%MgO-0.02%SiO ₂ -0.02%CaO-0.02%Fe ₂ O ₃
	Zircoa 1027	95.5%ZrO ₂ -Bal. Proprietary
	Titanium Carbide	94%TiC-4.25%WC-0.9%Ni-0.1%Fe-0.68%Co
	Titanium Diboride	98%TiB ₂ -0.39%Fe-0.30%C
E. Refractory Metal Bonded Carbided	TiC+5%W	90%TiC-4.79%WC-5%W-0.36%Fe
	TiC+10%Mo	85.4%TiC-10.5%Mo-3.99%WC-0.13%Fe
	TiC+10%Cb	83.6%TiC-9.54%Cb-5.85%WC-0.73%Co-0.33%Fe
	Grade 7178	85.6%W-6.9%Mo-1.8%Cb-0.3%Ti-5.7%C

requirements and the information obtained on the candidate bearing materials which were subject to both potassium and non-potassium testing, seven materials combinations listed below were selected in cooperation with the NASA Technical Manager. Potassium corrosion and wetting tests and friction and wear measurements in high vacuum and liquid potassium have proceeded with these combinations.

<u>Rotating Disc</u>		<u>Stationary Rider</u>
*1.	Grade 7178	Mo-TZM
2.	Mo-TZM	Grade 7178
*3.	Grade 7178	Grade 7178
4.	Carboloy 907	Mo-TZM
5.	Carboloy 907	Carboloy 907
6.	TiC+10%Cb	Mo-TZM
*7.	TiC+10%Cb	TiC+10%Cb

Those materials combinations marked with an asterick (*) were selected for friction and wear testing in both liquid potassium and high vacuum. Where significant differences in hardness exist, the softer material, i.e., Mo-TZM alloy, was selected as the rider material (stationary specimen) to facilitate wear-in during testing in liquid potassium. Couple No. 2 was selected to determine what affect a hard rider material would have on the wear pattern of a soft disc material in comparison with the reverse combination.

The decision to place considerable emphasis on the refractory metal bonded carbides was based on their excellent stability at the higher temperatures. Also, it was elected to test the hard carbide materials against themselves in order to obtain a direct comparison of the friction and wear behavior of hard/hard combinations vs hard/soft combinations where Mo-TZM alloy is one material in a pair. From investigations conducted by Coffin^(2,3), it was concluded that generally it is desirable to have one of the materials harder than the other in order to facilitate wear-in of the couple. If both materials are hard and brittle, the surface asperities on the weaker material can fracture and the resultant debris could cause severe surface damage by abrasion. However, recent friction and wear tests and full scale bearing tests conducted elsewhere⁽⁴⁾, using liquid potassium as a lubricant, have indicated superior performance of hard/hard combinations over hard/soft combinations because of the tendency of wear debris from the hard material to become imbedded in the soft material of hard/soft combinations and possibly resulting in a cutting action.

The ultimate product of this program will be a recommendation, substantiated with complete documentation, of the material or materials which have the greatest potential for use in alkali metal journal bearings in high speed, high temperature rotating machinery for space applications. Hopefully, the results will indicate the future course of alloy or material development specifically designed for alkali metal lubricated journal bearing and shaft combinations.

II. SUMMARY

During the eleventh quarter of the program, the topics abstracted below were covered and the results are interpretively presented in this report.

Evaluation of the compatibility of candidate bearing material combinations in a Cb-1Zr alloy/potassium system has been completed. Three material combinations, Mo-TZM/Carboloy 907, Mo-TZM/Grade 7178, and Mo-TZM/TiC+10%Cb contained in Cb-1Zr alloy capsules with high-purity potassium, had been tested isothermally for 1,000 hours at 800°, 1200°, and 1600°F. Metallographic examination of the TiC+10%Cb and Grade 7178 specimens which were tested at all three temperatures and Carboloy 907 specimens tested at 800°F revealed no surface reactions or evidence of attack. Examination of the Carboloy 907 specimens exposed to potassium liquid at 1200° and 1600°F revealed surface reactions to a depth of 0.00047 inch and 0.0006 inch, respectively, on the surfaces adjacent to the Mo-TZM alloy surface.

The X-ray diffraction analyses of the facing surfaces of the candidate bearing material combinations (0.020-inch separation) tested at 1600°F revealed major phase changes in the Carboloy 907, Grade 7178, and Mo-TZM alloy specimens. The dissociation of WC to tungsten on the surface of the two carbides and the formation of Mo₂C on the surface of the Mo-TZM alloy were observed. The formation of the complex carbides Co₂WC₄ and (Co,W)_xC also were observed on the surface of the Carboloy 907 tested at 1600°F. The presence of the Mo₂C layer on the surface of the Mo-TZM alloy was observed metallographically on the specimens tested with Carboloy 907 and Grade 7178 at 1600°F and with Carboloy 907 tested at 1200°F.

Spectrographic analyses of the potassium drained from the capsules containing the TiC+10%Cb specimens that were tested at 1600°F revealed a slight increase in titanium and aluminum content.

Microhardness surveys of all the corrosion test specimens that were tested individually or in combination with Mo-TZM alloy in potassium liquid and vapor for 1,000 hours at 800°, 1200°, and 1600°F have been completed. A change in hardness was observed only in the Zircoa 1027 specimens (decrease).

The elevated temperature, vacuum compression test facility has been assembled, checked-out and testing procedures established. From tests conducted with an instrumented specimen, the effective gauge length of the test specimen was determined to be equal to the measured gauge length plus 1.44X the wall thickness.

Potassium wetting tests were completed on Carboloy 907, Grade 7178, TiC+10%Cb and Mo-TZM alloy. Of the four materials investigated, the Mo-TZM alloy exhibited the best wetting characteristics with receding contact angles of essentially zero degrees. Carboloy 907 had the highest receding contact angle, i.e., 22°.

Friction and wear tests were conducted in high vacuum for Carboloy 907 vs Carboloy 907, Mo-TZM vs TiC+10%Cb, Mo-TZM vs Carboloy 907, Mo-TZM vs Grade 7178, and Grade 7178 vs Grade 7178. The tests were conducted at room temperature, at speeds of 800 SFM and at pressures in the 10^{-9} torr range. Values for the coefficient of friction ranged from 0.40 to 1.07.

Rotating tests in both the high vacuum and liquid potassium friction and wear testers have shown that some form of lubrication is required in the shaft bearings for sustained operation. Commercial MRC 7207 angular contact bearings with MoS_2 coated retainers have been selected and used successfully in the high vacuum friction tester; ITI 8089 special angular contact bearings with $\text{BaF}_2/\text{CaF}_2$ coated retainers have been selected for trial in the liquid potassium friction tester.

High speed water flow tests, conducted with the pump for the potassium friction and wear tester, showed that equal flow of about 43 cc/min could be achieved over the upper and lower test specimens at a speed of 5,000 SFM by restricting the area of the flow orifice to the lower specimen to 0.0008 sq in.

III. PROCUREMENT OF CANDIDATE BEARING MATERIALS COMBINATIONS FOR FRICTION AND WEAR TESTING

The delivery status of the test specimens required for friction and wear testing of selected candidate journal bearing material combinations is given in Table II. All the Mo-TZM alloy and Carboloy 907 specimens were on hand at the end of the reporting period and the Grade 7178 and TiC+10%Cb specimens were received as noted in Table II.

TABLE II. PROCUREMENT STATUS OF CANDIDATE BEARING MATERIALS TEST SPECIMENS

Material	Spec.	Vendors	Test Specimen Identity				MCN Series
			Qty.	Disc	Rider		
					Promise/ Delivery Date	Qty. Date	
Mo-TZM (Raw Stock)	SPPS-15	American Metal Climax	10-each		3-25-65	0.250" ϕ	1084
			0.187"			x 36"	1085
			thk. x				1086
			4.375"				
			x 4.375"				
Mo-TZM (Machining)	--	Dawson Carbide	10		5-3-65	74	1037
Carboloy 907	SPPS-23T	GE-MPD	17(a)		5-3-65	18	1036
			15(h)				
Grade 7178	SPPS-36T	Kennametal, Inc.	11		7-27-65	56(b)	1046
			19(c)		11-10-65	54(h)	
			15(c)		11-17-65		
			5		12-7-65		
			14		1-11-66		
			6		2-6-66		
			36(h)				
TiC+10%Cb	SPPS-35T	Kennametal, Inc.	11(d)		7-27-65	26(e)	1045
			16(f)		8-25-65	4	
			4		12-7-65	26(h)	
			18(g)		3-17-66		
			6		5-1-66		
			34(h)				

- (e) Four specimens rejected for surface defects.
 (f) Twelve specimens rejected for surface defects.
 (g) Three specimens rejected for surface defects.
 (h) Total specimens accepted.
- (a) Two specimens rejected for surface defects.
 (b) Two specimens rejected for surface defects.
 (c) Rejected for surface defects.
 (d) Six specimens rejected for surface defects.

IV. TEST FACILITIES

A. FRICTION AND WEAR IN HIGH VACUUM

1. Temperature Calibration of Test Specimens

In preparation for conducting friction and wear tests in high vacuum at temperatures of 400°F and higher, it was necessary to correlate the temperatures measured by the chromel-alumel thermocouples located in the thermowells, which are in contact with the rider specimens in the loading arms, with the true test temperature. Excessive heat losses through the Mo-TZM alloy loading arm and the tantalum coated stainless steel thermowells prevented direct reading of the specimen temperatures. The calibration system which was derived compares the temperature of a Mo-TZM alloy reference block with the temperature of the disc specimens and the rider specimens, Figure 1. The temperature of the disc specimen is considered the test temperature and was measured with Pt vs Pt+10%Rh thermocouples; the beaded ends of the thermocouples were clamped between the test specimens and the specimen holder washers. Because of the problems of transmitting the signals from the disc specimen thermocouples during rotation, these thermocouples are not present during friction and wear tests, nor was the shaft rotated during the calibration tests. The temperature of the Mo-TZM alloy reference block was measured with two beaded Pt vs Pt+10%Rh thermocouples. The thermocouples were inserted in 1/2-inch deep holes at opposite ends of the reference block.

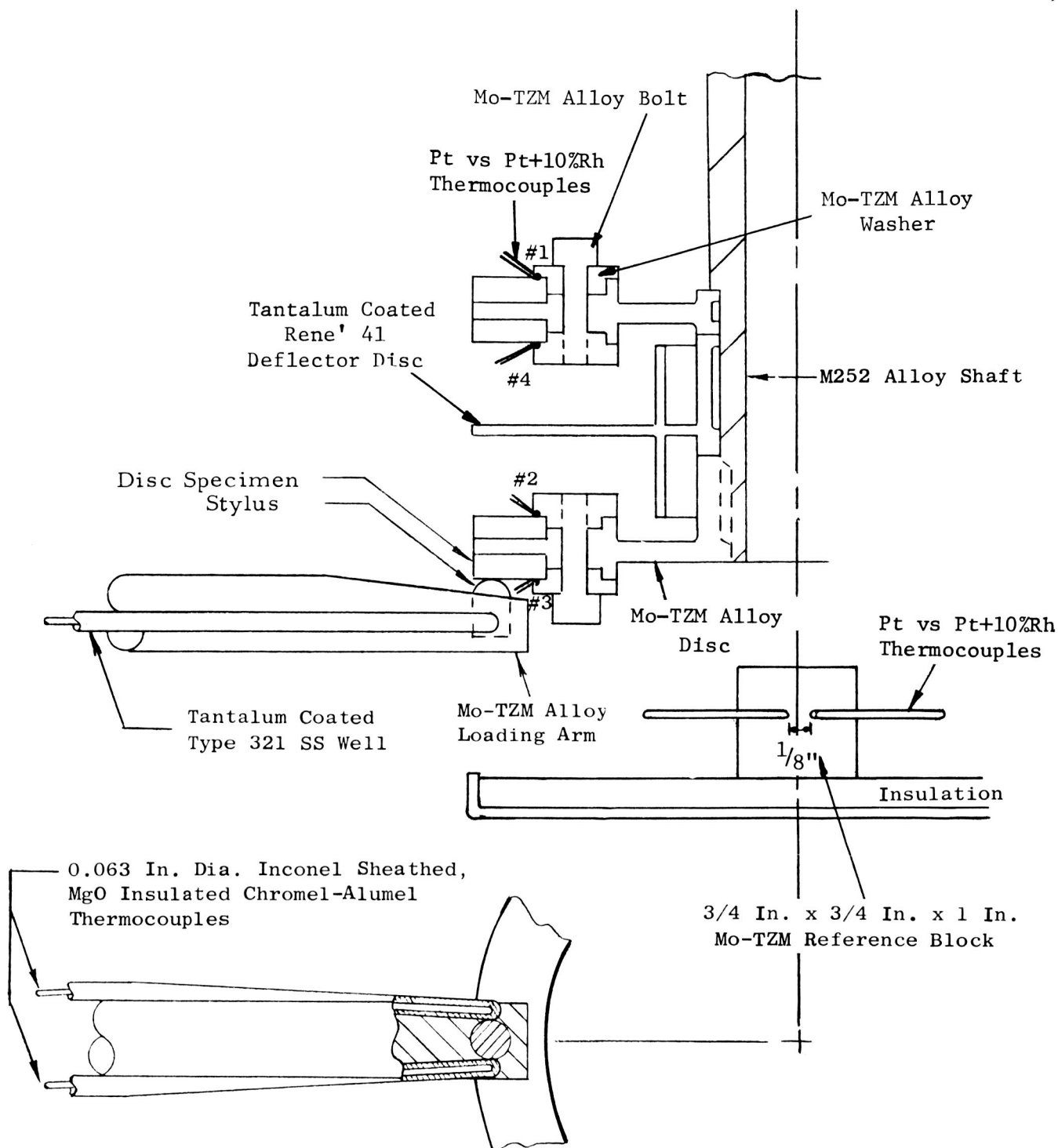
The specimen pairs used for the calibration tests were chosen to mate those of relatively high, medium, and low thermal conductivity in several different combinations and locations. No detectable differences in results were observed with respect to differences in thermal conductivity. However, the results were affected by specimen location and are so reported. The temperature differences due to specimen location in the tester are related to the geometry and temperature of the surfaces to which the specimens radiate heat.

The raw temperature data were plotted in the form $(T_{\text{block}} - T_{\text{disc}})$ vs $\left(\frac{\Delta T_{\text{disc}}}{\Delta \text{time}}\right)$ and, as would be expected, generally showed strong dependence of the temperature differential upon the rate of change of disc temperature. The $(T_{\text{block}} - T_{\text{disc}})$ temperatures were read at zero rate of change for purposes of calibration and are plotted against T_{disc} , as shown in Figure 2. The data are summarized in Table III. The arm thermocouple readings were correlated with T_{disc} and the results are plotted in Figure 3.

The reference block will be left permanently in the tester as the primary specimen temperature control device.

2. Evaluation of Ball Bearings for High Speed

The High Vacuum Friction and Wear Tester (HVFT) was loaded with specimens in preparation for the first room temperature, 5,000 SFM friction tests.



B1088-1

Figure 1. Location of Thermocouples in High Vacuum Friction and Wear Tester For Temperature Calibration of Reference Block.

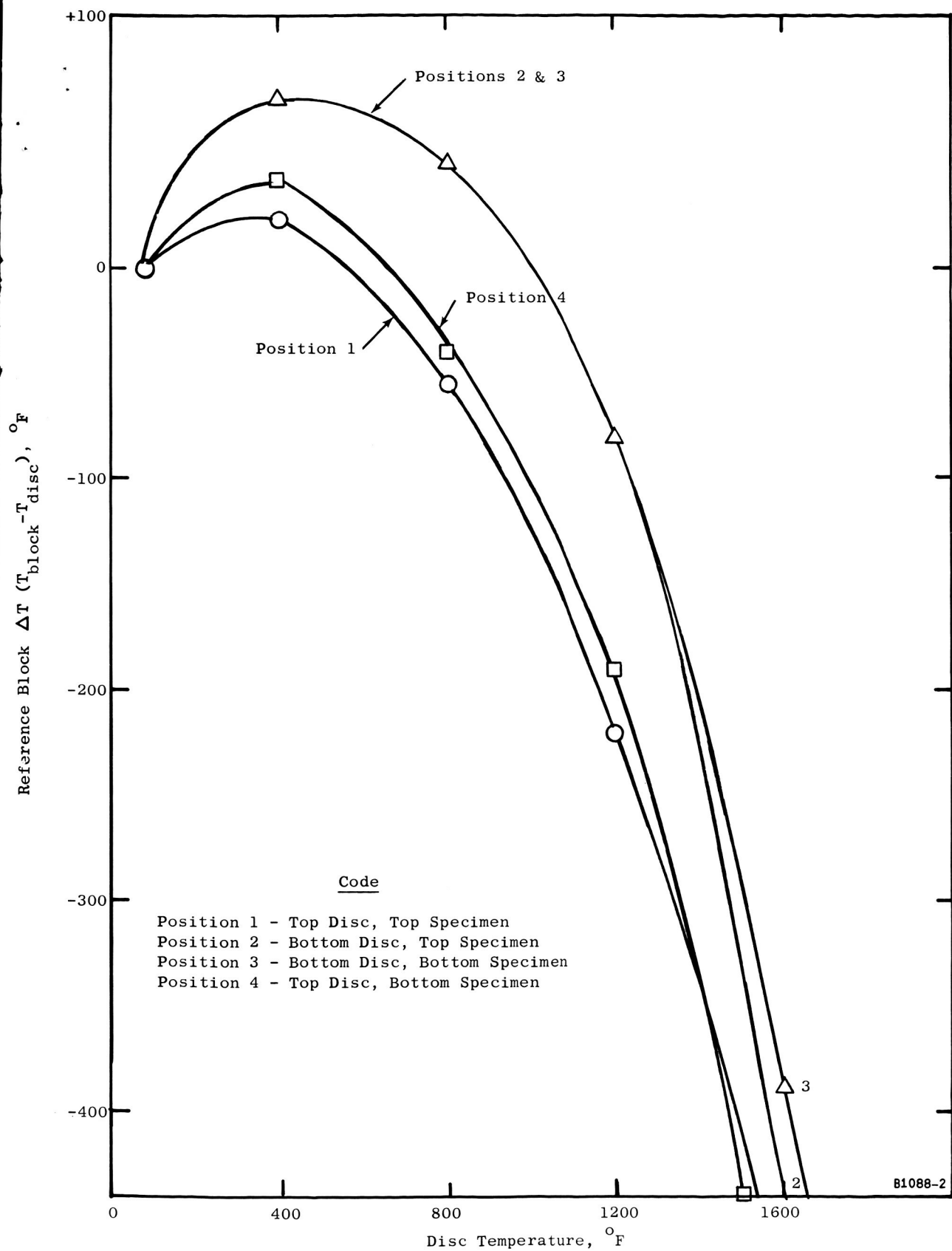


Figure 2. Reference Block ΔT Required to Obtain Disc Temperature.

TABLE III. TEST TEMPERATURE CALIBRATION FOR HIGH VACUUM FRICTION AND WEAR TESTS

Test Position	Reference Block Temperature			
	Test Temperature			
	400°F	800°F	1200°F	1600°F
Arm 1	376	856	1420	2092
Arm 2	320	750	1280	2040
Arm 3	320	750	1280	1988
Arm 4	359	840	1390	2130

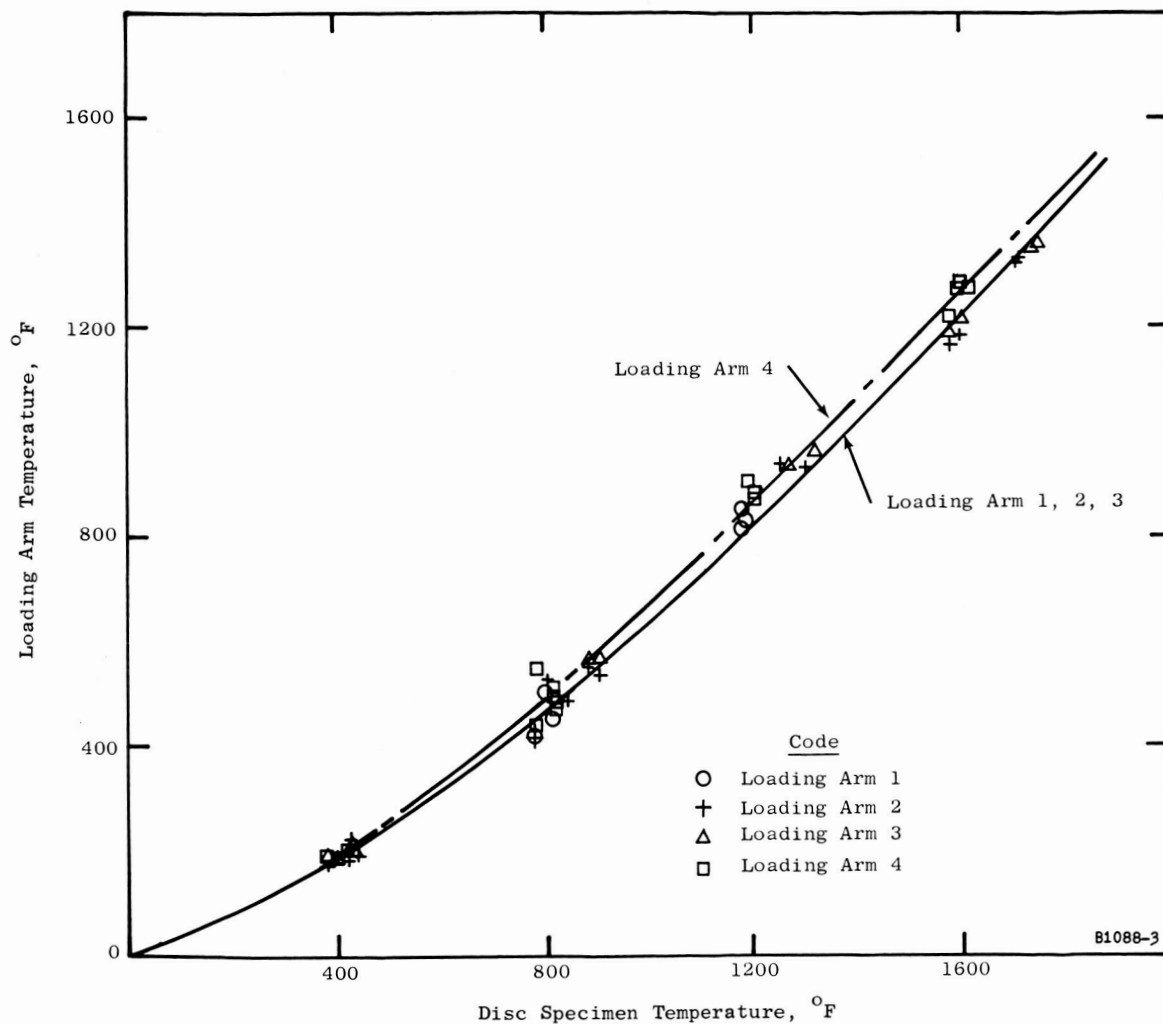


Figure 3. Comparison of Loading Arm Thermocouple Temperature vs Disc Specimen Temperature (Specimens in Contact).

The commercial angular contact bearings were replaced with the special ITI 8158 gold- and silver-plated bearings designed for high speed, high vacuum operation. The bearings failed without warning as the speed was being increased to the test speed. The operating conditions of this test are listed in Table IV, Test #2. Failure of these bearings could have occurred for a number of reasons, including improper assembly, poor housing geometry, improper axial play, insufficient clearance between the retainer and the inner race lands, and improper material selection. A program of investigation, using commercially-available bearings, was initiated to identify a bearing design suitable for high speed operation in high vacuum. All of the bearings that were tested were degreased, ultrasonically cleaned, and rinsed with acetone and/or alcohol before installation into the friction and wear test rig. The detailed conditions of the tests that were conducted are listed in Table IV; the main test variable and the test results are summarized below:

<u>Test No.</u>	<u>Bearing Identification</u>	<u>Test Variable/Objective</u>	<u>Results</u>
3	MRC 7207 (Angular Contact)	To determine whether commercial 7207 bearings would have sustained life at 4,780 RPM.	Bearings seized immediately.
4	MRC 7207 (Angular Contact)	Lower bearing outer face preloaded $\sim$ 23 lbs.	No improvement - bearings seized immediately.
5	MRC 7207 Upper SKF 207 Lower (Radial)	Changed lower bearing from angular contact to radial bearing.	No improvement - balls in radial bearing wedged in retainer.
6	MRC 7207 (Angular Contact)	Repeat of test 4 with bearings that operated successfully at 500 RPM for $\sim$ 50 hours.	No improvement - retainer failed.
7	Fafnir PM 207K (Radial)	Machined copper-bearing retainer.	No improvement - bearings seized.
8	ITI 8158 (Angular Contact)	Lower bearing outer face preloaded to $\sim$ 86 lbs.	No improvement - bearings seized.
9	Fafnir PM 207K (Radial)	Bearings lubricated with Lubriplate 130A to prove that bearing housing and shaft geometry and assembly procedures are satisfactory.	No failure - perfect operation.
10	Fafnir PM 207K (Radial)	Bearings lubricated with W Se ₂ powder.	Bearings seized

TABLE IV. SUMMARY OF BALL BEARING TESTS FOR 5000 SFM OPERATION IN
HIGH VACUUM FRICTION AND WEAR TESTER

Test No.	Bearing Identification	Ball and Race Material	Retainer Type & Material	Lubrication	Environment	Speed RPM	Thrust Load, lbs	Time, Hrs	Lower Bearing Outer Race Preload, lbs	Cause of Failure/Remarks
0	MRC 7207 (35 mm bore, angular contact)	52100	Pressed, low carbon steel	MoS ₂ dry powder	10 ⁻⁹ torr RT - 350°F	4800	80	~6	Axial play geometry controlled	No failure
1	MRC 7207	52100	Pressed, low carbon steel	None	10 ⁻¹⁰ torr RT - 350°F	4800	80	0.50 at low spd ~1 at 4800	Axial play geometry controlled	No failure - Removed to install ITI 8158 for high speed tests.
2	ITI 8158	M50 races Au-plated	Machined, Circle C, Ag Plated	None	10 ⁻⁹ torr RT - 350°F	2500	80	0.02	Axial play geometry controlled	Bearings seized due to galling of silver on retainer with gold on inner race lands.
3	MRC 7207	52100	Pressed, low carbon steel	None	Air RT	4800	80	0.05	Axial play geometry controlled	Bearings seized due to retainer deforming and wedging into outer race ball groove.
4	MRC 7207	52100	Pressed, low carbon steel	None	Air RT	4000	80	0.05	~23	Bore of bearing housing was increased in diameter to insure thrust washer loading of lower bearing. Seizure same as test 3.
5	Upper MRC 7207 Lower SKF 207 (radial)	52100	Pressed, low carbon steel	None	Air RT	4800	80	0.05	~23	Bearings so noisy that seizure appeared imminent. Balls in radial bearing wedged in retainer.
6	MRC 7207 (same as test 1)	52100	Pressed, low carbon steel	MoS ₂ dry powder	Air RT	4800	80	0.25	~23	Bearings so noisy that seizure appeared imminent. Retainer wedged into outer race ball groove.
7	Fafnir PM 207K (radial)	52100	Machined, yellow brass (a)	None	Air RT	4800	80	0.08	~23	Bearings seized due to wedging of brass flakes between balls and retainer.
8	ITI 8158	M50 races Au-plated	Machined, Circle C, Ag plated	None	Air RT	4800	80	0	~86	Bearings seized due to galling of silver on retainer with gold on inner race lands.
9	Fafnir PM 207K	52100	Machined, yellow brass (a)	Lubriplate 130 A	Air RT	4800	80	1.17	~86	No failure, perfect operation. This test and housing inspections eliminate geometry and assembly procedure as causes of failures. Lubrication is the problem.
10	Fafnir PM 207K	52100	Machined yellow brass (a)	WS ₂ dry powder	Air RT	4800	80	0.67	~86	Bearings seized due to galling of balls with retainer and retainer with inner race.
11	ITI 8158 (modified retainer 1)	M50 races Au-plated	Machined, Circle C, Ag plated	WS ₂ dry powder	Air RT	4800	80	1.17	~86	Silver removed from retainer I.D. Bearings so noisy that seizure appeared imminent. Balls fretted against retainer and retainer fretted against inner race.
12	N.D. V3207AH L85 (radial)	52100	Machined, riveted, bronze Ag plated	None	Air RT	5000	80	1.17	~86	Bearings so noisy that seizure appeared imminent. Could barely turn by hand after removal.
13	ITI 8158 (+.024 retainer) races Au-plated	M50 races Au-plated	Machined, Circle C, Ag plated	None	Air RT	780	80	6	~46	This bearing was upper bearing for KFT bearing Test 7. Noise level was very high after first 20 min and gold plating was worn through in ball grooves and lands.
14	MRC 7207 (MoS ₂ ret)	52100	Pressed, low carbon steel	MoS ₂ baked on retainers	10 ⁻⁷ torr RT	5000	80	4 +	~86	After 4 hr successful operation, bearings seized several times after about 20 min. at 5000 RPM, subsequently bearing operated successfully at 780 RPM. To be used for tests of Assembly X.

(a) For evaluation in air only; not to be tested in vacuum.

<u>Test No.</u>	<u>Bearing Identification</u>	<u>Test Variable/Objective</u>	<u>Results</u>
11	ITI 8158 (Angular Contact)	Silver removed from ID of retainer to provide Circle C vs gold surfaces in contact.	Some improvement - fretting occurred between retainer and inner race.
12	ND V3207AH LR5 (Radial)	Machined Ag plated bronze retainer.	Some improvement - seizure imminent after 1.17 hours.
13	ITI 8158 (Angular Contact)	Clearance between retainer and inner race increased to 0.012 inch.	Tested at low speed only (780 RPM) - Au wore through on ball grooves in races.
14	MRC 7207 (Angular Contact)	MoS ₂ coating applied to retainer and baked at 450°F.	Operated successfully at 780 RPM; seized intermittently at 5,000 RPM.

The above tests show that the tester design is satisfactory for high speed operation provided that the shaft bearings are lubricated. Therefore, a decision was made to install new MRC 7207 angular contact bearings with MoS₂ coated retainers in the high vacuum test rig, proceed with the low speed (780 RPM) testing and repeat the high speed test.

Ten sets of MRC 7207 bearings with DF-700 (molybdenum disulfide) baked on the retainers were received. One set was installed in the high vacuum friction and wear tester and operated successfully for 30 minutes at 780 RPM and 60 minutes at 4,780 RPM. The noise and vibration level was very low, and the systems could be evacuated into the 10⁻⁹ torr range. However, bearing seizure occurred several times during the high speed run. This test and others led to the conclusion that the full life of this type of bearing is barely over one hour at 4,780 RPM. Since the bearings will operate again after seizing at the high speeds if they are allowed to cool, and since they operate satisfactorily at lower speeds, a contributing factor to the failure may be the closing of internal bearing clearance due to differential thermal expansion of the bearing components. Undoubtedly, wearing of the MoS₂ coating also is greatly accelerated at the higher speed.

B. FRICTION AND WEAR IN LIQUID POTASSIUM

1. Low Speed Impeller Pump Tests

Because several changes were made in the water collection device to improve the accuracy of the water flow rate measurements, the previously reported

low speed water flow tests were repeated to determine the validity of the data. The data obtained from the second set of tests, Figure 4, show very little deviation from the previous results. However, it was found that a short run was required at a slightly higher speed than 778 RPM in order to achieve full loading of the impeller. This appeared to be more of a necessity at high liquid levels than at lower liquid levels.

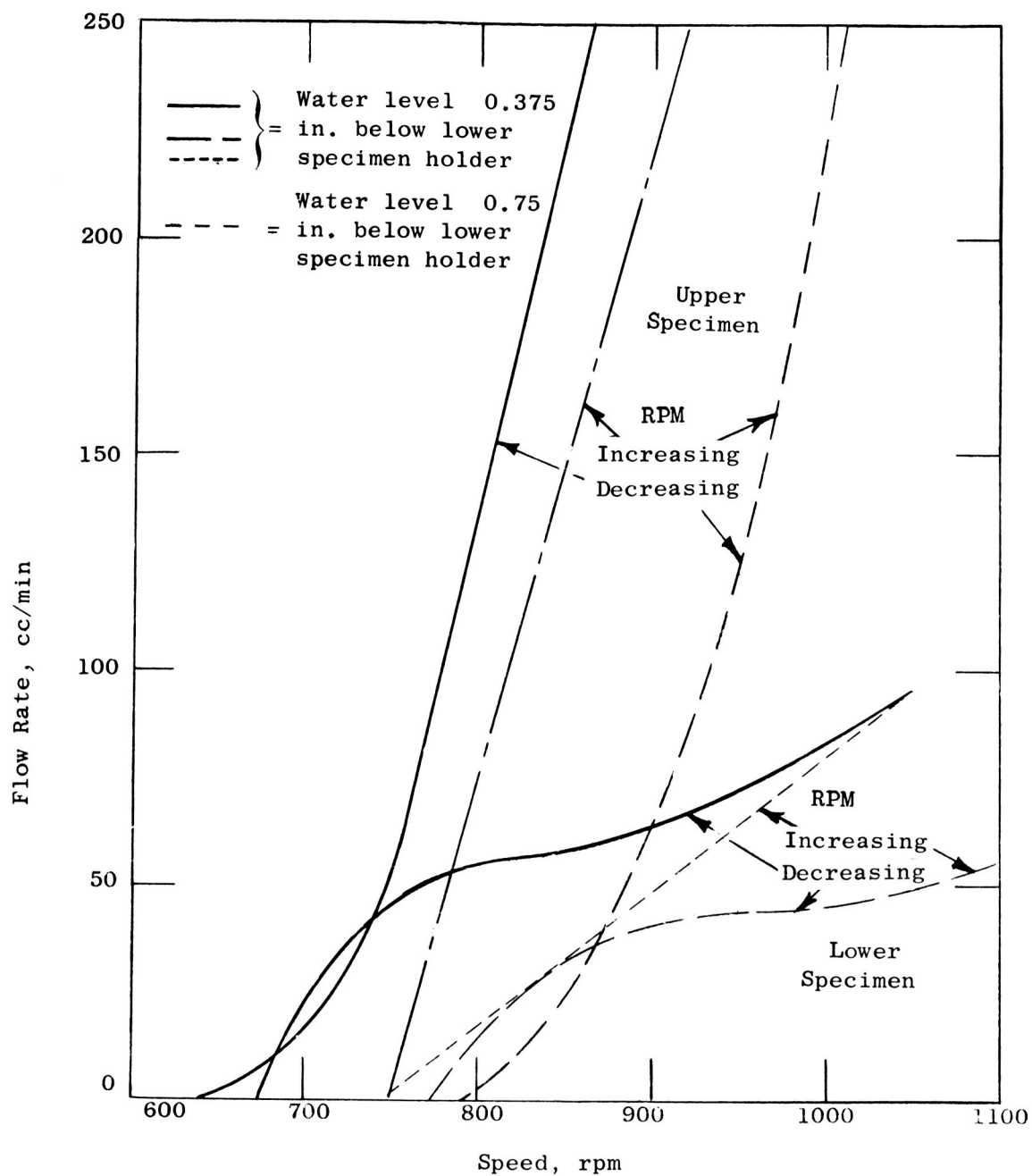
2. High Speed Impeller Pump Tests

Water flow tests were concluded with the high speed impeller. These tests showed that the achievement of balanced liquid flow across the top and bottom specimens is strongly dependent upon the flow area of the lower specimen orifices. By continuously decreasing the diameter of the 0.089-inch diameter ports which supply liquid to the channel adjacent to the inside diameter of the disc specimens, the flow of liquid over the lower specimen decreased and the flow over the upper specimens increased, Figure 5. A flow area of about 0.0008 in² was chosen to give an equal flow of about 43 cc/min across each specimen at a speed of 5,000 SFM which is approximately equivalent to the expected balanced flow at 800 SFM. The above flow area could be achieved by one hole of 0.032-inch diameter or four holes of 0.016-inch diameter. After the fabrication of a new specimen hold-down ring which achieves the necessary blockage of the lower supply ports, the flow rate will be rechecked.

3. Evaluation of Ball Bearings for High Speed Tests

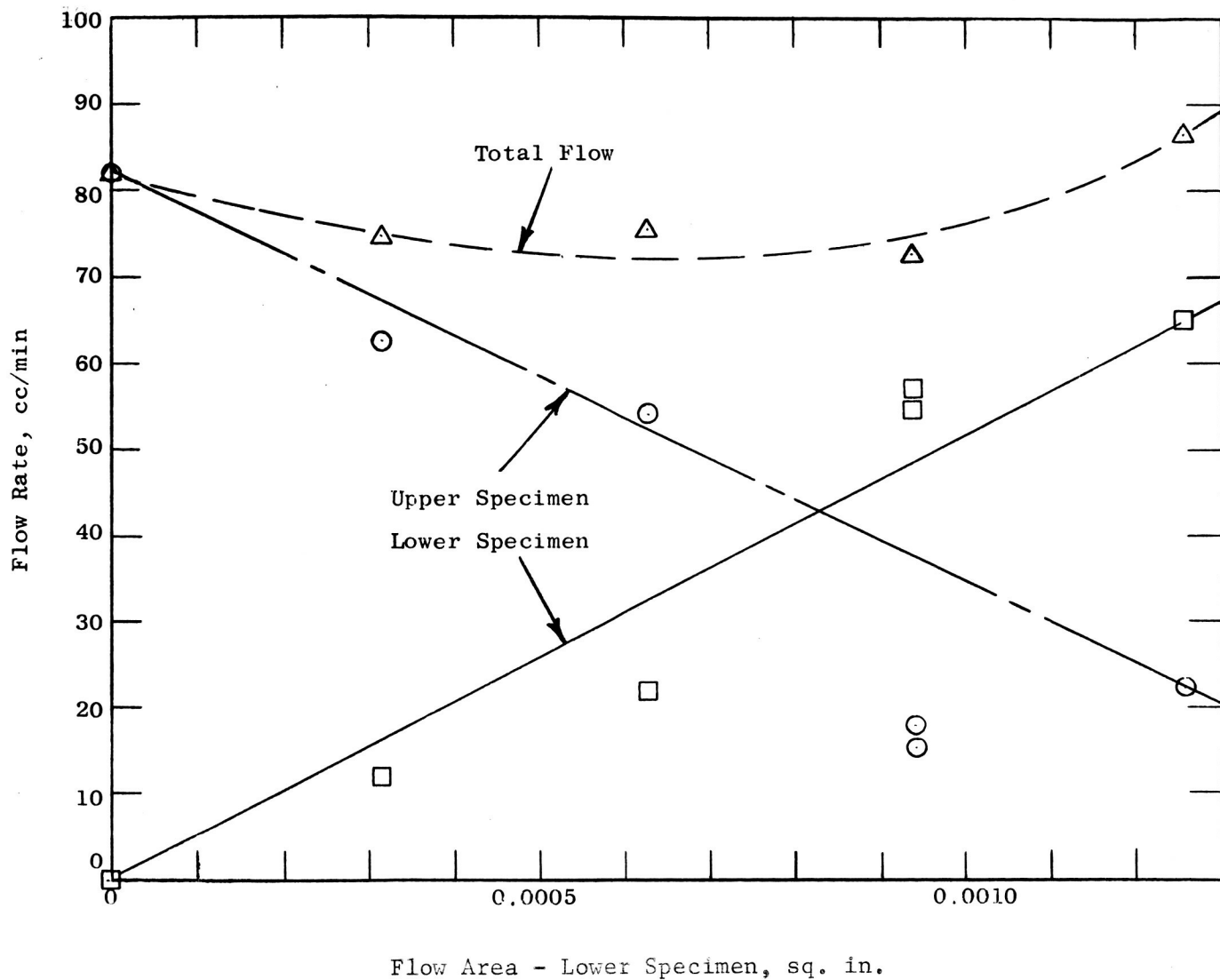
The lubricated commercial MRC 7207 angular contact bearings were removed and the special ITI 8089 bearings installed in order to prove the high speed capability of the bearings before the system was seal welded (Test #2, Table V). Bearing seizure appeared imminent after a short period of running at speeds of 1,000 RPM. Other bearings were evaluated concurrently with those being evaluated in the high vacuum friction and wear tester. The detailed conditions of the tests are listed in Table V and the main test variable and results are summarized below:

<u>Test No.</u>	<u>Bearing Identification</u>	<u>Test Variable/Objective</u>	<u>Results</u>
3	ITI 8089 (Angular Contact)	Change 440C balls to WC.	No improvement - retainer galled inner race lands.
4	MRC 7207 (Angular Contact)	To evaluate commercial bearings for low speed tests.	No improvement.
5	MRC 7207 (Angular Contact)	Retainer removed from lower bearing.	Bearing seized at 770 RPM.



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Figure 4. Water Flow Achieved With the Low-Speed Impeller of the Potassium Friction and Wear Tester.



B1088-5

Figure 5. Liquid Flow Across Potassium Friction and Wear Tester Disc Specimens at 4800 RPM as a Function of Lower Specimen Supply Port Area. (Liquid Level 0.75 In. Below Lower Specimen Holder).

TABLE V. SUMMARY OF BALL BEARING TESTS FOR 5000 SFM OPERATION IN LIQUID
POTASSIUM FRICTION AND WEAR TESTER

Test No.	Bearing Identification	Ball and Race Material	Retainer Type & Material	Lubrication	Environment	Speed RPM	Thrust Load, lbs.	Time, Hrs.	Lower Bearing Outer Race Preload, lbs.	Cause of Failure/Remarks
1	MRC 7207 (Angular Contact)	52100	Pressed, low carbon steel	Lubriplate 130A	Air RT	4800 500	80	0.15 0.15	Axial play geometry controlled	No failure - removed to prove capability of ITI 8089 bearings.
2	ITI 8089	440C	Machined, 440C, Ni plated	None	Air RT	1800 1000	80 80	0.02 0.25	Axial play geometry controlled	Bearings so noisy that seizure appeared imminent. Retainer galled against inner race lands and balls showed skid marks.
3	ITI 8089	440C races WC balls	Machined, 440C, Ni plated	None	Air RT	1000	80	0.33	0.46 3	Bearings so noisy that seizure appeared imminent, noise worse at 46-lb. preload. Balls perfect, but retainer galled inner race lands and deposits appeared in ball grooves.
4	MRC 7207	52100	Pressed, low carbon steel	None	Air RT	1000	80	0.05	0.46	Bearings so noisy that seizure appeared imminent.
5	Upper MRC 7207 Lower MRC 7207 (retainerless)	52100 52100	Pressed, low carbon steel None	Lubriplate 130A None	Air RT	1000 2000 1500 780	80	0.50 0.50 1.00 1.00	0.46	Bearings noisy without deterioration in noise or vibration level. Seizure occurred several times at 770 RPM before uninterrupted 1 hour run. Success attributed to lubrication from oxidation of balls.
6	ITI 8089 (+0.015 retainers)	440C races WC balls	Machined, 440C, 0.015 dia. clearance	None	Air RT	780	80	2	0.46	Diametral clearance between retainer and inner race lands was 0.012 in. in tests 2 & 3. Bearings so noisy that failure appeared imminent after first 20 min. of test.
7	ITI 8089 (+0.018 retainer)	440C races WC balls	Machined, 440C, 0.018 dia. clearance	None	Air RT	780	80	6	0.46	This bearing was lower bearing in HVFT bearing test 13. Noise level was very high after first 20 min.; wear mainly in HVFT bearing - this bearing in very good condition.
8	ITI 8089 (+0.026 retainer)	440C races WC balls (upr) 440C balls (lwr)	Machined, 440C, 0.026 dia. clearance	None	Air RT	780	80	0.25	0.46	Noise level high; test stopped and tester assembled for trial run.

<u>Test No.</u>	<u>Bearing Identification</u>	<u>Test Variable/Objective</u>	<u>Results</u>
6	ITI 8089 (Angular Contact)	Clearance between retainer and inner race increased to 0.0075 inch.	Some improvement at low speed (780 RPM)
7	ITI 8089 (Angular Contact)	Clearance between retainer and inner race increased to 0.009 inch.	Significant improvement at low speed (780 RPM); no damage after six hours.
8	ITI 8089 (Angular Contact)	Clearance between retainer and inner race increased to 0.013 inch.	Assembly to be used in trial run of test rig.

Again, the above tests show that the basic test rig design is satisfactory for high speed operation provided that the shaft bearings are adequately lubricated. However, in the case of the liquid potassium friction tester, the selection of materials for possible use as a lubricant for the shaft bearings is extremely limited. From a review of the possible materials considered for application as a lubricant in the potassium friction tester, the fluorides appear to be most promising. Several fluorides have shown good lubricating characteristics in tests conducted in sodium and vacuum (10^{-8} torr) at NASA-Lewis Research Center⁽⁵⁾. The thermodynamic properties of the fluorides, shown in Table VI, also indicate that they may be stable in liquid potassium. Subsequently, the decision was made to build up a set of ITI 8089 angular contact bearings incorporating Type 440C stainless steel races, Type 440C or WC balls, and BaF₂/CaF₂ coated Type 440C retainers for evaluation in the liquid potassium friction tester. Concurrently, it was decided to install the bearings from Test #8 in the tester and to prepare the tester for the first test run in order to identify other possible problem areas.

C. COMPRESSION PROPERTIES

The elevated temperature vacuum compression test facility, Figure 6, has been assembled, checked out and testing procedures have been established.

1. Test Facility

A schematic of the load train and vacuum chamber is shown in Figure 7 and photographs of the load train with a specimen and the extensometer in place and of the completed assembly with the tantalum strip heater and shielding in place are shown in Figures 8 and 9, respectively.

The vacuum chamber is of double-wall construction, utilizing water cooling, and is fabricated from Type 304 SS. The dimensions of the chamber are 11-3/8-inch ID x 14-1/2-inch deep. Entry into chamber is accomplished by means of a front-mounted, double-wall door. The load train that was used

TABLE VI. THERMODYNAMIC PROPERTIES OF FLUORIDES

Compound	$-\Delta H_{298^\circ K}$	$S_{298^\circ K}$	$-\Delta F_{300^\circ K}$	$-\Delta F_{500^\circ K}$	$-\Delta F_{1000^\circ K}$	$-\Delta F_{1500^\circ K}$
BaF ₂	143.0	11.5	136.9	132.9	123.7	113.8
CaF ₂	145.1	8.2	138.8	134.8	124.9	115.3
K F	134.5	15.9	127.2	121.9	108.9	--
FeF ₂	84.0	10.4	78.8	75.6	67.6	--
CbF ₅	86.4	7.7	81.0	--	--	--
ZrF ₄	114.2	--	108.7	--	--	--

NOTE: Data given in kcal for one gram atom of F.

Data Source - Smithells, C. J., Metals Reference Book, Vol. 2, Third Edition, Butterworth, 1962.

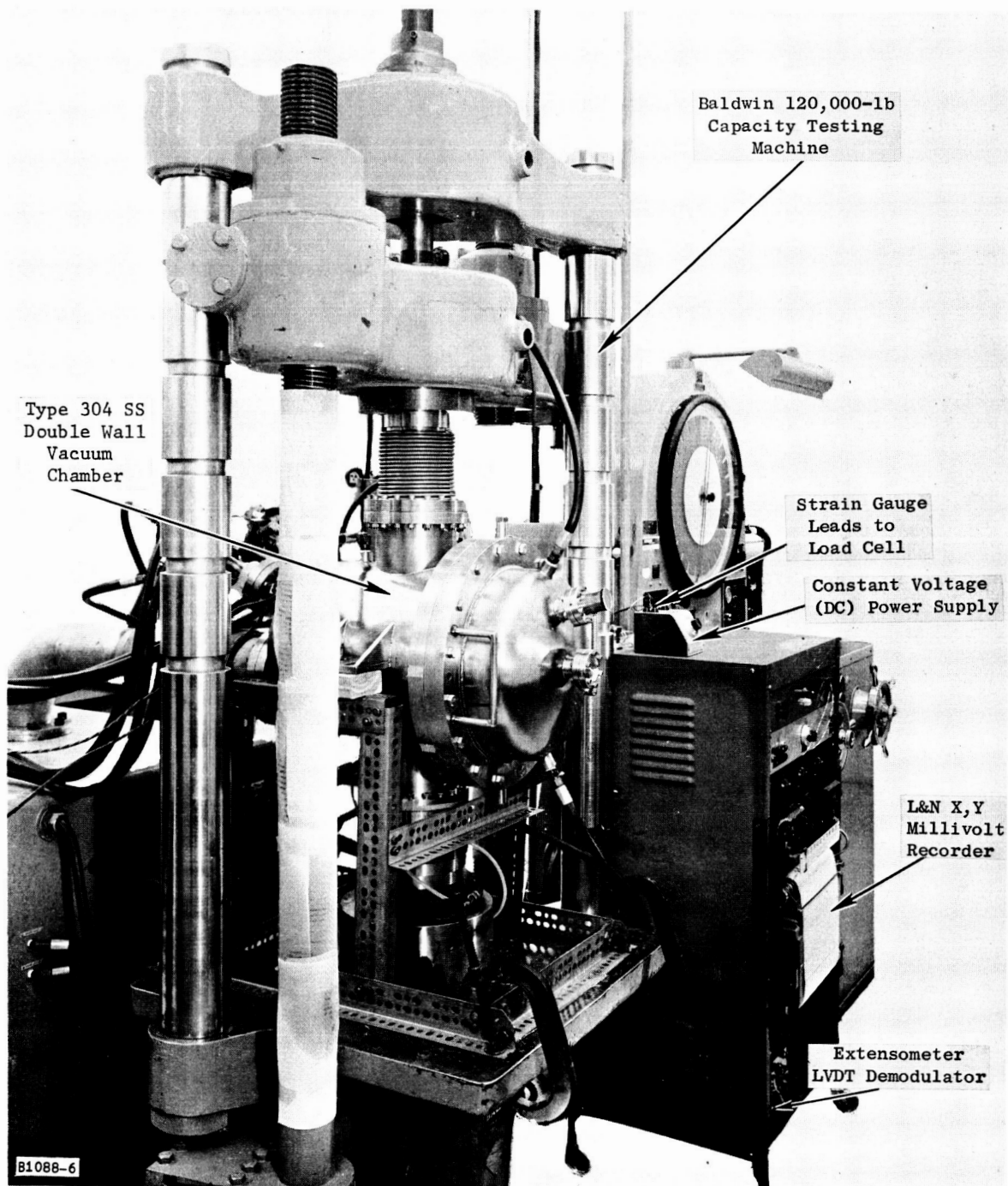
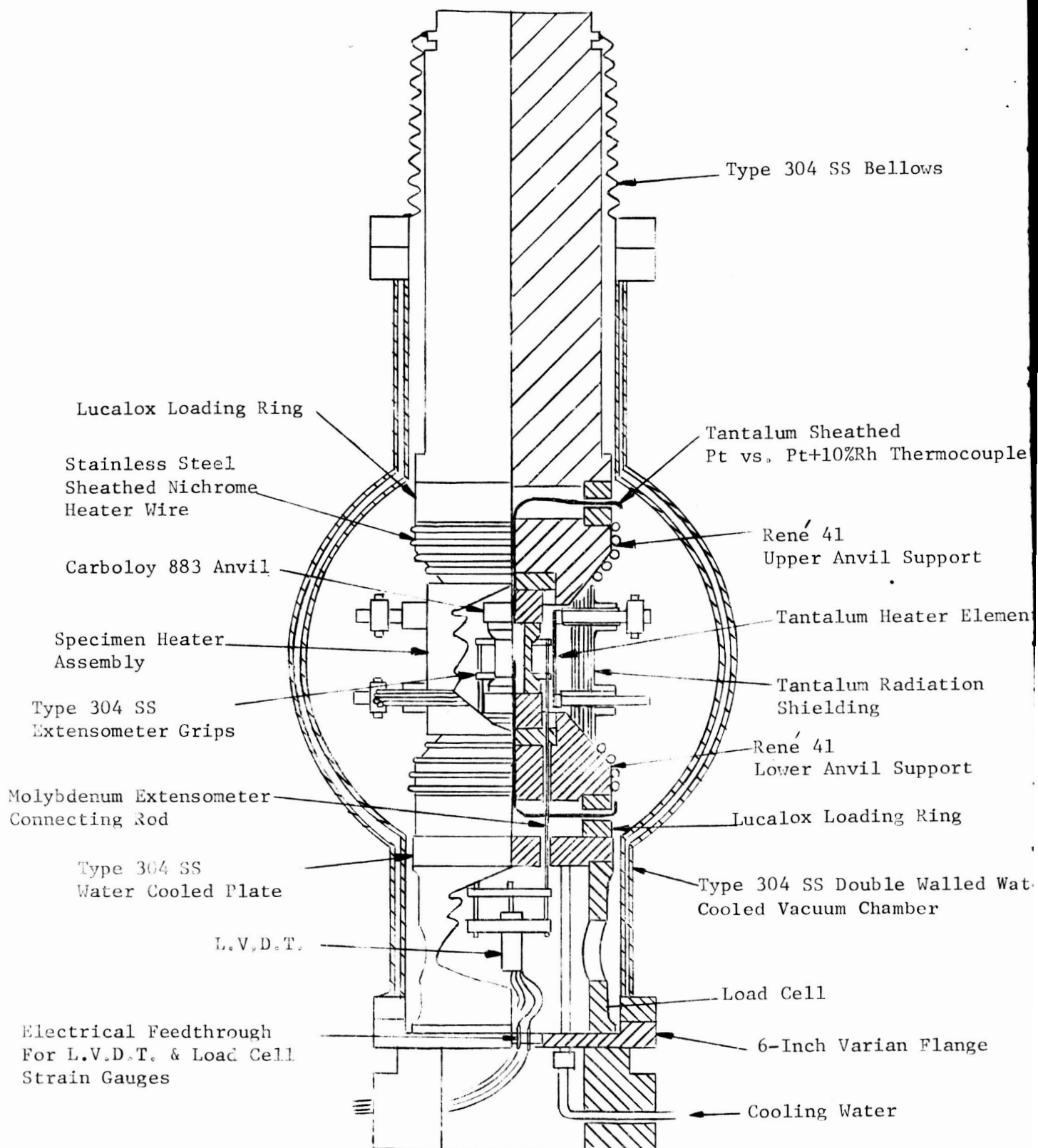


Figure 6. Elevated Temperature Vacuum Compression Testing Facility.
(C65102525)



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Figure 7. Schematic of Elevated Temperature Vacuum Compression Testing Facility.

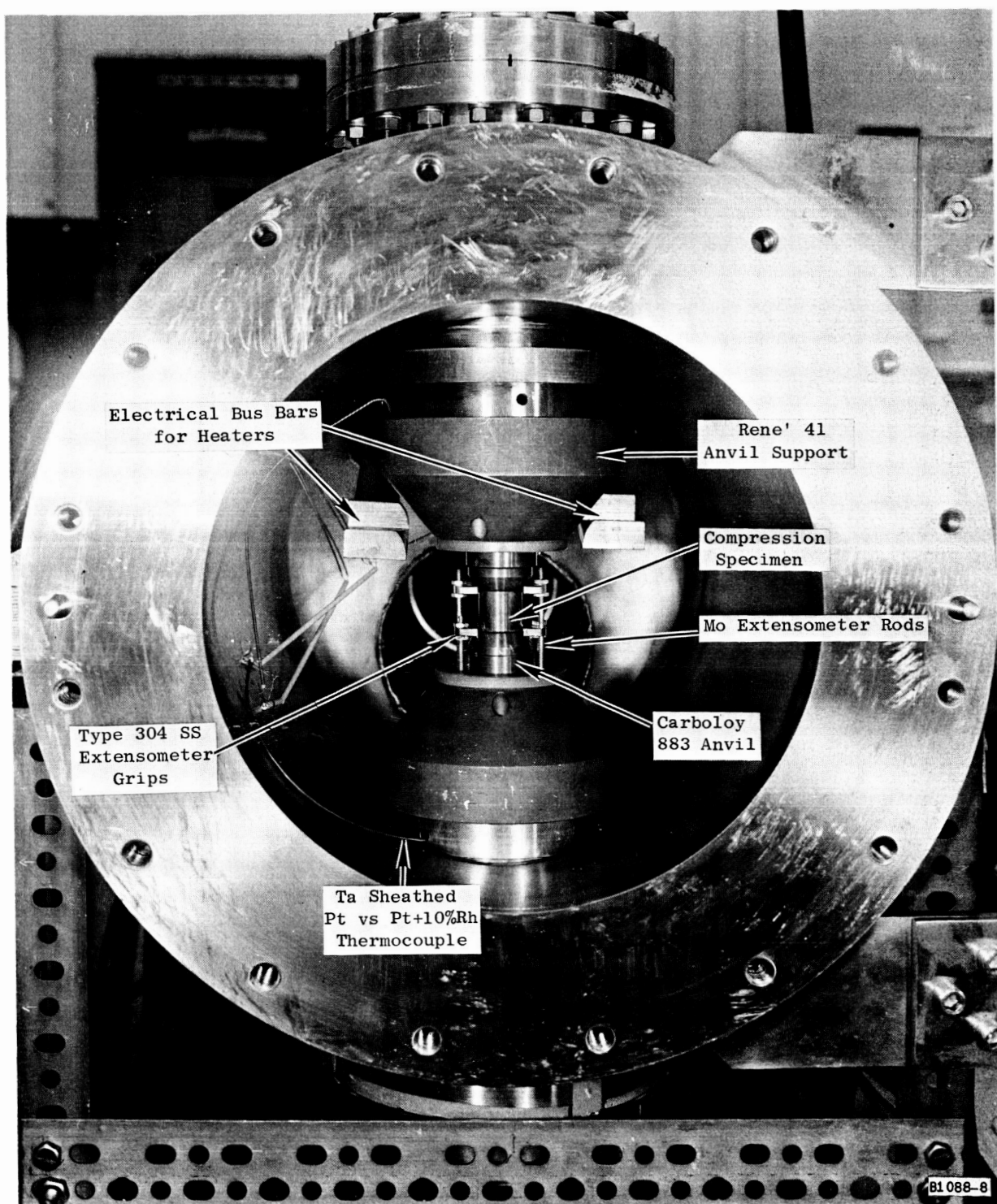


Figure 8. Inside View of Elevated Temperature Vacuum Compression Test Facility Showing Load Train Assembly. (C65102523)

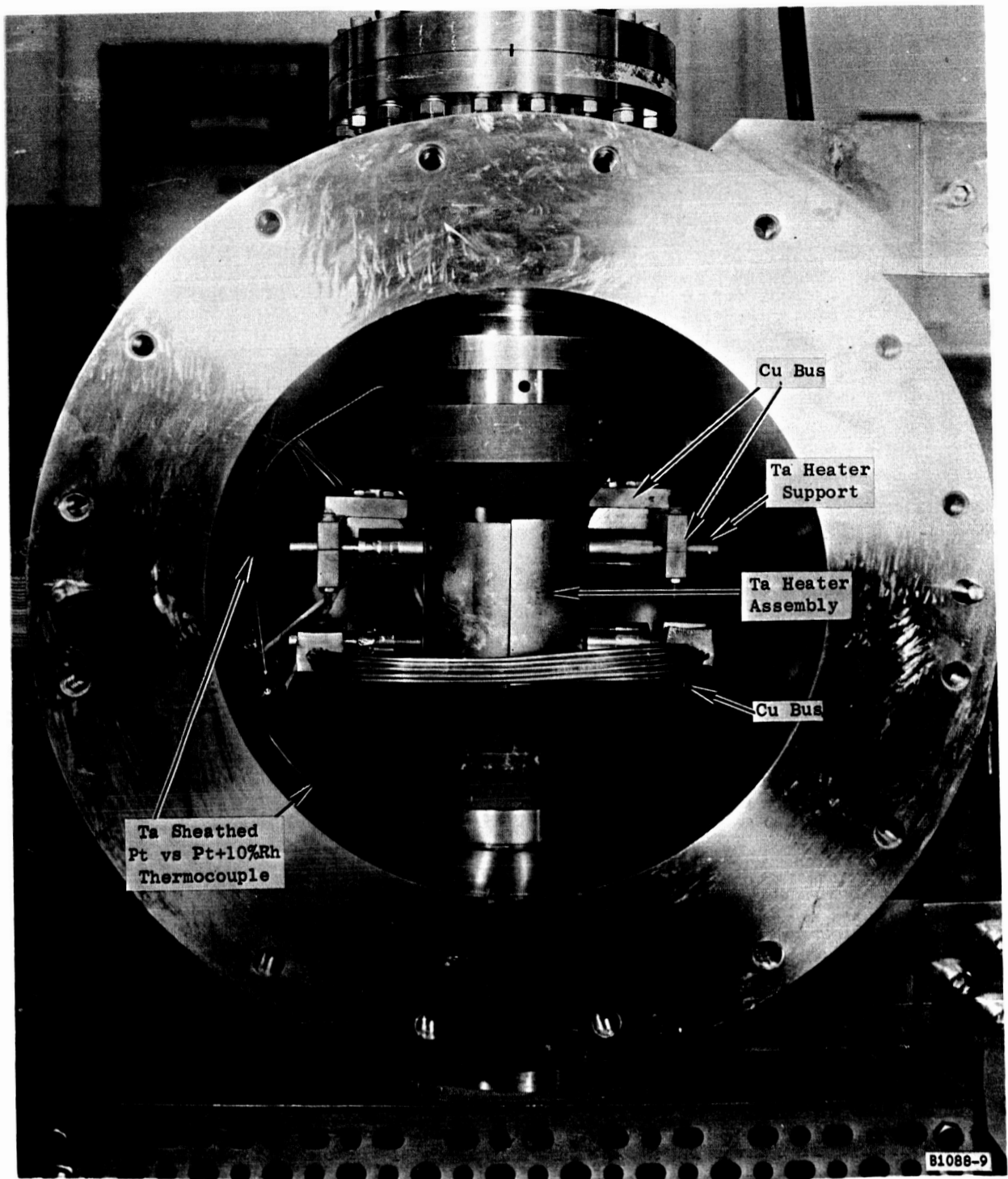


Figure 9. Inside View of Elevated Temperature Vacuum Compression Test Facility Showing Specimen Heater Assembly. (C65102524)

for the room temperature tests conducted previously⁽⁶⁾ is incorporated in the vacuum facility. Details of the load cell and the extensometer system used in the load train and their calibration have been reported in Quarterly Progress Report No. 7⁽⁷⁾. The bottom half of the load train rests on a 6-inch diameter flange which is bolted to the bottom of the chamber. An eight prong electrical feedthrough for the load cell and extensometer leads is welded into the bottom flange and a Veeco, C-25, vacuum fitting for water cooling tubulation is brazed into the bottom flange. Water cooling is required for the plate directly above the load cell. The vacuum seal for the top ram is made by means of a 6-inch ID, Type 304 SS bellows. The gasket used to seal the door is constructed of silver-plated, argon-filled stainless steel tube, and with the exception of the Veeco, C-25, fitting, all other gaskets are constructed of copper.

A standard CVC PS-40A packaged pumping system is used to evacuate the chamber and includes a PMC-720 oil diffusion pump, with a rated pumping speed of about 660 ℓ /sec at 10^{-5} torr, a Welch Duo-Seal mechanical pump, and a Type BCR Chevron ring cryogenic baffle. Pressures are measured with a Type CP-140 CVC Pirani gauge, which has a range of 2 torr to 1×10^{-3} torr, and with a CVC Type GPH-100A discharge gauge which has a range of 2.5×10^{-2} torr to 1×10^{-7} torr.

The electrical feedthroughs which supply the power for the specimen heater extend through the back of the vacuum chamber as shown in Figure 10. The water cooled copper bar feedthroughs also serve as supports for the heater assembly, Figure 9. The heater assembly is constructed in two halves to facilitate installation of the test specimens. The heating element is fabricated from 0.005-inch thick tantalum sheet, is 2-1/2 inches ID x 2-1/2 inches long, and is shielded with 5 layers of 0.005-inch thick tantalum sheet and one outer layer of Type 304 SS sheet.

The power for the tantalum heater is provided by means of a 15 KVA step-down transformer which reduces the line voltage from 220 V to 10 V. Temperature is controlled with a West JSCR controller and is measured by means of two 1/8-inch diameter tantalum-sheathed, Al_2O_3 -insulated, Pt vs Pt+10%Rh thermocouples. One thermocouple extends up through the bottom anvil so that it is in contact with the inside surface of the hollow specimens at the center of the gauge section; the second thermocouple extends down through a hole in the top anvil so that it is flush with the bottom surface of the anvil. The temperature indicated by the thermocouple at the center of the gauge section on the inside surface of the specimen during the test is recorded on a single channel Brown potentiometer recorder. Lucalox loading rings located adjacent to the anvil supports and external Inconel-sheathed, Nichrome heating elements attached to the anvil supports are incorporated into the load train to minimize heat losses from the specimen during testing.

2. Determination of Effective Gauge Length

In order to establish the effective gauge length of the compression test specimen being used in the test program, Figure 11, true strain in a

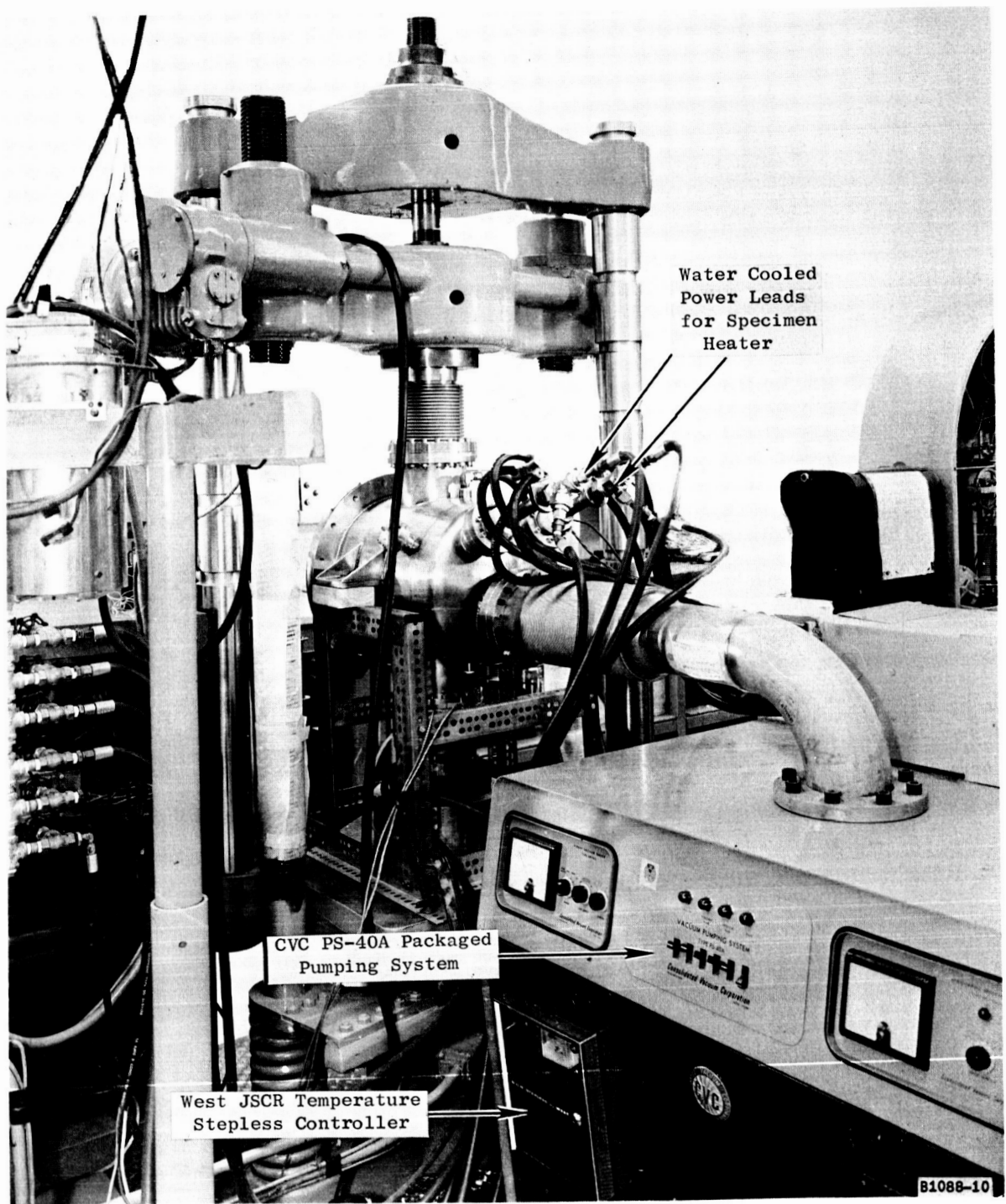
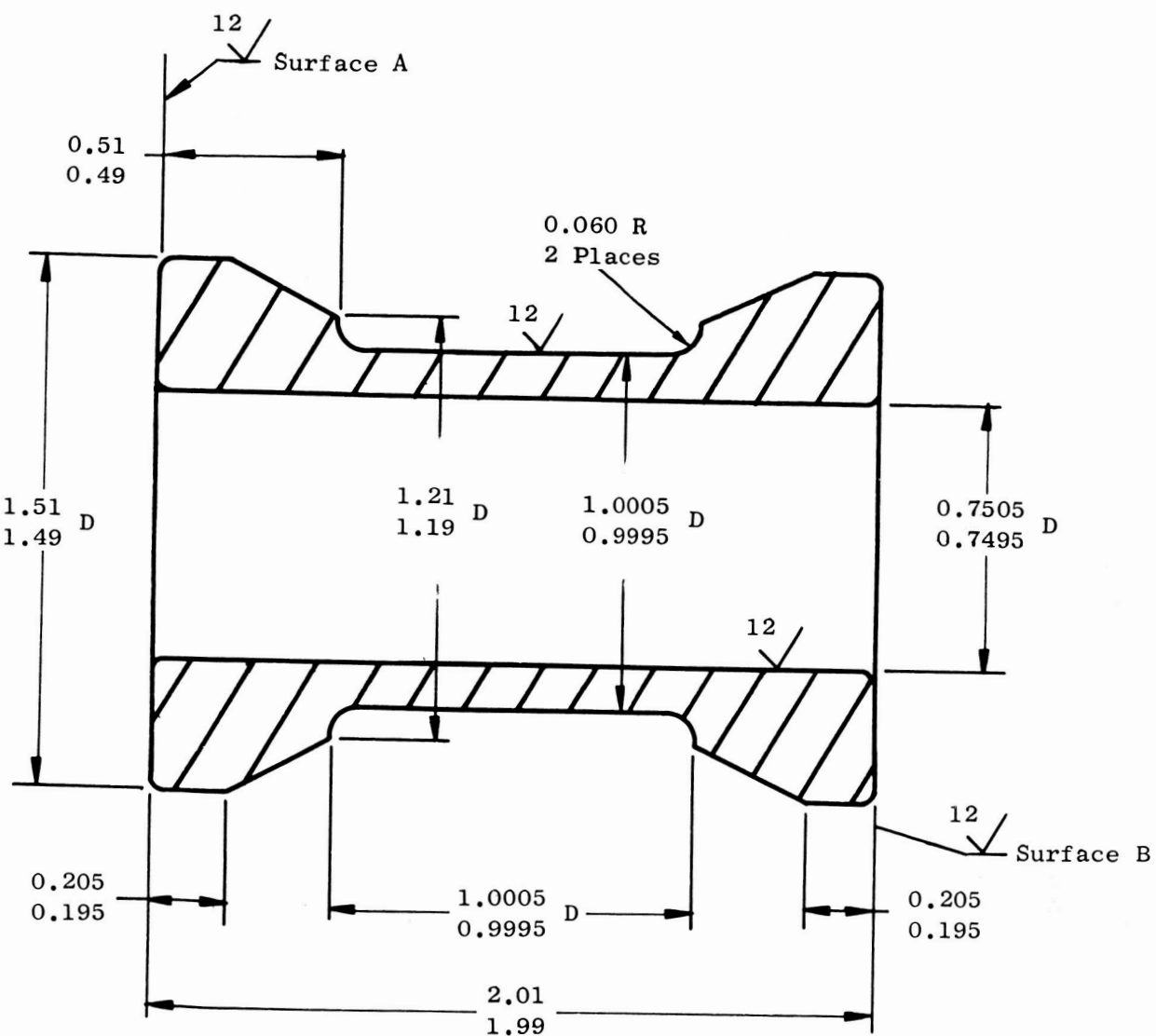


Figure 10. View of Elevated Temperature Vacuum Compression Testing Facility Showing Vacuum System and External Power Leads for Specimen Heater. (C65102526)



81088-11

Figure 11. Compression Test Specimen.

Mo-TZM alloy specimen was measured at known total extensions and loads within the elastic limit. The true strain measurements were made by means of strain gauges and total extension measurements were made with the extensometer/LVDT assembly shown in Figure 7.

Two sets of two Type C6121 strain gauges were attached to the OD surface of the specimen at the center of the gauge length and at a radial interval of 90°. One strain gauge of each set was attached parallel to the specimen axis and the other strain gauge of each set was attached perpendicular to the specimen axis, Figure 12. The signals from the strain gauges and from the LVDT of the extensometer assembly were fed into a Leeds and Northrup "Azore" two channel millivolt X-Y recorder. From the data, the effective gauge length can be calculated using the following relationship (Equation 1):

$$L = \frac{l}{\epsilon_1} \quad (1)$$

where: L = Effective gauge length, in.

l = Total extension at true strain ϵ_1 , in.

ϵ_1 = True strain, in./in.

The instrumented specimen was stressed in compression within the elastic limit at room temperature three times. Using Equation 1, the effective gauge length, L , was calculated for each test, Table VII, and found to be an average of 1.024 inches. These data confirm the original assumption^(7,8) that the effective gauge length extends into the radius of the specimen shoulder a distance equal to approximately one half the specimen wall thickness.

3. Test Procedures

Prior to any attempt to conduct compression tests in vacuum at elevated temperature, two Mo-TZM alloy specimens were tested at room temperature and at a pressure of approximately $2-5 \times 10^{-6}$ torr. The purpose of the tests was to demonstrate proper alignment of the load train and to verify the expected performance of the load cell and extensometer system. The testing procedures used were the same as those used for the previous room temperature compression tests conducted at ambient pressures^(6,7). Results of the tests are given in Table VIII. After it was determined that the test facility was functioning properly, tests to establish techniques for elevated temperature compression tests in vacuum were initiated with Mo-TZM alloy specimens. Upon completion of these tests, the following test procedures were established:

1. Inspect specimen for defects using ultrasonic and penetrant techniques
2. Dimensionally measure specimen, i.e., gauge length, wall thickness, and parallelism of end faces.

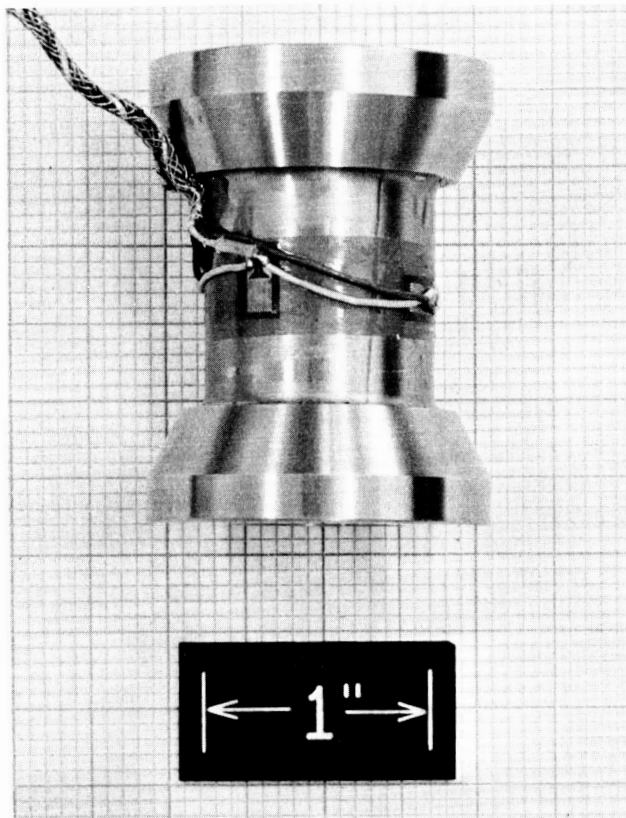


Figure 12. Mo-TZM Alloy Compression Test Specimen With C6121 Strain Gauges Attached to Determine Effective Gauge Length. (C66063031).

TABLE VII. EFFECTIVE GAUGE LENGTH OF COMPRESSION TEST SPECIMEN

(1) Specimen Identification	(2) Test No.	(3) Measured Gauge Length Inch	(4) Calculated ^(a) Effective Gauge Length Inch	(5) Difference Column (4)- Column (3) Inch	Measured Wall Thickness Inch
MCN-1037-G-11 ^(b)	1	0.880	1.040	0.160	0.100
	2	0.880	1.017	0.137	0.100
	3	0.880	1.014	0.134	0.100
Average		0.880	1.024	0.144	0.100

(a) $L = \frac{\ell}{\epsilon_1}$; ϵ_1 = true strain; ℓ = total extension at ϵ_1 .

(b) Mo-TZM alloy.

TABLE VIII. ROOM TEMPERATURE COMPRESSION PROPERTIES OF Mo-TZM
ALLOY^(a) IN AIR AND VACUUM (10^{-6} TORR)

Specimen Identity	Pressure Torr	0.02% CYS psi ^(b)	0.2% CYS psi ^(b)	Strength ^(c) 1% Strain psi	Strength ^(c,d) 5% Strain psi
MCN-1037-G-1	760	89,400	102,000	102,000	107,200
MCN-1037-G-2	760	86,000	104,000	104,300	111,000
MCN-1037-G9	10^{-6}	99,600	115,500	115,500	119,000
MCN-1037-G10	10^{-6}	83,800	112,500	112,500	112,500

(a) Stress-relieved 2350°F/one hour.

(b) Strain rate 0.005 in./in./min through 0.2% offset.

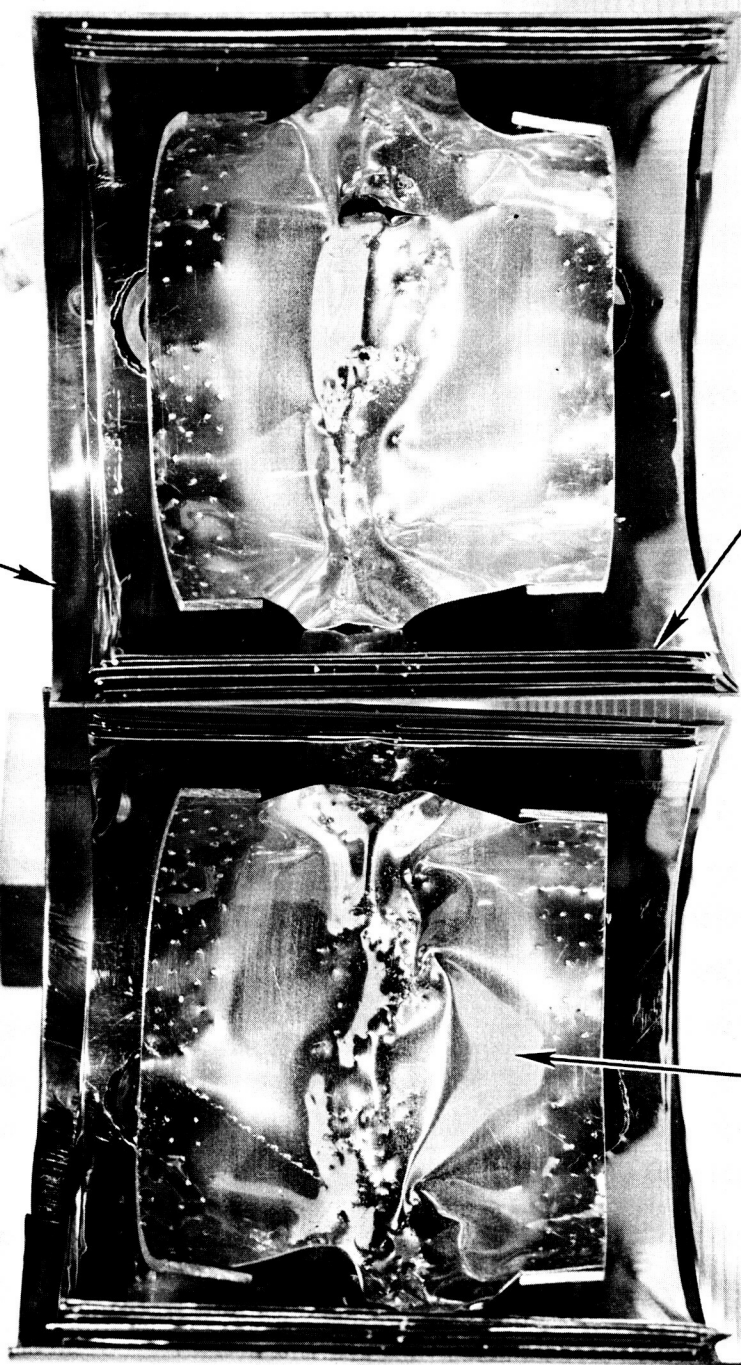
(c) Average effective gauge length 0.977 inch.

(d) Test terminated at 5% total strain.

3. Check alignment and install specimen in load train with 0.005-inch thick tantalum shim between specimen and anvil.
4. Install tantalum strip heater assemblies.
5. Evacuate chamber to a pressure of $\leq 1 \times 10^{-5}$ torr.
6. Apply power to anvil support heaters to attain maximum temperature of approximately 450°F.
7. Apply power to tantalum strip heaters and heat specimen to test temperature (800°, 1200°, 1600°F). Maintain pressure below 1×10^{-5} torr during heat-up cycle.
8. Apply 1,000-pound load on the specimen as the test temperature is reached. The load is controlled by the Baldwin load maintainer and the temperature is automatically controlled by a West stepless controller. The 1,000-pound load is maintained on the specimen at the test temperature for approximately 1-1/2 hours in order to permit the system to reach equilibrium temperatures.
9. Switch the method of temperature control from the automatic stepless controller to a constant voltage input to the tantalum heater. It is necessary to maintain a constant heater temperature in order to prevent the temperature of the extensometer rods, which are located within the heater area, from varying and producing a false strain indication due to thermal expansion or contraction.
10. Apply load to the specimen at a strain rate of 0.005 in./in./min. through the 0.2% yield point. The valve setting on machine to achieve the 0.005 in./in./min. strain rate in the elastic region is left unchanged for the duration of the test.
11. Terminate the test at 5% total strain or 115,000 pounds load if fracture of the specimen has not occurred.
12. Calibrate the load cell and extensometer/LVDT system as described in Quarterly Progress Report No. 7(7).

Several problems were uncovered in the testing of specimens in compression at temperatures to 1600°F and at loads to 115,000 pounds. The major problem is the destruction or damage of test components due to fracture of the specimens. Particles from the fractured specimen will virtually destroy the heater, Figure 13. Also, the fine debris from the fractured specimen will lodge

Type 304 SS
Radiation
Shield



0.005-Inch Thick
Ta Heater Element

Ta Radiation
Shields (5)

81088-13

Figure 13. Impact Damage to Tantalum Heater Element Due to Fracture of Specimen at High Loads. (C65122389)

between the extensometer rods and the anvil supports requiring disassembly of the load train prior to conducting the next test, Figure 14. Bending of the extensometer rods may also occur due to the sudden shock from the fracture. Although these problems do not occur with each failure, the degree of the damage is greater at the higher loads and temperatures. Finally, even though the anvil supports are heated externally and Lucalox insulators are employed, significant heat losses occur through the load train resulting in a reduction in specimen temperature of as much as 75°F. To compensate for the drop in test temperature, the temperature of the specimen, prior to initiating the test, was equilibrated at a temperature slightly higher than the desired test temperature. As mentioned previously, power to the heater cannot be augmented without affecting the strain measurement. Major design changes of the test facility would be required to further reduce the magnitude of these problems.

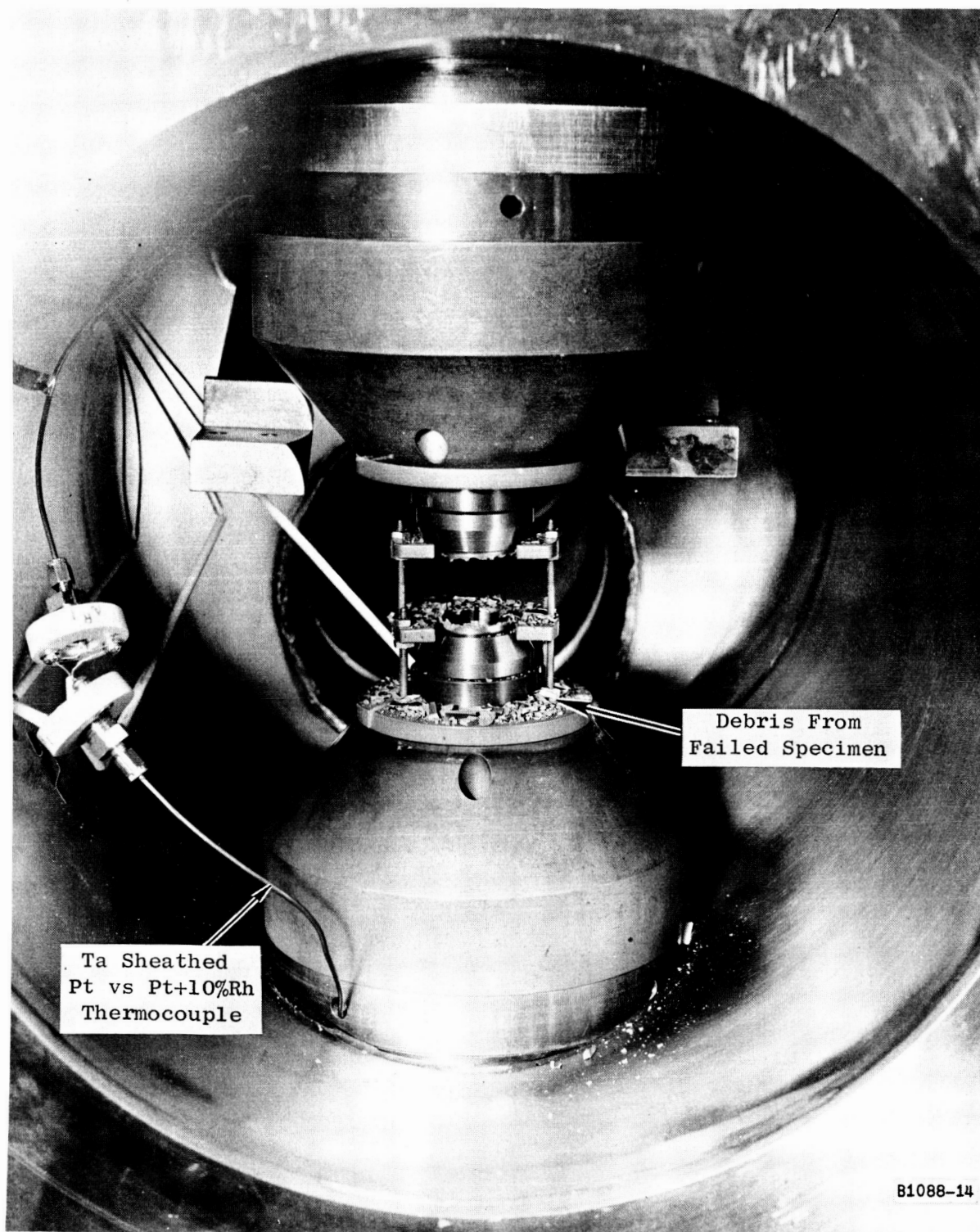


Figure 14. Inside View of Elevated Temperature Vacuum Compression Test Facility Showing Debris from Failed Specimen. (C65122390)

V. TEST PROGRAMS

A. CORROSION - CANDIDATE BEARING MATERIAL COMBINATIONS

In order to evaluate the compatibility of the candidate bearing material combinations in a Cb-1Zr alloy/potassium system, 1,000-hour isothermal capsule corrosion tests were conducted with the following three material combinations at 800°, 1200°, and 1600°F:

Mo-TZM vs Carboloy 907

Mo-TZM vs Grade 7178

Mo-TZM vs TiC+10%Cb

The experimental procedures of the test are described in Quarterly Progress Report No. 9⁽⁹⁾; visual examination and dimensional and weight change measurements of the test specimens and chemical analyses of the Cb-1Zr alloy capsule wall for carbon, oxygen, nitrogen, and hydrogen are reported in Quarterly Progress Report No. 10⁽¹⁰⁾. The test specimens have been evaluated further by X-ray diffraction techniques, metallographic examination, micro-hardness, and chemical analyses. The potassium from the capsules was sampled and chemically analyzed for metallic impurities.

1. X-Ray Diffraction Analysis

The X-ray diffraction analyses of the candidate bearing material specimens Carboloy 907, TiC+10%Cb, and Grade 7178 exposed to potassium liquid and vapor at 1600°F for 1,000 hours in combination with Mo-TZM alloy specimens have been completed. The phases detected on the surface of the specimens adjacent to the Mo-TZM alloy specimens before and after testing are shown in Table IX. No phase change was observed after testing in the TiC+10%Cb material as was the case for the TiC+10%Cb specimens that were tested individually⁽¹⁰⁾.

The specimens of both the Carboloy 907 and Grade 7178 materials which were exposed to potassium liquid exhibited major surface phase changes from WC to tungsten. Only a minor amount of WC was detected on the surfaces after exposure. Similar specimens of Carboloy 907 and Grade 7178 which were exposed in the vapor zone exhibited major phases of both WC and tungsten after testing. The transfer of carbon in this case is most likely accomplished through a liquid film of potassium maintained between the specimens and the adjacent Mo-TZM alloy specimens.

The formation of $\text{Co}_2\text{W}_4\text{C}$ and $(\text{Co}, \text{W})_x\text{C}$ was detected on the surface of the Carboloy 907 specimen exposed to K liquid; only $(\text{Co}, \text{W})_x\text{C}$ was detected on the surface of the Carboloy 907 specimen exposed in the potassium vapor zone.

TABLE IX. SURFACE PHASE CHANGES^(a) OF CANDIDATE BEARING MATERIALS EXPOSED TO POTASSIUM LIQUID AND VAPOR FOR 1,000 HOURS AT 1600°F ADJACENT TO Mo-TZM ALLOY

Material	(b)					
	Phases Present			Post Test		
	Pre-Test		Specimen in Liquid		Specimen in Vapor	
	Major	Minor	Major	Minor	Major	Minor
Carboloy 907	WC	TaC	W	WC, TaC, Co ₂ W ₄ C, (Co, W) _x C	WC, W	TaC, (Co, W) _x C
TiC+10%Cb	(Ti, Cb)C	--	(Ti, Cb)C	---	(Ti, Cb)C	--
Grade 7178	WC	(Mo, Cb) ₂ C	W	(Mo, Cb) ₂ C, WC	WC, W	(Mo, Cb) ₂ C

(a) X-ray diffraction analyses performed on specimen surface adjacent to Mo-TZM alloy specimen.

(b) Identified by x-ray diffraction (Cu K α).

2. Metallographic Examination

The metallographic examination of the candidate bearing material specimen combinations tested at 800°, 1200°, and 1600°F for 1,000 hours has been completed. The Carboloy 907 specimen exposed to liquid potassium at 1600°F exhibited surface roughness, excessive porosity to a depth of approximately 0.001 inch and a surface reaction to a depth of 0.0006 inch on the surface adjacent to the Mo-TZM alloy specimen, Figure 15. Excessive porosity (<0.0001 inch from the surface) also was detected on the surface of the Carboloy 907 specimen exposed to potassium vapor at 1600°F and adjacent to the Mo-TZM alloy specimen, Figure 15.

The Mo-TZM alloy specimen tested with the Carboloy 907 specimen in liquid potassium at 1600°F exhibited a uniform surface layer 0.0003 inch thick on the surface adjacent to the Carboloy 907 specimen, Figure 16. An X-ray diffraction analysis of the layer on the surface of the Mo-TZM alloy showed it to be Mo₂C. The formation of this layer is the result of carbon transfer from the adjacent Carboloy 907 specimen. Thermodynamically, this reaction is possible⁽¹⁰⁾. The Mo-TZM alloy specimen tested with the Carboloy 907 specimen in potassium vapor at 1600°F also exhibited a uniform surface layer of Mo₂C (0.00017-inch thick) on the surface adjacent to the Carboloy 907 specimen, Figure 16. The transfer of carbon in this case is probably made possible through a liquid film of potassium maintained in the 0.020-inch space between the specimens during the test. It is interesting to note the considerably slower rate of transfer which existed between the specimens in the vapor zone as compared to the specimens in the liquid zone.

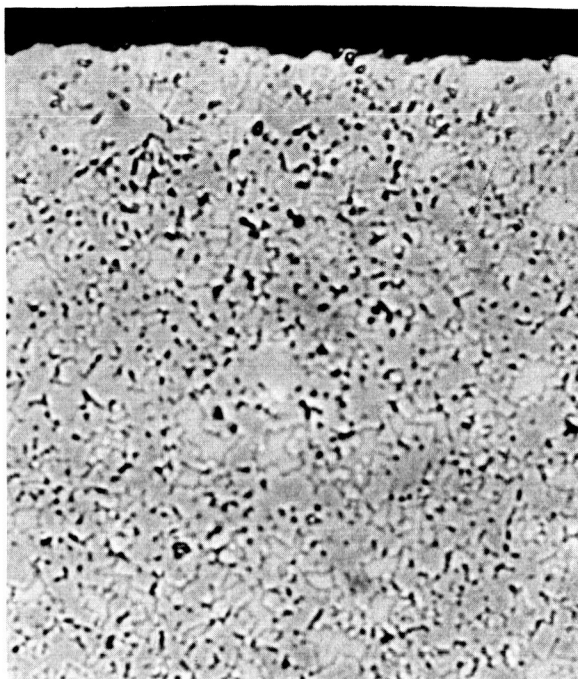
The Carboloy 907 specimen exposed to liquid potassium at 1200°F exhibited surface roughness and a surface reaction to a depth of 0.00047 inch on the surface adjacent to the Mo-TZM alloy specimen, Figure 17. The Carboloy 907 specimen exposed to potassium vapor at 1200°F showed a similar surface reaction to a depth of 0.00033 inch, Figure 17.

The Mo-TZM alloy specimen tested with the Carboloy 907 specimen in liquid potassium at 1200°F exhibited an extremely thin layer (i.e. 0.00003-inch thick) on the surface adjacent to the Carboloy 907 specimen. No significant changes were observed in the Mo-TZM alloy exposed with the Carboloy 907 specimen in the vapor zone at 1200°F.

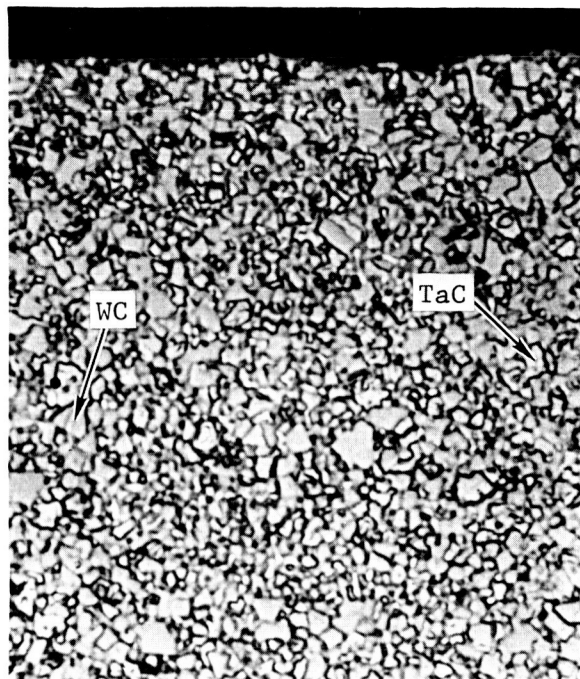
The Carboloy 907 specimens tested at 800°F showed no significant changes, Figure 18.

The Mo-TZM alloy specimens tested at 800°F with Carboloy 907 also showed no significant changes.

The Grade 7178 specimens tested in combination with the Mo-TZM alloy specimens at 800°, 1200°, and 1600°F exhibited no serious surface attack or reactions. However, an agglomeration of the eutectic phase was apparent in all the specimens, Figures 19, 20, and 21. Similar changes were observed



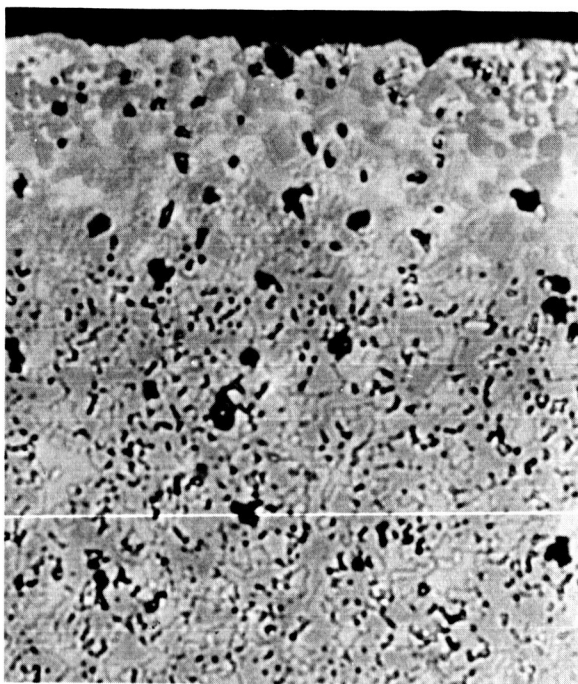
As Polished (A980321)



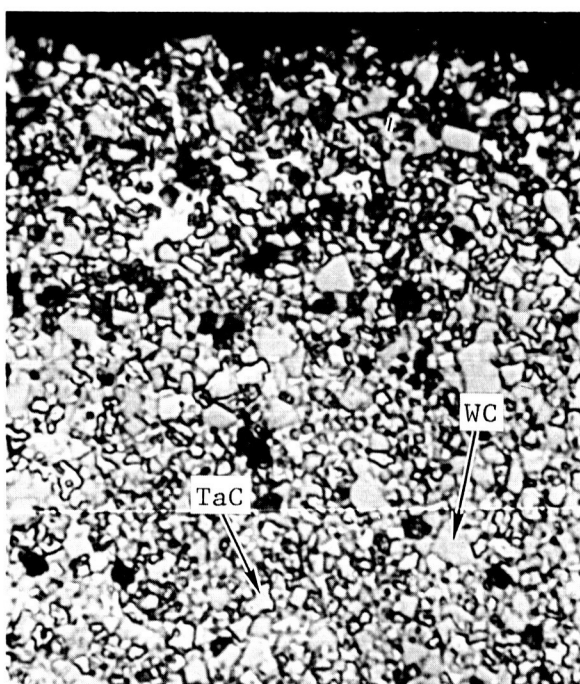
Etched (A980322)

Vapor Zone Note Apparent Surface Attack to a Depth of 0.0001 Inch.

0 0.0005 0.001
Inch



As Polished (A980311)



Etched (A980312)

Liquid Zone Note Apparent Surface Attack to a Depth of 0.001 Inch.

B1088-15

Figure 15. Microstructure of Transverse Sections of Carboloy 907 After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 1600°F.

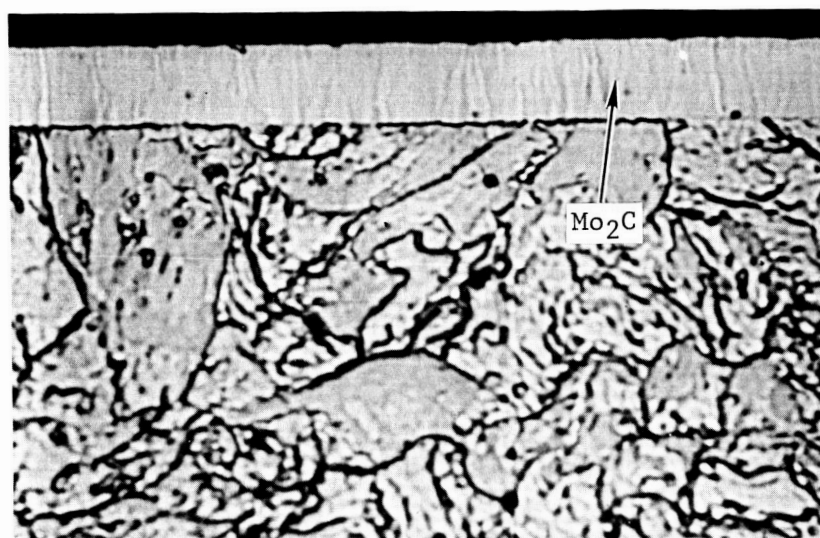
Etchant: 10%NaOH, Electrolytic

Magnification: 2000X
N.A.: 1.30



Vapor Zone (A981621)

0 0.0005 0.001
Inch



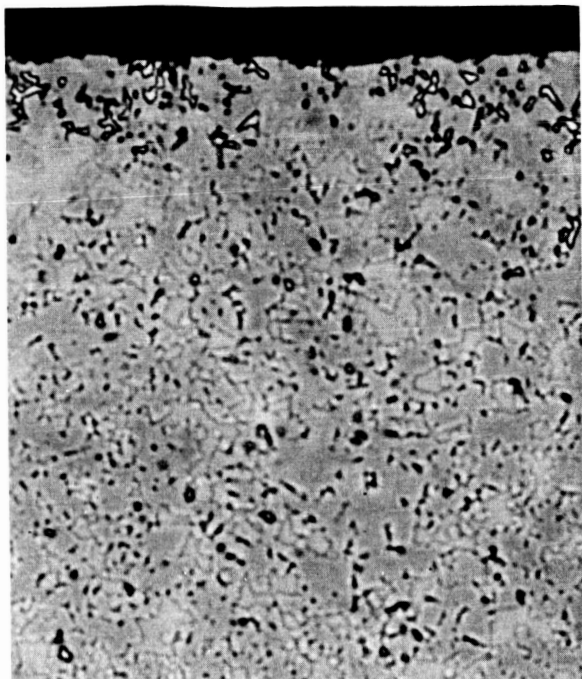
Liquid Zone (A981611)

B1088-16

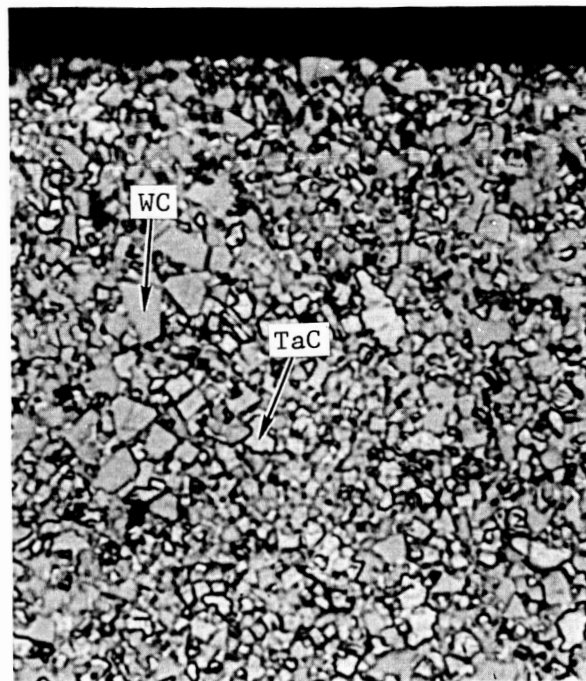
Figure 16. Mo_2C Layer on Surface of Mo-TZM Alloy Specimen Adjacent to Carboloy 907 Specimen After Exposure to Potassium for 1000 Hours at 1600°F .

Etchant: Murakamis

Magnification: 1500X
N.A.: 0.95

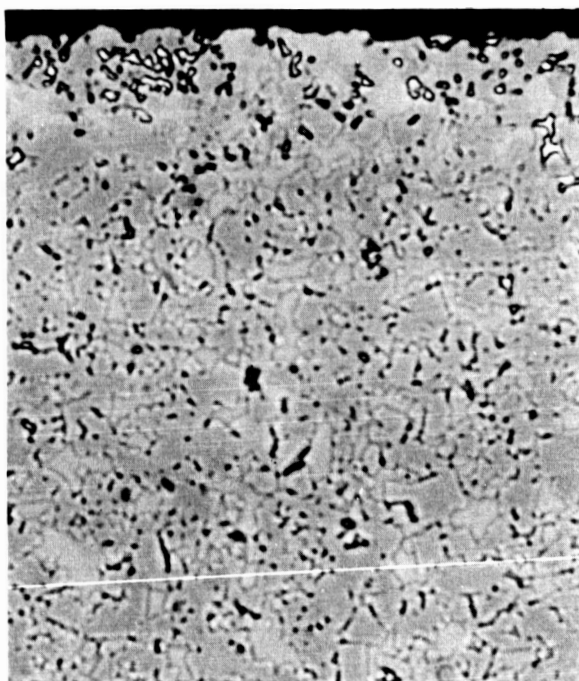
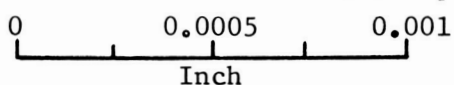


As Polished (A980221)

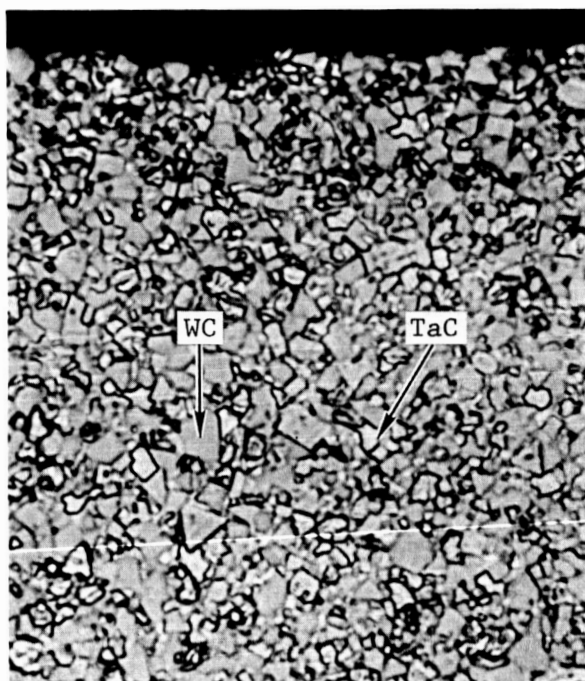


Etched (A980222)

Vapor Zone Note Apparent Surface Attack
to a Depth of 0.00033 Inch.



As Polished (A980211)



Etched (A980212)

Liquid Zone Note Apparent Surface Attack
to a Depth of 0.00047 Inch.

81088-17

Figure 17. Microstructure of Transverse Sections of Carboloy 907 After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 1200°F.

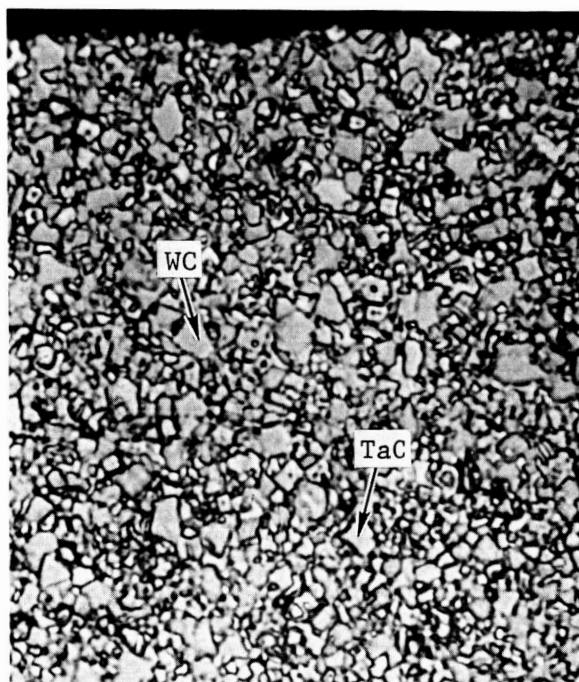
Etchant: 10%NaOH, Electrolytic

Magnification: 2000X
N.A.: 1.30



Vapor Zone (A980121)

0 0.0005 0.001
Inch



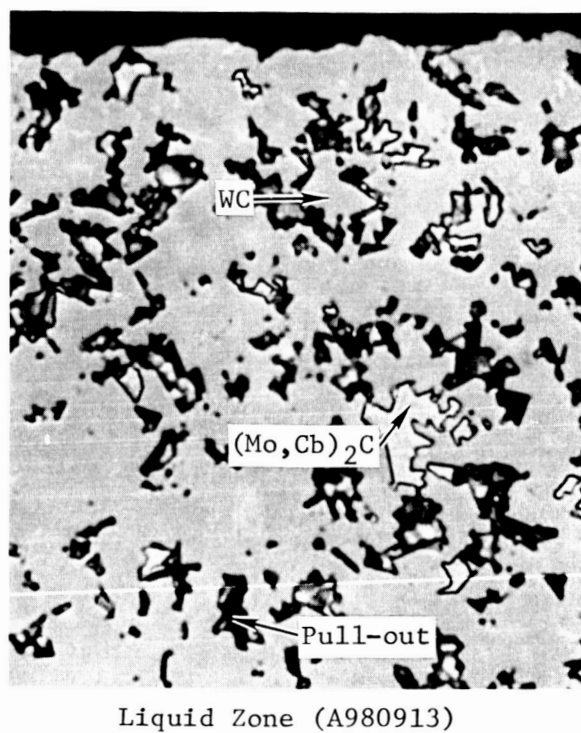
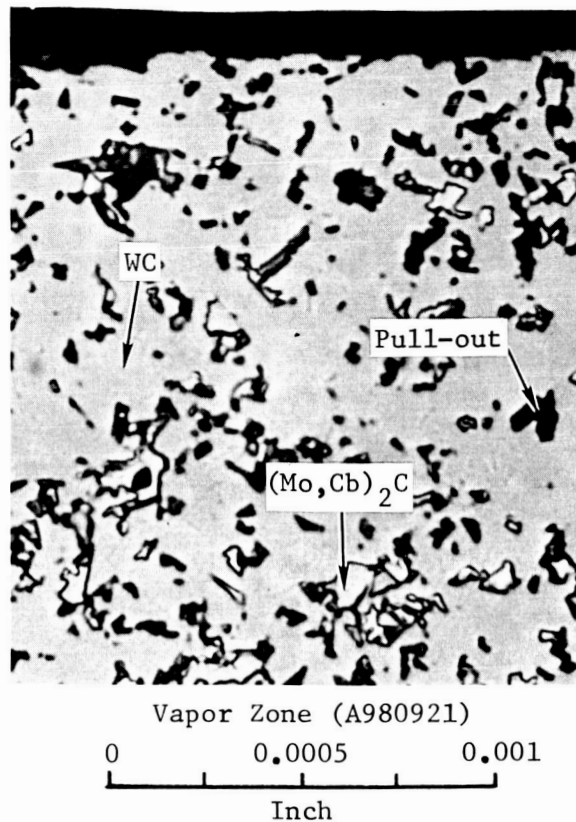
Liquid Zone (A980112)

B1088-18

Figure 18. Microstructure of Transverse Sections of Carboloy 907 After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 800°F.

Etchant: 10%NaOH, Electrolytic

Magnification: 2000X
N.A.: 1.30

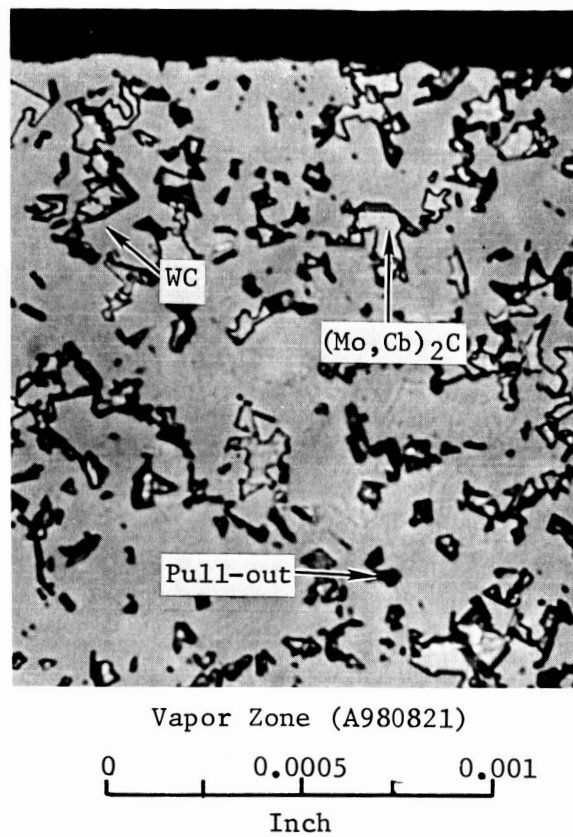


B1088-19

Figure 19. Microstructure of Transverse Sections of Grade 7178 After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 800°F.

Etchant: 10%NaOH, Electrolytic

Magnification: 2000X
N.A.: 1.30



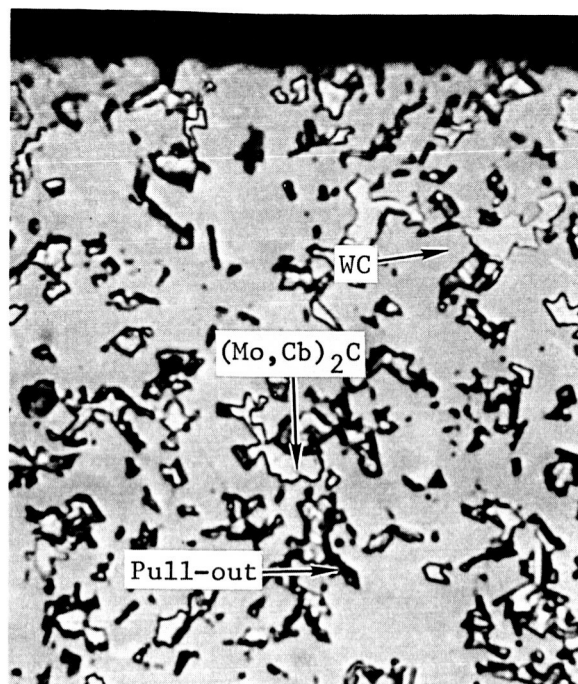
B1088-20

Figure 20. Microstructure of Transverse Sections of Grade 7178 After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 1200°F.

Etchant: 10%NaOH, Electrolytic

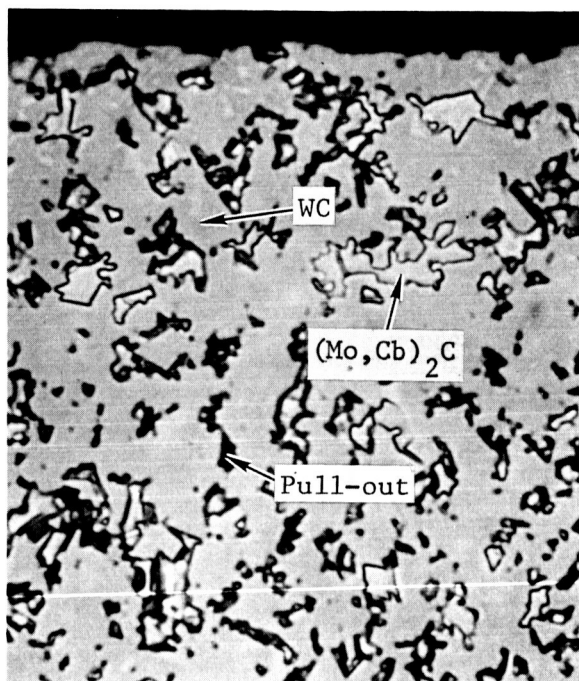
Magnification: 2000X

N.A.: 1.30



Vapor Zone (A980722)

0 0.0005 0.001
Inch



Liquid Zone (A980713)

B1088-21

Figure 21. Microstructure of Transverse Sections of Grade 7178 After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 1600°F.

Etchant: 10%NaOH, Electrolytic

Magnification: 2000X
N.A.: 1.30

in the Grade 7178 specimens tested individually, and as was discussed in Quarterly Progress Report Nos. 9(9) and 10(10), these apparent changes could be the result of inhomogeneity in the specimens.

The Mo-TZM alloy specimens tested in combination with Grade 7178 at 1600°F in liquid potassium exhibited a layer of Mo₂C 0.00015-inch thick on the surface adjacent to the Grade 7178 specimen, Figure 22, similar to the layer observed on the surface of the Mo-TZM alloy specimens that were tested in combination with Carboloy 907. Undoubtedly, this is the result of carbon transfer from the Grade 7178 specimen. The remaining Mo-TZM alloy specimens tested in combination with Grade 7178 showed no significant changes.

No significant changes were observed in the TiC+10%Cb specimens tested in combination with Mo-TZM alloy specimens at 800°, 1200°, and 1600°F in potassium liquid and vapor, Figures 23, 24, and 25. Also, no significant changes were observed in the Mo-TZM alloy specimens tested with the TiC+10%Cb specimens.

3. Chemical Analyses of Test Specimens

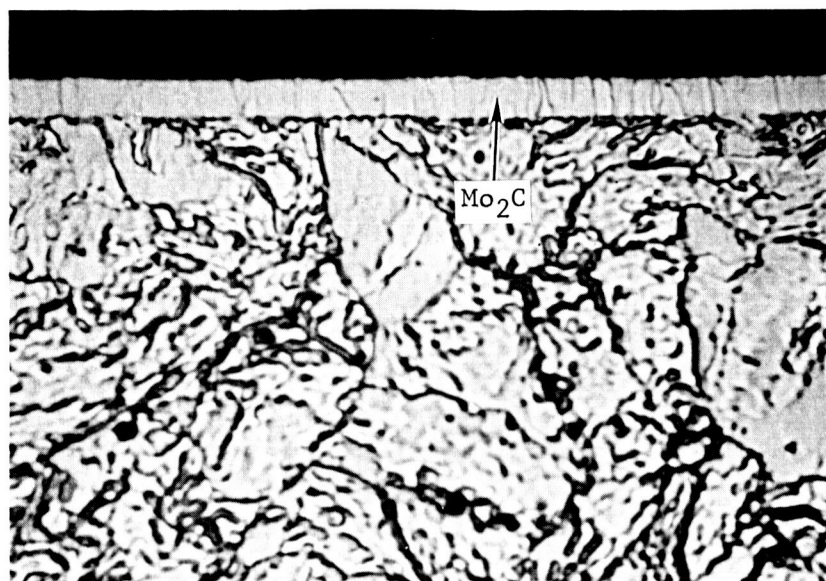
The chemical analyses of the Mo-TZM alloy specimens tested in combination with Carboloy 907, Grade 7178, and TiC+10%Cb in potassium liquid and vapor at 1600°F for 1,000 hours have been completed, and the results are shown in Table X. The slight increase in carbon content observed in the Mo-TZM specimen MCN 1037-A25 which was tested with Carboloy 907 in liquid potassium may be the result of the Mo₂C layer found metallographically on the surface of the specimen. Because of the sampling technique used(6) in which the entire 0.250-inch x 0.250-inch cross section was analyzed, the Mo₂C layers observed on the Mo-TZM specimens tested with Carboloy 907 and Grade 7178 would comprise only a small portion of the total carbon in the samples. Little significance is attached to the other slight variations observed.

4. Chemical Analyses of Potassium

The potassium which was contained in the Cb-1Zr alloy capsules tested at 1600°F for 1,000 hours was transferred to platinum dishes under argon in an electron beam weld chamber and was reacted with HCl to form the KCl salt. Subsequently, the KCl was analyzed for metallic impurities using spectrographic techniques; the data are presented in Table XI. No significant changes in metallic content of the potassium were observed except for the potassium which was exposed with the TiC+10%Cb/Mo-TZM alloy specimen combination. In this case, the potassium exhibited an increase in both aluminum and titanium contents.

5. Microhardness Determinations

Microhardness surveys on all of the corrosion specimens, tested individually and in combination in potassium at 800°, 1200°, and 1600°F for 1,000 hours, have been completed. Three sets of diamond pyramid indentations were made at 0.001 inch, 0.020 inch, and 0.125 inch from the edge using



(A981011)

0 0.005 0.001
Inch

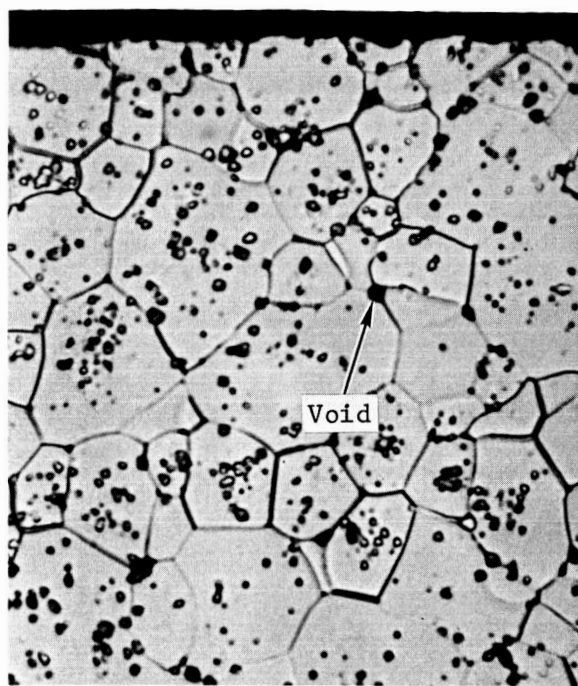
B1088-22

Figure 22. Mo_2C Layer on Surface of Mo-TZM Alloy Specimen Adjacent to Grade 7178 Specimen After Exposure to Potassium Liquid for 1000 Hours at 1600°F .

Etchant: Murakamis

Magnification: 1500X

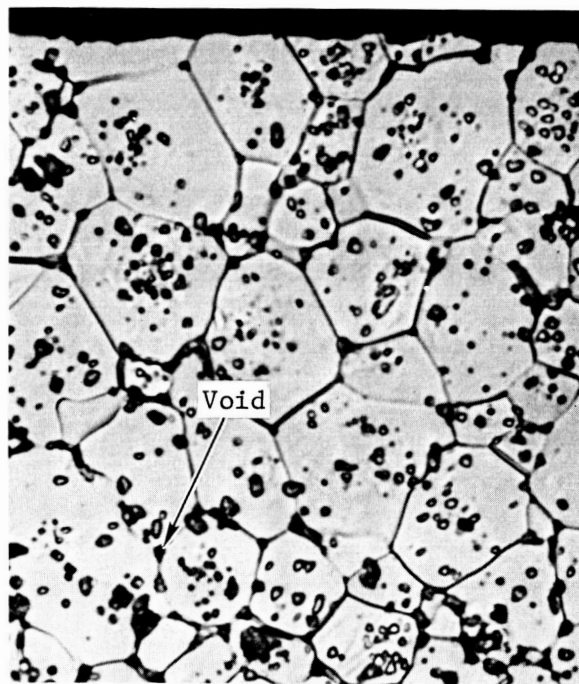
N.A.: 0.95



Vapor Zone (A980421)

0 0.0005 0.001

Inch



Liquid Zone (A980411)

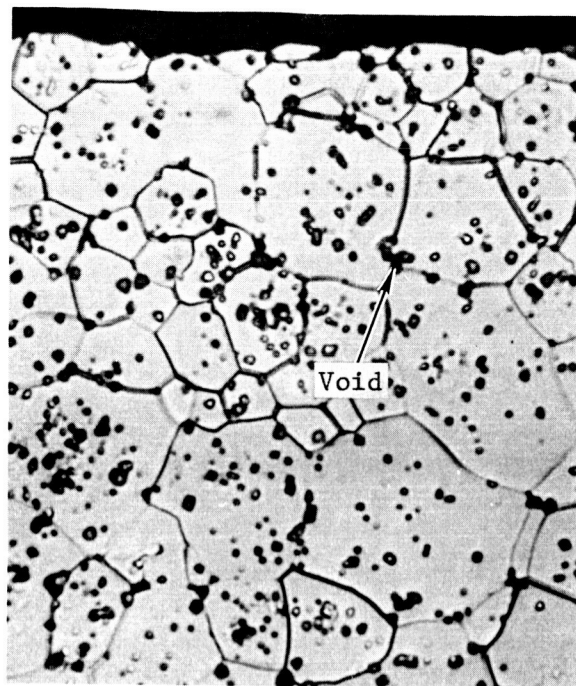
B1088-23

Figure 23. Microstructure of Transverse Sections of TiC+10%Cb After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 800°F.

Etchant: 80HNO₃+20HF

Magnification: 2000X

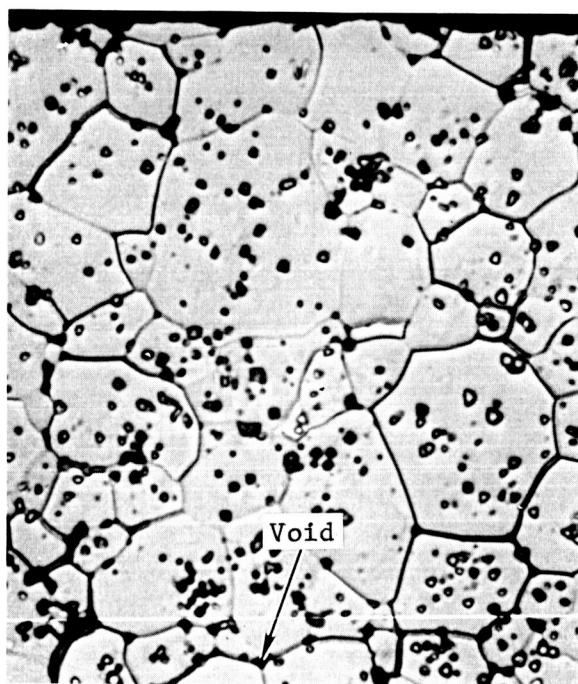
N.A.: 1.30



Vapor Zone (A980521)

0 0.0005 0.001

Inch



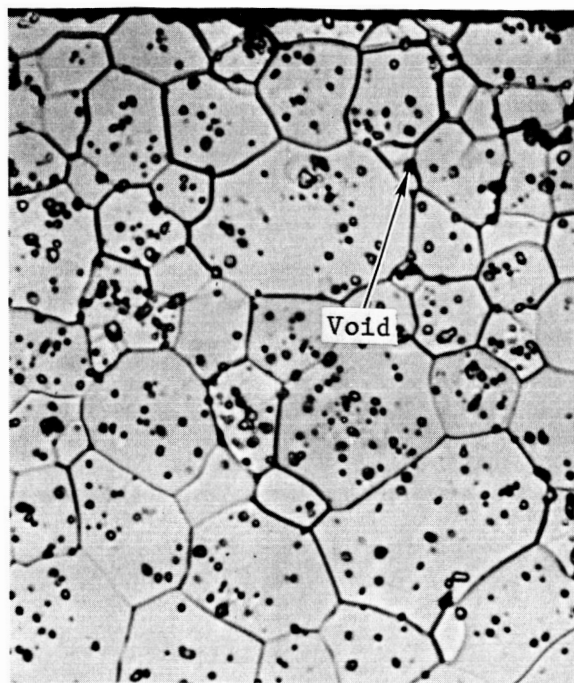
Liquid Zone (A980511)

B1088-24

Figure 24. Microstructure of Transverse Sections of TiC+10%Cb After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 1200°F.

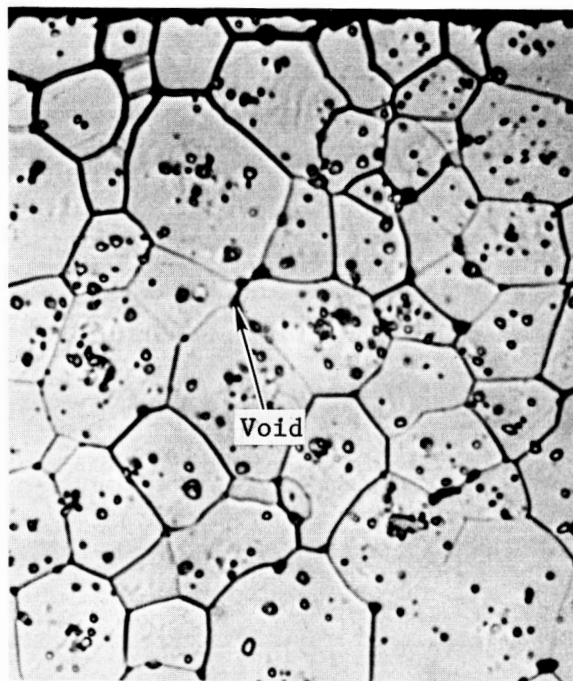
Etchant: $80\text{HNO}_3 + 20\text{HF}$

Magnification: 2000X
N.A.: 1.30



Vapor Zone (A980621)

0 0.0005 0.001
Inch



Liquid Zone (A980612)

B1088-25

Figure 25. Microstructure of Transverse Sections of TiC+10%Cb After Exposure With Mo-TZM Alloy to Potassium for 1000 Hours at 1600°F.

Etchant: 80HNO₃+20HF

Magnification: 2000X
N.A.: 1.30

TABLE X. CHEMICAL ANALYSES OF Mo-TZM ALLOY SPECIMENS TESTED WITH CARBOLOY 907,
GRADE 7178, AND TiC+10%Cb SPECIMENS BEFORE AND AFTER EXPOSURE TO POTASSIUM FOR 1,000 HOURS AT 1600°F

Capsule No.	Material Combination	Mo-TZM Alloy Specimen No.	Lab No.	Specimen Location	Chemical Analysis (a,b), ppm		
					C	O	N
BIC-53	Mo-TZM Alloy vs Grade 7178	1037-A7	3006	Liquid	180/240	18/32	1/3
		1037-A8	3007	Vapor	180/190	19/26	2/2
BIC-58	Mo-TZM Alloy vs Carboloy 907	1037-A25	2816	Liquid	280/280	5/16	<1/4
		1037-A26	2817	Vapor	210/220	27/10	5/3
BIC-61	Mo-TZM Alloy vs TiC+10%Cb	1037-A19	2820	Liquid	170/220	40/42	7/7
		1037-A20	2815	Vapor	200/180	11/10	2/2
Control	---	1037-A16	1668	Before Test	230/180	9/22	<1/5

(a) All analyses in duplicate; gas analyses by vacuum fusion techniques; carbon analyses by combustion conductometric.

(b) Entire specimen cross section (0.250 inch x 0.250 inch) was analyzed.

TABLE XI. CHEMICAL ANALYSES OF POTASSIUM TRANSFERRED FROM Cb-12r ALLOY CORROSION CAPSULE CONTAINING CANDIDATE BEARING MATERIAL COMBINATIONS AND EXPOSED FOR 1,000 HOURS AT 1600°F

Capsule No.	Specimen Materials	Date Analyzed	Lab No.	Chemical Analyses (a)																				
				ppm																				
				Al	Ag	Be	Ca	Cb	Co	Cr	Cu	Fe	Mg	Mn	Mo	Na	Ni	Si	Sn	Ti	V	Zr	Pb	B
BIC-51 to 54	---	6-22-65	193	<1	<1	--	5	<1	<1	<1	<1	1	<1	<1	<1	15	<1	5	<5	<1	-	<5	<1	-
BIC-53	Grade 7178 vs Mo-TZM Alloy	9-8-65	222	1	<1	<1	25	5	<1	<1	<1	1	<1	<1	<1	<5	<1	5	<5	<1	<5	<5	<1	-(d)
BIC-58	---	--	-	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
BIC-58	Carboloy 907 vs Mo-TZM Alloy	9-8-65	224	1	<1	<1	25	<5	<1	<1	<1	1	<1	<1	<1	<5	<1	25	<5	<1	<5	<5	<1	-(d)
BIC-55,61,62	---	6-22-65	196	1	<1	--	1	<1	<1	<1	<1	<1	<1	<1	<1	<1	5	<1	1	<5	<1	--	<5	<1
BIC-61	TiC+10%Cb vs Mo-TZM Alloy	9-8-65	223	25	<1	<1	25	5	<1	<1	<1	1	<1	<1	<1	<5	<1	5	<5	25	<5	<5	<1	-(d)

- (a) Metallic impurities in KCl analyzed by spectrographic techniques.
 (b) Pre-test analyses; samples cast inside EB tank under vacuum at same time indicated capsules were filled.
 (c) Sample cast inside EB tank under vacuum at same time indicated capsule was filled; pre-test analyses not performed.
 (d) Below detectable limit.

a 100-gram load on a Tukon hardness machine. The Zircoa 1027 specimens tested at 1200° and 1600°F were the only specimens in which a significant change could be detected. A decrease in hardness was observed in the specimens exposed both to the potassium liquid and vapor, Figure 26. No significant changes in the hardness of Zircoa 1027 are believed to have occurred as a result of the exposure to potassium at 800°F.

B. POTASSIUM WETTING STUDY

All potassium wetting measurements have been completed on the four candidate materials.

1. Test Facility Modification

Several modifications have been made to the potassium wetting apparatus in order to minimize accidental transfer of potassium from one portion of the apparatus to another and to facilitate formation of the potassium drop. The first of these changes was the addition of a stainless steel sheathed chromel-alumel thermocouple for measurement of the temperature of the condenser. The 0.063-inch OD sheath of the thermocouple is sealed into the condenser cooling tube with a Swagelok fitting; the thermocouple junction is positioned approximately at the level of the center of the condenser. This couple is equipped with an ice junction and its output is read, through a switch, on the same potentiometer used for the specimen thermocouple.

The seat of the valve between the main test chamber and the pumping system has been found to be at a slightly lower temperature than the specimen and undoubtedly some potassium has condensed at that location in previous tests. Apparently, appreciable heat is lost through the clamp holding this valve in position. An auxiliary heater has been placed around the valve seat and the power to this heater is adjusted during a wetting run to maintain the seat at a higher temperature than the specimen.

Some difficulty had been encountered in the formation of the potassium drop due to the tendency of the potassium to flow around the tip of the condenser and form a fillet at the braze joint between the stainless steel cooling tube and the Cb-1Zr alloy condenser plate. In an attempt to overcome this problem, the edges of the lower portion of the condenser were sharpened by filing the back side. Although this reduced the tendency of the potassium to flow around the tip, it did not completely eliminate the problem. A convenient solution was found by increasing the distance between the braze joint and the tip of the condenser so that any potassium that flowed around the tip of the condenser could not reach the brazed joint. The cooling tube was shortened by 0.37-inch so that it extended only to about the center of the condenser; that is, to a point 0.63 inch from the tip of the condenser. Details of this modification are obvious by referring to Figures 35 and 36 of Quarterly Progress Report No. 8(6). The orientation of the condenser with respect to the potassium inlet tube has, of course, been maintained with this modification. Since this change was made, very little difficulty has been encountered in the formation of the potassium drop.

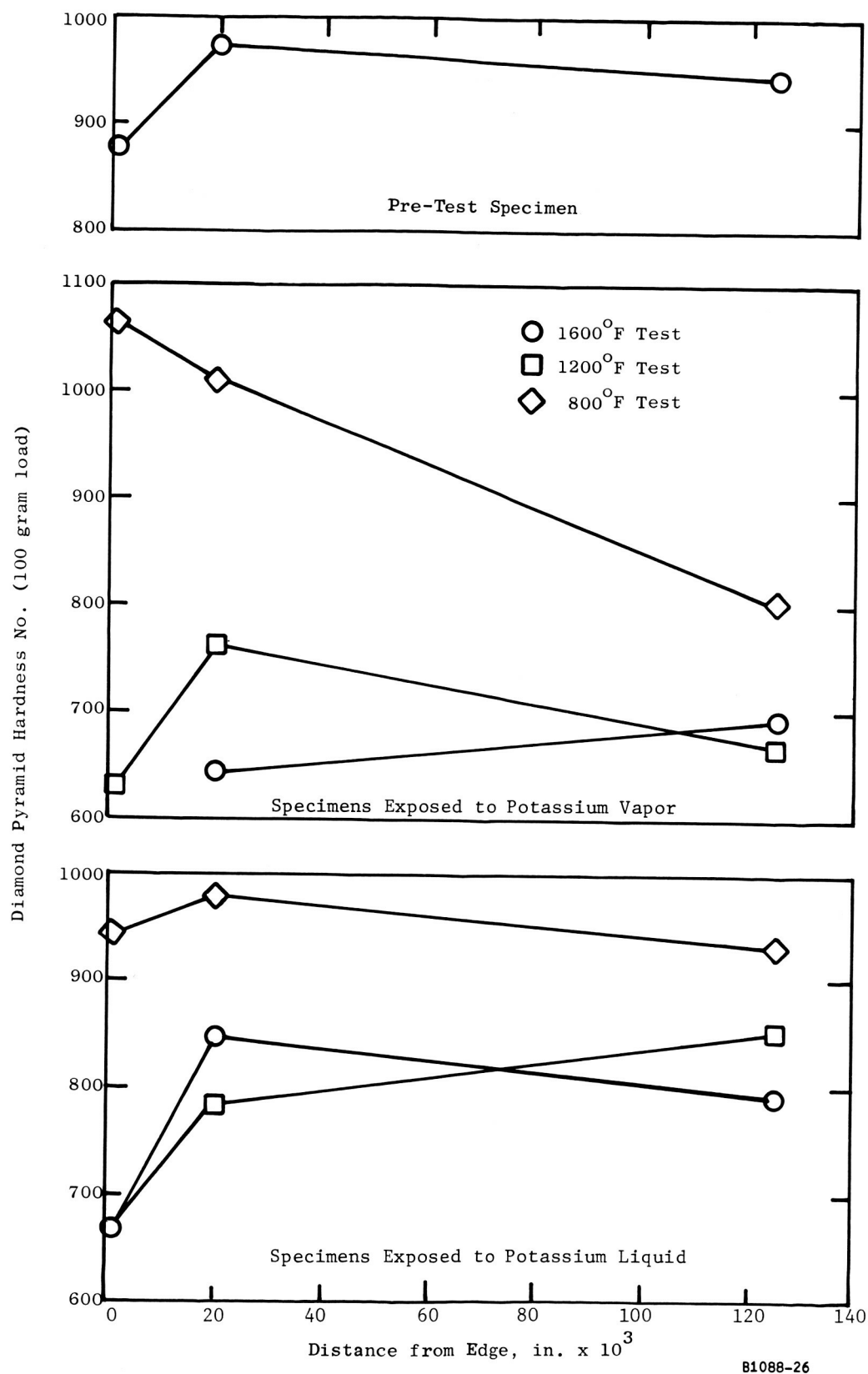


Figure 26. Microhardness Traverses of Transverse Sections of Zircoa 1027, Exposed to Potassium for 1000 Hours.

A photograph of the potassium wetting apparatus is shown in Figure 27. The variable autotransformers, which are located in the upper portion of the instrument cabinet in Figure 27, are to control the power to the auxiliary heaters located at the potassium reservoir, the valve to the main test, the vacuum manifold and the getter-ion pump. The latter two heaters are used during bake-out of the vacuum system. The thermocouple vacuum gauge is used to measure the pressure during the initial evacuation of the system. The pyrometer monitors the temperature as indicated by twelve chromel-alumel thermocouples at various locations on the system. The specimen temperature and the condenser temperature are obtained from two swaged chromel-alumel thermocouples equipped with ice junctions. The outputs of these two thermocouples are measured by the potentiometer through the thermocouple switch. Further details of the construction of the system have been given in Quarterly Progress Report No. 8(6).

A photograph of the apparatus with the oven and camera in place is shown in Figure 28.

2. Experimental Procedure

The upper surface of each of the four wetting test specimens was metallographically polished before conducting the wetting measurements. The surfaces were first ground with 600-grit silicon carbide paper followed by hand polishing on 15, 9, 6 and 0.5-micron diamond paste on micro cloth. Final polishing was accomplished with a Syntron polisher utilizing 0.05-micron Al_2O_3 on a Pre-K paper. The specimens were ultrasonically cleaned using Bendix BIRL solution and given a final rinse with alcohol.

After a specimen was mounted in position and the initial evacuation was concluded, the system was baked out with the valve to the potassium reservoir closed. The bakeout extended for about 30 hours and concluded with a period of six hours with the main test chamber at 350°C . At the end of this period, the discharge gauge indicated a pressure of about 1×10^{-6} torr. Upon cooling to room temperature, the pressure decreased rapidly into the low 10^{-9} torr range. With extended pumping time at room temperature, the pressure could be reduced to about 5×10^{-10} torr.

After the bakeout was completed and with the pressure in the main test chamber in the 10^{-9} torr range, the valve to the potassium reservoir was opened and the valve to the main test chamber was closed. Tap water was passed through the condenser cooling tube at a rate sufficient to maintain the condenser at about 16°C . Then, the oven temperature was increased in order to heat the entire main test section. Subsequently, the potassium in the reservoir was heated by applying power to the auxiliary heater and at the same time maintaining the potassium vapor flow path at a higher temperature than that of the condensed potassium. Under these conditions, potassium was distilled from the reservoir onto the condenser plate. Since the previously described modifications have been made to the condenser, drops have been consistently formed after distilling times of two hours with the potassium reservoir at 220° to 230°C . The calculated quantity of potassium

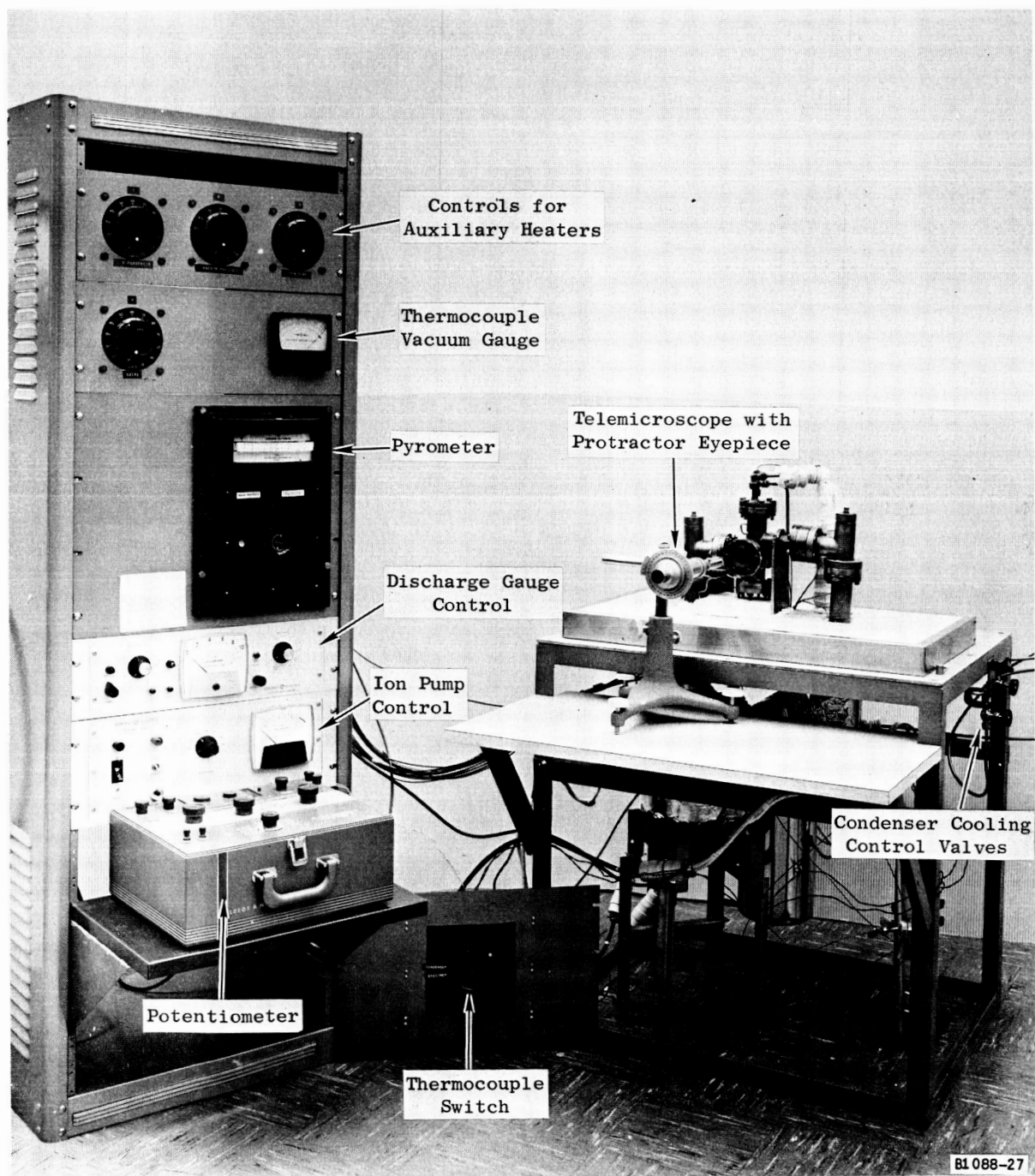


Figure 27. Potassium Wetting Apparatus. (C66011121)

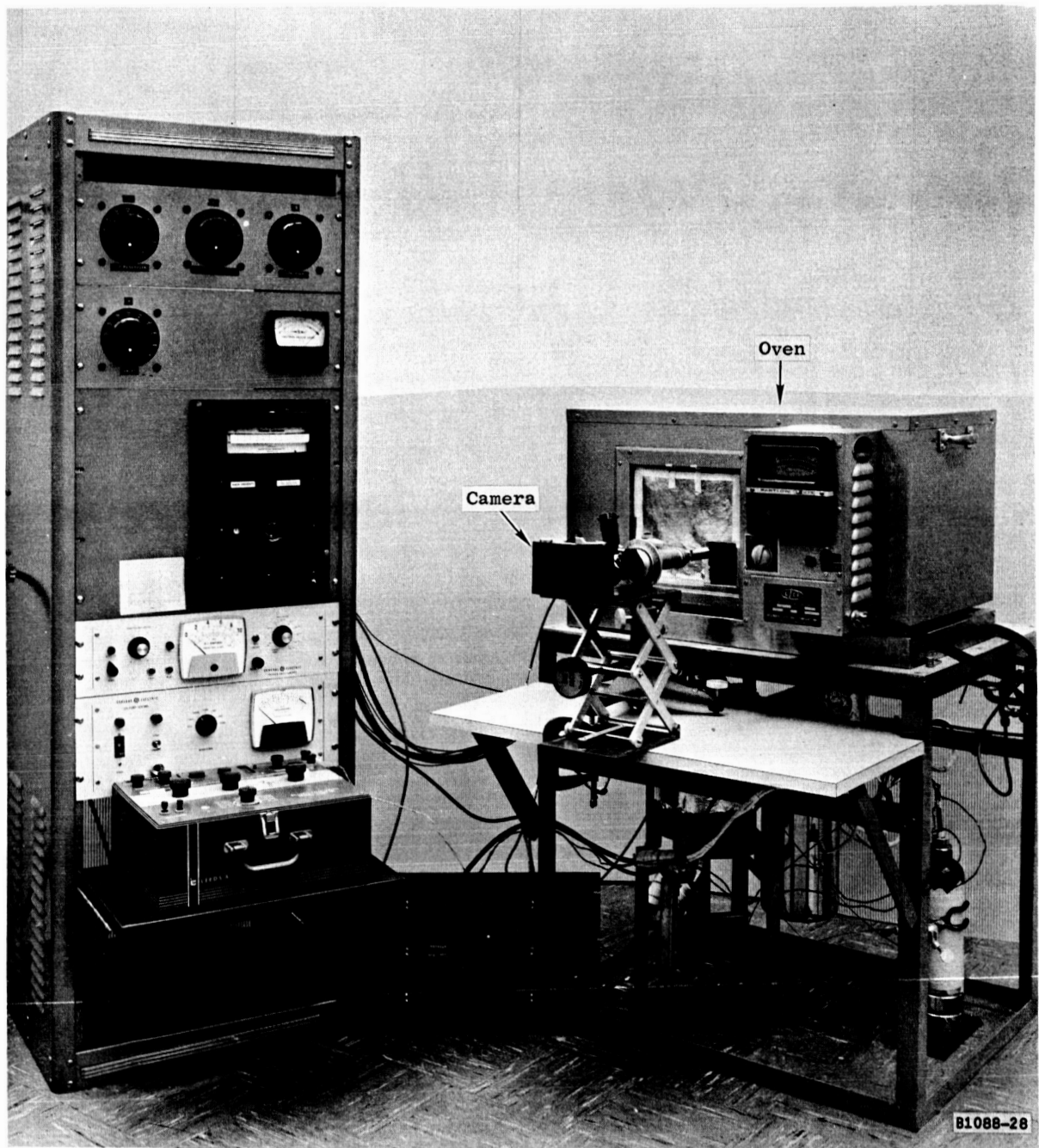


Figure 28. Potassium Wetting Apparatus with Oven and Camera in Place.
(C66011122)

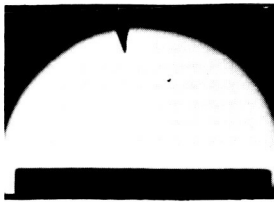
transferred under these conditions was about 0.34 gram. The drops subsequently formed on the specimen have had volumes of 0.2 to 0.3 cc (0.17 to 0.25 gram).

After potassium was distilled to the condenser, the entire system was cooled to room temperature. Then, the condenser cooling water was shut off and room temperature air was passed through the condenser cooling tube. Subsequently, the oven was heated and the air flow to the condenser was controlled at a rate sufficient to maintain the potassium solidified. When the specimen temperature reached about 50°C, the cooling air flow was decreased, and the condenser was allowed to warm up to melt the potassium and form the drop on the specimen.

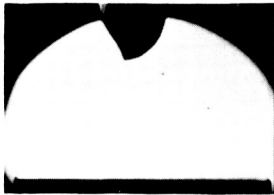
Several photographs obtained during the process of a drop formation are shown in Figure 29. Figure 29a shows the specimen surface with the tip of the condenser just visible at the top of the photograph. At this point, the potassium is condensed on the front surface of the upper portion of the condenser which is not visible. Figure 29b shows the potassium moving toward the tip of the condenser as the potassium melts. In Figure 29c, the potassium has flowed to the rear of the condenser forming a symmetrical agglomeration just prior to dropping. Figure 29d, shows the potassium dropping to the surface of the specimen and in Figure 29e, the potassium has separated from the condenser and solidified on the specimen. It has been found that if the potassium on the condenser is melted rather slowly, the drop will make contact with the specimen while it is still attached to the condenser as shown in Figure 29d; the resulting drop has a sharp tip as in Figure 29e. However, if the potassium is melted quickly, it detaches from the condenser before making contact with the specimen and the solidified drop has a more spherical shape as shown in Figure 31a.

After formation of the drop on the specimen, the specimen temperature continued to increase and melting of the potassium drop was generally observed between 66° and 70°C as compared to the accepted melting point of 63°C. This discrepancy is due to the fact that the thermowell in which the specimen thermocouple is located does not make sufficiently good thermal contact with the specimen and considerable heat flows through the thermowell from the lower flange. The result is that the specimen thermocouple indicates a value between the temperature of the lower flange and that of the specimen. The difference between the thermocouple temperature and the true temperature of the specimen is greater at higher heating rates and also is greater at the lower temperatures. At higher temperatures, significant heat is transferred by radiation between the thermowell and the specimen so that the thermocouple temperature is closer to the true specimen temperature. All specimen temperatures reported here are the temperatures indicated by the specimen thermocouple. No correction has been applied since wetting has not been found to be especially sensitive to temperature.

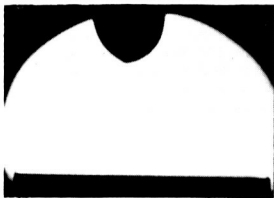
The temperature of the specimen was increased initially at a rate of about 30°C per hour. At temperatures above 190°C and to a maximum temperature of about 230°C, the heating rate was somewhat slower. The total duration of a wetting run from the time the drop melted on the specimen to the time the potassium was completely returned to the reservoir was six to eight hours.



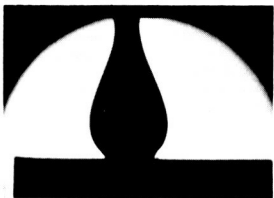
- a. Potassium Condensed as Uniform Film on Condenser, Tip of Condenser is Visible at Top of Photograph.



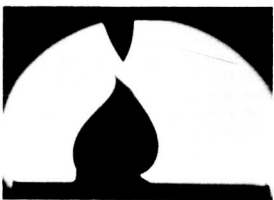
- b. Potassium Melts and Flows to Tip of Condenser.



- c. Liquid Potassium Flows to Backside of Condenser Forming Symmetrical Agglomeration Just Prior to Dropping.



- d. Potassium Drop Forms and Falls to Surface of Specimen.



- e. Potassium Separates from Condenser and Solidifies on Specimen.

B1088-29

Figure 29. Photographic Sequence Showing Formation of Potassium Drop

During the wetting runs, contact angles of the drop were measured with the protractor eyepiece on the telemicroscope at temperature intervals of approximately 10°C. Whenever significant changes in contact angle were observed, the drop was photographed. The optical system distances were set at the same value for each photograph so that the magnification was constant; the magnification of the optical system was found to be 1.57 X.

The temperature of the condenser, as indicated by the condenser thermocouple, was compared frequently to the specimen temperature during each wetting run. The condenser temperature was adjusted by varying the air flow through the condenser cooling tube. Although transfer of potassium between the specimen and condenser could be reduced more or less, depending upon how much care was exercised, it was impossible to completely prevent the transfer of potassium by employing manual control. As a consequence, the wetting run involved alternating periods during which either potassium was condensing on the specimen or the drop was evaporating depending upon whether the condenser temperature fluctuated above or below the specimen temperature. In some cases, the condenser was allowed to warm-up in order to completely coat the specimen with potassium. When this occurred, spreading of the drop and a decrease in contact angle generally was observed.

3. Wetting of Carboloy 907

Three separate wetting runs have been made on Carboloy 907. Photographs from the first run are shown in Figure 30 and complete wetting data for this run are given in Table XII. The first column of Table XII gives the time in minutes from the point at which the drop melted on the specimen; the second column lists the specimen temperature; the third column lists the contact angle; and the fourth column shows the diameter of the liquid-solid interface. The entry in the latter column is obtained by measuring the diameter on the photograph and dividing by the magnification. The interfacial diameter indicates whether the drop is spreading or the interface is receding. The fifth column of Table II gives the drop volume as calculated from the maximum height of the drop and the interfacial diameter. In this calculation, the drop is assumed to be a spherical segment in order to simplify the calculation. Actually, the drop is slightly flattened due to the gravitational force and, thus, the calculated volume is slightly less than the true volume. However, the entries in the fifth column give some idea of the extent of evaporation. The sixth column of Table XII shows the figure number from which the drop dimensions were obtained.

A photograph of the solid drop on the surface of the Carboloy 907 specimen taken during Run #1 at 25°C is shown in Figure 30a. At the melting point, a contact angle of 115° was attained and this angle remained essentially constant until the temperature reached 160°C, (Figures 30b and 30c). Between 160° and 193°C, the contact angle decreased to 101° as shown in Figures 30d, 30e, and 30f. Although the decrease in contact angle was due mainly to evaporation of the drop, slight spreading was observed as noted in Table XII. At about 200°C, a sudden decrease in the contact angle to 72° was observed,

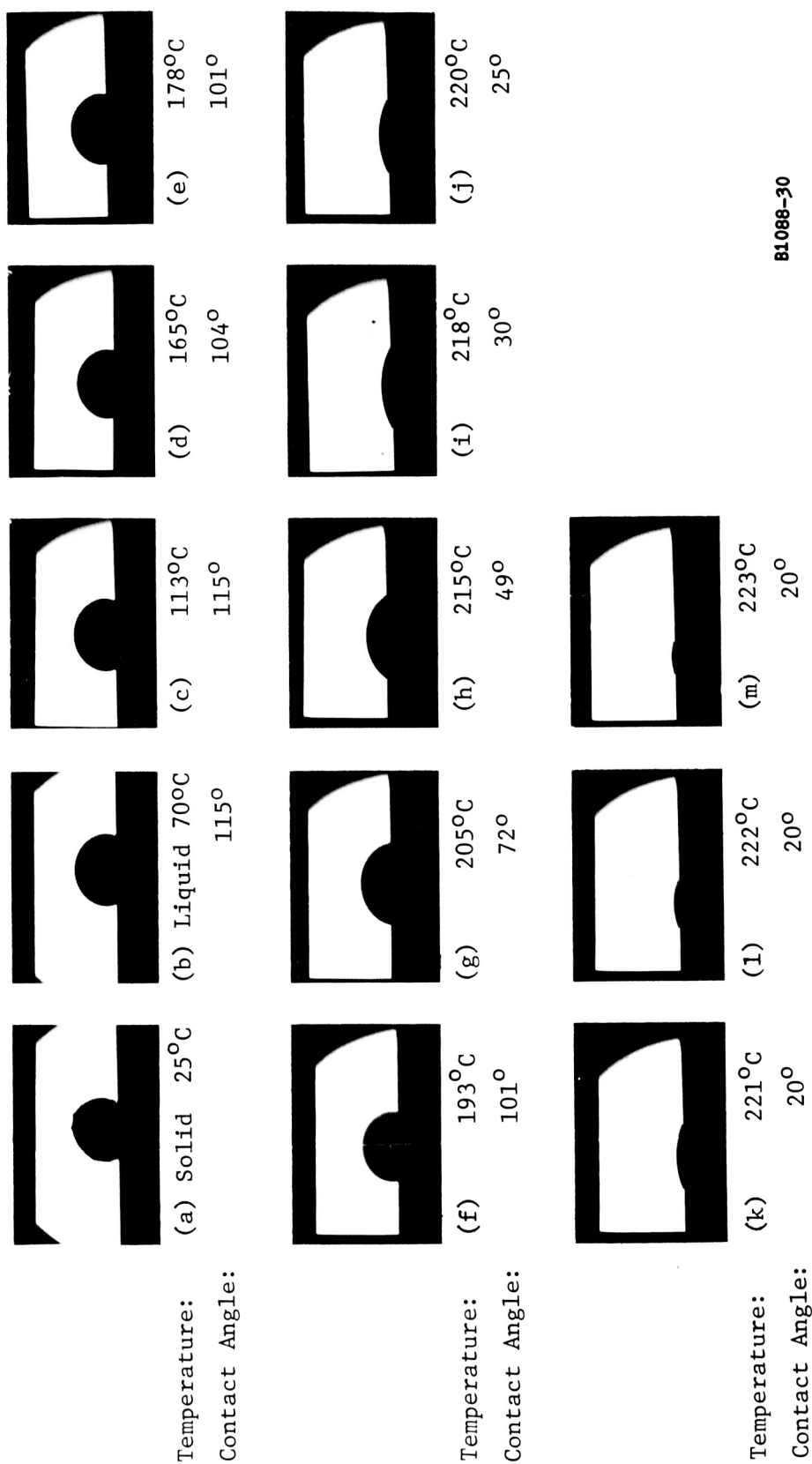


Figure 30. Sequence of Photographs of Initial Potassium Drop on Carboloy 907.
(Run #1).

Magnification, 1.57X

TABLE XII. WETTING DATA FOR CARBOLOY 907 - RUN #1

<u>Time From Drop Melting Minutes</u>	<u>Specimen Temperature °C</u>	<u>Contact Angle Degrees</u>	<u>Interface^(a) Diameter cm</u>	<u>Drop^(b) Volume cc</u>	<u>Figure Number</u>	<u>Remarks</u>
10	70	115	0.66	0.108	30b	
25	79	115				
60	97	115				
90	113	115	0.66	0.108	30c	
110	124	115				
140	136	121				
175	157	122				
190	165	104	0.66	0.084	30d	Evaporation
195	169	104				
210	175	101				
220	178	101	0.67	0.082	30e	Spreading
265	193	101	0.67	0.081	30f	
295	205	72	0.79	0.080	30g	Spreading
310	209	68				
325	213	60				
332	215	49	0.08	0.051	30h	Evaporation
340	218	30	0.78	0.024	30i	Evaporation
348	220	25	0.71	0.018	30j	Receding
352	221	20	0.65	0.011	30k	Receding
357	222	20	0.48	0.005	30l	Receding
361	223	20	0.34	0.002	30m	Receding

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated assuming drop is spherical segment.

Figure 30g. This decrease in contact angle is due to spreading of the drop, as indicated by the increase in diameter of the liquid-solid interface, with little change in volume. As the temperature increased further, evaporation of the drop became quite rapid and resulted in a decrease in contact angles to a minimum value of 20°.

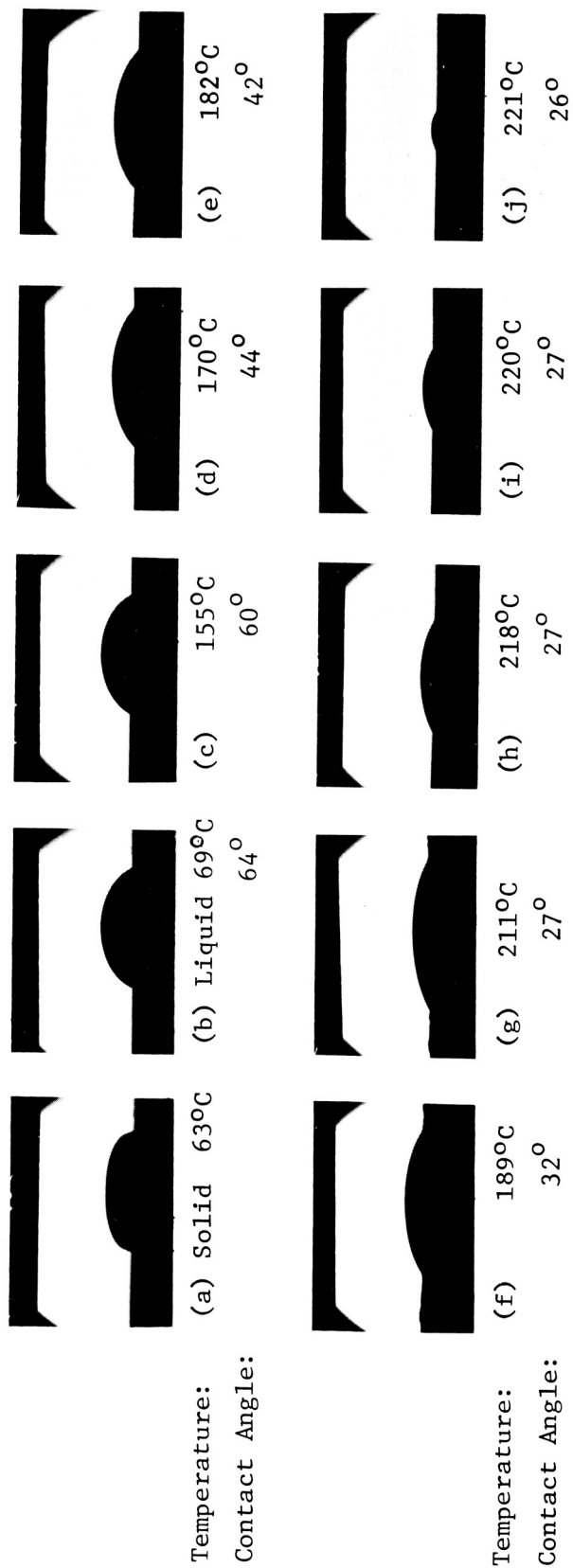
A similar series of photographs taken during Run #2 on the Carboloy 907 specimen are shown in Figure 31; complete data for this run are given in Table XIII. Figure 31a shows the solid drop at 63°C. When the drop melted, the contact angle was 64°, Figure 31b. The contact angle remained essentially constant as the temperature was increased until the temperature reached about 160°C. At this temperature, potassium condensation was observed on the specimen surface and the contact angle decreased to 44° as a result of spreading of the drop as shown in Figure 31d. The condenser was allowed to heat-up in order to thoroughly coat the specimen with potassium. The small droplets of potassium are clearly visible on the surface in Figure 31f. Further spreading of the drop occurred until the maximum interfacial diameter was reached at 211°C. As the temperature continued to increase, the interface receded and a constant contact angle of 27° was maintained.

Representative photographs from Run #3 for Carboloy 907 are shown in Figure 32 and Table XIV; these results are very similar to the results obtained in Run #2. The advancing contact angle is 54° and is constant between the melting point and about 150°C. Potassium condensation on the specimen surface at a temperature of 183°C is visible in Figure 32e. The minimum contact angle observed during evaporation of the drop was 20°.

4. Wetting of TiC+10%Cb

Two wetting test runs were made on a metallographically prepared surface of a TiC+10%Cb specimen. Photographs from the first wetting test run on TiC+10%Cb are shown in Figure 33 and complete data for this run are given in Table XV. As the specimen temperature was increased for the TiC+10%Cb Run #1, the contact angle remained essentially constant at 66° until the temperature reached 120°C. At this temperature, the drop spread out resulting in a reduction in the contact angle to 46°. As the temperature was increased above 120°C, gradual spreading of the drop occurred until, at a temperature of 173°C, potassium from the condenser began condensing on the specimen resulting in a significantly greater spreading of the drop. The maximum interfacial diameter was reached at 173°C and remained essentially constant, as the temperature increased and as evaporation continued, until the minimum contact angle of 6° was reached. Subsequently, the interface began receding and continued to do so until the drop completely evaporated.

Photographs obtained during the TiC+10%Cb Run #2 are shown in Figure 34 and the data for this run are given in Table XVI. Figure 34a shows the solid drop at 63°C and Figures 34b and 34c show the drop spreading as it melts. The initial advancing contact angle was 36° and remained constant until the temperature reached 131°C. A further increase in temperature resulted in spreading of the drop until the maximum interfacial diameter was obtained at 196°C. As the drop completely evaporated, the minimum receding contact angle was 6°.



81088-31
 Figure 31. Sequence of Photographs of Potassium Drop on Previously Wet Surface of Carboloy 907. (Run #2).

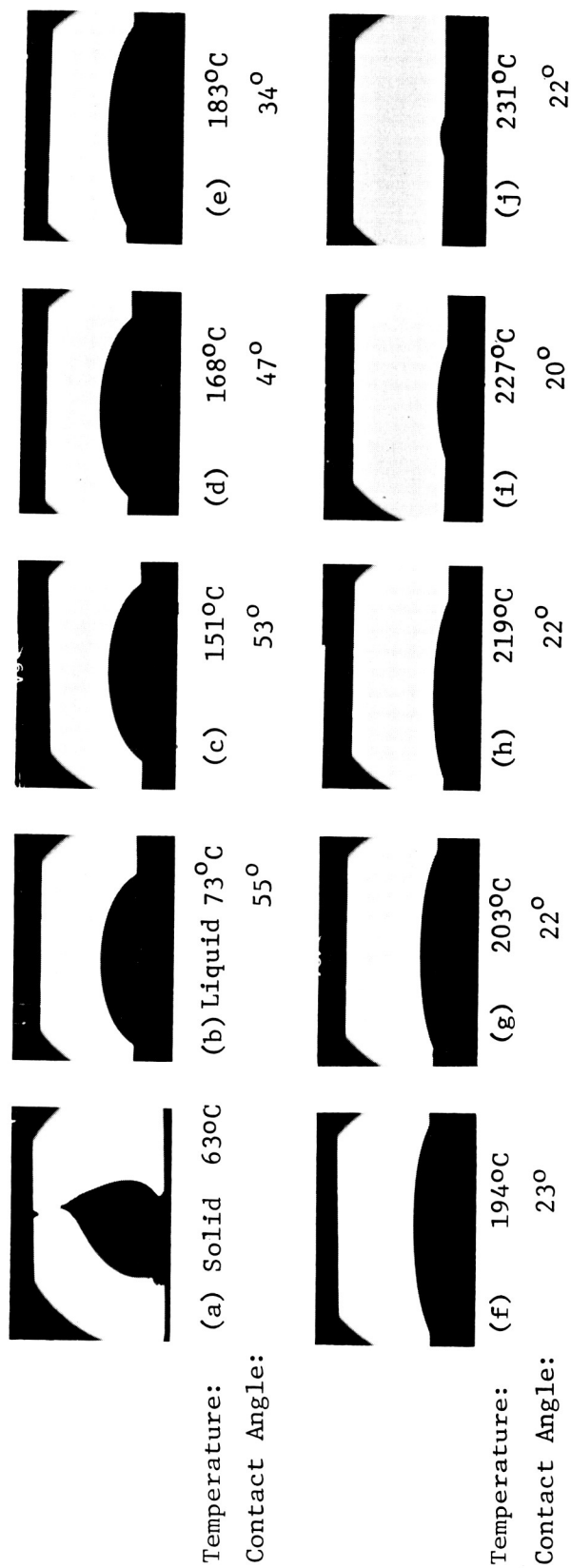
Magnification: 1.57X

TABLE XIII. WETTING DATA FOR CARBOLOY 907 - RUN #2

Time From Drop Melting Minutes	Specimen Temperature °C	Contact Angle Degrees	Interface ^(a) Diameter cm	Drop ^(b) Volume cc	Figure Number	Remarks
10	69	64	1.08	0.134	31b	
20	74	61				
50	87	59				
80	103	59				
110	121	59				
140	137	59				
170	155	60	1.08	0.125	31c	
200	170	44	1.25	0.122	31d	Condensation on specimen - spreading
230	182	42	1.25	0.125	31e	
240	187	34				
247	189	32	1.28	0.102	31f	
255	192	32				
267	196	32				
290	204	36				
320	211	27	1.35	0.102	31g	
350	218	27	1.02	0.046	31h	Receding
365	220	27	0.72	0.017	31i	Receding
375	221	26	0.36	0.002	31j	Receding

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated assuming drop is spherical segment.



81088-32

Figure 32. Sequence of Photographs of Potassium Drop on Previously Wet Surface of Carboloy 907. (Run #3).

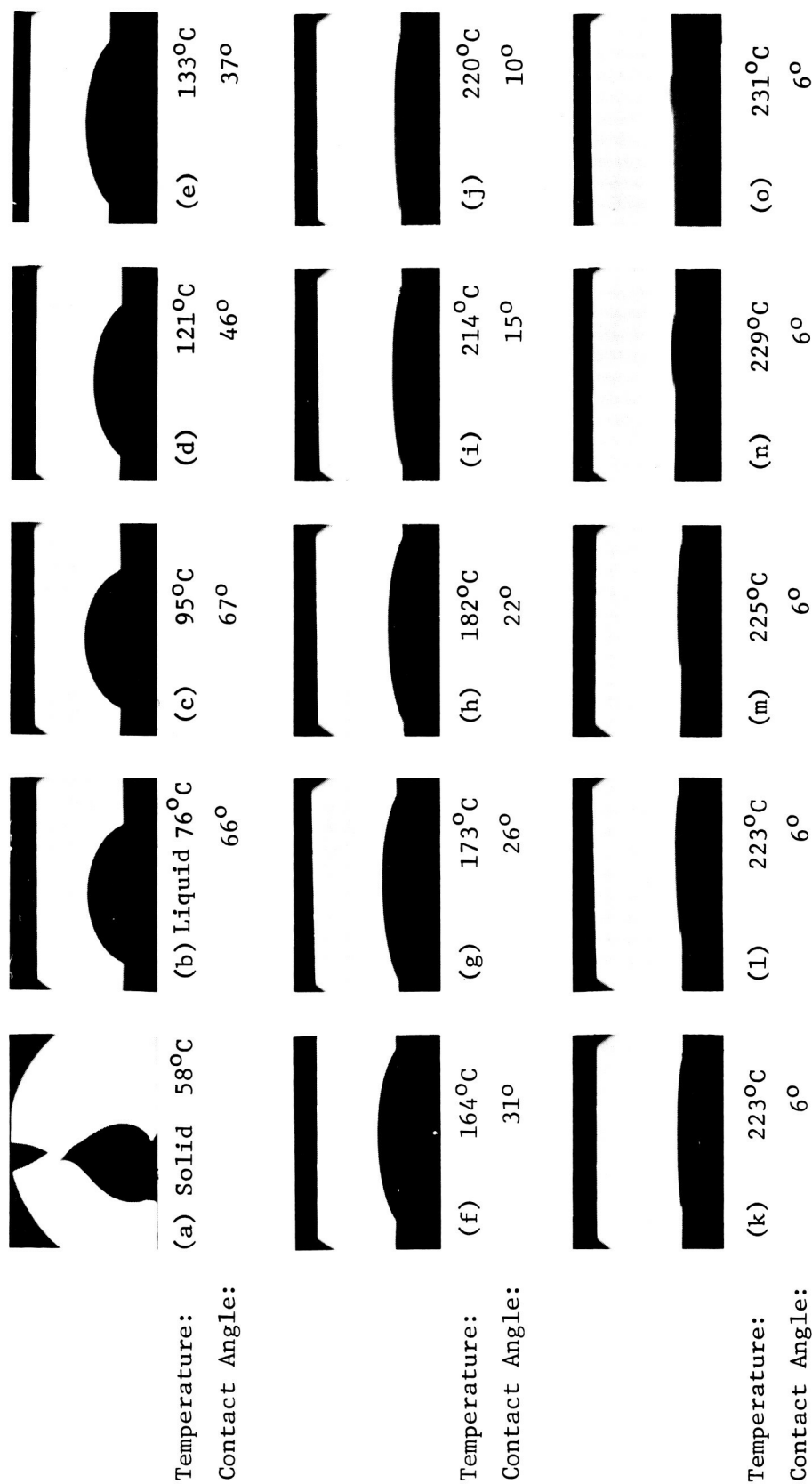
Magnification: 1.57X

TABLE XIV. WETTING DATA FOR CARBOLOY 907 - Run #3

<u>Time From Drop Melting Minutes</u>	<u>Temperature °C</u>	<u>Contact Angle Degrees</u>	<u>Interface^(a) Diameter cm</u>	<u>Drop^(b) Volume cc</u>	<u>Figure Number</u>	<u>Remarks</u>
10	73	55	1.52	0.29	32b	
35	85	55				
90	111	56				
125	134	53				
165	151	53	1.59	0.30	32c	
195	168	47	1.59	0.27	32d	
225	183	34	1.78	0.25	32e	Condensation on specimen - spreading
255	194	23	1.85	0.19	32f	Specimen clea
285	203	22	1.77	0.17	32g	Condensation on specimen
315	211	22				
345	219	22	1.59	0.109	32h	Receding
375	227	20	1.01	0.034	32i	Receding
405	231	22	0.36	0.002	32j	Receding

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated assuming drop is spherical segment.



81088-33

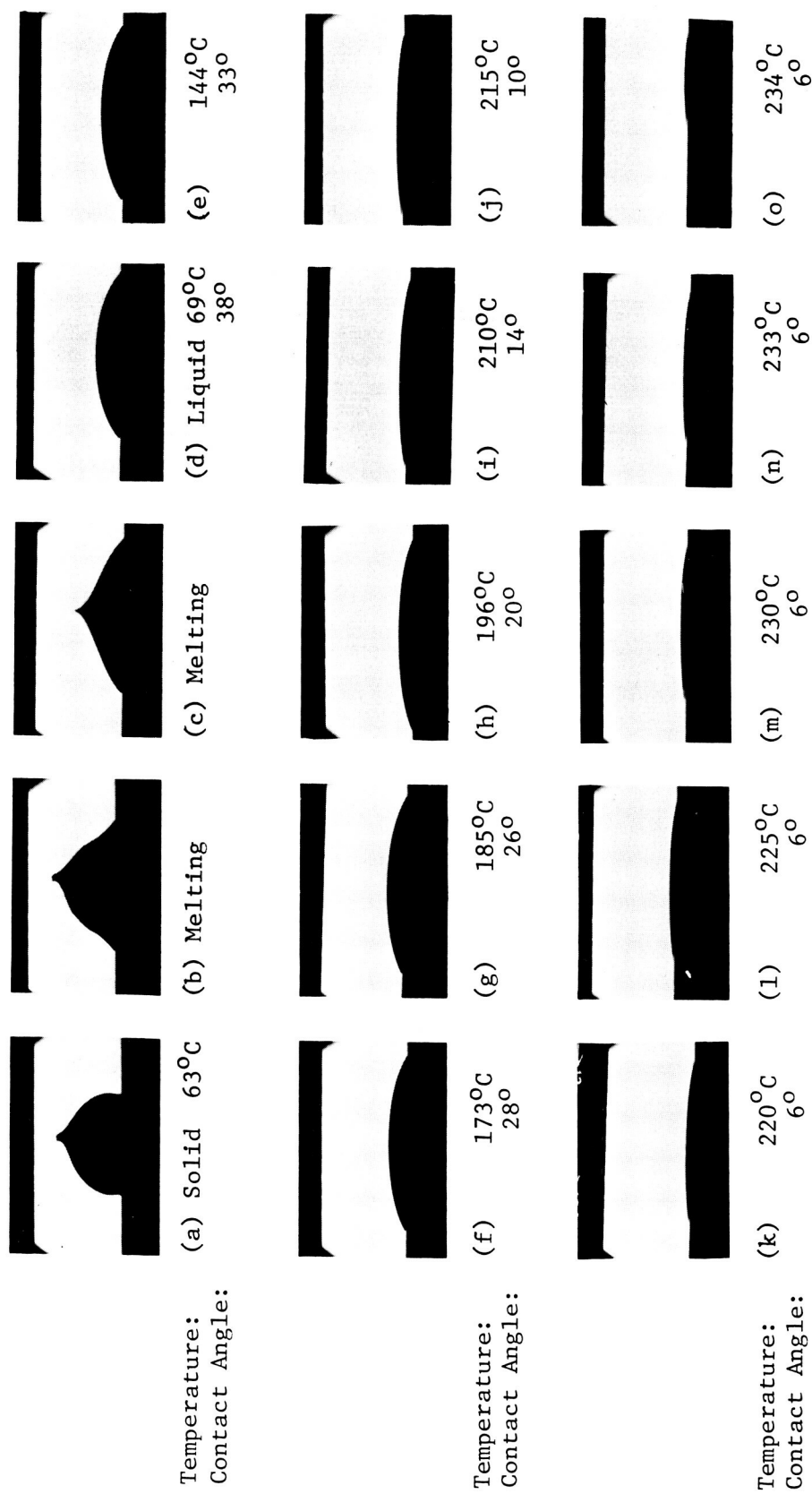
Figure 33. Sequence of Photographs of Initial Potassium Drop on TiC+10%Cb (Run #1).
Magnification, 1.57X

TABLE XV. WETTING DATA FOR TiC+10%Cb - RUN #1

Time From Drop Melting Minutes	Temperature °C	Contact Angle Degrees	Interface ^(a) Diameter cm	Drop ^(b) Volume cc	Figure Number	Remarks
15	76	66	1.31	0.25	33b	
40	95	67	1.31	0.25	33c	
75	114	66				
85	121	46	1.41	0.21	33d	Spreading
105	133	37	1.55	0.21	33e	Spreading
145	149	38				
170	159	38				
180	164	31	1.64	0.19	33f	Spreading
210	173	26	1.77	0.18	33g	Condensation specimen - spreading
240	182	22	1.77	0.18	33h	Condensation specimen
270	196	23				Condensation specimen
290	204	22				Specimen clea
320	214	15	1.75	0.107	33i	Evaporation
340	220	10	1.75	0.069	33j	Evaporation
350	223	6	1.52	0.040	33k	Receding
355	223	6	1.41	0.035	33l	Receding
360	225	6	1.27	0.026	33m	Receding
380	229	6	0.74	0.007	33n	Receding
390	231	6	0.48	0.002	33o	Receding

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated by assuming drop is spherical.



81088-34

Figure 34. Sequence of Photographs of Potassium Drop on Previously Wet Surface of TiC+10%Cb (Run #2).
Magnification, 1.57X

TABLE XVI. WETTING DATA FOR TiC+10%Cb - RUN #2

Time From Drop Melting Minutes	Specimen Temperature °C	Contact Angle Degrees	Interface ^(a) Diameter cm	Drop ^(b) Volume cc	Figure Number	Remarks
5	69	38	1.59	0.24	34d	
35	80	34				
70	100	36				
100	115	37				
130	131	37				
160	144	33	1.67	0.23	34e	Spreading
190	156	31				
230	173	28	1.67	0.19	34f	
260	185	26	1.71	0.19	34g	Spreading
285	194	20				
290	196	20	1.81	0.17	34h	Spreading
310	202	21				
330	210	14	1.81	0.103	34i	Evaporation
350	215	10	1.80	0.083	34j	Evaporation
370	221	8	1.54	0.053	34k	Receding
390	225	6	1.52	0.049	34l	Receding
410	230	6	1.49	0.047	34m	Receding
420	233	6	1.47	0.038	34n	Receding
425	234	6	0.92	0.010	34o	Receding

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated by assuming drop is spherical.

5. Wetting of Grade 7178

Two wetting runs also were made on a metallographically prepared surface of a Grade 7178 specimen. Photographs from the first run are shown in Figure 35 and complete data are given in Table XVII. The initial advancing contact angle was 52° and remained constant until the temperature reached 157°C . Between 157° and 192°C , a slight evaporation of the drop occurred resulting in a decrease in the contact angle to 42° . No spreading occurred until, at 197°C , the remaining potassium on the condenser was allowed to evaporate and condense on the specimen. This resulted in considerable spreading with the drop interface moving nearly to the edge of the specimen as shown in Figure 35i. The maximum interfacial diameter was reached at 227°C . As the drop evaporated, a minimum receding contact angle of 3° was observed.

Photographs obtained during the Grade 7178 Run #2 are shown in Figure 36 and complete data for this run are given in Table XVIII. The initial advancing contact angle was 31° . In this run, the drop was maintained at a temperature of about 82°C for 16 hours. During this period, slight spreading and evaporation occurred. No further spreading was observed as the temperature was increased and, as evaporation of the drop occurred, a minimum receding contact angle of 5° was observed.

6. Wetting of Mo-TZM Alloy

Preliminary measurements on the Mo-TZM alloy surface have been previously reported⁽¹⁰⁾. These measurements showed an initial advancing contact angle of 135° and a receding contact angle which was estimated to be less than 10° . Since these initial measurements were performed, several modifications to the apparatus have been made to facilitate control of the experimental variables and a consistent operating procedure has been established as described in the present report. Therefore, two additional wetting runs have been made on Mo-TZM alloy.

Prior to these additional measurements, the specimen was thoroughly cleaned and the upper surface was repolished by hand using 0.05-micron alumina on microcloth. Photographs from the first run on the repolished surface (Mo-TZM alloy Run #4) are shown in Figure 37 and complete data for this run are given in Table XIX. The initial advancing contact angle was 47° as shown in Figure 37b. As the temperature was increased, the contact angle remained constant until, at 130°C , the drop suddenly spread out and attained a contact angle of 27° . This spreading was due to a small amount of potassium which condensed on the upper surface. As the temperature was increased further, the contact angle decreased due to slow evaporation of the drop with the interfacial diameter remaining constant. When the specimen temperature reached 201°C , the condenser temperature was permitted to increase and the residual potassium evaporated from the condenser and condensed on the specimen. This caused the drop to spread nearly to the edge of the specimen and the contact angle was so small that the location of the liquid-solid interface could not be accurately determined. In Figures 37g

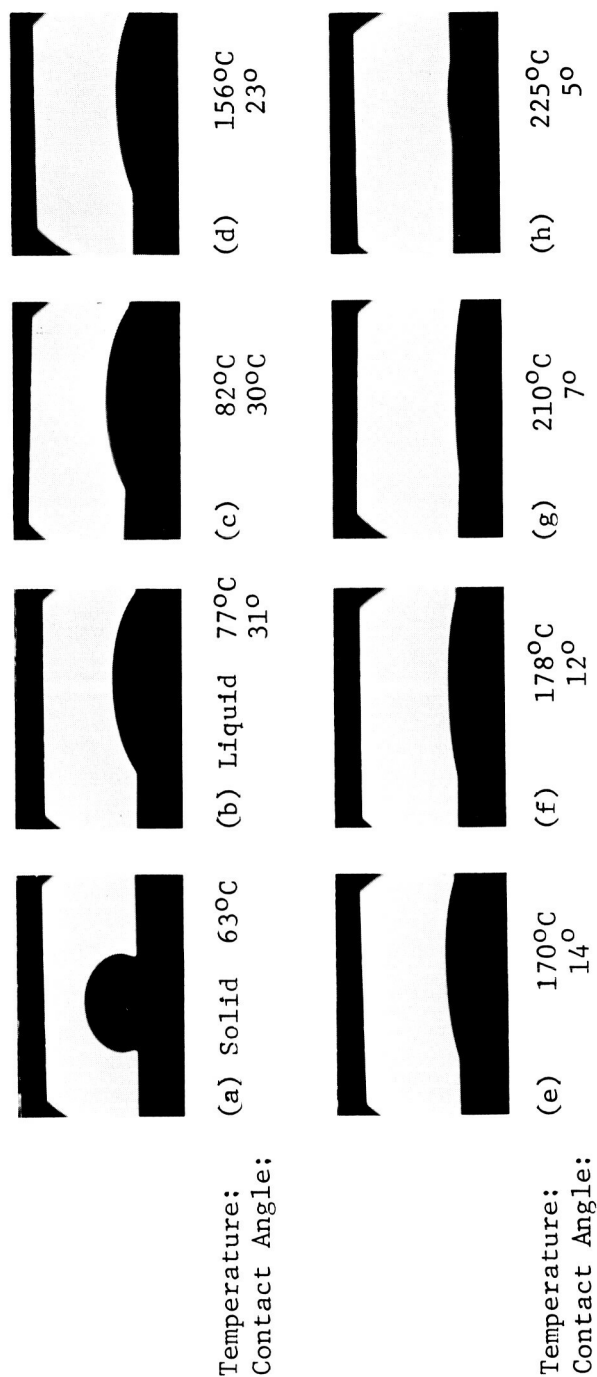
Figure 35. Sequence of Photographs of Initial Potassium Drop on Grade 7178 (Run #1).
Magnification, 1.57X

TABLE XVII. WETTING DATA FOR GRADE 7178 - RUN #1

Time From Drop Melting Minutes	Specimen Temperature °C	Contact Angle Degrees	Interface ^(a) Diameter cm	Drop ^(b) Volume cc	Figure Number	Remarks
10	72	51	1.50	0.31	35a	
30	80	52				
60	97	52				
85	111	53				
110	126	53	1.50	0.31	35b	
135	141	53				
175	157	52	1.50	0.29	35c	Evaporation
205	172	49				
240	187	44	1.50	0.25	35d	Evaporation
252	192	42	1.49	0.22	35e	Evaporation
265	197	25	1.77	0.17	35f	Condensation on specimen - spreading
275	200	17	1.87	0.15	35g	Spreading
280	202	16	1.97	0.17	35h	Spreading
305	211	17				
335	220	16				
350	225	13				
360	227	12	2.03	0.13	35i	Spreading
370	231	9				
380	232	9	2.00	0.070	35j	Evaporation
390	234	3	1.62	0.039	35k	Receding

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated by assuming drop is spherical.



B1088-36

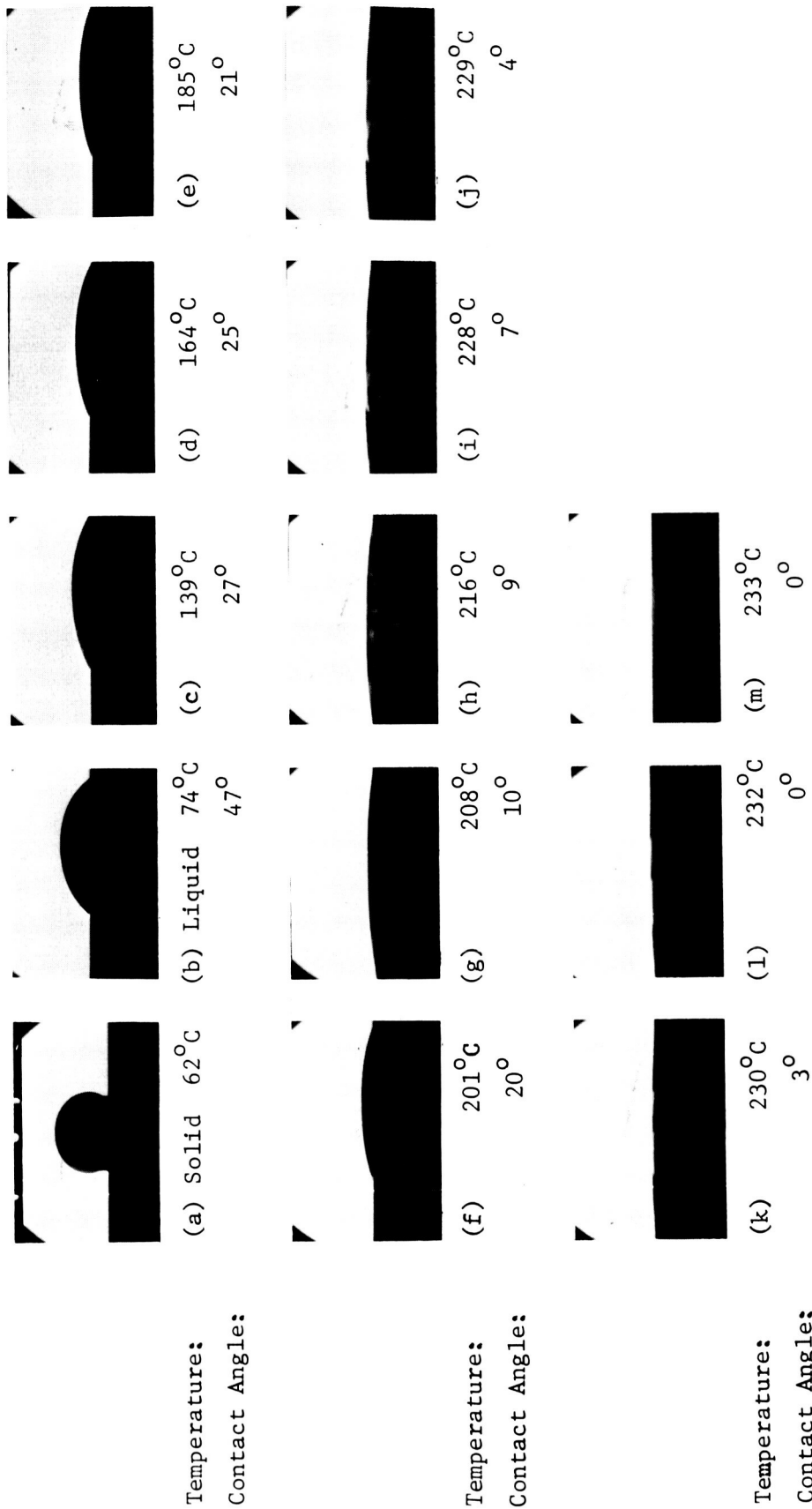
Figure 36. Sequence of Photographs of Potassium Drop on Previously Wet Surface of Grade 7178 (Run #2).
Magnification, 1.57X

TABLE XVIII. WETTING DATA FOR GRADE 7178 - RUN #2

Time From Onset of Melting Minutes	Specimen Temperature °C	Contact Angle Degrees	Interface ^(a) Diameter cm	Drop ^(b) Volume cc	Figure Number	Remarks
70	77	31	1.54	0.19	36b	
90	78	31				
1040	82	30	1.57	0.18	36c	Slight spread- ing after 16 hours
1135	97	29				
1160	108	29				
1210	130	29				
1265	156	23	1.53	0.12	36d	Evaporation
1310	170	14	1.53	0.088	36e	Evaporation
1330	178	12	1.53	0.065	36f	Evaporation
1360	189	9				
1380	200	9				
1420	210	7	1.45	0.039	36g	Receding
1460	225	5	0.070	0.004	36h	Receding

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated by assuming drop is spherical.



81088-37

Figure 37. Sequence of Photographs of Initial Potassium Drop on Mo-TZM Alloy (Run #4)
After Repolishing the Surface.

Magnification: 1.57X

TABLE XIX. WETTING DATA FOR Mo-TZM - RUN #4 - REPOLISHED SURFACE

Time From Drop Melting Minutes	Specimen Temperature °C	Contact Angle Degrees	Interface ^(a) Diameter cm	Drop ^(b) Volume cc	Figure Number	Remarks
5	74	47	1.31	0.201	37b	
35	88	48				
50	98	47				
75	113	47				
100	129	48				
117	139	27	1.48	0.148	37c	Condensation on specimen - spreading
128	145	27				
155	157	26				
175	164	25	1.48	0.131	37d	Evaporation
205	175	24				
230	185	21	1.48	0.110	37e	Evaporation
260	195	20				
275	201	20	1.48	0.091	37f	Evaporation
290	208	10	Liquid-Solid Interface Could Not Be Located		37g	Condensation on specimen - spreading
325	216	9			37h	
375	228	7			37i	
380	229	4			37j	
387	230	3			37k	
395	232	0			37l	
400	233	0			37m	

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated assuming drop is spherical.

and 37h, the condensed potassium is visible on the front surface and on the upper surface of the specimen. Since the interface could not be accurately located, the last seven tabulated contact angles in Table XIX are approximate. The receding contact angle is essentially zero, as shown in Figure 37m. When the quantity of potassium on the surface became small, it was very difficult to determine whether or not there was any potassium present.

Photographs obtained during the subsequent wetting measurements on the previously wet Mo-TZM surface (Mo-TZM alloy Run #5) are shown in Figure 38 and complete data for this run are given in Table XX. When the drop melted, the contact angle was 31° as shown in Figure 38b. As the temperature was increased, the interfacial diameter remained constant with a small decrease in contact angle due to evaporation. At 168°C , a small amount of potassium condensed on the surface and the drop suddenly spread with a concurrent decrease in contact angle to 19° , Figure 38d. As the temperature increased further, no additional spreading of the drop occurred and the contact angle decreased as the drop evaporated, Figures 38e through 38m. Similar to the previous run, the liquid-solid interface was difficult to locate as the contact angle approached zero.

7. Summary of Results and Discussion

From a consideration of all measurements made on the four candidate bearing materials, the general behavior of wetting of these surfaces by potassium drops is as follows:

1. The largest contact angle for a particular material is observed when the drop first melts on a freshly prepared surface (advancing contact angle).
2. As the specimen is heated, there is a definite tendency for spreading to occur. Spreading can often be induced by allowing additional potassium to condense on the surface. In a few cases, spreading was apparently spontaneous. In general, the increase in interfacial diameter was sudden rather than a gradual spreading of the drop.
3. Evaporation of the drop results in a decrease in contact angle with little change in interfacial diameter until a minimum angle is reached at which point the interface recedes with continued evaporation.
4. When the first potassium drop is removed from the surface and a second drop is placed without exposure of the surface to the atmosphere, the initial advancing contact angle for the second drop is considerably less than that observed for the first drop.

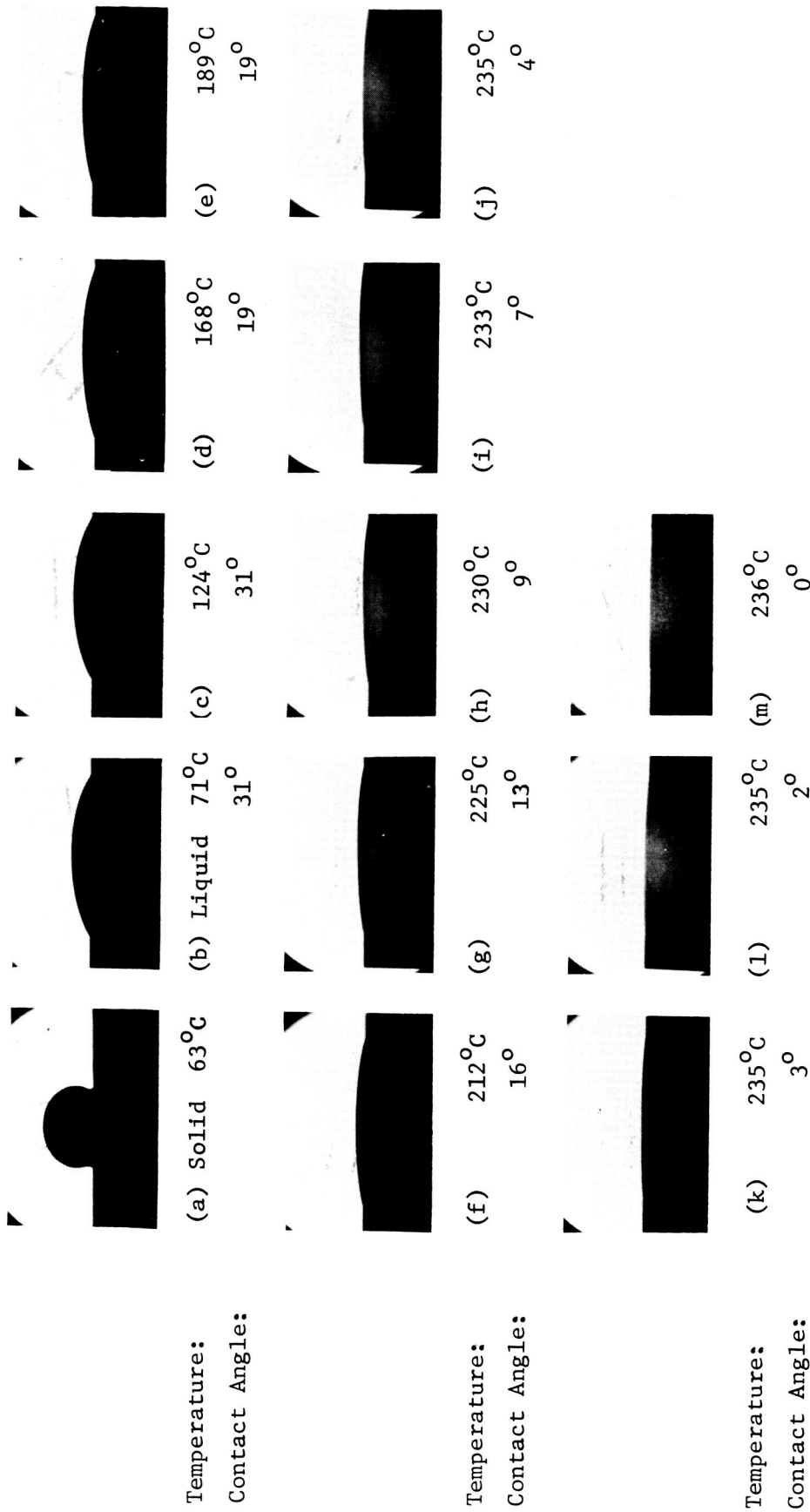


Figure 38. Sequence of Photographs of Potassium Drop on Previously Wet Surface of Mo-TZM Alloy (Run #5).

Magnification: 1.57X

TABLE XX. WETTING DATA FOR Mo-TZM - RUN #5

<u>Time From Drop Melting Minutes</u>	<u>Specimen Temperature °C</u>	<u>Contact Angle Degrees</u>	<u>Interface^(a) Diameter cm</u>	<u>Drop^(b) Volume cc</u>	<u>Figure Number</u>	<u>Remarks</u>
5	71	31	1.57	0.178	38b	
35	87	31				
55	97	31				
75	107	31				
100	124	31	1.57	0.175	38c	
125	135	31				
155	147	31				
180	156	29				
200	164	28				
212	168	19	1.64	0.129	38d	Condensation on specimen - spreading
220	172	19				
240	179	18				
260	189	19	1.64	0.116	38e	Evaporation
280	197	17				
300	204	17				
320	212	16	1.64	0.085	38f	Evaporation
340	218	14				
360	225	13	1.61	0.061	38g	Evaporation
375	230	9	1.61	0.052	38h	Evaporation
385	233	7	1.59	0.041	38i	Evaporation
392	235	4	1.59	0.040	38j	Evaporation
397	235	3	1.57	0.031	38k	Evaporation
398	235	2	1.50	0.017	38l	Receding
400	236	0	1.31	0.006	38m	Receding

(a) Drop dimensions obtained from photograph. Mag. 1.57X.

(b) Drop volume calculated assuming drop is spherical.

The four different materials may be compared on the basis of the initial advancing contact angle on a freshly prepared surface, the initial advancing contact angle on a previously wet surface and the receding contact angle. This comparison is illustrated in the data presented in Table XXI. The initial advancing contact angles were observed shortly after the drop melted, the surface temperature being about 65°C. Receding contact angles were obtained over a range of temperature near 230°C. It may be seen from the data of Table XXI that the materials with the lower receding contact angle also exhibit lower advancing contact angles. The outstanding exception to this is the initial advancing contact angle observed on the first run on Mo-TZM alloy⁽¹⁰⁾. The advancing contact angle measured for this run (135°) was the largest contact angle observed in these tests, yet the receding angle was essentially zero. However, this specimen had been subjected to repeated bakeouts and exposures to the atmosphere during the early checkout of the system and visible discoloration of the surface had occurred. After repolishing of the surface, a considerably lower advancing contact angle was obtained (Mo-TZM alloy Run #4).

Of the four materials investigated, the Mo-TZM alloy specimen exhibited the best wetting characteristics, with receding contact angles of essentially zero degrees; Carboloy 907 had the highest receding contact angles (about 22°). There probably is some significance to the fact that the three carbide compositions had higher receding contact angles than Mo-TZM alloy, the only metal alloy in the group.

The relatively large differences in advancing contact angle between a potassium drop initially wetting a freshly prepared surface and a potassium drop on a previously wet surface indicates that some modification of the surface occurs upon the initial contact with potassium. A photograph of the surface of the TiC+10%Cb specimen after it was removed from the system following the two wetting runs is shown in Figure 39. The central portion of the surface where the drops had been located is relatively clean and the surrounding area is coated with a deposit. The circular areas within the central region are light reflections from the camera lens.

A possible explanation for the surface appearance of the TiC+10%Cb specimen is that the surface is initially coated with an invisible oxide film which is reduced upon contact with liquid potassium. When the drop is subsequently evaporated, the oxygen is deposited as potassium oxide at the point where the last bit of potassium evaporates as indicated in Figure 39. Since the area surrounding the central region has been uniformly coated with potassium, a fairly uniform deposit of potassium oxide remains in this area after evaporation. A further indication that the deposit is potassium oxide (or at least contains potassium) is that a basic indication was obtained when moist Ph paper was touched to the surface. The entire surface (six sides) of the TiC+10%Cb specimen subsequently was washed with distilled water to remove all visible deposit. Analysis of the water with a flame photometer showed that the deposit contained 200 micrograms of potassium.

TABLE XXI. COMPARISON OF POTASSIUM WETTING DATA ON FOUR
CANDIDATE JOURNAL BEARING MATERIALS

<u>Material</u>	<u>Run #</u>	Initial Advancing Contact Angle (T ~ 65°C)		Receding Contact Angle (T ~ 230°C)
		<u>Surface Polished and Baked</u>	<u>Previously Wet Surface</u>	
Carboloy 907	1	115		20
	2		64	26
	3		55	22
TiC+10%Cb	1	66		6
	2		38	6
Grade 7178	1	51		3
	2		31	5
Mo-TZM	1	135		0
	4	47		0
	5		31	0

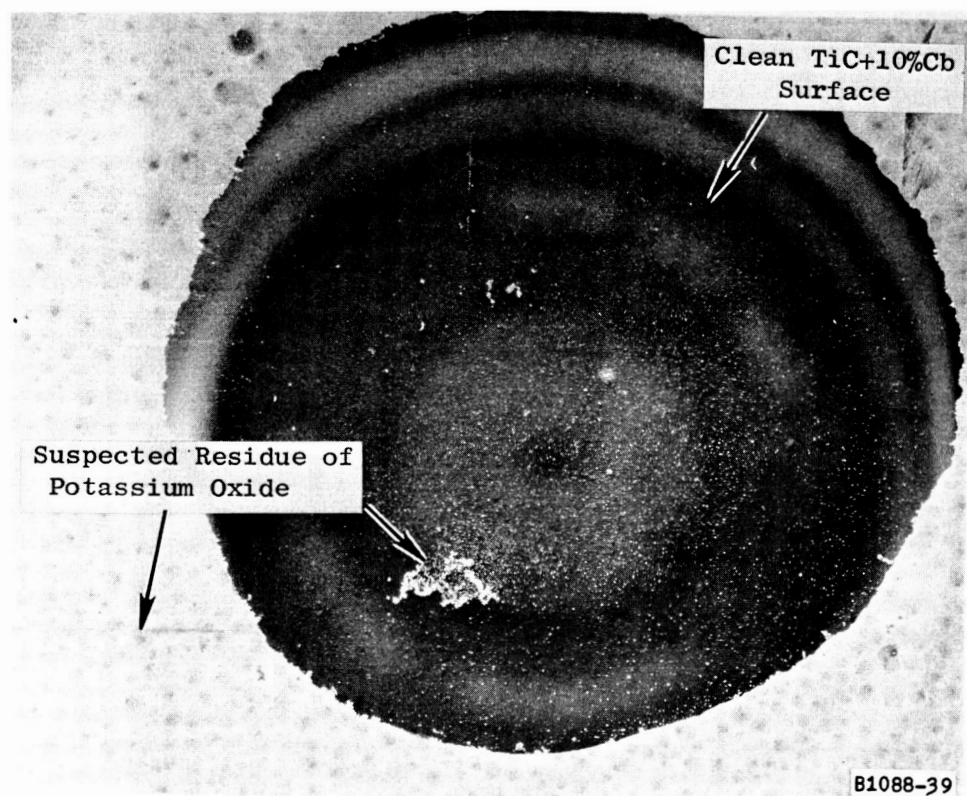


Figure 39. Upper Surface of TiC+10%Cb Specimen after Wetting Runs #1 and #2. (C65121719)

Magnification: 5.5X

Similar behavior has been reported for sodium wetting iron, cobalt and nickel⁽¹¹⁾ and chromium, molybdenum and tungsten⁽¹²⁾. In these studies, it was concluded that the invisible oxide film present on the metal surfaces after abrasion in air, was reduced by sodium. As a result of this, these metals exhibit a critical wetting temperature below which receding contact angles remain above 90°. Above the critical wetting temperature, complete wetting occurs (zero receding contact angle).

In the presently reported study, there is no definite evidence of a critical wetting temperature and, in fact, in only one instance (Mo-TZM alloy) was complete wetting observed. However, it is interesting to note that although completely different systems were studied and different measurement techniques were utilized, both the Addison-Iberson measurements and those reported in this work indicate the existence of an oxide film on the solid surface which tends to increase the contact angle and which can be reduced by the liquid alkali metal.

C. FRICTION AND WEAR IN HIGH VACUUM

Seven friction and wear tests were conducted during the report interim, four in Test Assembly VIII and three in Test Assembly IX. The following material combinations were tested.

<u>Assembly No.</u>	<u>Rider</u>	<u>Disc</u>	<u>Load</u>
VIII	Carboloy 907	Carboloy 907	P ₇
VIII	Mo-TZM	TiC+10%Cb	K
VIII	Mo-TZM	Carboloy 907	K
VIII	Mo-TZM	Grade 7178	K
IX	Grade 7178	Grade 7178	K
IX	Grade 7178	Mo-TZM	K
IX	Mo-TZM	TiC+10%Cb	K

All the tests were conducted at a temperature of 75°F, at speeds of 800 SFM, and at initial pressures in the 10⁻¹⁰ to 10⁻⁹ torr range. The pressure in the chamber remained in the 10⁻⁹ torr range during the tests with the exception of test 500708A (Carboloy 907 vs Carboloy 907). In this test, the high load (6.90 pounds) resulted in a relatively high wear rate (-335.0 in.³/10¹⁰ ft for the rider) and a temperature rise in the test specimens (432°F on the rider away from the interface) which caused the pressure to rise to 3.0 x 10⁻⁸ torr.

The results of these tests are summarized in Table XXII. A short testing time of <60 minutes is the result of high friction between the specimens which leads to slippage in the magnetic drive. The static breakaway torque of the tester is regularly measured at 50 in.-lb. The Sanborn traces showing the changes in friction coefficients with time, photographs of the test specimens illustrating wear patterns, weight change data, and surface finish measurements are presented in the Appendix. An analysis of the test results will be presented at the termination of the existing test program.

TABLE XXII. SUMMARY OF FRICTION AND WEAR TESTS CONDUCTED IN HIGH VACUUM

Assembly No.	Arm No.	Test No.	Test Material		Test Temp, °F	Increase in Rider Specimen Temp, °F (1)	Compressive Load			Test Duration Minutes	Chamber Pressure, Torr		Average Coefficient of Friction	Wear Rate, in ³ /10 ¹⁰ ft.		Remarks
			Rider	Disc			Hertzian Stress, Psi	lbs.	(%0.2%YS or UCS) Rider		Start	Maximum		Rider	Disc	
VIII (2)	1	500708A	Carboloy 907	Carboloy 907	RT	432	6.90	485,410	69	69	7.0 x 10 ⁻¹⁰	3.0 x 10 ⁻⁸	0.53	-355.0	+59.4	Test terminated due to slippage in magnetic drive.
VIII (2)	2	600K08A	Mo-TZM	TiC+10%Cb	RT	70	0.080	89,120	82	26	8.0 x 10 ⁻¹⁰	1.0 x 10 ⁻⁹	1.07	-0.980	-31.4	--
VIII (2)	3	400K08A	Mo-TZM	Carboloy 907	RT	27	0.081	93,820	86	13	7.6 x 10 ⁻¹⁰	2.0 x 10 ⁻⁹	0.40	-1.19	-0.075	--
VIII (2)	4	100K08B	Mo-TZM	7178	RT	44	0.071	91,020	83	13	1.5 x 10 ⁻⁹	2.9 x 10 ⁻⁹	0.94	-0.431	-0.992	--
IX (2)	1	300K08A	7178	7178	RT	440	1.26	286,060	41	41	5.0 x 10 ⁻¹⁰	7.0 x 10 ⁻⁹	0.65	-- (3)	-- (3)	--
IX (2)	2	200K08A	7178	Mo-TZM	RT	30	0.081	95,180	14	87	1.0 x 10 ⁻⁹	1.6 x 10 ⁻⁹	0.79	-- (3)	-- (3)	--
IX (2)	3	600K08B	Mo-TZM	TiC+10%Cb	RT	60	0.082	89,530	82	26	7.0 x 10 ⁻¹⁰	1.1 x 10 ⁻⁹	0.51	-- (3)	-- (3)	--

(1) Measured with Sheathed Chromel-Alumel Thermocouples Located in Thermowell in Contact with Side of Rider Specimen (1/4 inch Away from Interface.

(2) Main Shaft Angular Contact Bearings (MRC-7207) Ultrasonically Cleaned and Unlubricated.

(3) Specimens Damaged After Test; No Weight Data Available.

VI. FUTURE PLANS

The summary which follows enumerates the steps to be pursued during the succeeding quarter to implement this study.

A. Conduct elevated temperature compression tests of candidate bearing materials at 800°, 1200°, and 1600°F.

B. Conduct friction and wear tests of Mo-TZM vs. Grade 7178, Grade 7178 vs. itself, Mo-TZM vs. TiC+10%Cb, TiC+10% vs. itself, Mo-TZM vs. Carboloy 907, and Carboloy 907 vs. itself in high vacuum (10^{-9} torr) and at ambient temperatures of 400°, 800°, and 1200°F.

C. Conduct trial friction and wear test in liquid potassium at 400°F.

VII. REFERENCES

- (1) "Materials for Potassium Lubricated Journal Bearings," Quarterly Progress Report No. 1, ctr. NAS 3-2534 (July 22, 1963), SPPS, MSD, General Electric Company; Report NASA-CR-54006.
- (2) Coffin, L. F., Jr., "Theory and Application of Sliding Contact of Metals in Sodium," Report KAPL-828, Knolls Atomic Power Laboratory, General Electric Company, October 1955.
- (3) Zeman, K. P., Young, W. R., and Coffin, L. F., Jr., "Friction and Wear of Refractory Compounds," Report 59GC-23, General Electric Company, May 1959.
- (4) AiResearch Manufacturing Company of Arizona, Quarterly Technical Progress Report for Period Ending September 30, 1965, SNAP 50/SPUR Contract AF33(615)-2289 BPSN; 5(6399-675A) 63409124, ADS-5152-R4, p 7.
- (5) Sliney, H. E., Strom, T. N., and Allen, G. P., "Fused Fluoride Coatings as Solid Lubricants in Liquid Sodium, Hydrogen, Vacuum and Air," NASA TN D-2348, August 1964.
- (6) "Materials for Potassium Lubricated Journal Bearings," Quarterly Progress Report No. 8, ctr. NAS 3-2534 (April 22, 1965), SPPS, MSD, General Electric Company, Report NASA-CR-54646.
- (7) "Materials for Potassium Lubricated Journal Bearings," Quarterly Progress Report No. 8, ctr. NAS 3-2534 (January 22, 1965), SPPS, MSD, General Electric Company; Report NASA-CR-54345.
- (8) DeMoney, F. W., "Static and Dynamic Creep-Rupture Properties of B50R207 and B50R311 Alloys," General Electric Company, Contract No. X241982-2, University of Minnesota, June 15, 1954.
- (9) "Materials for Potassium Lubricated Journal Bearings," Quarterly Progress Report No. 9, ctr. NAS 3-2534 (July 22, 1965), SPPS, MSD, General Electric Company, Report NASA-CR-54892.
- (10) "Materials for Potassium Lubricated Journal Bearings," Quarterly Progress Report No. 10, ctr. NAS 3-2534 (October 22, 1965), SPPS, MSD, General Electric Company.
- (11) Addison, C. C., Iberson, E., and Manning, J. A., Journal Chem. Soc., p 2699 (1962).
- (12) Addison, C. C., and Iberson, E., Journal Chem. Soc., p 1437 (1965).

PUBLISHED REPORTS

Quarterly Progress Reports

For Quarter Ending

Report No. 1 (NASA-CR-54006)

July 22, 1963

Report No. 2 (NASA-CR-54007)

October 22, 1963

Report No. 3 (NASA-CR-54073)

January 22, 1964

Report No. 4 (NASA-CR-54113)

April 22, 1964

Report No. 5 (NASA-CR-54169)

July 22, 1964

Report No. 6 (NASA-CR-54264)

October 22, 1964

Report No. 7 (NASA-CR-54345)

January 22, 1965

Report No. 8 (NASA-CR-54646)

April 22, 1965

Report No. 9 (NASA-CR-54892)

July 22, 1965

Report No. 10 (NASA-CR-72027)

October 22, 1965

APPENDIX

FRICITION AND WEAR DATA - HIGH VACUUM FRICTION AND WEAR TEST PROGRAM

FRICITION AND WEAR TEST DATA FOR CARBOL

Test No. - 500708A

Rider

Test Temperature, °F - 80

Material - Carboloy 907

Max. ΔT of Rider, $^{\circ}\text{F}$ - 4

Specimen No. - 1036-E-8

Assembly No. - VIII

Speed, SFM - 800

Loading Arm No. - 1

Disc

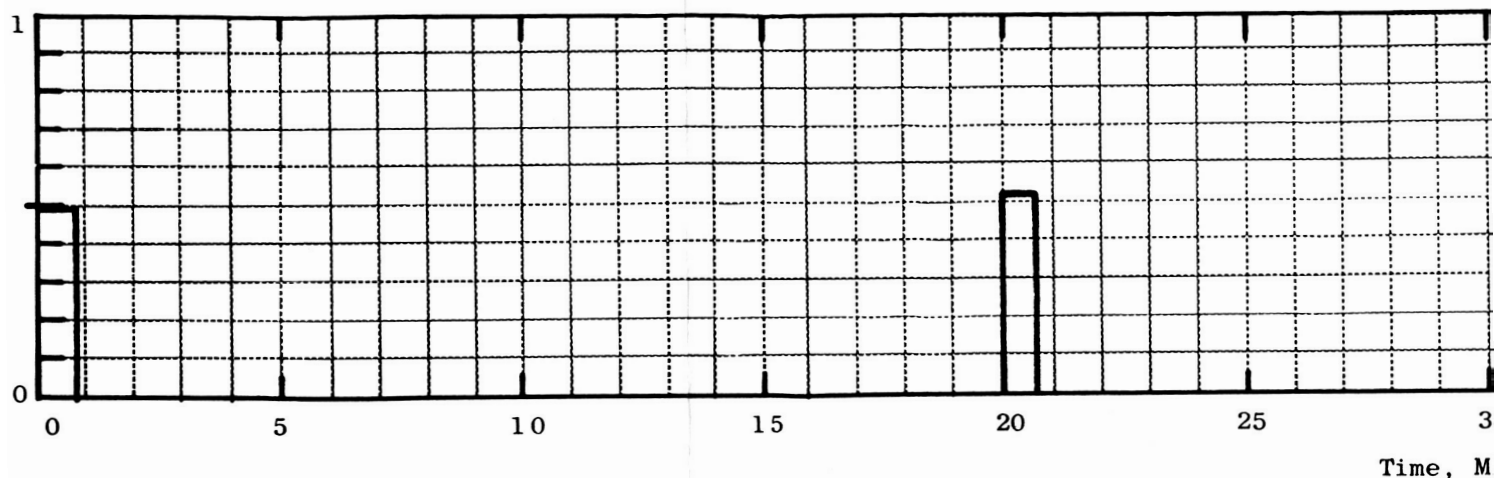
Test Duration, Min. - 3.2

Material - Carboloy 907

Test Date - 11/6/65

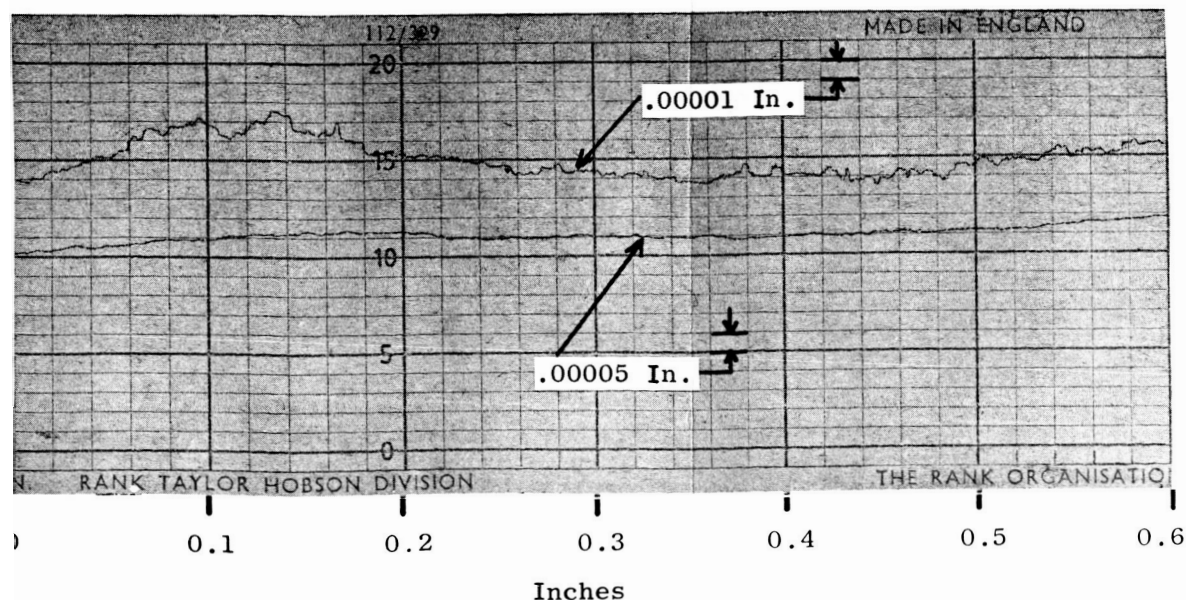
Specimen No. - 1036-F-16A

Change in Coefficient of Friction with Time -



WEAR - RIDER

Initial Surface Finish -



Wear Scar Dia., In

Weight, Gms -

Start - 3.5154

Finish - 3.4927

Change - -0.0227

Material Density,

14.684

Volume Change, Mm^3

-1.546

Wear Rate, $\text{In}^3/10^5$

-355

Y 907 VS CARBOLOY 907 IN HIGH VACUUM

Compressive Load, Lbs - 6.90(P₇)

Chamber Pressure, Torr -
Start - 7.0 x 10⁻¹⁰
Max. - 3.0 x 10⁻⁸

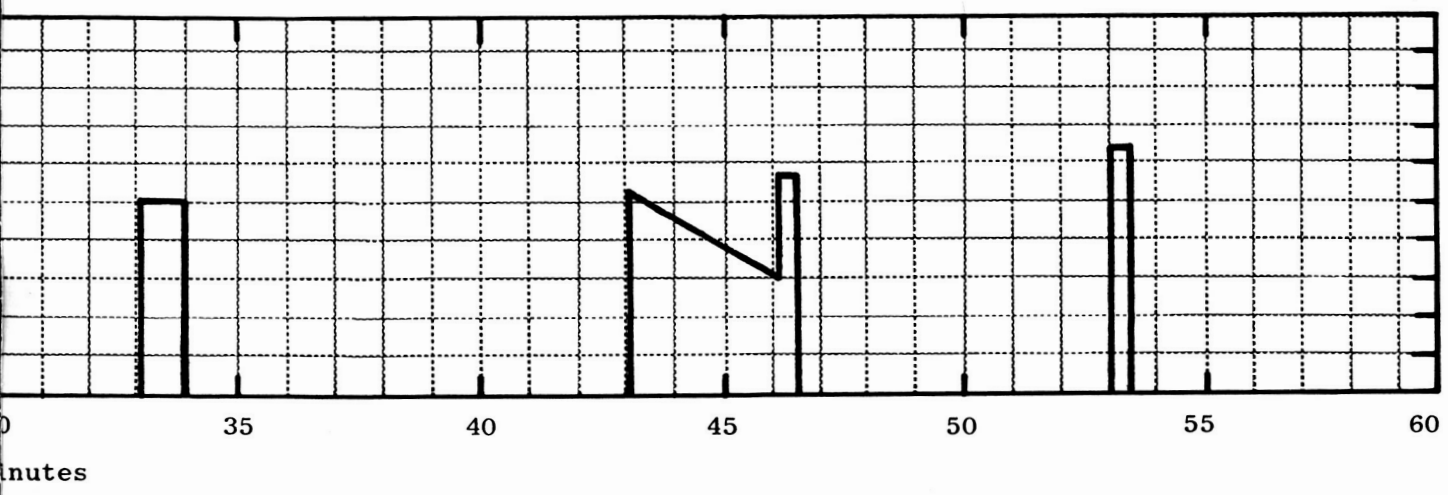
Compressive Stress, psi - 485,410

Load/Material UCS or 0.2%CYS -

Rider - 69%
Disc - 69%

Remarks - Test terminated due
to slippage in magnetic
drive.

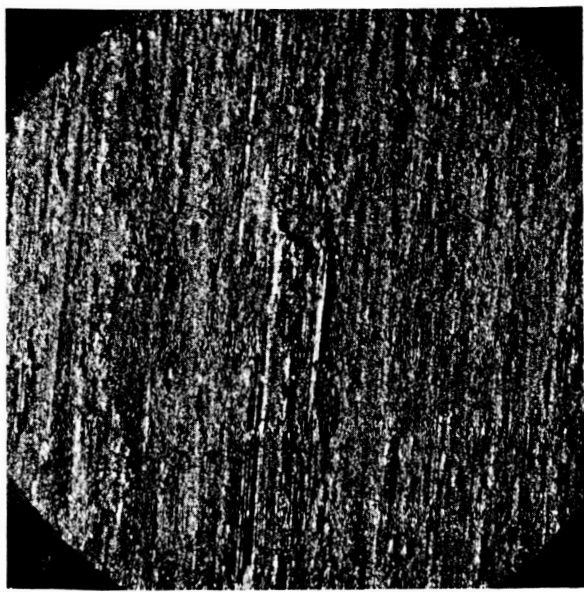
Average Coefficient of Friction - 0.53



- 0.124

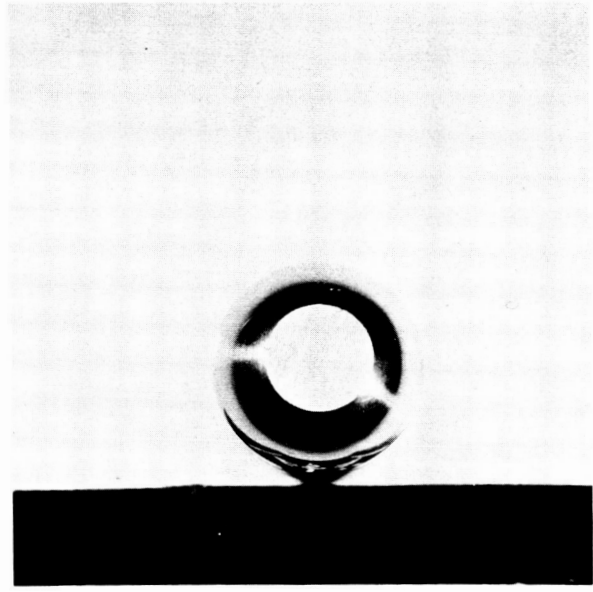
Gms/Cm³ -

0 ft -



(C66081668)

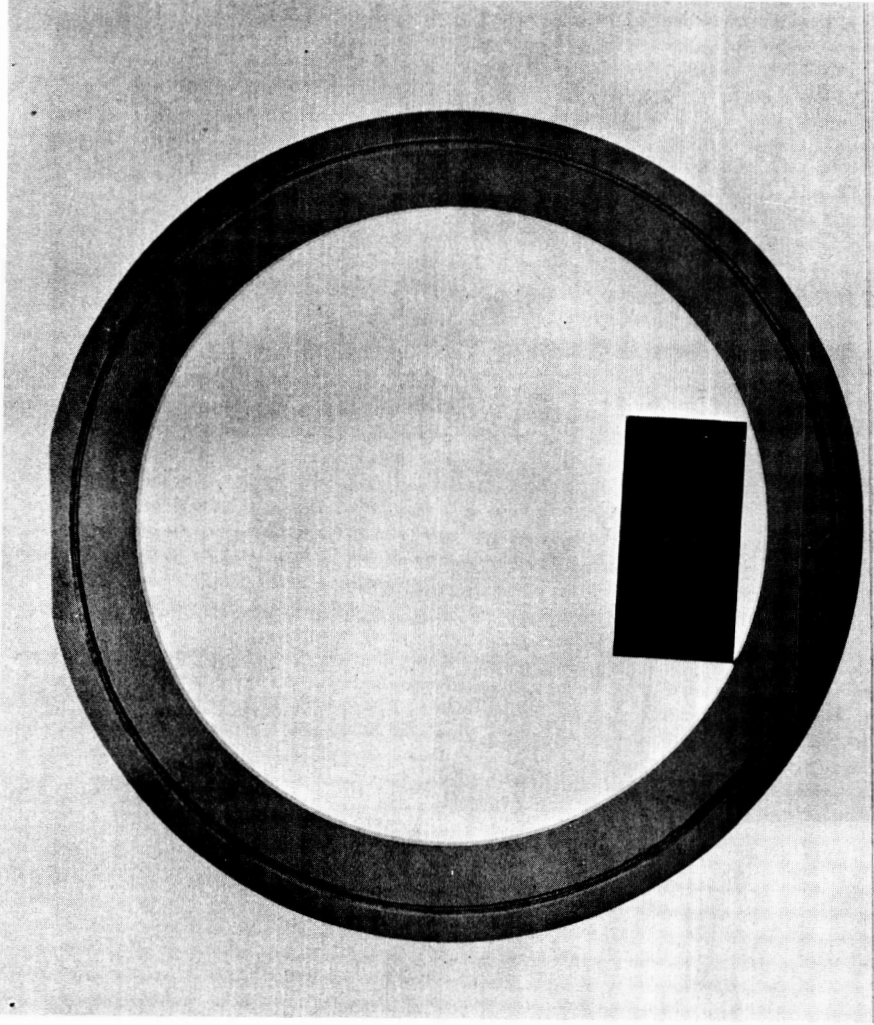
Mag.: 28.5X



(C651223155)

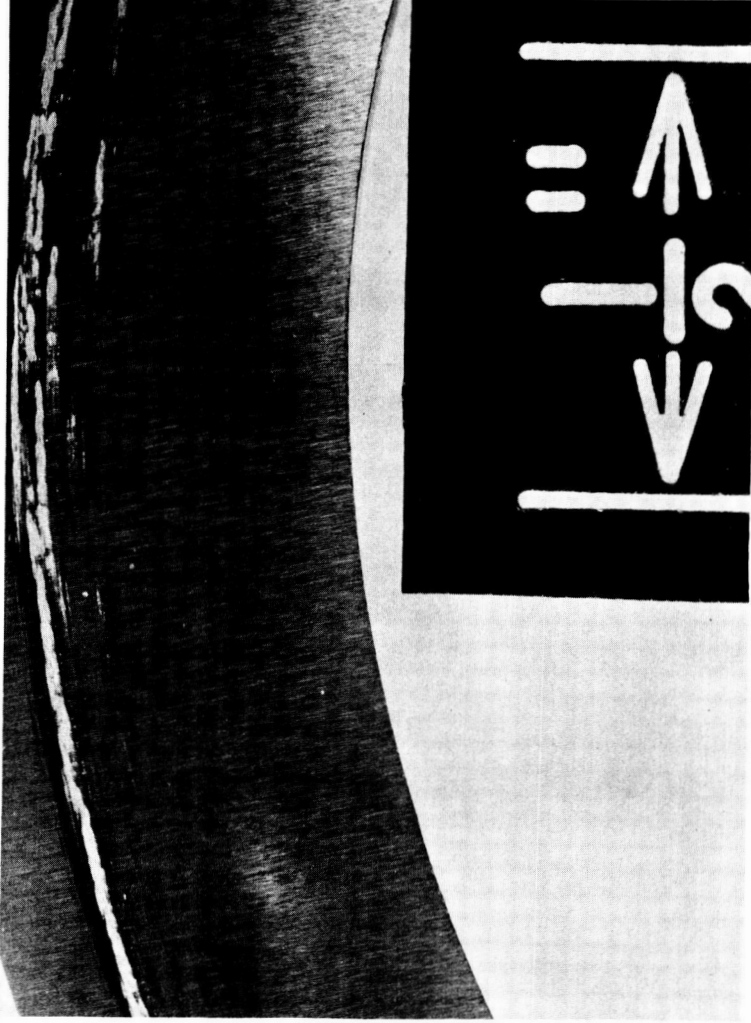
Mag.: 5X

100-2



(C66060172)

Mag.: 1X



(C651223146)

Mag.: 5X

Initial Surface Finish, Avg. RMS - 1-2

Wear Scar Width, In. - 0.109

Weight, Gms /Cm³ -

Start - 226.3745

Finish - 226.3783

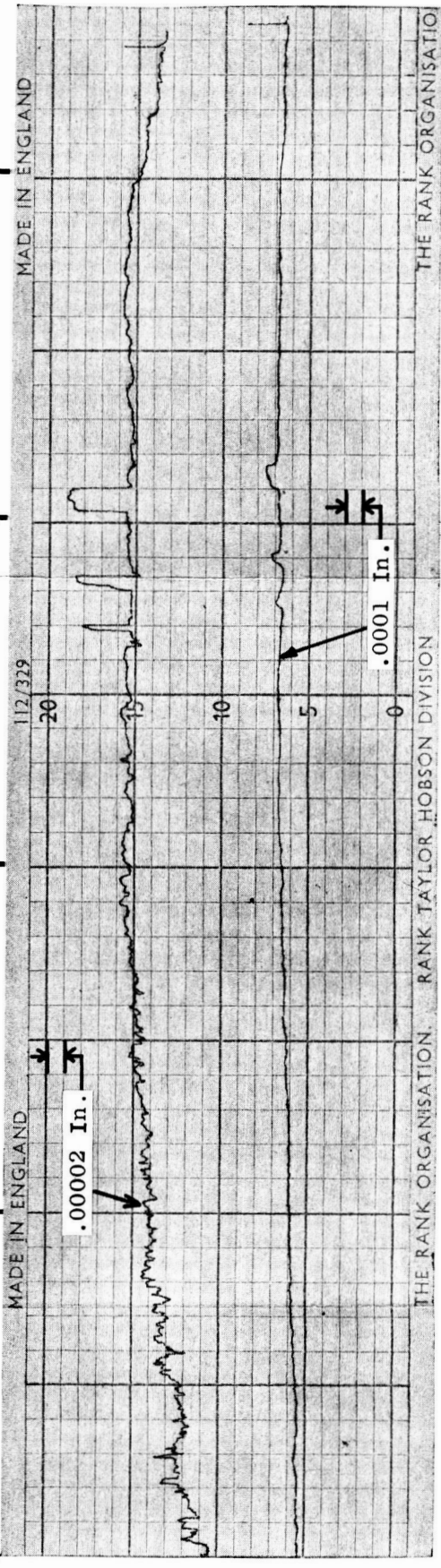
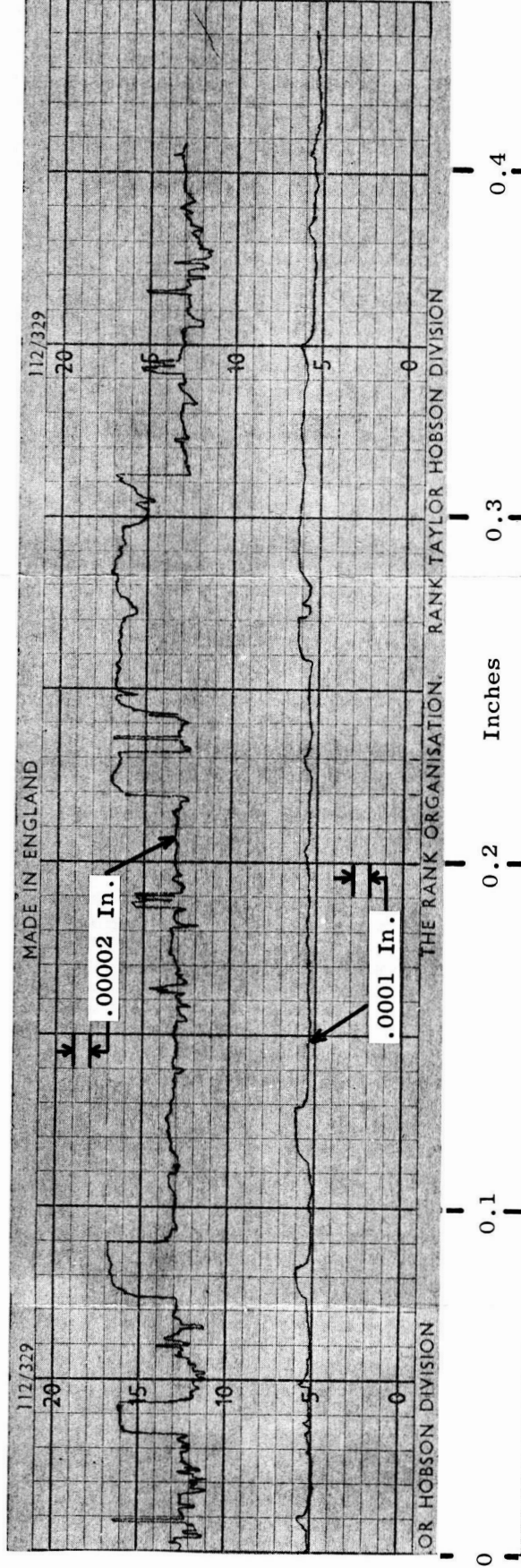
Change - +0.0038

Material Density, Gms /Cm³ - 14.684

Volume Change, Mm³ - +0.259

Wear Rate, In³/10¹⁰ft - +59.4

Post-Test Profilometer Trace - Circumferential at 90° CW from S/N

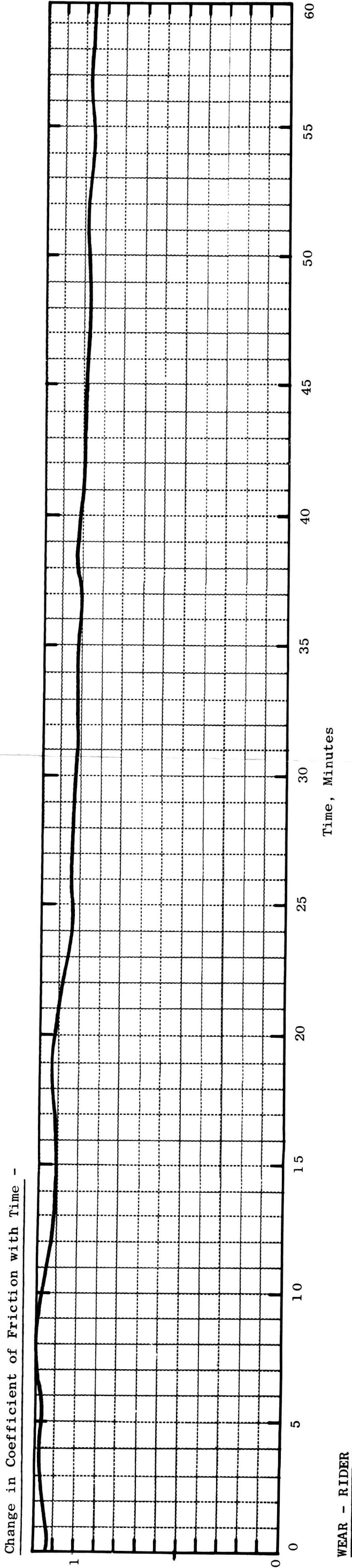


Post-Test Profilometer Trace - Radial at 90° CW from S/N

101-1

FRICION AND WEAR TEST DATA FOR Mo-TZM ALLOY VS TiC+10%Cb IN HIGH VACUUM

Test No. - 600K08A	Rider	Test Temperature, °F - 80	Compressive Load, Lbs - 0.080 (K)	Chamber Pressure, Torr -
Assembly No. - VIII	Material - Mo-TZM Specimen No. - 1037-E-8	Max. ΔT of Rider, °F - 70	Compressive Stress, psi - 89,120	Start - 8.0×10^{-10} Max. - 1.0×10^{-9}
Loading Arm No. - 2	Disc	Speed, SFM - 800	Load/Material UCS or 0.2%CYS -	Remarks -
Test Date - 11/5/65	Material - TiC+10%Cb Specimen No. - 1045-F-5A	Test Duration, Min. - 60.00	Rider - 82% Disc - 26%	
Average Coefficient of Friction - 1.07				



Initial Surface Finish - <6 RMS

Profilometer trace not available.

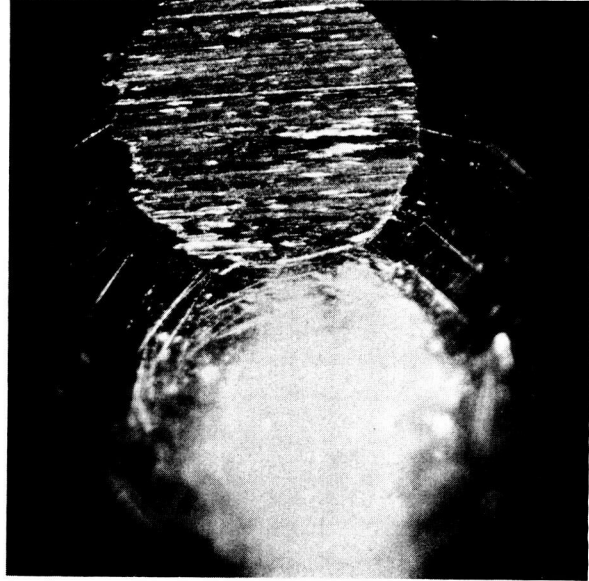
Wear Scar Dia., In. - .056

Weight, Gms -
Start - 2.3810
Finish - 2.3801
Change - -0.0009

Material Density, Gms/Cm³ -
10.139

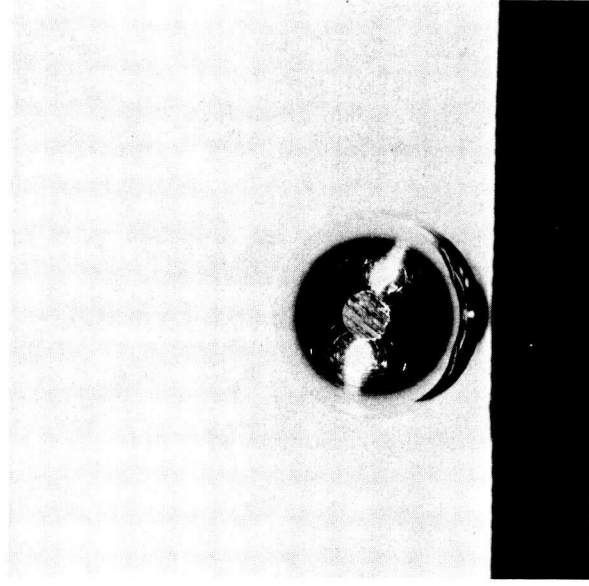
Volume Change, Mm³ -
-0.089

Wear Rate, In³/10¹⁰ft -
-0.98



(C66081635)

Mag.: 28.5X

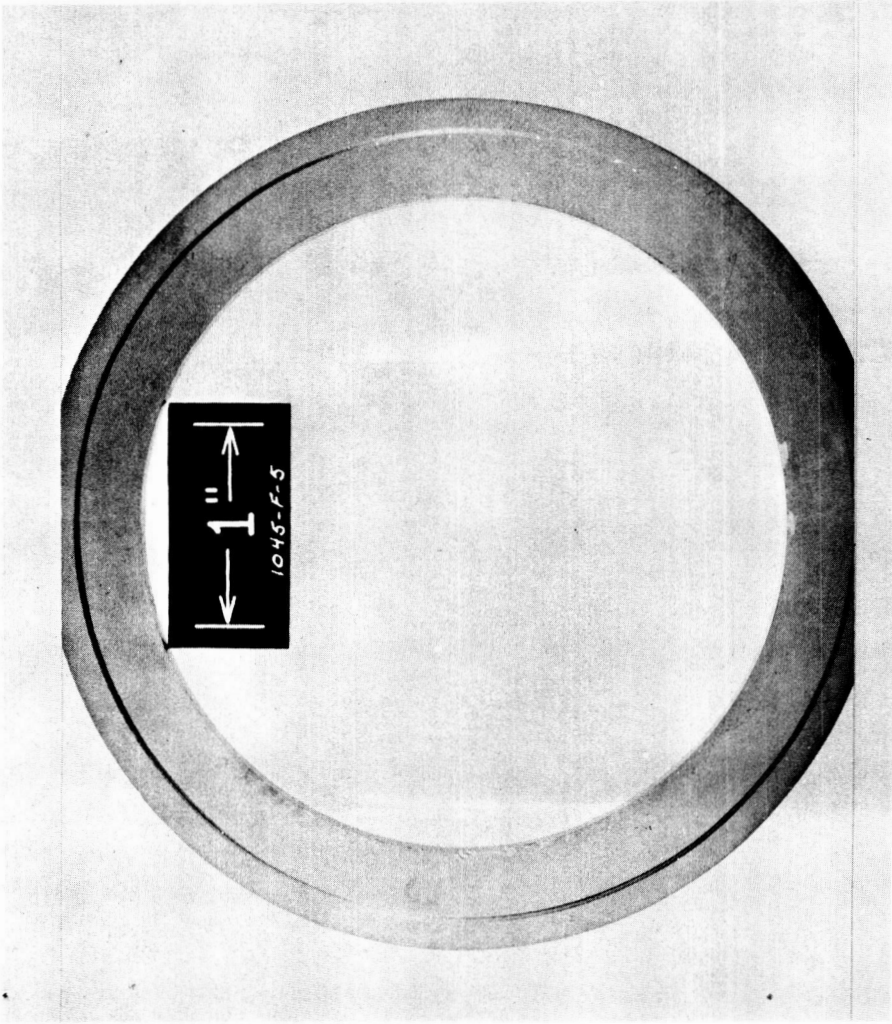


(C651223151)

Mag.: 5X

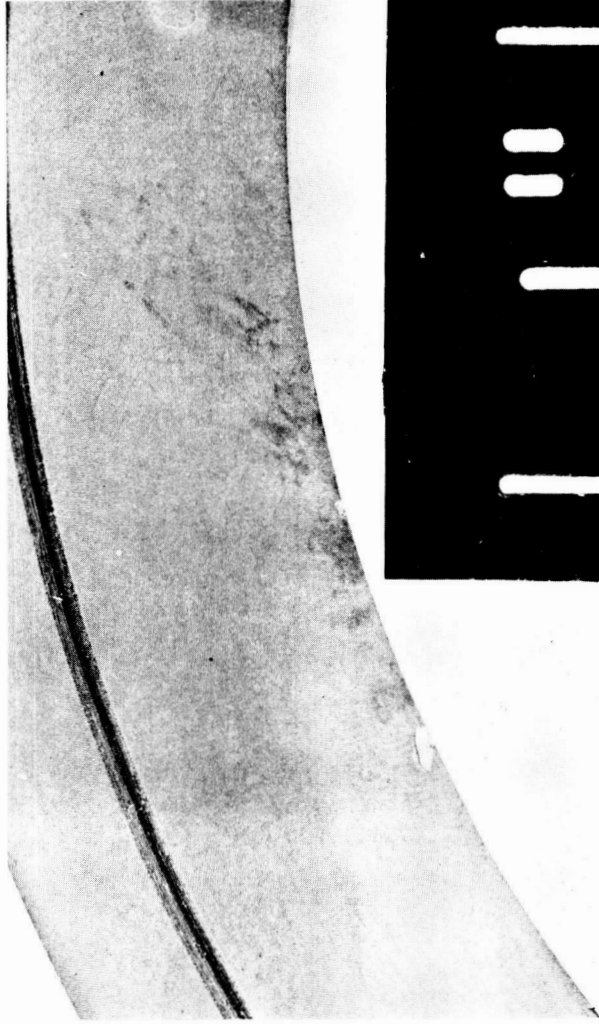
102-1

102-2



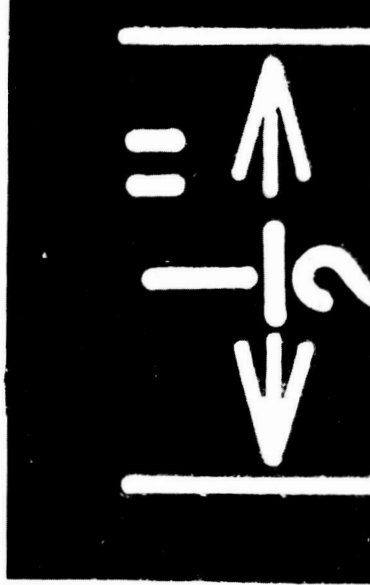
(C651223137)

Mag.: 1X



(C651223136)

Mag.: 5X



Initial Surface Finish, Avg. RMS - 3.5-4.5

Wear Scar Width, In. - 0.031

Weight, Gms /Cm³ -

Start - 81.9859

Finish - 81.9710

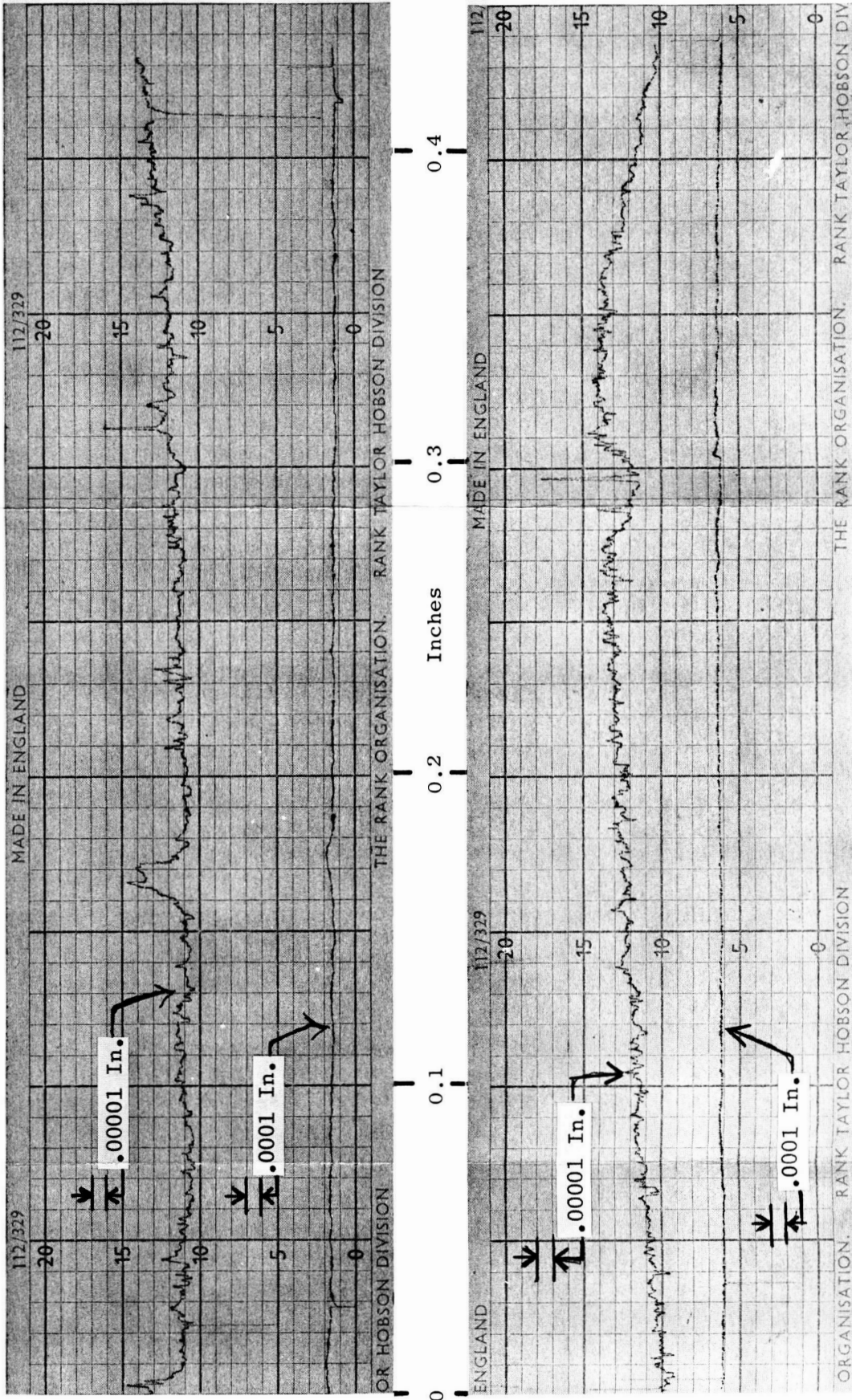
Change - -0.0149

Material Density, Gms /Cm³ - 5.239

Volume Change, Mm³ - -2.844

Wear Rate, In³/10¹⁰ft - -31.4

Post-Test Profilometer Trace - Circumferential at S/N



Post-Test Profilometer Trace - Radial at S/N

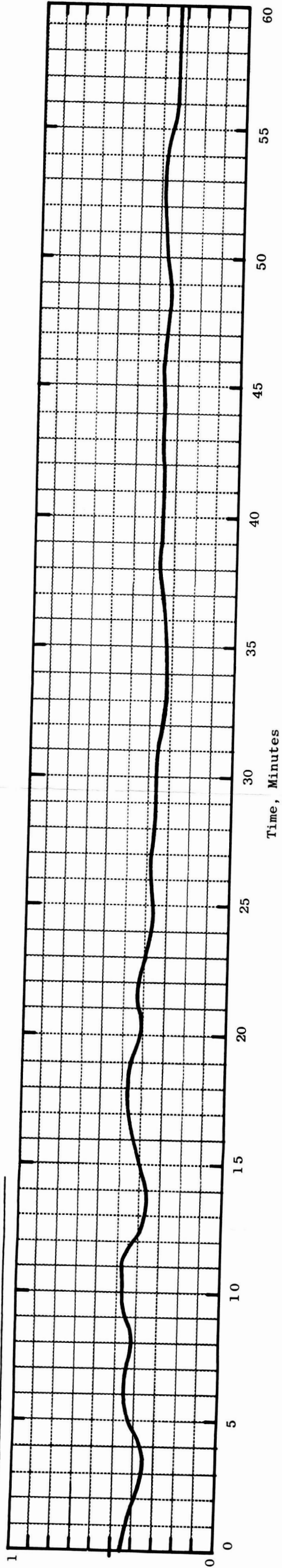
103-1

FRICITION AND WEAR TEST DATA FOR Mo-TZM ALLOY VS CARBOLOY 907 IN HIGH VACUUM

Test No. - 400K08A	Rider	Test Temperature, °F - 80	Compressive Load, Lbs - 0.081 (K)	Chamber Pressure, Torr -
Assembly No. - VIII	Material - Mo-TZM	Max. ΔT of Rider, °F - 27	Compressive Stress, psi - 93,820	Start - 7.6×10^{-10}
Loading Arm No. - 3	Specimen No. - 1037-E-7	Speed, SFM - 800	Load/Material UCS or 0.2%CYS -	Max. - 2.0×10^{-9}
Test Date - 11/4/65	Disc	Test Duration, Min. - 60.00	Rider - 86%	Remarks -
	Material - Carboloy 907		Disc - 13%	
	Specimen No. - 1036-F-17A			

Average Coefficient of Friction - 0.40

Change in Coefficient of Friction with Time -



WEAR - RIDER

Initial Surface Finish - <6 RMS

Profilometer trace not available.

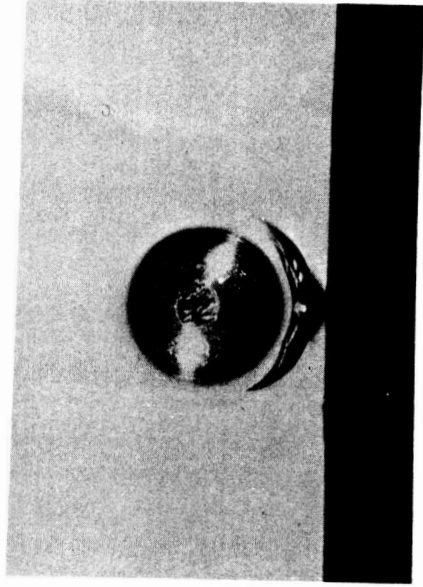
Wear Scar Dia., In. - 0.051

Weight, Gms -
Start - 2.3763
Finish - 2.3752
Change - -0.0011
Material Density, Gms/Cm ³ -
10.139
Volume Change, Mm ³ -
-0.108
Wear Rate, In ³ /10 ¹⁰ ft -
-1.19



(C66081602)

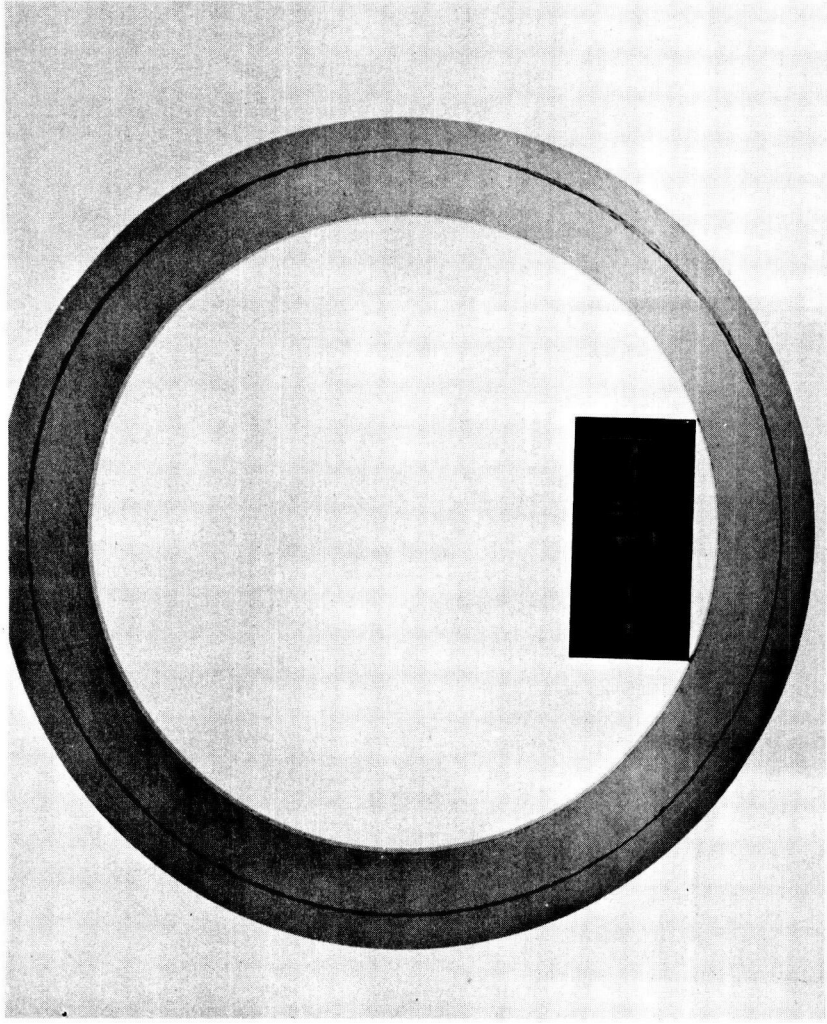
Mag.: 28.5X



(C651223124)

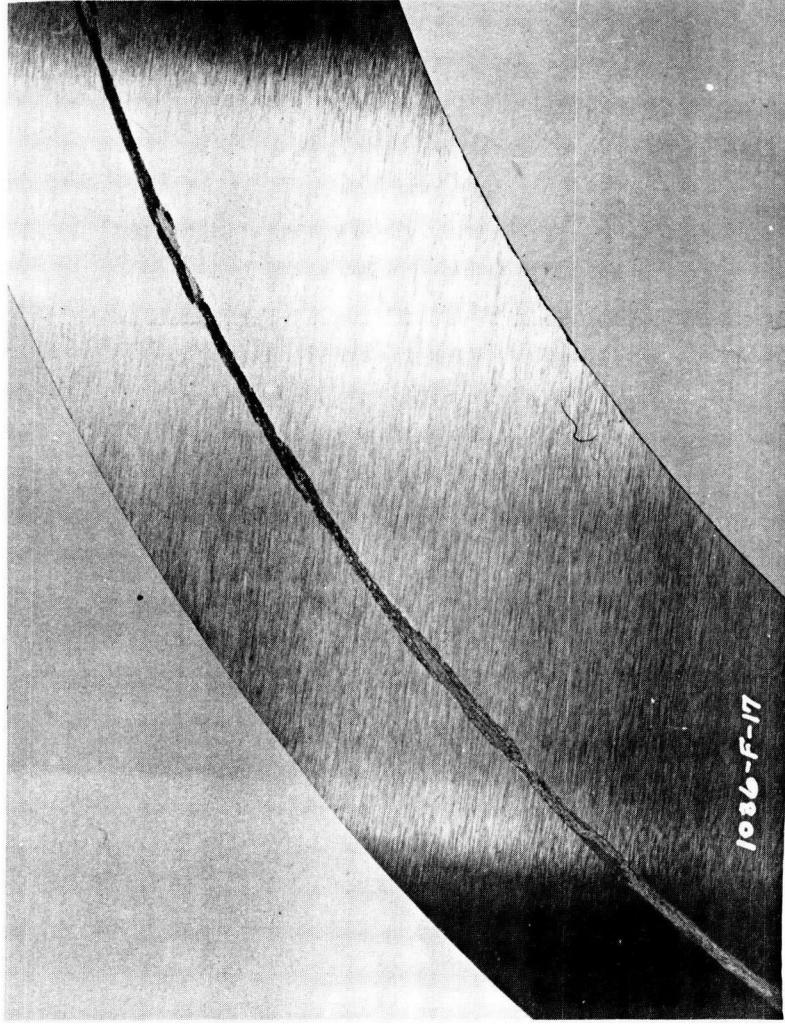
Mag.: 5X

104-1



(C66060173)

Mag.: 1X



(C66060178)

Mag.: 5X

Initial Surface Finish, Avg. RMS - 1.5-2

Wear Scar Width, In. - 0.031

Weight, Gms /Cm³ -

Start - 226.1342

Finish - 226.1341

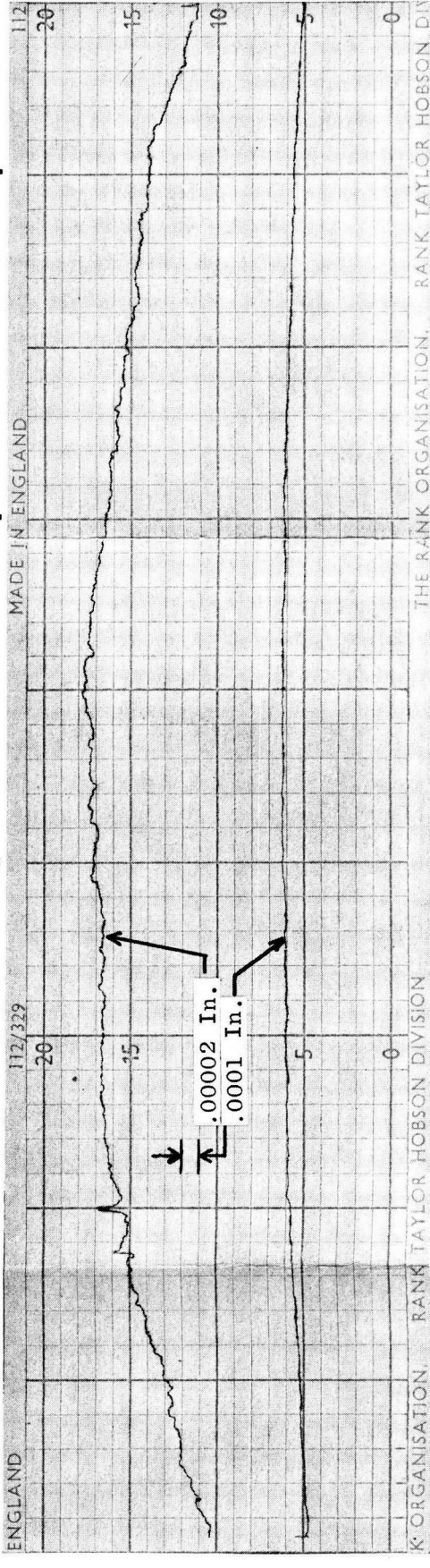
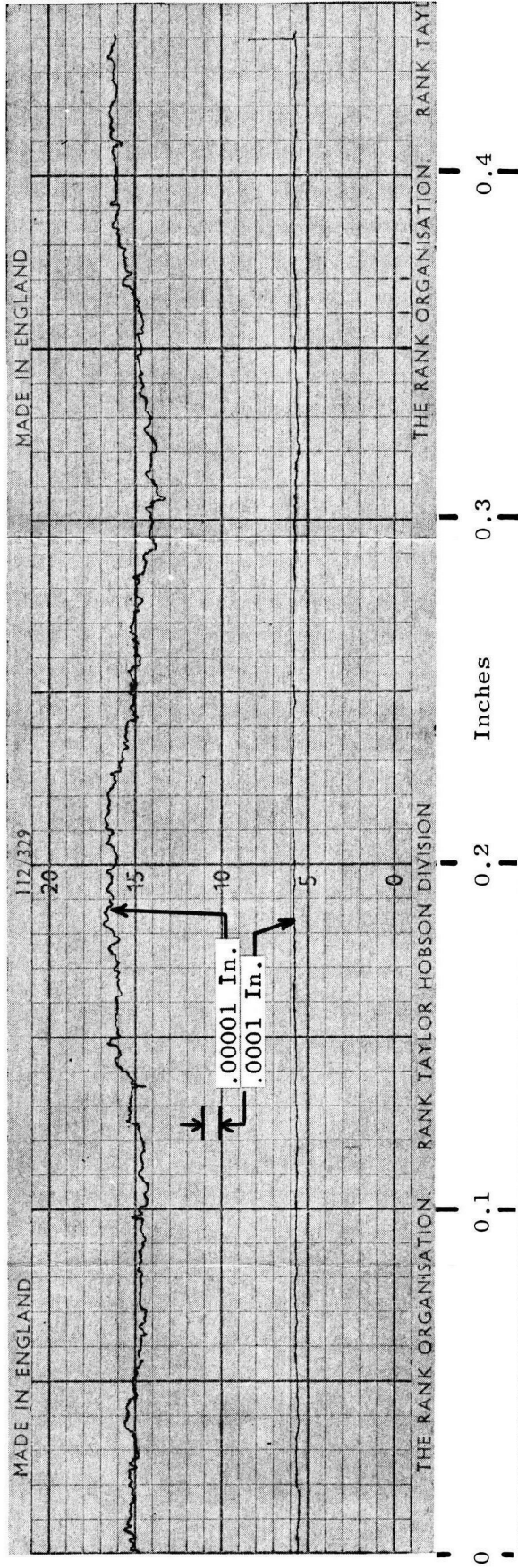
Change - -0.0001

Material Density, Gms /Cm³ - 14.684

Volume Change, Mm³ - -0.007

Wear Rate, In³/10¹⁰ft - -0.07

Post-Test Profilometer Trace - Circumferential at 90° CW from S/N



Post-Test Profilometer Trace - Radial at 90° CW from S/N

FRICION AND WEAR TEST DATA FOR MO-TZM ALLOY VS GRADE 7178 IN HIGH VACUUM

Test No. - 100K08B

Assembly No. - VIII

Loading Arm No. - 4

Test Date - 11/3/65

Rider

Material - Mo-TZM

Specimen No. - 1037-E-9

Disc

Material - Grade 7178

Specimen No. - 1046-F-4A

Test Temperature, °F - 80

Max. ΔT of Rider, °F - 44

Speed, SFM - 800

Test Duration, Min. - 60.00

Compressive Load, Lbs - 0.071 (K)

Compressive Stress, psi - 91,020

Load/Material UCS or 0.2%CYS -

Rider - 83%

Disc - 13%

Chamber Pressure, Torr -

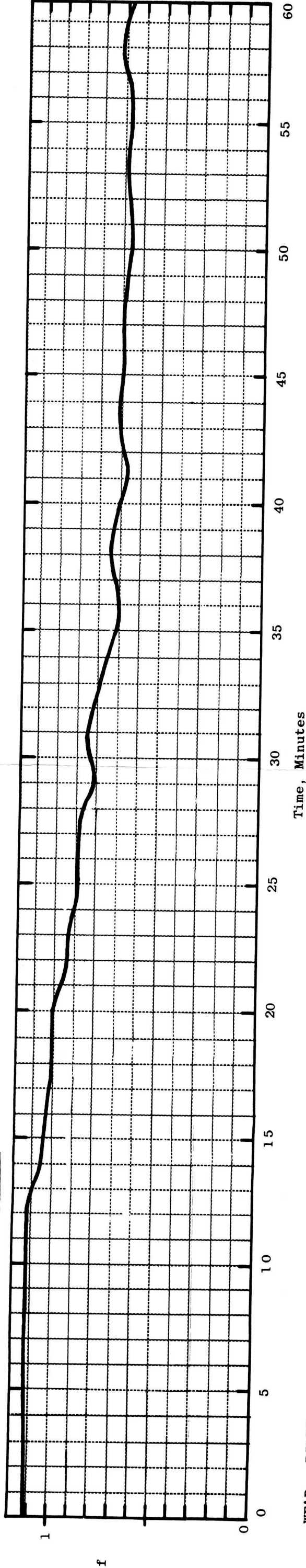
Start - 1.5×10^{-9}

Max. - 2.9×10^{-9}

Remarks -

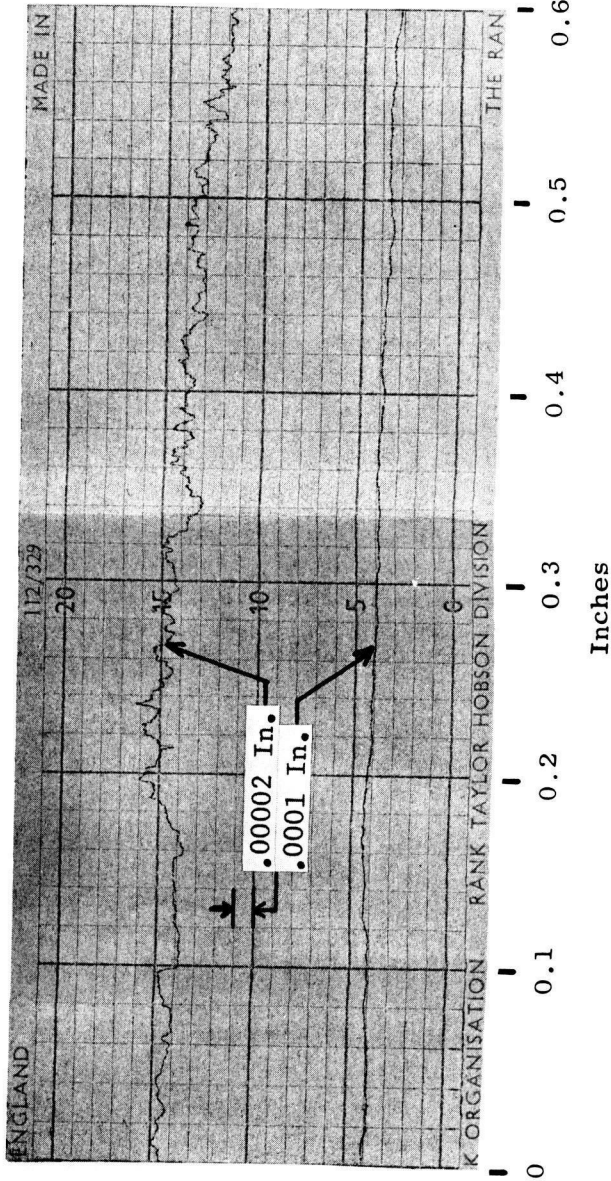
Average Coefficient of Friction - 0.94

Change in Coefficient of Friction with Time -



WEAR - RIDER

Initial Surface Finish -



Wear Scar Dia., In. - .058

Weight, Gms -

Start - 2.3850

Finish - 2.3846

Change - -0.0004

Material Density, Gms/Cm³ -

10.139

Volume Change, Mm³ -

-0.039

Wear Rate, In³/10¹⁰ft -

-0.43

Inches

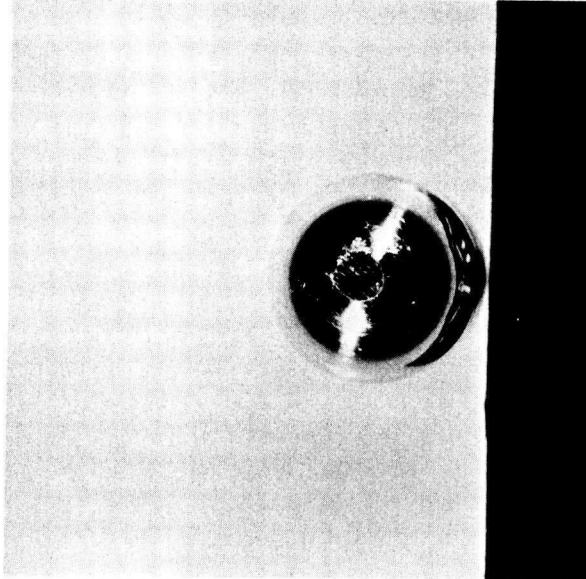
-106-

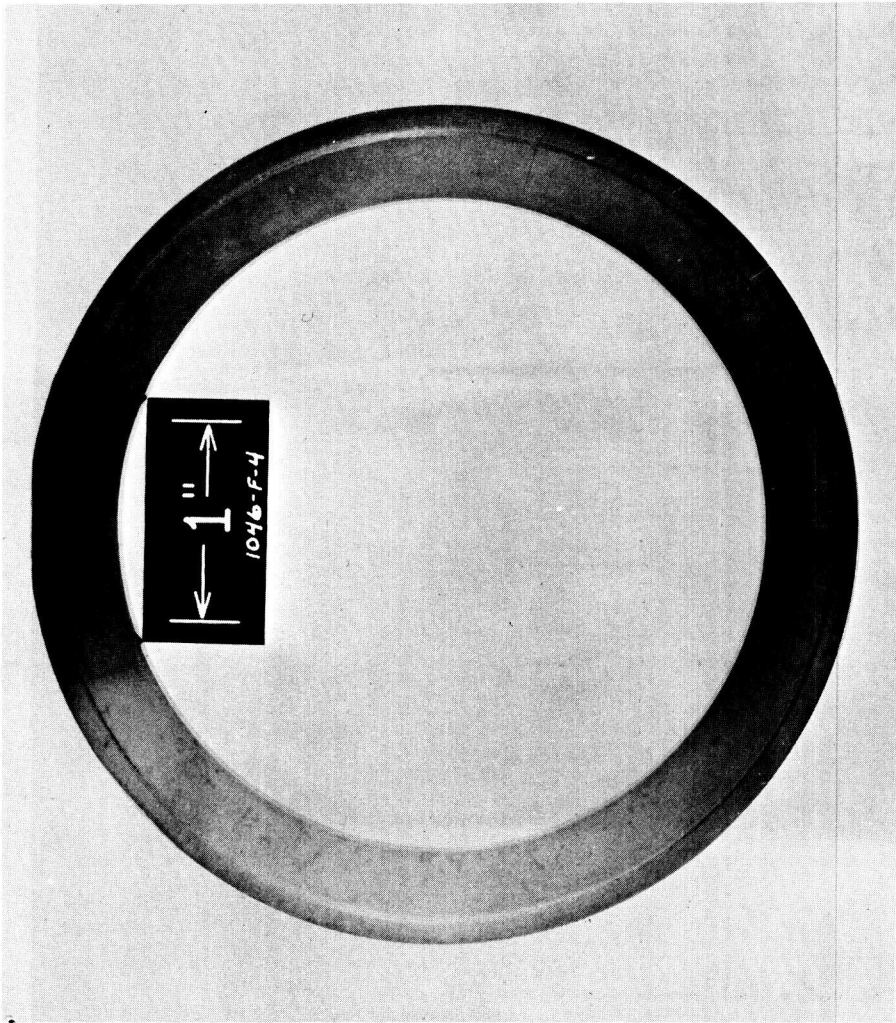
Mag.: 28.5X

(C66081649)

(C651223149)

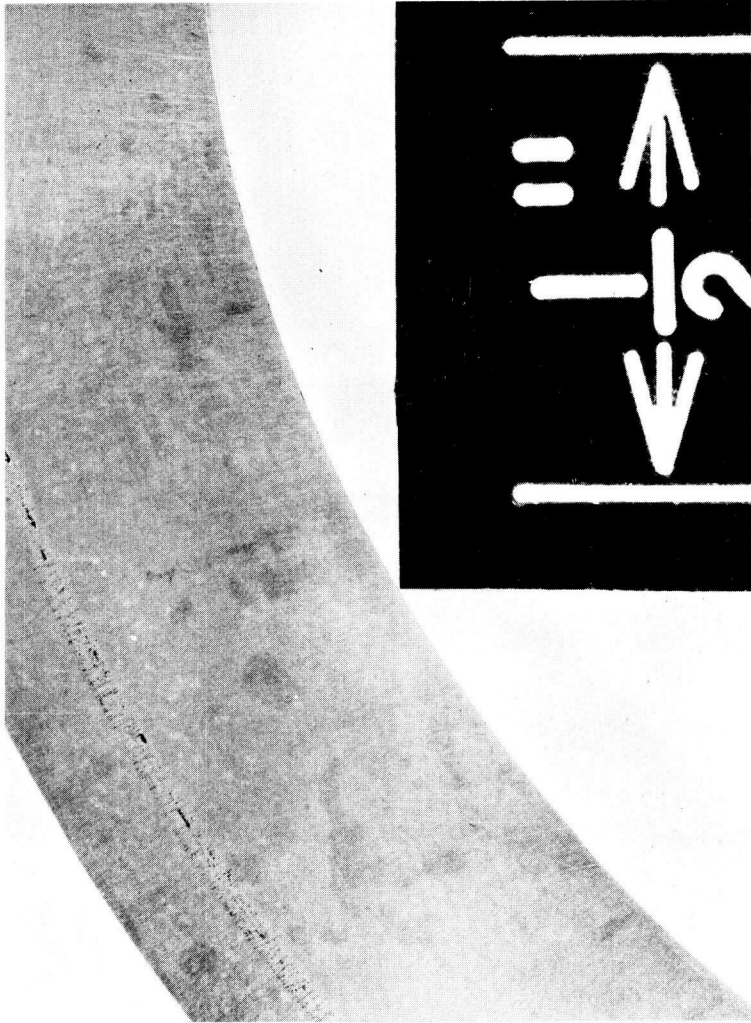
Mag.: 5X





(C651223123)

Mag.: 1X



(C651223122)

Mag.: 5X

Initial Surface Finish, Avg. RMS - 2.5-4

Wear Scar Width, In. - 0.031

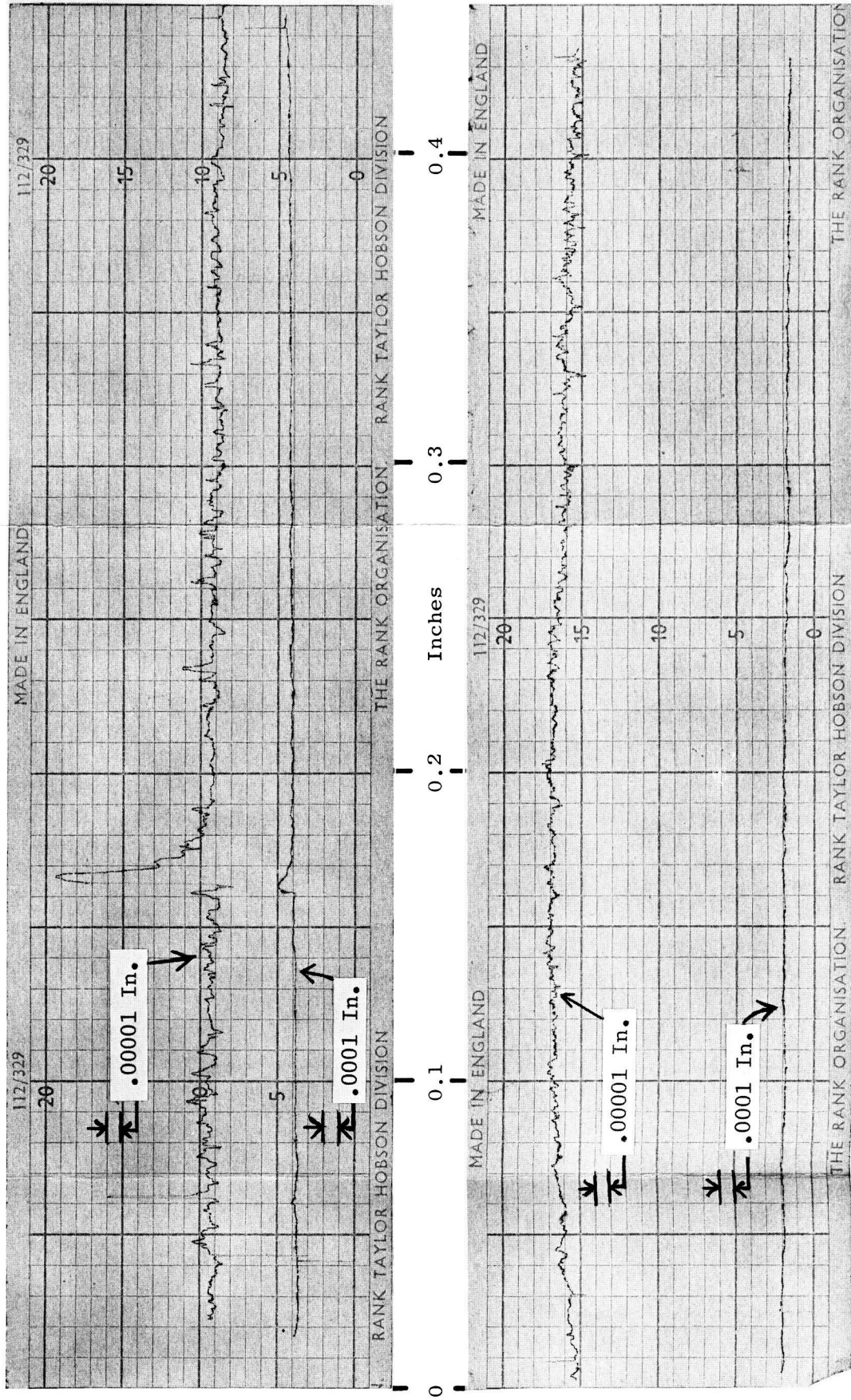
Weight, Gms /Cm³ -
Start - 209.3283
Finish - 209.3270
Change - -0.0013

Material Density, Gms /Cm³ - 14.301

Volume Change, Mm³ - -0.091

Wear Rate, In³/10¹⁰ft - -0.99

Post-Test Profilometer Trace - Circumferential at S/N



Post-Test Profilometer Trace - Radial at S/N

FRICITION AND WEAR TEST DATA FOR GRADE 7178 VS GRADE 7178 IN HIGH VACUUM

Test No. - 300H08A

Assembly No. - IX

Loading Arm No. - 1

Test Date - 11/13/65

Rider

Material - Grade 7178
Specimen No. - 1046-E-8

Disc

Material - Grade 7178
Specimen No. - 1046-F-5A

Test Temperature, °F - 80

Max. ΔT of Rider, °F - 432

Speed, SFM - 800

Test Duration, Min. - 60.00

Compressive Load, Lbs. - 1.26 (H)

Compressive Stress, psi - 286,060

Load/Material UCS or 0.2%CY\$ -

Rider - 41%

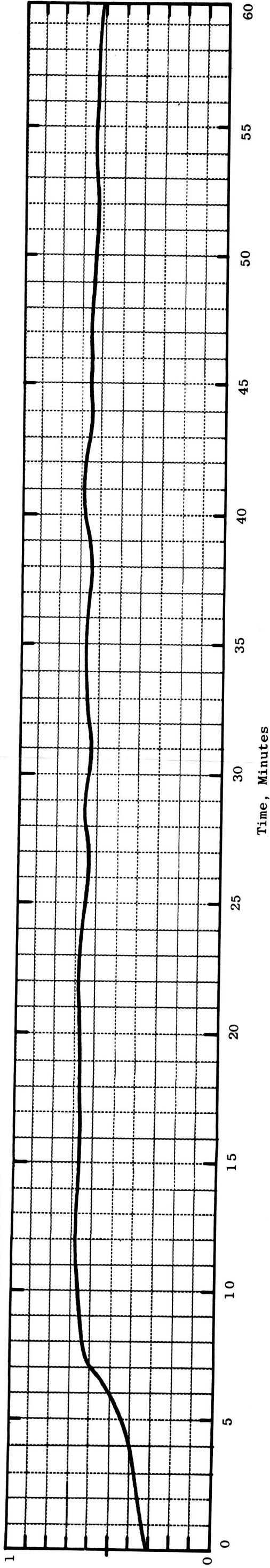
Disc - 41%

Average Coefficient of Friction - 0.65

Chamber Pressure, Torr -
Start - 5.0 x 10⁻¹⁰
Max. - 7.0 x 10⁻⁹

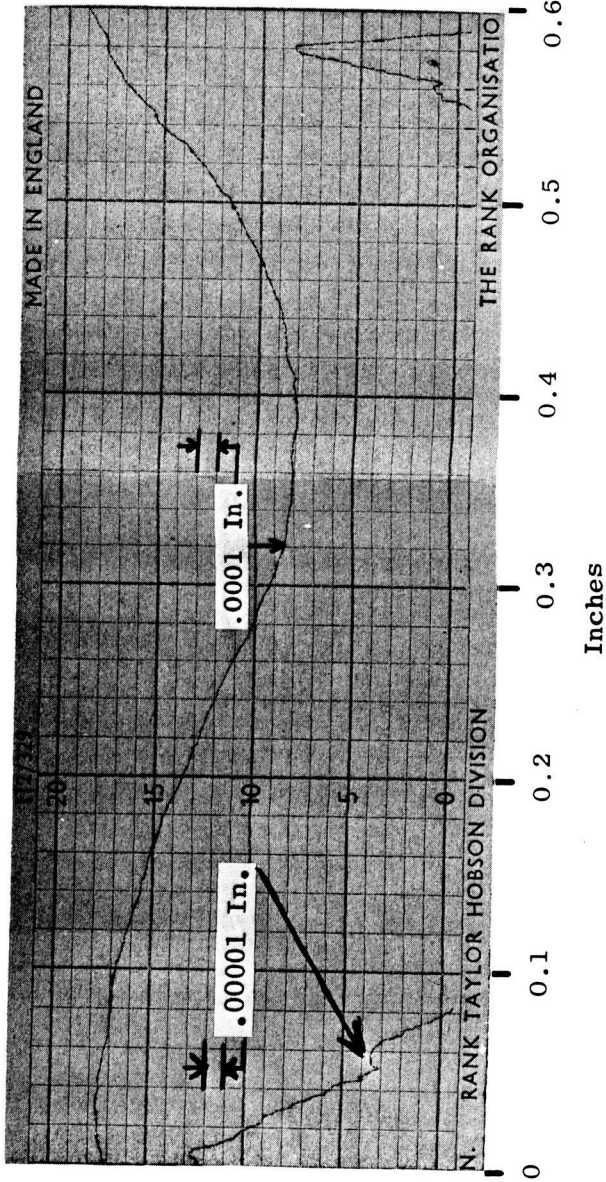
Remarks - Fracture of another specimen after this test was completed damaged these specimens and voided wear measurements.

Change in Coefficient of Friction with Time -



WEAR - RIDER

Initial Surface Finish -



Wear Scar Dia., In. - 0.043

Weight, Gms -

Start - 3.1176

Finish - (See remarks)

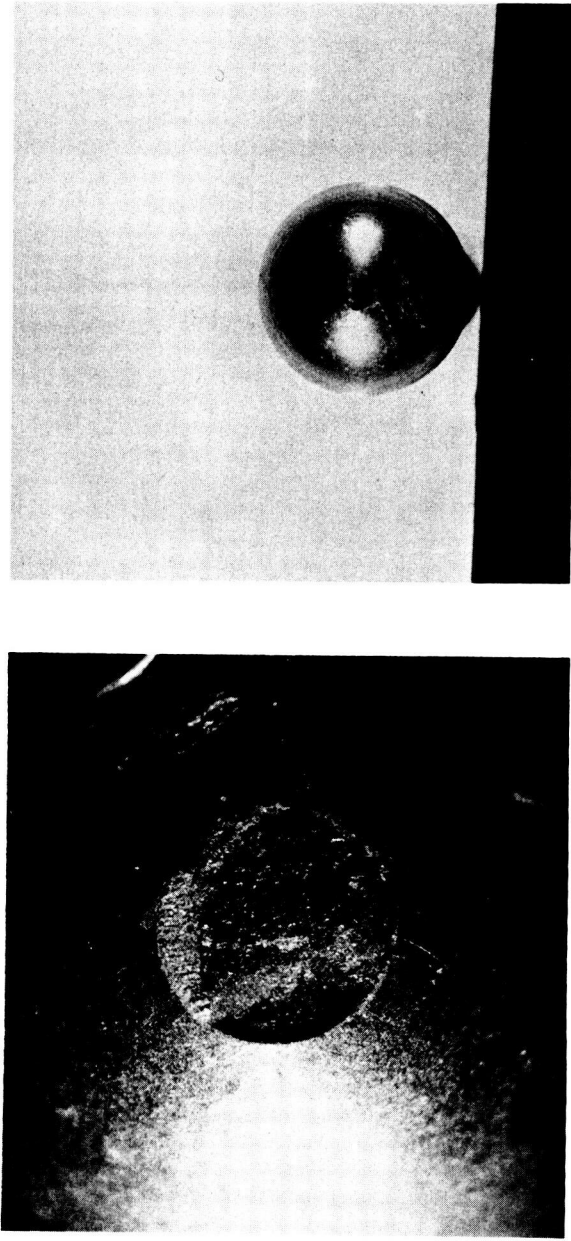
Change - -

Material Density, Gms/Cm³ -

14.301

Volume Change, Mm³ -

Wear Rate, In³/10¹⁰ft -



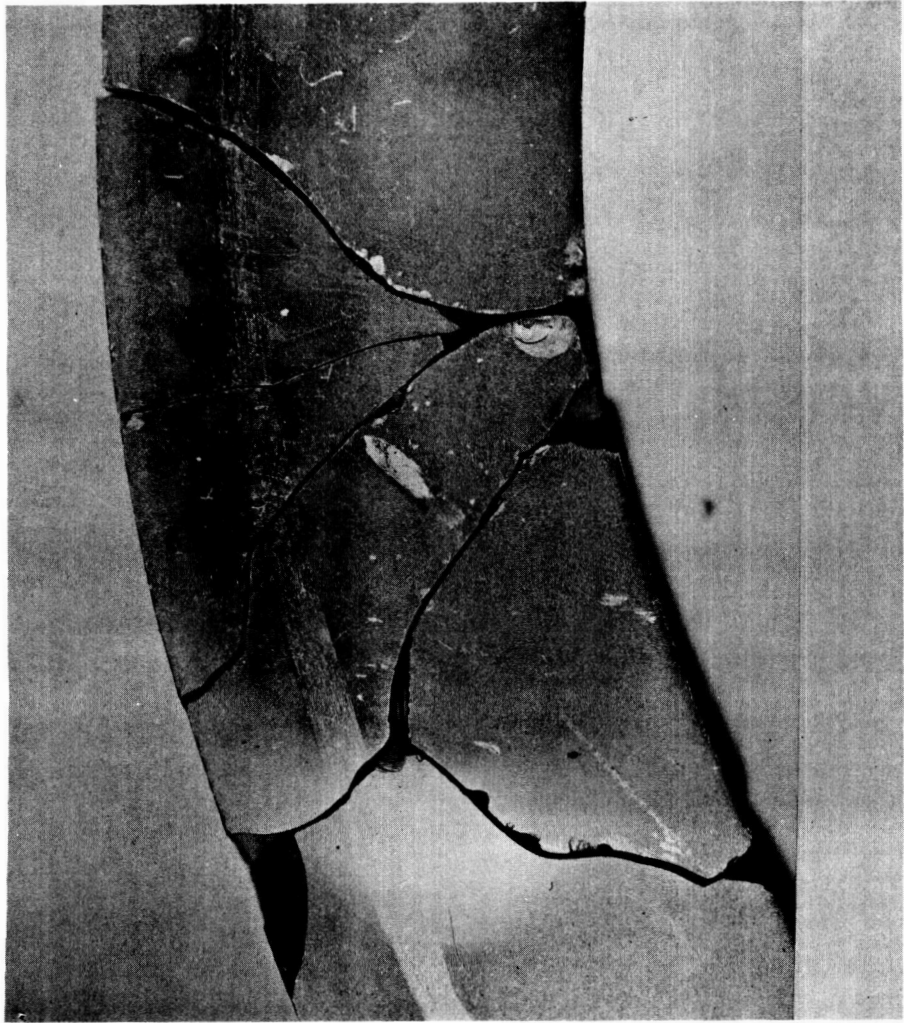
(C66081672)

Mag.: 28.5X

(C651223119)

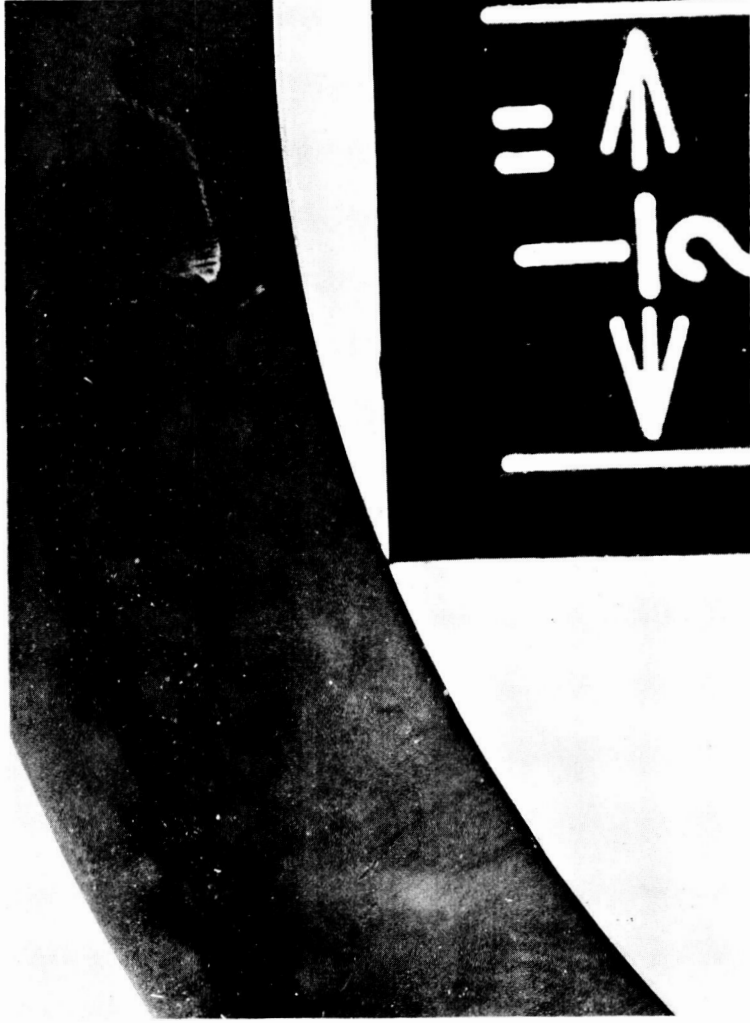
Mag.: 5X

108-1



(C65112630)

Mag.: 5X



(C651223157)

Mag.: 5X

Initial Surface Finish, Avg. RMS - 1

Wear Scar Width, In. - 0.094

Weight, Gms /Cm³ -

Start - 211.2902

Finish - (See remarks)

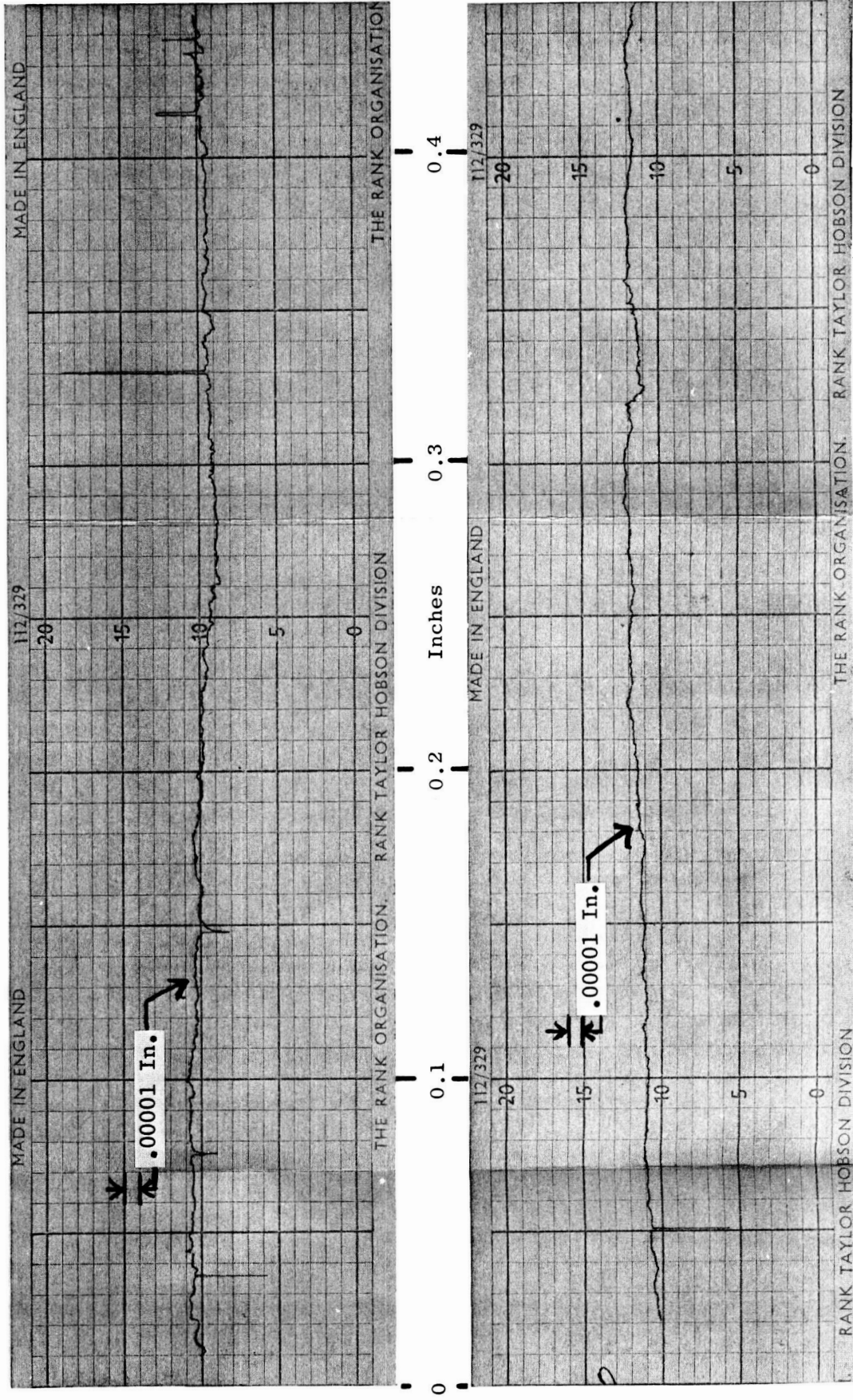
Change - -

Material Density, Gms /Cm³ - 14.301

Volume Change, Mm³ - -

Wear Rate, In³/10¹⁰ft - -

Post-Test Profilometer Trace - Circumferential at S/N



Post-Test Profilometer Trace - Radial at S/N

109-1

FRICION AND WEAR TEST DATA FOR GRADE 7178 VS Mo-TZM ALLOY IN HIGH VACUUM

Test No. - 200K08A

Assembly No. - IX

Loading Arm No. - 2

Test Date - 11/11/65

Rider

Material - Grade 7178

Specimen No. - 1046-E-9

Disc

Material - Mo-TZM

Specimen No. - 1037-F-5A

Test Temperature, °F - 80

Max. ΔT of Rider, °F - 30

Speed, SFM - 800

Test Duration, Min. - 60.00

Compressive Load, Lbs - 0.081(K)

Compressive Stress, psi - 95,180

Load/Material UCS or 0.2%CYS -

Rider - 14%

Disc - 87%

Chamber Pressure, Torr -

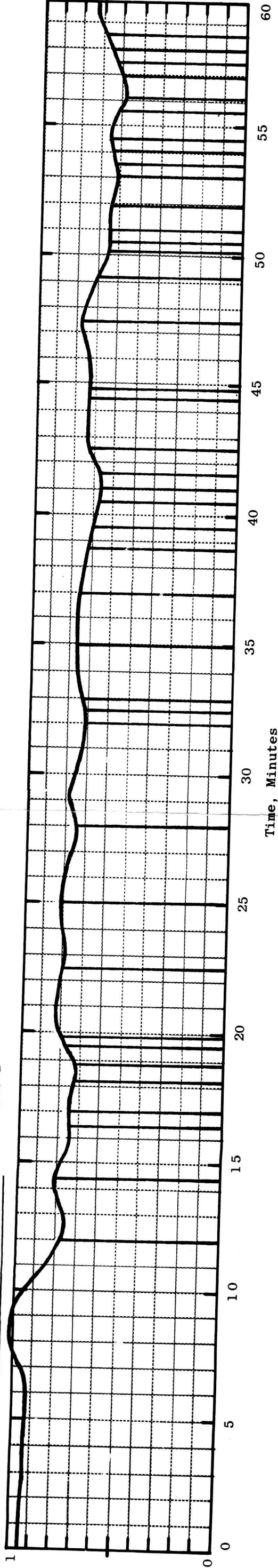
Start - 1.0×10^{-9}

Max. - 1.6×10^{-9}

Remarks - Fracture of another specimen after this test was completed damaged these specimens and voided wear measurements.

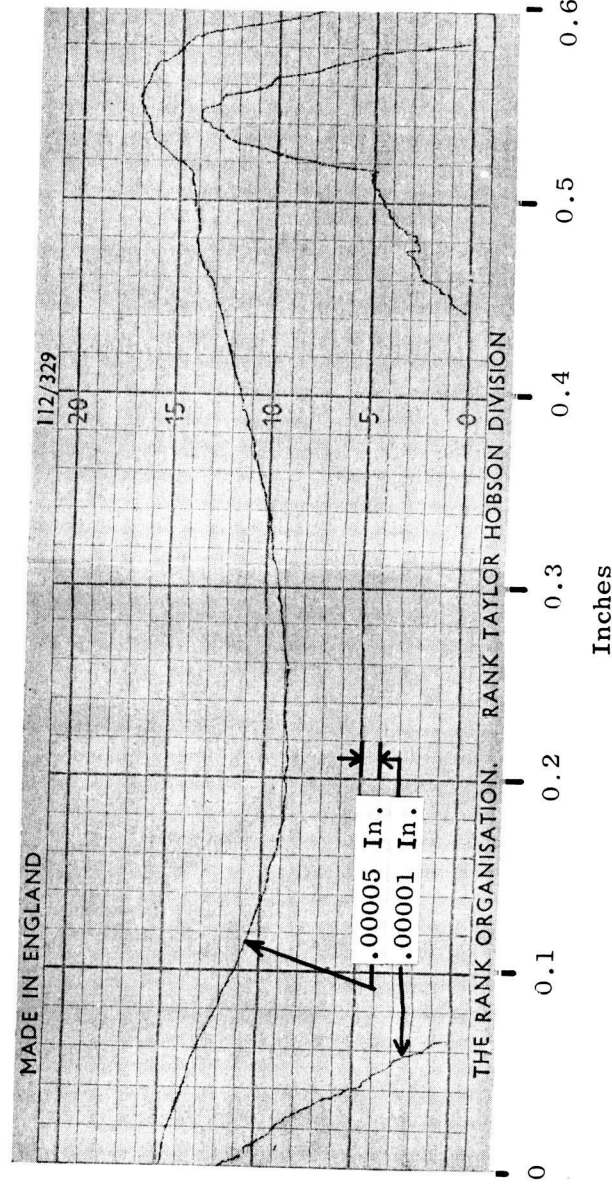
Average Coefficient of Friction - 0.79

Change in Coefficient of Friction with Time - See Note 1



WEAR - RIDER

Initial Surface Finish -



Wear Scar Dia., In. - 0.094

Weight, Gms -

Start - 3.0957

Finish - (see remarks)

Change -

Material Density, Gms/Cm³ -

14.301

Volume Change, Mm³ -

Wear Rate, In³/10¹⁰ft -

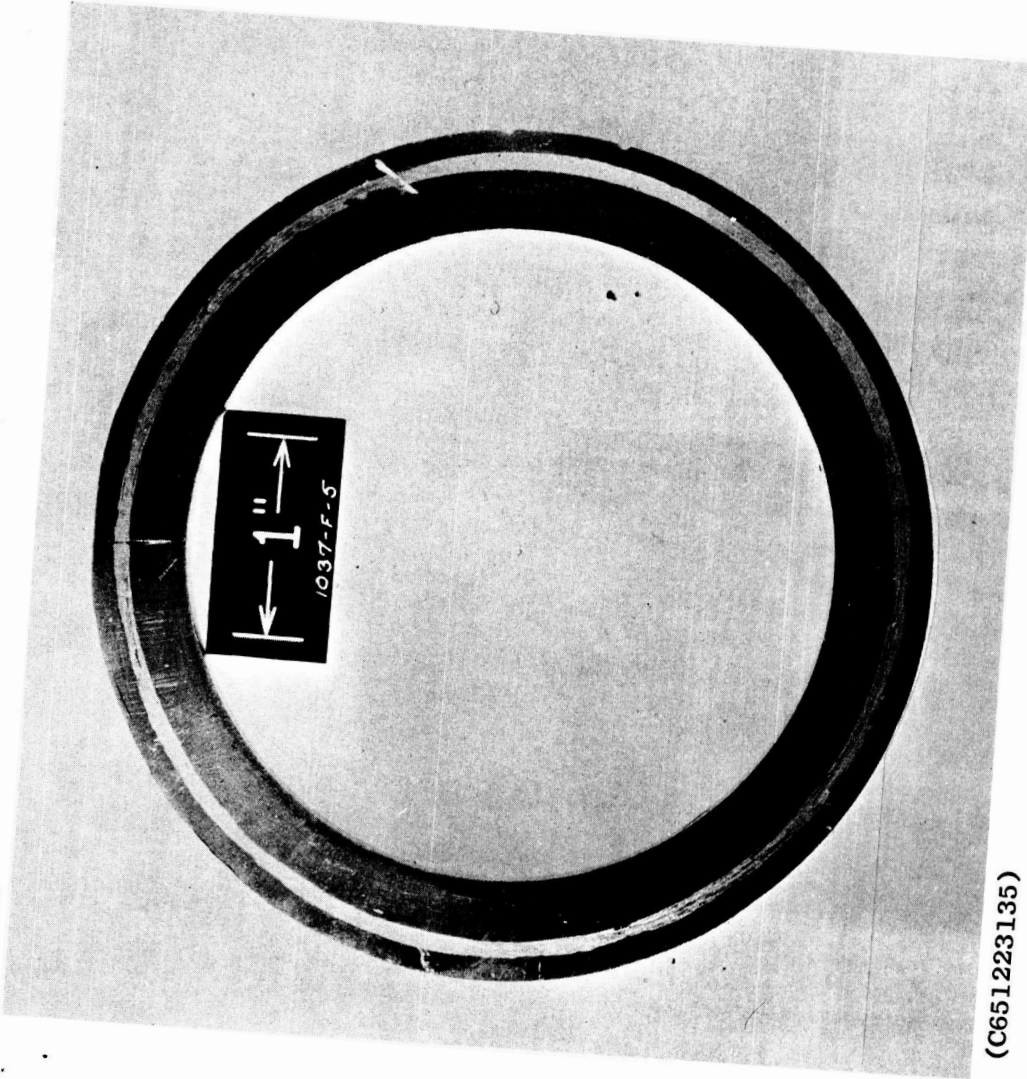
(C66081613)

Mag.: 28.5X

(C651223127)

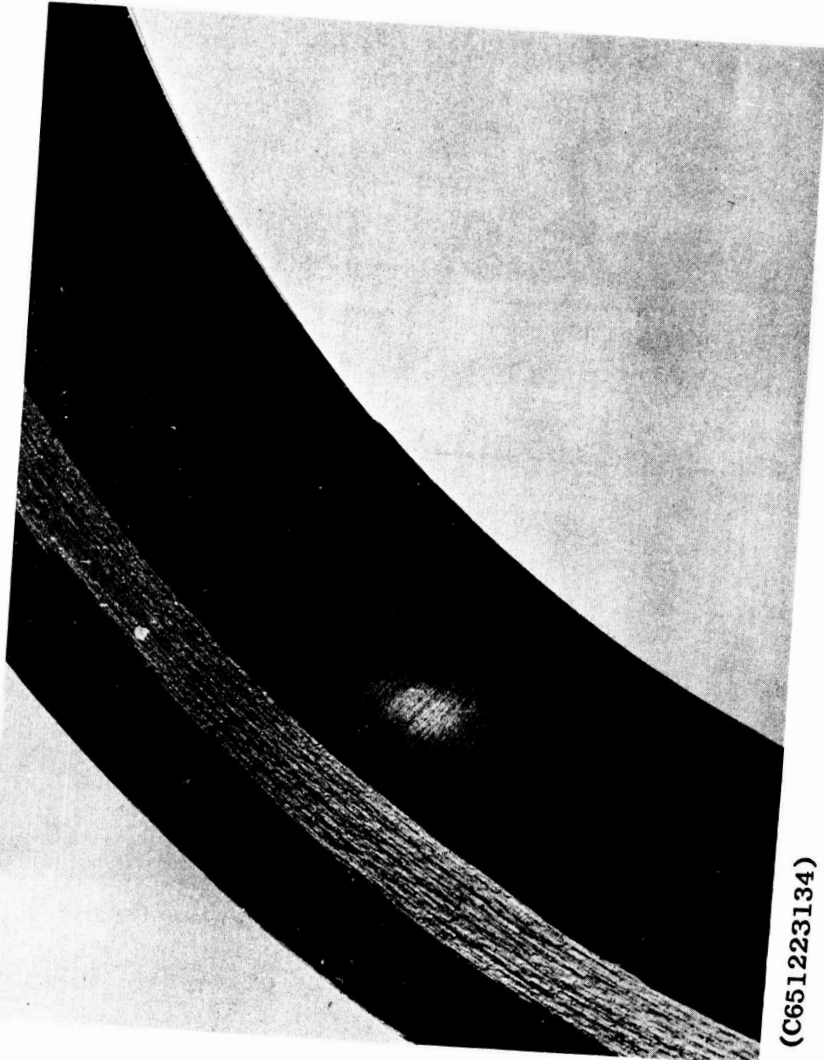
Mag.: 5X

WEAR - DISC



(C651223135)

Mag.: 1X



(C651223134)

Mag.: 5X

Test No. - 200K08A

Initial Surface Finish, Avg. RMS - 1-3

Wear Scar Width, In. - 0.094

Weight, Gms /Cm³ -

Start - 156.4966

Finish - (see remarks)

Change -

Material Density, Gms /Cm³ - 10.139

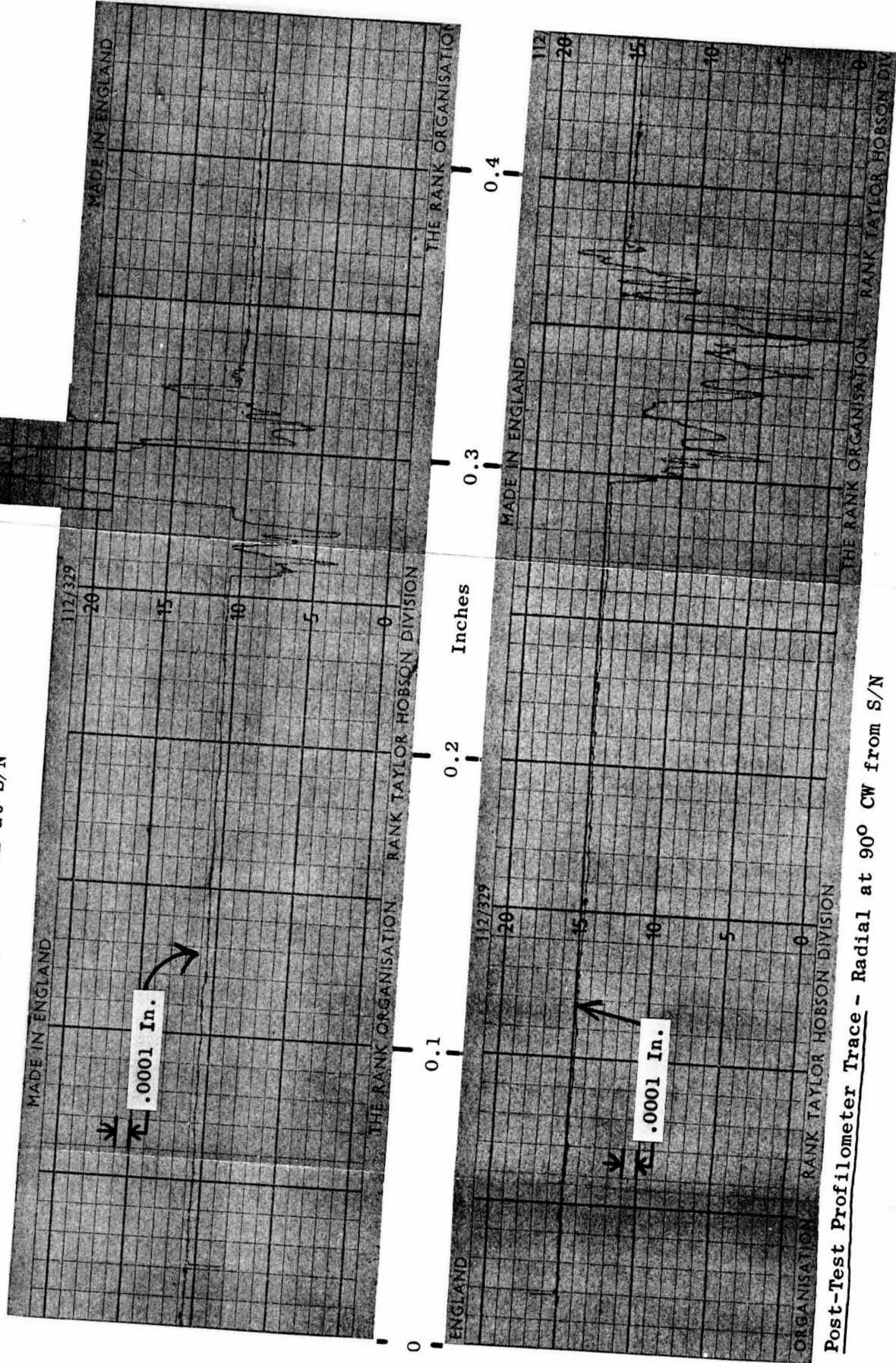
Volume Change, Mm³ -

Wear Rate, In³/10¹⁰ft -

Notes

- (1) Vertical lines indicate momentary zero readings believed due to violent bouncing of the loading arm which caused it to contact and stick to other hardware.

Post-Test Profilometer Trace - Radial at S/N



Post-Test Profilometer Trace - Radial at 90° CW from S/N

FRICION AND WEAR TEST DATA FOR Mo-TZM ALLOY VS TiC+10%Cb IN HIGH VACUUM

Test No. - 600K08B

Assembly No. - IX

Loading Arm No. - 3

Test Date - 11/12/65

Rider

Material - Mo-TZM

Specimen No. - 1037-E-10

Disc

Material - TiC+10%Cb

Specimen No. - 1045-F-7A

Test Temperature, °F - 80

Max. ΔT of Rider, °F - 60

Speed, SFM - 800

Test Duration, Min. - 60.00

Compressive Load, Lbs - 0.082 (K)

Compressive Stress, psi - 89,530

Load/Material UCS or 0.2%CYS -

Rider - 82%

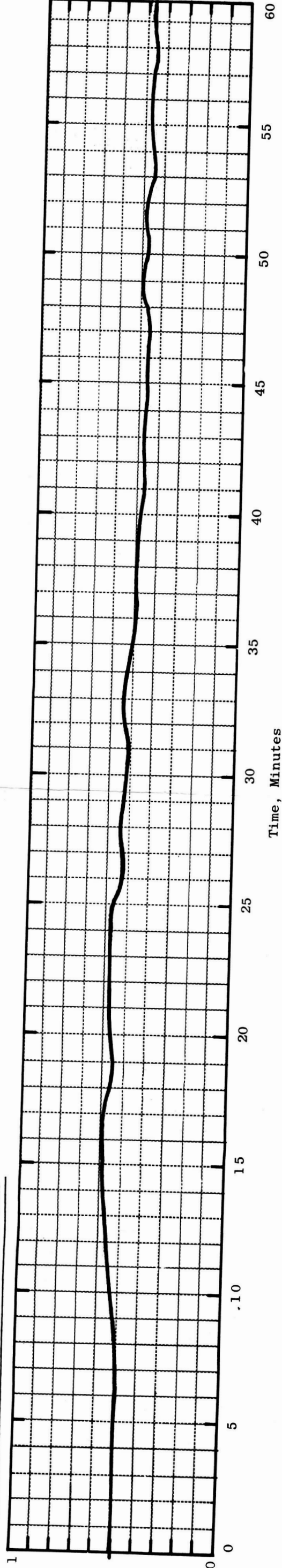
Disc - 26%

Average Coefficient of Friction - 0.51

Chamber Pressure, Torr -
Start - 7.0×10^{-10}
Max. - 1.1×10^{-9}

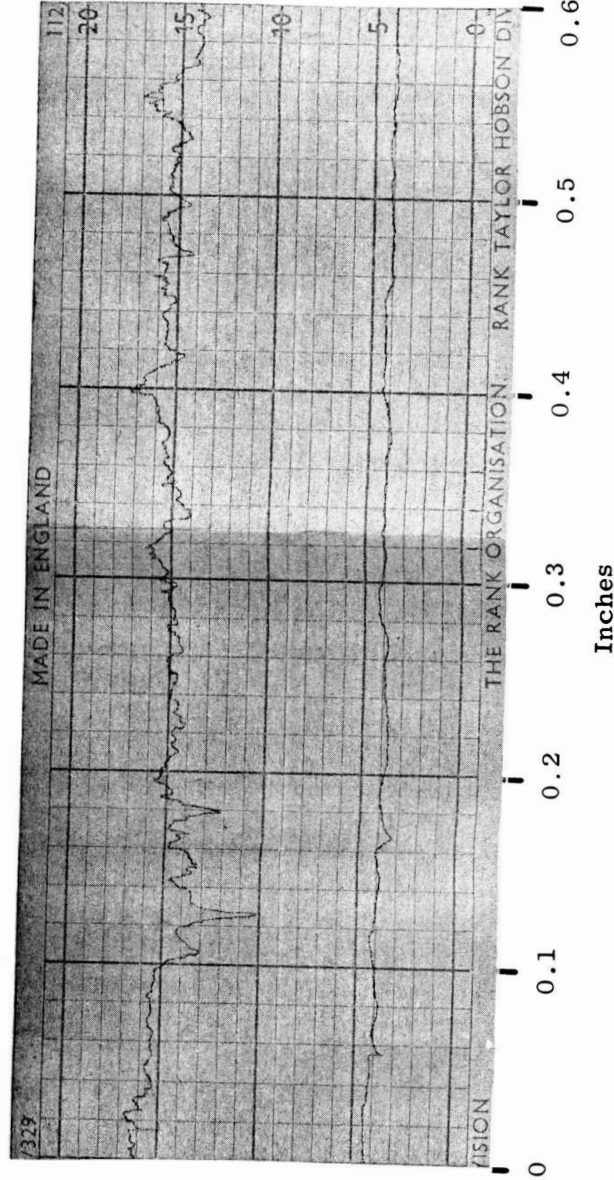
Remarks - Fracture of another specimen after this test was completed damaged these specimens and voided wear measurements.

Change in Coefficient of Friction with Time -



WEAR - RIDER

Initial Surface Finish -



Wear Scar Dia., In. - 0.047

Weight, Gms -

Start - 2.3870

Finish - (see remarks)

Change -

Material Density, Gms/Cm³ -

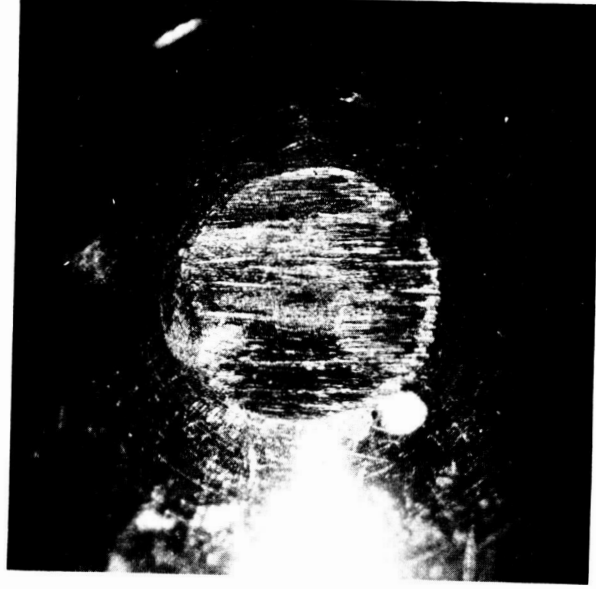
10.139

Volume Change, Mm³ -

-

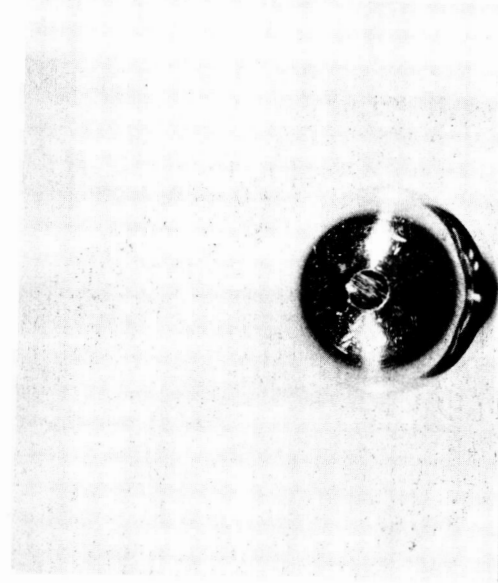
Wear Rate, In³/10¹⁰ft -

-



(C66081612)

Mag.: 28.5X

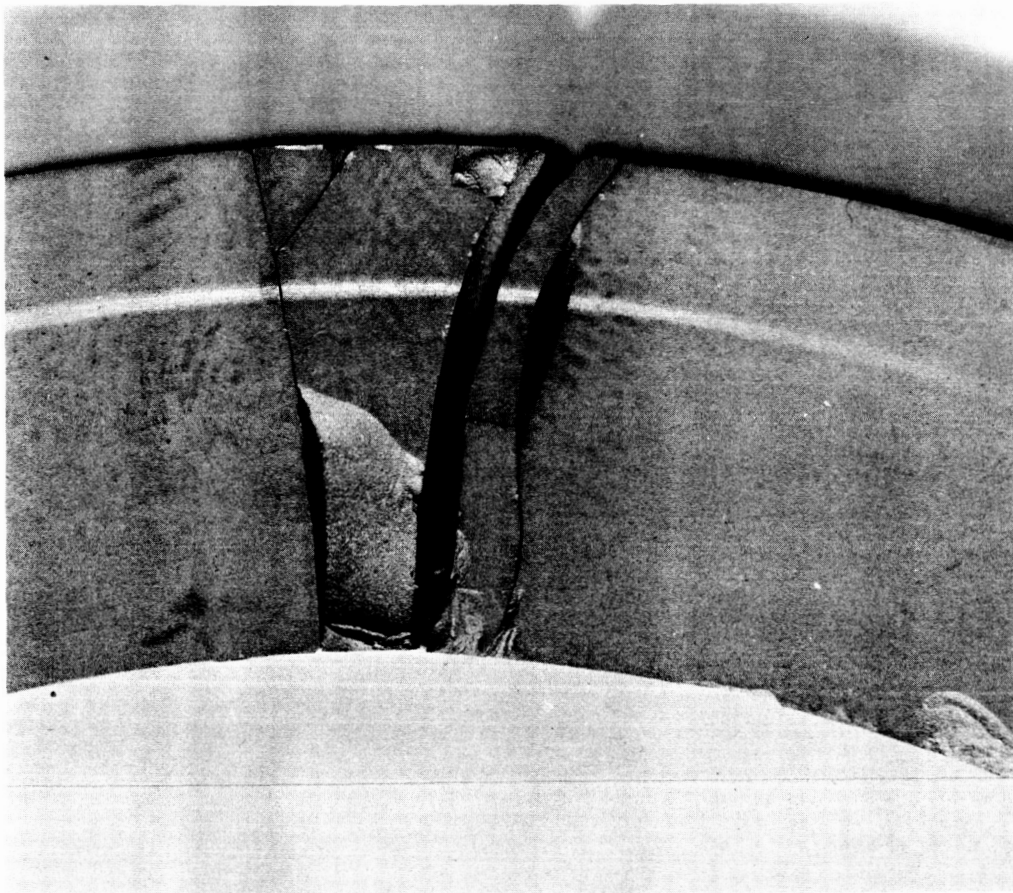


(C651223116)

Mag.: 5X

-112- 2

WEAR - DISC



(C65112629)

Mag.: 5X



(C651223156)

Mag.: 5X

Initial Surface

Wear Scar Width

Weight, Gms /C

Start - 82.2

Finish - (see

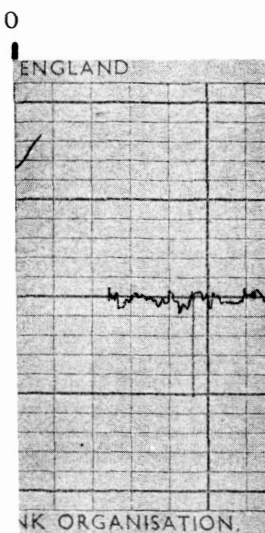
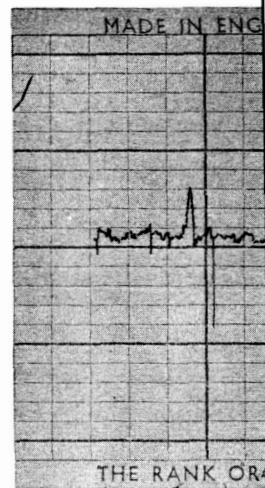
Change -

Material Densi

Volume Change,

Wear Rate, In³

Post-Test Prof



Post-Test Profi

113-1

sh, Avg. RMS - 3.5-5

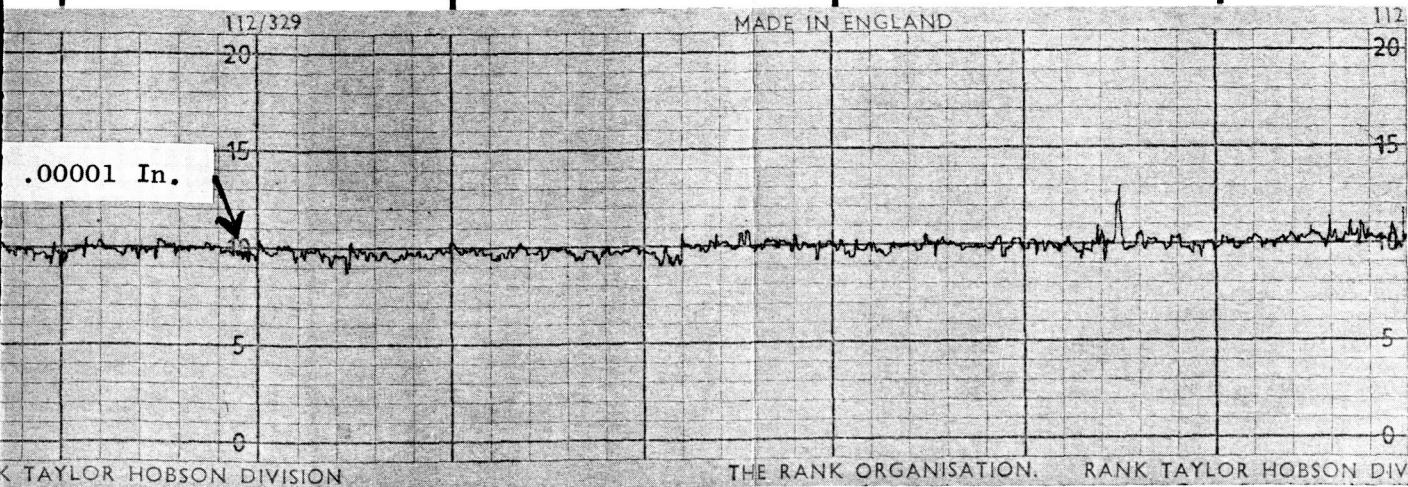
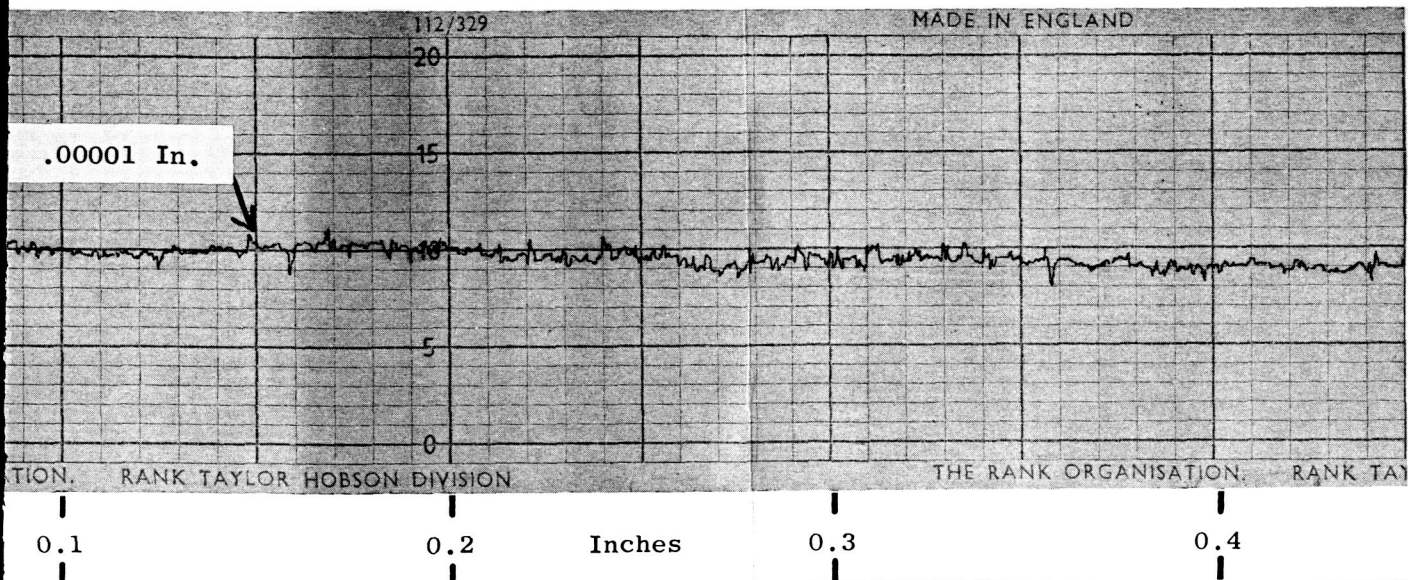
- 0.016

cks)

s /Cm³ - 5.239

t - -

ter Trace - Circumferential at S/N



ter Trace - Radial at S/N

113-2

~~113-2~~ ~~113-2~~

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