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SOME EXPERIENCES IN THE WELD FABRICATION OF REFRACTORY METALS

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FOR PRESENTATION AT THE
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THE MARQUARDT CORPORATION
VAN NUYS, CALIFORNIA

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I. INTRODUCTION

Information presented in this paper was obtained from contributing engineering studies conducted at The Marquardt Corporation in support of Air Force Contract AF 33(600)-40636.

The discussion of welding fabrication of refractory metals will, in this paper, be restricted to alloys of the four elements: tungsten, molybdenum, tantalum, and columbium. These are all transition elements, columbium and tantalum being elements of group 5b and molybdenum and tungsten, elements of 6b.

Some of the properties which make the refractory metals important are listed in Table 1. Included in this tabulation for comparison is titanium. Only the element, carbon, exceeds the melting point of tungsten. Tantalum has the fourth highest melting point of any metal and molybdenum, the fifth. All four metals have low thermal coefficients of expansion. The thermal conductivities of molybdenum and tungsten are excellent, being exceeded only by those commonly known metals; aluminum, magnesium, copper, silver, and gold. Densities of the refractory metals are high. Only platinum, osmium, and irridium of the precious metals group and rhenium have greater densities than tantalum and tungsten. The specific heats of tantalum and tungsten are very low. Uranium and some members of the precious metals group are the elements having lower specific heat values. In a consideration of moduli only rhenium and again, members of the precious metals group; osmium, ruthenium, irridium, and rhodium are superior to molybdenum and tungsten.

The melting points and densities of this group of metals contribute to a high heat input for fusion. This requirement is tempered by the low specific heat characteristics of the metals. High thermal conductivities and low thermal coefficients of expansion result in good heat impact resistance. High thermal conductivity also assists in rapid quench of the weld, promoting grain refinement.

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However, when preheat is required for welding molybdenum and tungsten, maintaining a desired temperature becomes a problem with good thermal conductivity. The refractory metals have two other well known characteristics which make them difficult to fabricate by welding. All are reactive to air at slightly elevated temperatures and all except tantalum undergo a ductile-brittle transition.

As shown in Figure 1,[1] the refractory metals oxidize rapidly at high temperatures. Since the oxide of molybdenum becomes volatile at about 1400°F, loss of metal above this temperature occurs at a catastrophic rate. The influences of oxygen and nitrogen on the ductility of molybdenum have been the subject of many reports, among them articles by Wulff[2] and Rengstorff, et al[3]. With respect specifically to the effects of these elements on molybdenum weldments, Perry, et al[4] postulated that Mo-MoO_2 eutectic which forms above 2100°C is responsible for poor weld ductility by forming brittle grain boundary films. Platte[5,6] has made quantitative studies of the embrittling effects of oxygen and nitrogen on molybdenum welds. More recently, Begley[7] reported on the embrittlement of columbium welds by oxygen and nitrogen.

An excellent review of the knowledge on ductile-brittle transition in refractory metals has been compiled by Schwartzberg, et al[8]. The actual temperature of transition varies depending on the type of test used for evaluation. Using a slow bend test, it may be stated generally that tungsten has a transition temperature of 400°F or higher, molybdenum undergoes transition in behavior around room temperature and columbium has a transition temperature considerably below room temperature. No transition temperature for tantalum has been found.

The reactive nature of the refractory metals and their pronounced embrittlement by oxygen, nitrogen, and hydrogen in the ppm range require that material to be welded be chemically cleaned, inert shielding gases be of maximum obtainable purity, and inert-gas, arc shielding techniques be highly refined. For those

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elements and their alloys which have ductile-brittle transition temperatures at or above room temperature, imposition of welding stresses and a weak, extremely brittle, cast structure further require the use of preheat to avoid weld cracking.

Among the refractory metals and alloys whose welding characteristics have been studied are those listed in Table 2. Programs have been primarily concerned with sheet materials, although molybdenum plate, bar, and tubing have also been welded.

Extensive evaluation has been conducted using the gas, tungsten arc welding process employing both manual and machine welding and is reported herein. Design data has been obtained exclusively from machine welded sheet materials. Flash welding, resistance spot welding and brazing, electron beam welding, and high frequency resistance welding processes have also been applied to molybdenum alloys.

II. WELDING STUDIES

A. Molybdenum Base Materials

Experience with molybdenum, especially material processed from arc-cast ingots, is extensive. Early in the refractory metals program, some effort was concentrated on sintered materials, but their generally unfavorable welding characteristics soon led to discontinuance of studies. Some interesting facts were learned about welding the sintered materials, however, and these will be presented.

Both the hydrogen sintered and vacuum sintered, pure molybdenum grades were found unweldable using the inert arc process without filler additions. Excessive weld bead porosity, and centerbead cracks shown in Figure 2b were evidence of the gaseous content of these materials. By the use of a filler wire of arc-cast, molybdenum-0.5% titanium alloy, sufficient deoxidation of the molten metal by carbon and titanium could be obtained to eliminate cracking as shown in Figure 2a. Open pits caused by evolution of gases during welding were still present at the edges of the weld bead however. The maximum degree of quality was obtained by electron beam welding such material. Not only were cracks eliminated, but porosity was greatly reduced as shown in Figure 2c.

The sintered molybdenum-titanium alloy which has a nominal analysis identical to the commercial arc-cast alloy, was found to be weldable. However, it had several markedly different characteristics. During welding the arc gave off a soft, crackling sound and fine blue lines appeared perpendicular to, and at the edges of the weld bead. Since it was expected that the interstitial content of this material was high, the occurrences were assumed to be evidence of deoxidation of the molten metal during welding. Analyses of parent and weld metals proved this was true, and oxygen content had been reduced from 280 ppm to 220 ppm. The weld profile was also different, the edges of the bead being sharply undercut. Reference to Figure 3 allows a visual comparison of welds made under identical conditions in sintered pure, sintered alloy, and arc-cast, alloy molybdenum sheet all of 0.040 inch thickness. In Figure 3a are shown the open pits caused by gas evolution and the gross weld cracking resulting from the high residual oxygen and nitrogen content of the weld metal in sintered, pure molybdenum. The blue discoloration adjoining the weld bead and the undercut condition at the bead edges typical of a weld in the sintered alloy material are shown in Figure 3b. A photograph of the clean, sound weld obtained in the arc-cast alloy grade is shown in Figure 3c.

The sintered alloy was found to be more susceptible to weld center bead cracking and less susceptible to transverse weld cracking than the arc-cast alloy. Welds were also found to have no or negligible porosity. Property data on welded tensile specimens of the sintered, molybdenum-0.5% titanium alloy are reported in Table 3. These specimens were fully recrystallized during application of an oxidation-resistant coating (Chromalloy W2) by the pack cementation process.

Arc-cast, commercially pure molybdenum traces its weldability to deoxidation by carbon during melting and to melting under vacuum. These factors lower the oxygen plus nitrogen content to the order of 15 ppm. No improvement in weldability of arc-cast material has been noted as a result of the titanium alloy addition. Apparently the deoxidation reaction has proceeded to its limits during ingot melting. Tensile properties of welded arc-cast, molybdenum-0.5% titanium alloy are reported in Table 4.

The recently commercialized TZM alloy has proved more difficult to weld than the titanium alloy. Photomicrographs of weld grain boundaries in these two alloys reveal the reason. Figure 4 shows grain boundary contamination in TZM to be greatly in excess of that in molybdenum-0.5% titanium alloy accounting for the increased weld cracking susceptibility of the former. While grain boundary contaminants of the TZM alloy have not been analyzed, it is assumed they are associated with the zirconium addition. Oxygen and nitrogen analyses presented in Table 5 of both TZM and molybdenum-titanium alloys have proved the effects cannot be correlated with a marked difference in ppm of these impurities in the parent material. Welding evaluation of this alloy is limited to a single heat of material. Studies of additional heats are required to verify initial results.

B. Columbium - Base Materials

Both Fansteel 80 and Fansteel 82 alloys have been welded. These alloys are both ductile materials which suffer only slight embrittlement when welded. Weld bead cracking is not encountered unless inert gas protection is poor.

Sheet 0.040 inch thick has been welded at speeds of 10 ipm and 100 ipm without filler addition or preheat. Transverse weld macrostructures showing the effect of welding speed on fusion zone grain orientation and weld profile are presented in Figure 5.

Some limited data has been obtained on welded tensile specimens and is presented in Table 6. Strength-wise, these alloys fall well below molybdenum-0.5% titanium alloy.

C. Tantalum - Base Materials

Hydrogen sintered, commercially pure tantalum and both arc-cast and electron-beam melted tantalum-10% tungsten alloy sheet have been machine welded without difficulty. If anything, these alloys are even more ductile than the columbium base alloys mentioned. Fine edge bead porosity has been found in welds in the sintered, pure tantalum. This is a common occurrence and is attributed to the interstitial content of the material. It has been reported by Haslip, et al[9] that such porosity does not occur in welds in arc-cast, pure tantalum. Because of its low elevated temperature strength, only a few tensile tests were conducted on welded specimens of the pure material. The data is presented in Table 7. The tantalum-10% tungsten alloy appears to have high temperature strength properties competitive with the molybdenum-0.5% titanium alloy but studies have not advanced to the stage of obtaining weld property data.

D. Tungsten - Base Materials

The only available commercial sheet material is processed from hydrogen-sintered, tungsten bars. This product has the high interstitial analysis typical of sintered molybdenum sheet. This factor in addition to its inherently higher ductile-brittle transition temperature makes it much more difficult to weld than molybdenum. Success has been limited to manual welds made in an inert gas, purge chamber and to a few automatically welded panels of 0.020-inch thick

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sheet. The automatic welds were made at speeds of 20,60, and 80 ipm and preheats of 400-450°F. While a number of welds were produced which were free from cracks, visual inspection revealed the presence of tiny pits in the weld bead surface. Radiographs confirmed the presence of gross weld bead porosity shown in Figure 6.

From the experience gained thus far, it appears that high quality manual welding of tungsten will be more easily accomplished than automatic welding. The chief drawback to automatic welding is preheat temperature requirements which are estimated to be 1000°F. At this temperature, tungsten oxidizes rapidly and complicates inert gas shielding of the weld joint.

III. FABRICATION

Fabrication thus far has been restricted primarily to molybdenum or tantalum components. In addition, a number of simple forming tests have been conducted to demonstrate the elevated temperature ductility of molybdenum welds.

Figure 7 shows sections from a molybdenum weld which were reduced at temperatures of 400-600°F. A pair of cracked molybdenum cups which were successfully repaired by manual welding techniques adding 50 Mo-50 Re or molybdenum-0.5% titanium filler wire is shown in Figure 8. These cups were later spun to the shapes shown in Figure 9. Molybdenum-rhenium welds failed by ductile tearing; molybdenum-titanium weldments cracked. These tests showed molybdenum weldments capable of accepting a surprising amount of working at elevated temperatures before failure.

A tantalum tube fabricated by forming and manually welding sheet 1/16 inch thick is shown in Figure 10. While some oxidation occurred as indicated by discolored areas the weld was free from cracks. The part is now in service. Another tantalum assembly is shown in Figure 11. Fabrication consisted of welding tantalum tubes having wall thicknesses of 0.015-inch and 0.030-inch to a tantalum "test tube" having a 1/8-inch thick wall. The unit brazed to the top of the "test tube" is a copper water cooling system.

Figure 12 is an example of tungsten hardware. A pair of tungsten emitters were fabricated by forming and manually welding 1/16-inch diameter to 1/8-inch diameter wire.

The sequence of assembly of the most complicated manually welded molybdenum component yet attempted is shown in Figure 13. All welds were made using molybdenum-0.5% titanium filler wire.

Figure 14 is a molybdenum cylinder made from 0.040-inch thick sheet rolled up and machine welded. No filler wire was added to the weld. A molybdenum hyperjet exit nozzle produced from a pair of spun sections by machine welding a circumferential joint is the subject of Figure 15. This assembly was later incorporated into the hyperjet tailpipe of Figure 16. The tailpipe consists of a combustion chamber fabricated from two longitudinally welded cylinders joined by a circumferential weld and then attached by another circumferential weld to the nozzle assembly. Sheet thicknesses vary from 0.040-inch to 0.050-inch. This item represents the most advanced application of welded molybdenum hardware produced by The Marquardt Corporation.

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TABLE 1

Physical Properties of the Refractory Metals

Element	Melting Point (°F)	Density (g/cc)	Thermal Conductivity ($Cal/Cm/Cm^2/sec/°C$)	Specific Heat Cal/G/°C	Coefficient of Linear Thermal Expansion Coefficient/°C$\times 10^{-6}$
Tungsten	6152	19.3	0.31	0.034	4.45
Molybdenum	4752	10.2	0.382	0.062	5.35
Tantalum	5425	16.6	0.130	0.036	6.5
Columbium	4379	8.57	0.13	0.065	7.1
Titanium[1]	3300	4.54	—	0.126	8.5

1. Included for comparison

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TABLE 2

Materials Welding Experience

Base Metal	Production Process	Alloy Designation	Nominal Analysis (Weight Percent)						
			C	Ti	Zr	Ta	W	Cb	Mo
Molybdenum	Hydrogen-sintered	Commercially pure							100
	Vacuum sintered	Comercially pure							100
	Hydrogen sintered	Ti-Mo		0.5					bal.
	Arc cast	Ti-Mo	0.02	0.5					bal.
	Arc cast	TZM	0.02	0.5	0.07				bal.
	Arc cast	Carbon Deoxidized	0.02						bal.
Columbium	Arc cast	Fansteel 80			0.9			bal.	
	Arc cast	Fansteel 82			0.4	40		bal.	
Tantalum	Hydrogen sintered	Commercially pure				100			
	Arc cast	Ta-10W				bal.	10		
	Electron beam melted	Ta-10W				bal.	10		
Tungsten	Hydrogen sintered	Commerically pure					100		

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TABLE 3

Tensile Properties of Welded Specimens of Hydrogen-Sintered, Molybdenum-0.5% Titanium Alloy Sheet

Test Temperature (°F)	UTS (ksi)	0.2% YTS (ksi)	Elongation (% in 2-in.)	Failure Location
2000	34.1	20.8	8.8	Parent Metal
2400	20.0	14.2	5.0	Parent Metal
2600	18.6	13.6	6.2	Parent Metal
3000	14.3	11.7	5.8	Parent Metal

Test Conditions

Heating - Resistance

Hold Time - 2 minutes

Oxidation Resistant Coating - Chromalloy W-2

Test Atmosphere - Air

Strain Rate - 0.001 in/in/sec to yield

0.01 in/in/sec to rupture

TABLE 4

Tensile Properties of Welded Specimens of Arc-Cast, Molybdenum-0.5% Titanium Sheet

Test Temperature (°F)	UTS (ksi)	0.2% YTS (ksi)	Elongation (% in 2 in.)	Location of Failure
80[1]	130.0	119.0	1.0	Weld Fusion Zone
400[1]	71.0	66.0	1.3	Weld Fusion Zone
2000	44.5	44.5	1.4	Weld Heat Affected Zone
2200	36.6	35.0	1.2	Weld Heat Affected Zone
2400	29.5	18.9	1.9	Parent Metal
2600	21.0	11.6	7.0	Parent Metal
2800	15.7	8.6	6.7	Parent Metal
3000	11.5	6.7	9.4	Parent Metal
3200	8.0	4.2	8.8	Parent Metal

1. Uncoated Specimens

Test Conditions

Heating - Resistance

Hold Time - 2 Minutes

Oxidation Resistant Coating - Durak MG

Test Atmosphere - Air

Strain Rate - 0.001 in/in/sec to yield
0.01 in/in/sec to rupture

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TABLE 5

Comparison of Interstitial Analyses of Sheet and Weld Bend Transition Temperatures

Material	Heat Identity	Oxygen	Nitrogen	Weld Bend Transition Temperature
TZM	BZ	7.7 ppm	3.6 ppm	350 - 400°F
Molybdenum-0.5% titanium	U	11.0	4.6	225 - 250°F
Molybdenum-0.5% titanium	J	16.0	5.0	300

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TABLE 6

Tensile Properties of Welded Specimens of Arc-Cast, Fansteel 80 and Fansteel 82 Sheet

Material	Temperature (°F)	UTS (ksi)	0.2% YTS (ksi)	Elongation (% in 2 in.)	Location of Failure
Fansteel 82	2000	32.8	27.0	6.2	Parent metal and HAZ
	2400	14.6	9.1	11.5	Parent Metal
	2800	11.9	8.5	12.5	Parent Metal
Fansteel 80	2000[1]	26.6	24.2	2.0	Weld HAZ
	2400	15.9	12.3	13.5	Parent Metal
	2800	5.6	3.6	16.0	Parent Metal

1. Single test value. All others average of two tests.

Test Conditions

Heating - Resistance

Hold Time - Two minutes

Oxidation Resistant Coating - none

Atmosphere - Welding grade argon

Strain Rate - 0.001 in./in./sec. to yield
0.01 in./in./sec. to rupture

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TABLE 7

Tensile Properties of Welded Specimens of Hydrogen-Sintered, Commercially Pure, Tantalum Sheet

Temperature (°F)	UTS (ksi)	0.2% YTS (ksi)	Elongation (% in 2 in)	Location of Failure
Room temperature (80°)	45.0 47.0	42.0 41.0	2.0 4.6	Weld Parent Metal
2400	14.5 16.7	4.5 2.4	23.5 28.0	Parent Metal Weld
3000	8.9 9.6	0.6 1.4	53 24	Parent Metal Parent Metal

Test Conditions

Heating - Resistance

Hold Time - 2 minutes

Oxidation Resistant Coating - Chromalloy W-2

Test Atmosphere - Air

Strain Rate - 0.001 in./in./sec. to yield
0.01 in./in./sec. to rupture

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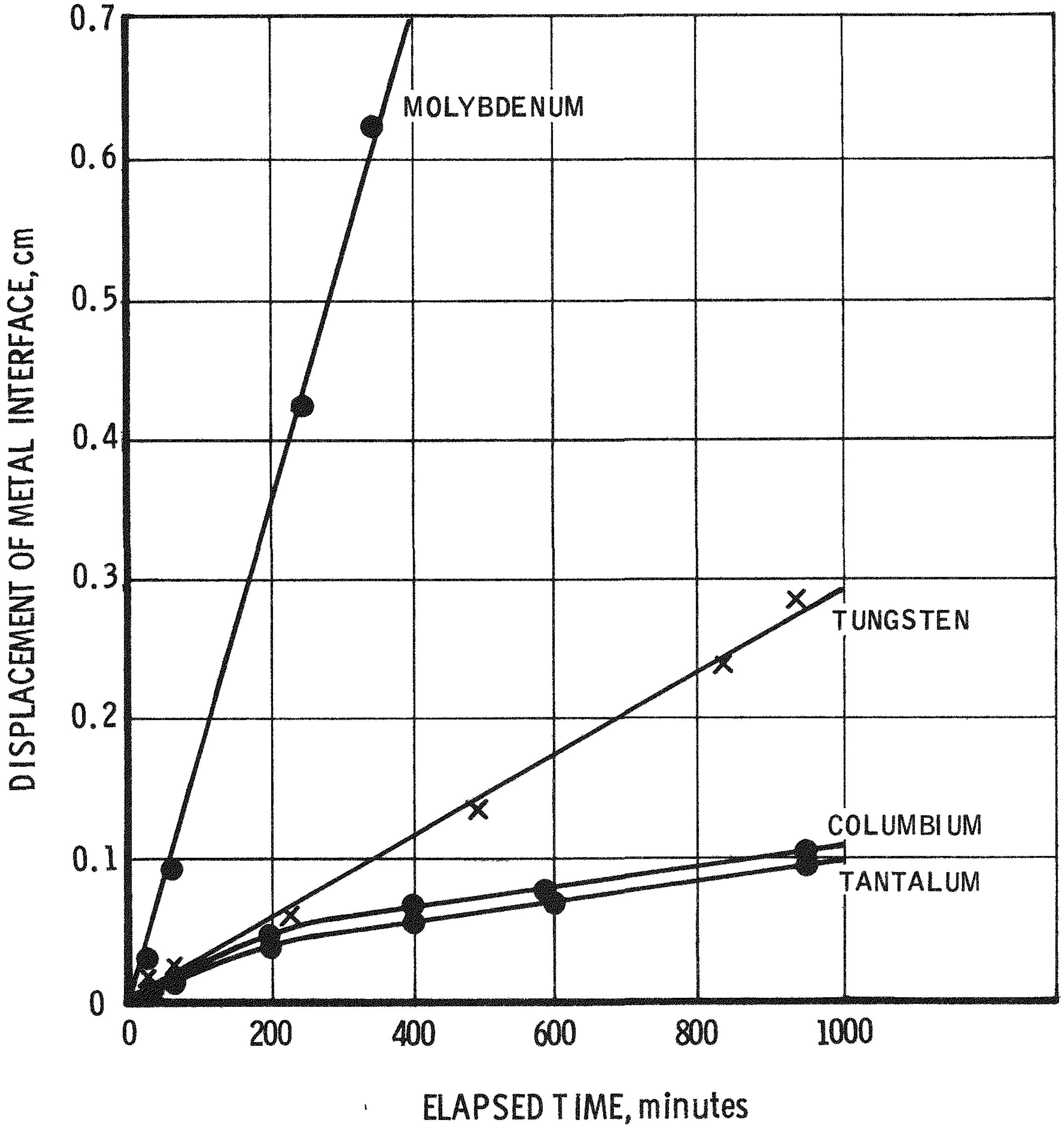
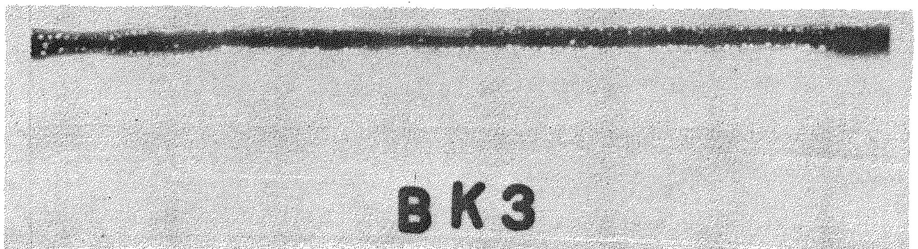
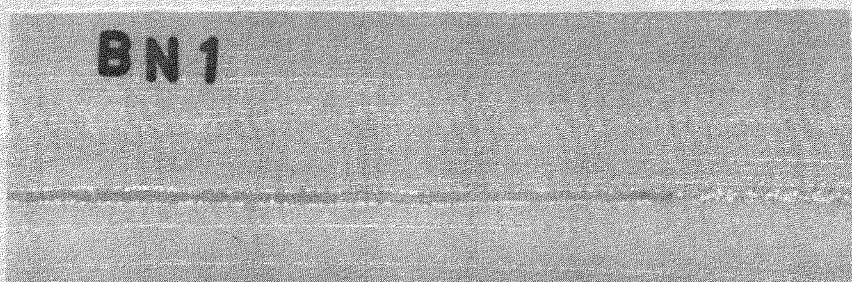


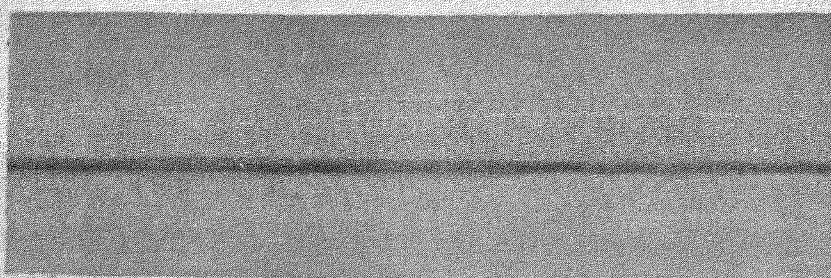
OXIDATION OF Mo, Ta, AND Cb IN FLOWING AIR AT 2000°F
(AFTER MICHAELS) (1)



a) HYDROGEN SINTERED, COMMERCIALLY PURE MOLYBDENUM TUNGSTEN-ARC INERT GAS WELDED WITH MOLYBDENUM - 0.5% TITANIUM FILLER



b) VACUUM SINTERED, COMMERCIALLY PURE MOLYBDENUM TUNGSTEN-ARC, INERT GAS WELDED WITHOUT FILLER



c) VACUUM SINTERED, COMMERCIALLY PURE MOLYBDENUM ELECTRON BEAM WELDED WITHOUT FILLER

RELATIVE POROSITIES ENCOUNTERED IN WELDING SINTERED PURE MOLYBDENUM

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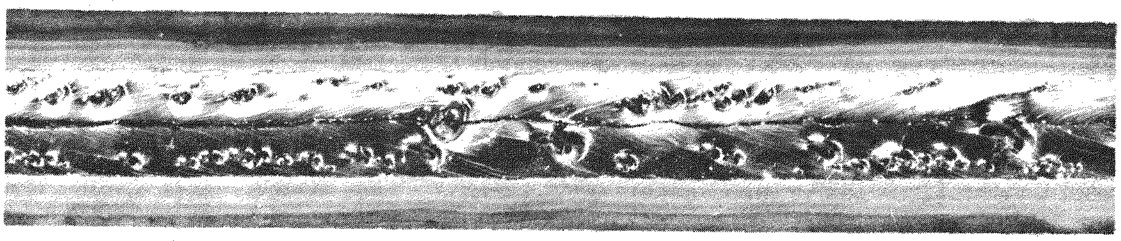
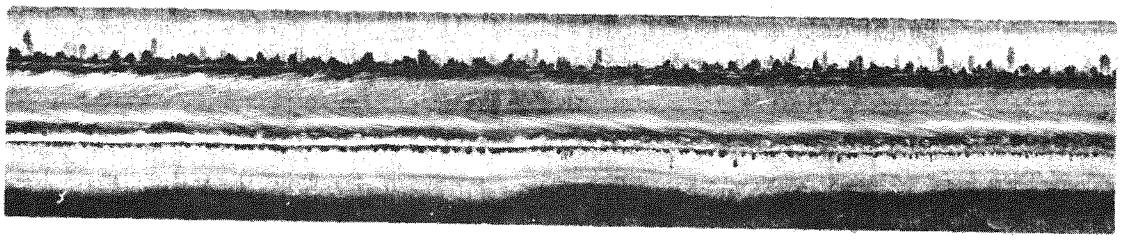
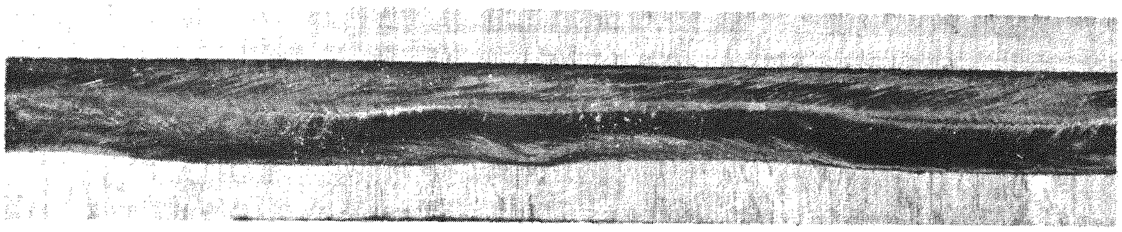


(a) HYDROGEN SINTERED, COMMERCIALLY PURE MOLYBDENUM



(b) HYDROGEN SINTERED, MOLYBDENUM - 0.5% TITANIUM



(c) ARC CAST, MOLYBDENUM - 0.5% TITANIUM

VISUAL WELD APPEARANCE FOR THREE MOLYBDENUM - BASE SHEET MATERIALS WELDED UNDER IDENTICAL CONDITIONS

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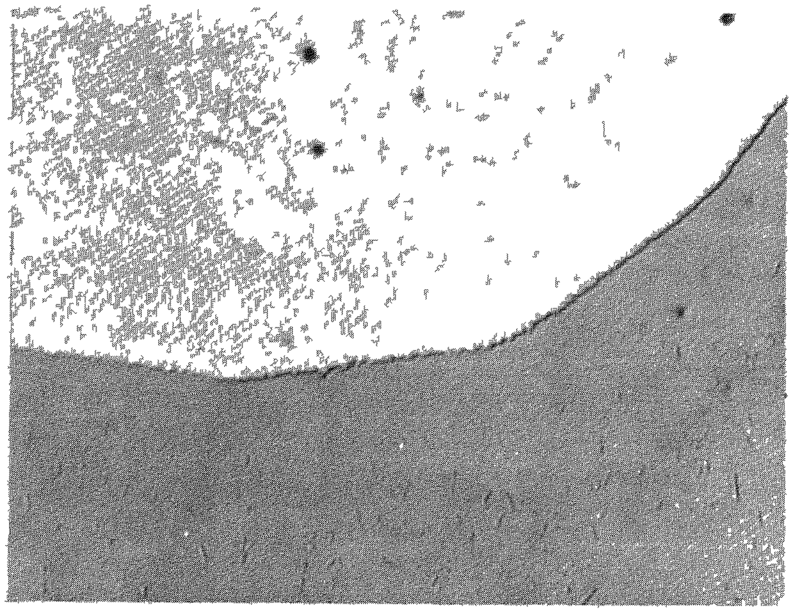
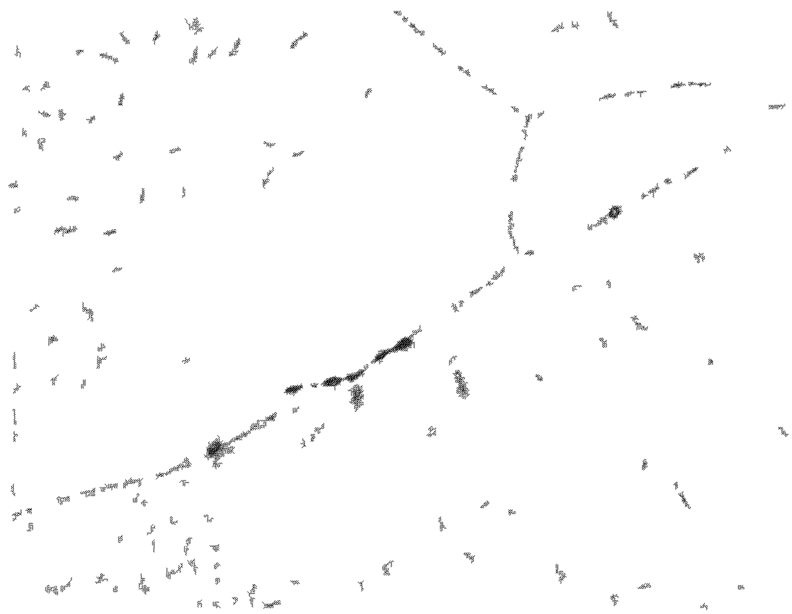


(a) ARC-CAST, MOLYBDENUM-0.5% TITANIUM ALLOY X2000



(b) ARC-CAST, TZM ALLOY X1000

GRAIN BOUNDARY CONTAMINATION IN MOLYBDENUM WELDS
ELECTROLYTICALLY POLISHED AND ETCHED WITH $NaOH+K_3Fe(CN)_6+H_2O$

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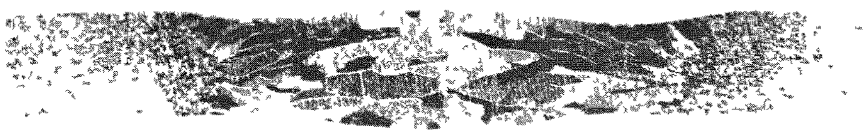
(a) WELDED AT 10ipm



(b) WELDED AT 100ipm

WELD MACROSTRUCTURES OF FANSTEEL 82 ALLOY SHEET X15
MECHANICALLY POLISHED AND ETCHED WITH HNO_3+HF+GLYCERINE

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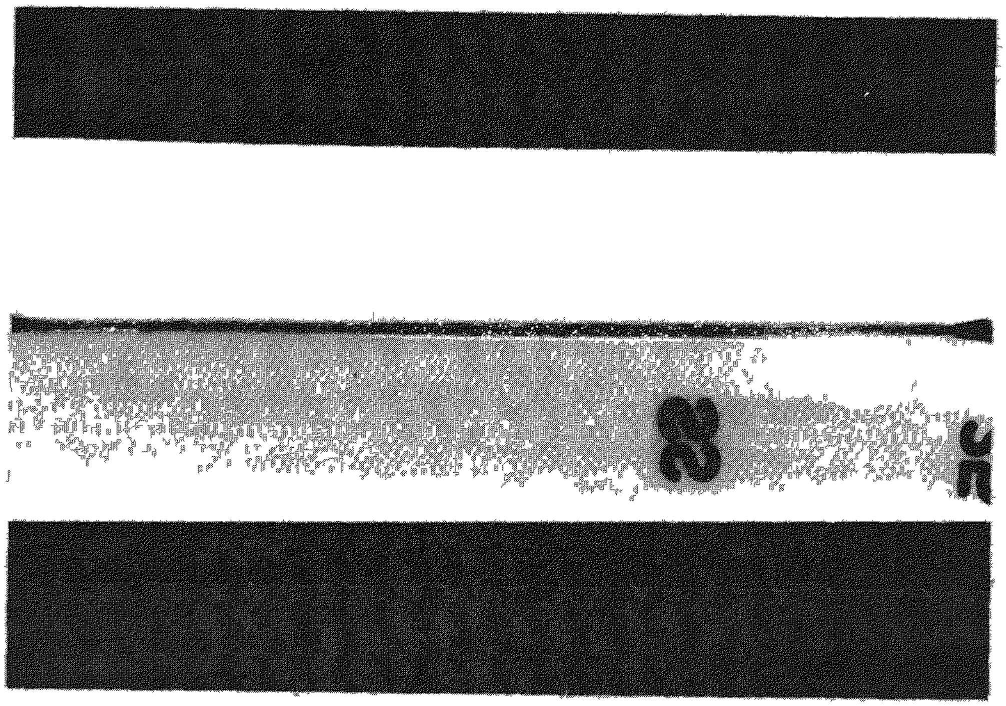
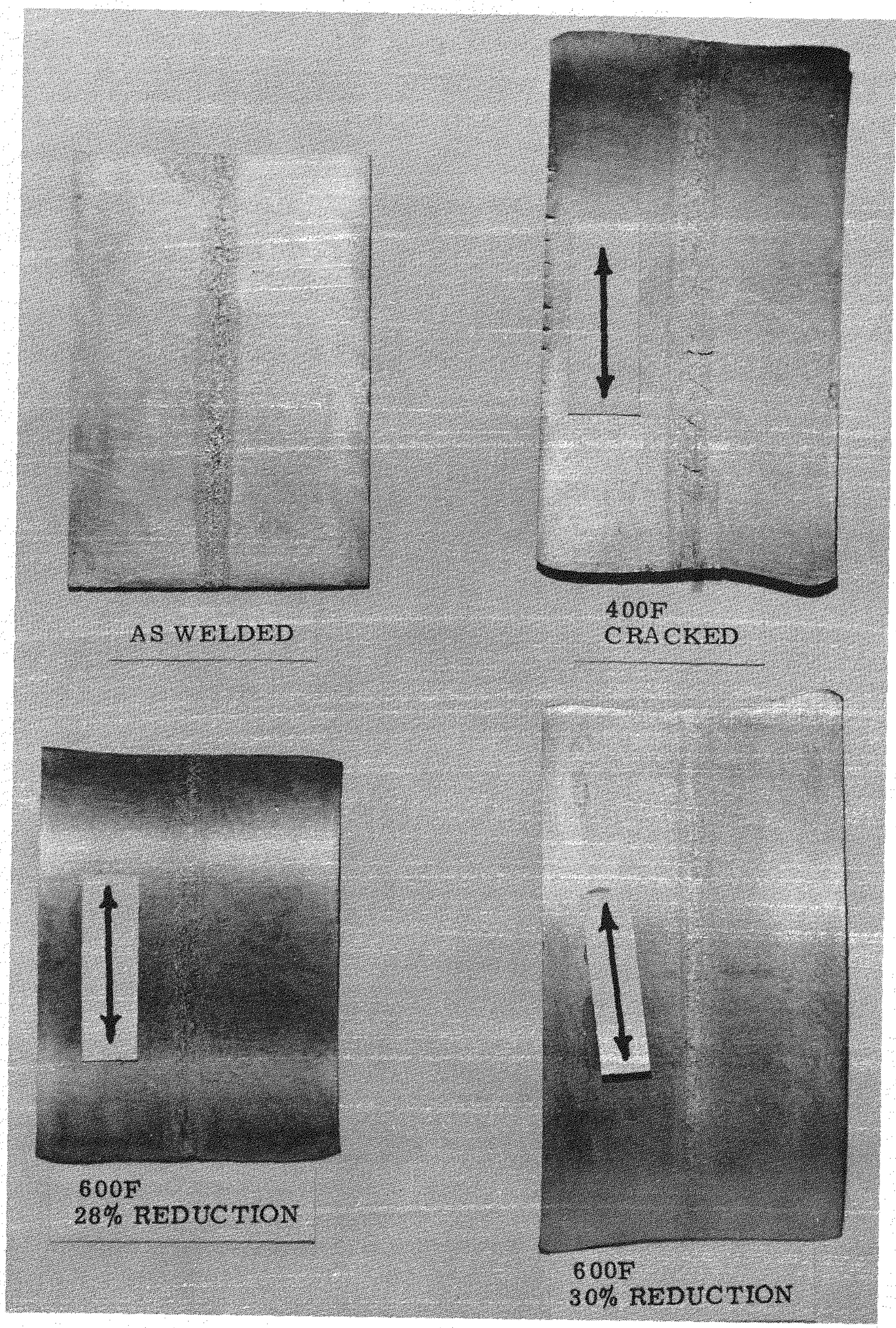
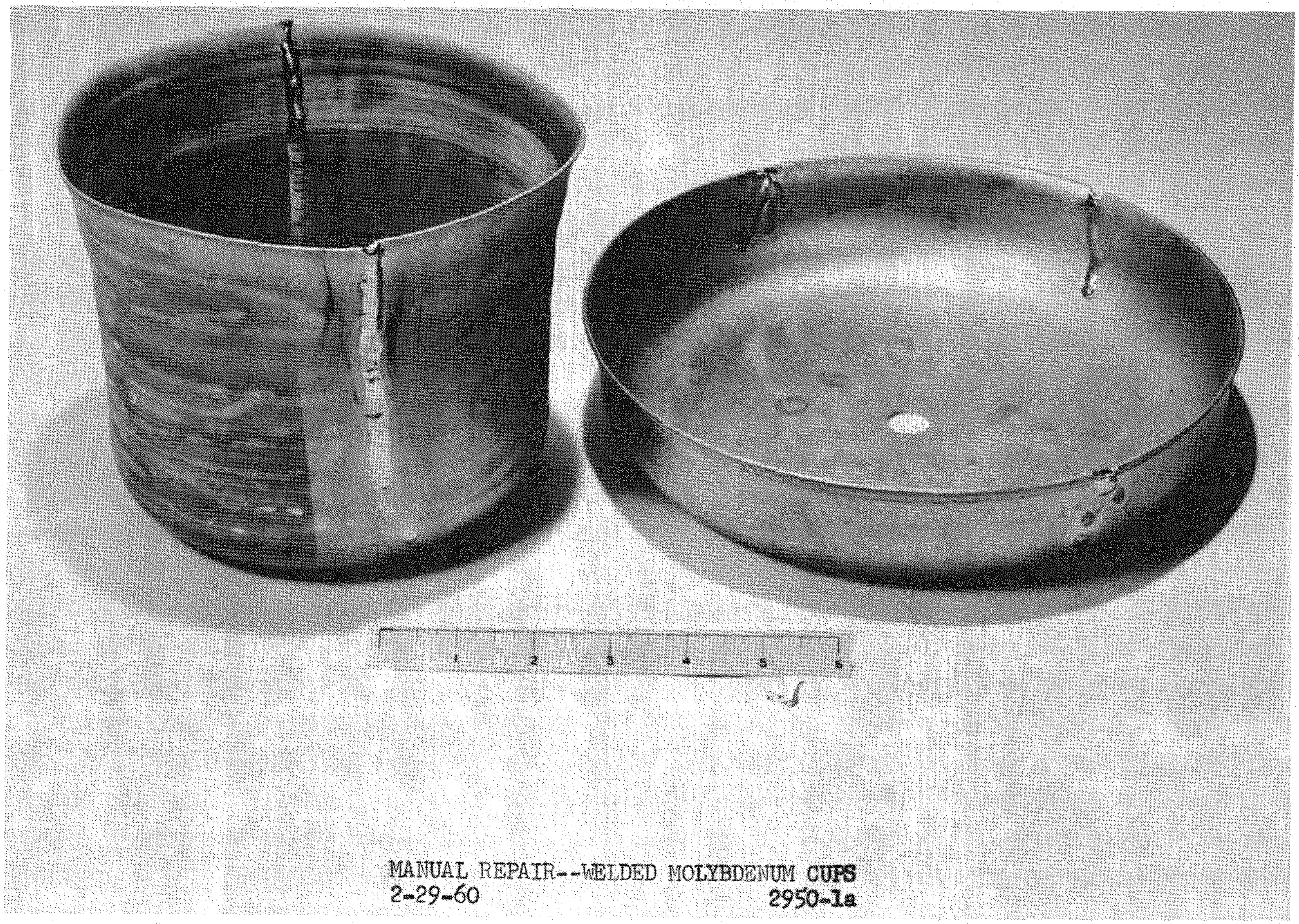


WELD POROSITY IN HYDROGEN-SINTERED TUNGSTEN SHEET



SECTIONS OF WELD **U71 WARM** ROLLED AT INDICATED TEMPERATURES AND IN **INDICATED** DIRECTIONS
2-13-59 2213-1a

421 026



MANUAL REPAIR--WELDED MOLYBDENUM CUPS
2-29-60 2950-1a

FIGURE 8

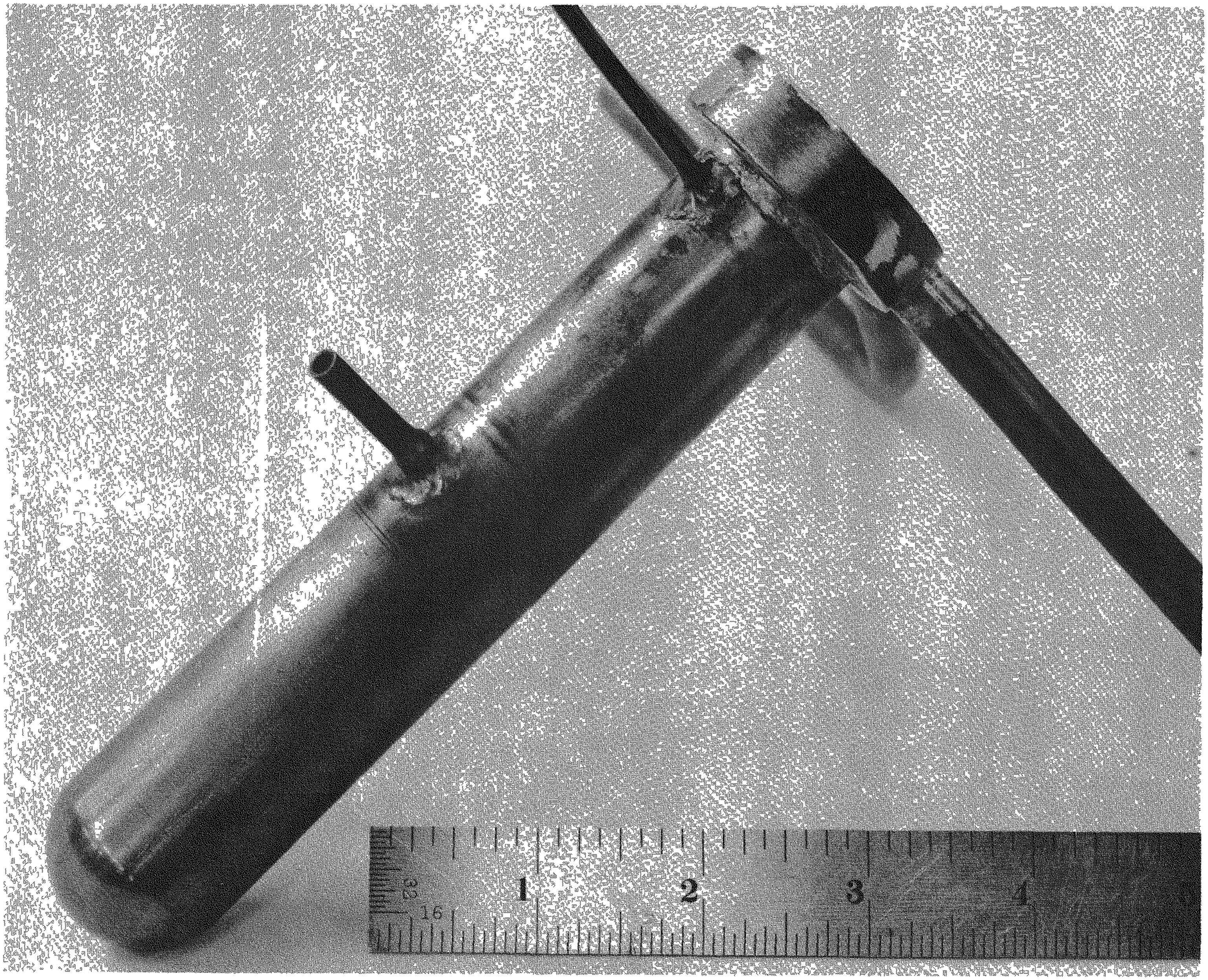
CRACKED MOLYBDENUM-0.5% TITANIUM ALLOY
CUPS--REPAIR WELDED AND MANUALLY SPUN
6-3-60 3207-1a

FIGURE 9

FIGURE 10

UNCLASSIFIED

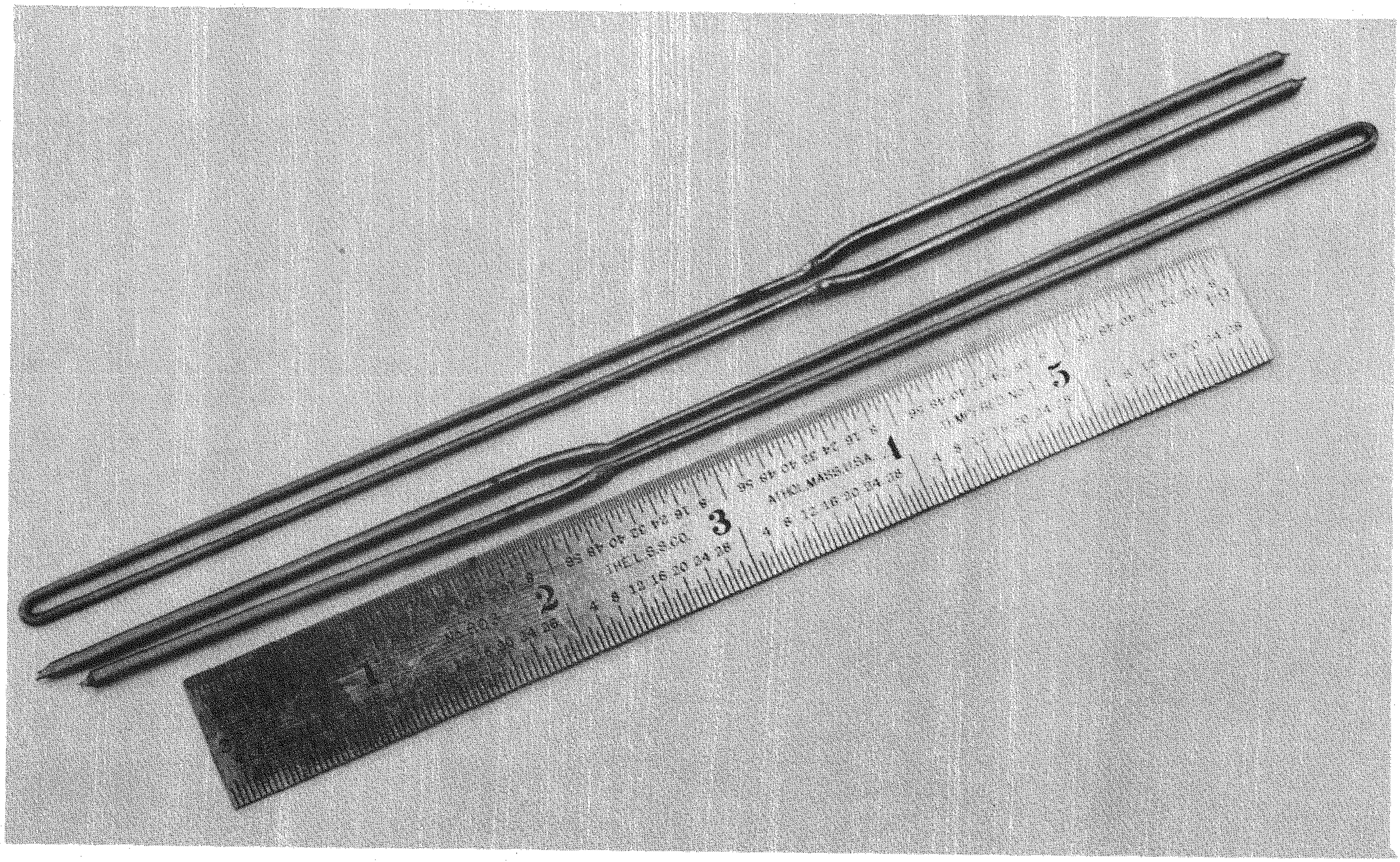
UNCLASSIFIED



FUSION WELDED TANTALUM ASSEMBLY
5-26-60 3197-1a

FIGURE 11

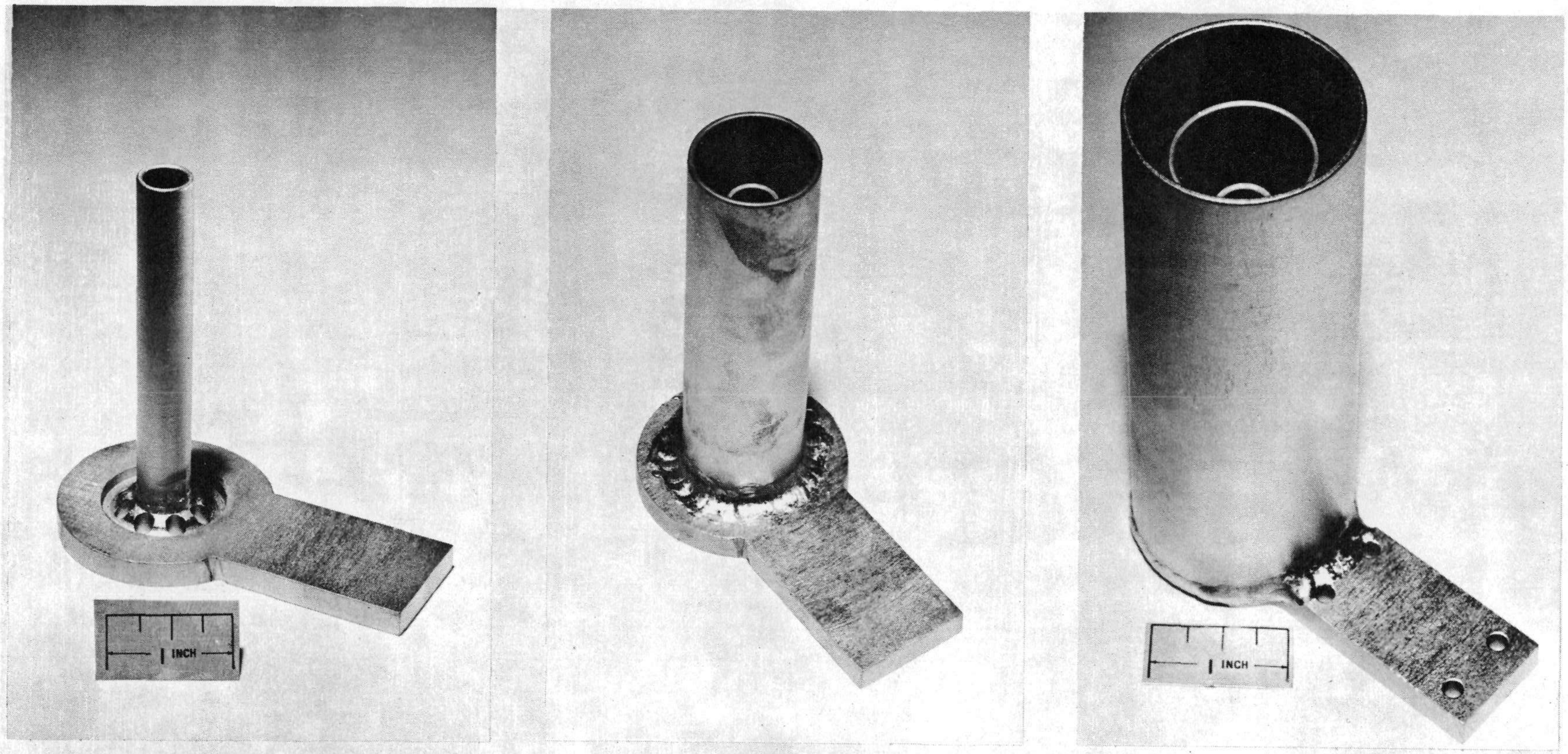
UNCLASSIFIED



WELDED TUNGSTEN EMITTER ASSEMBLY
5-26-60 3197-2a

FIGURE 12

421 031



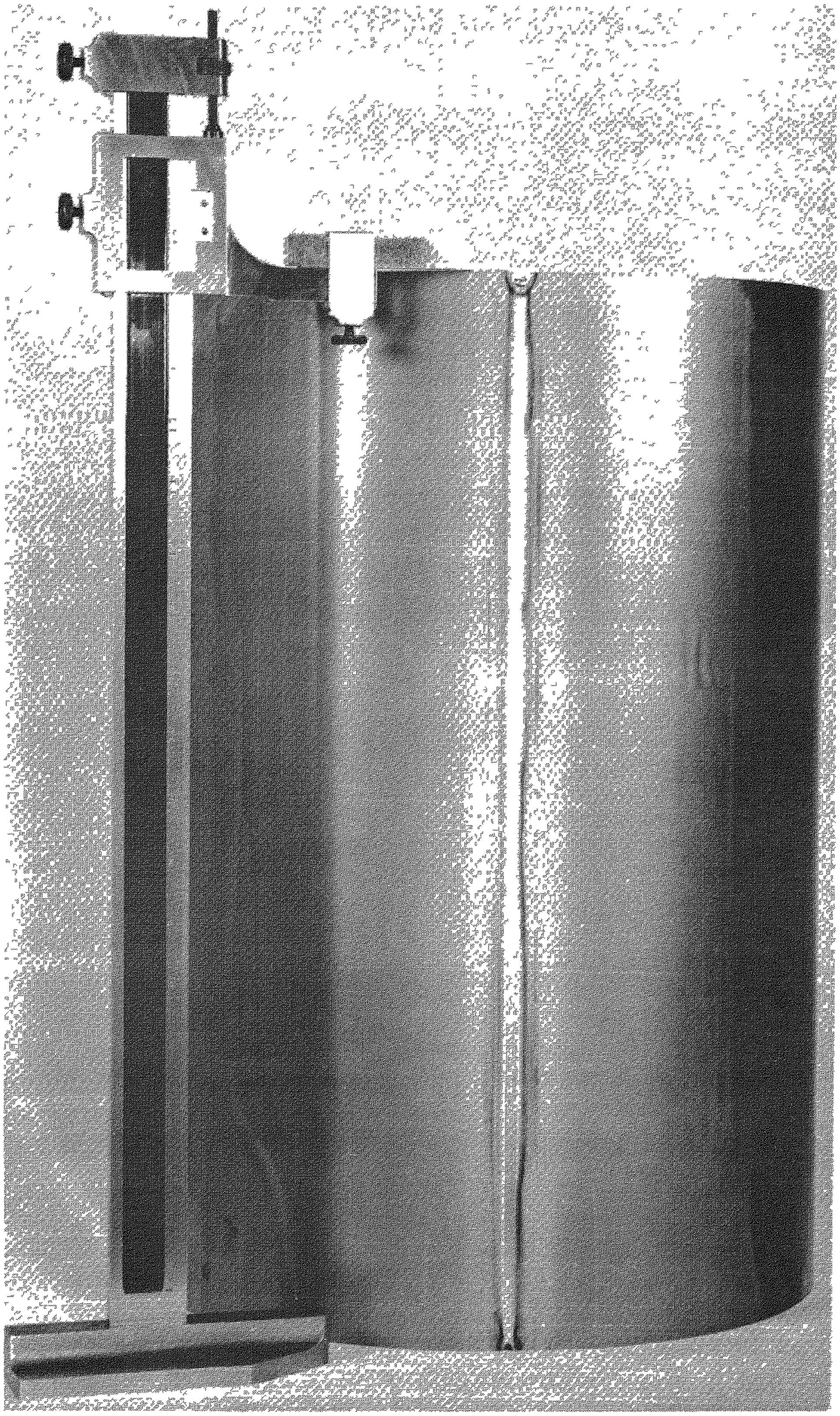
PHASE 1 PHASE 2 PHASE 3

WELD FABRICATION OF MOLYBDENUM - 0.5% TITANIUM
TEMPERATURE AND PRESSURE SENSING PROBE

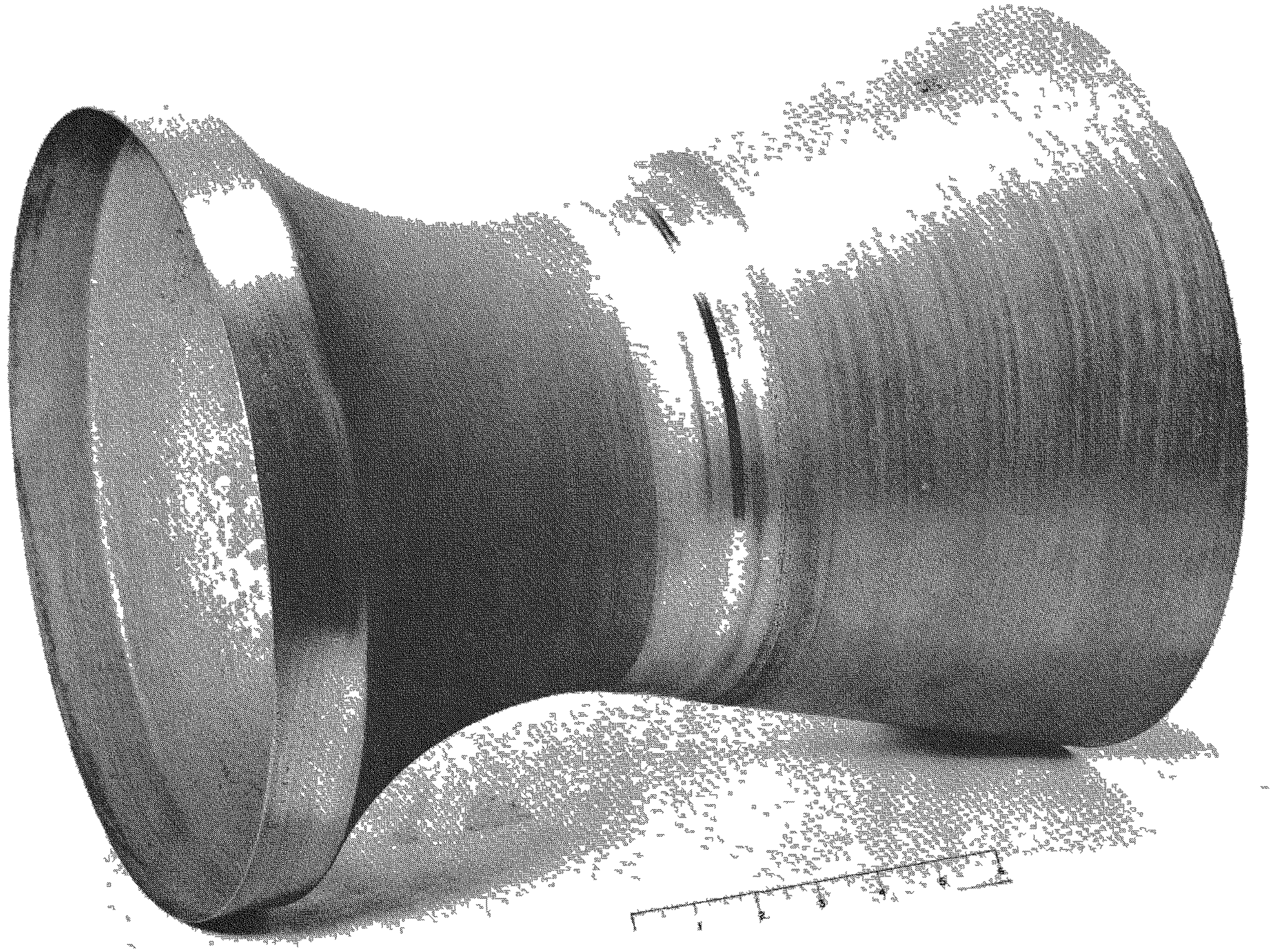
FIGURE 13

421 032

UNCLASSIFIED

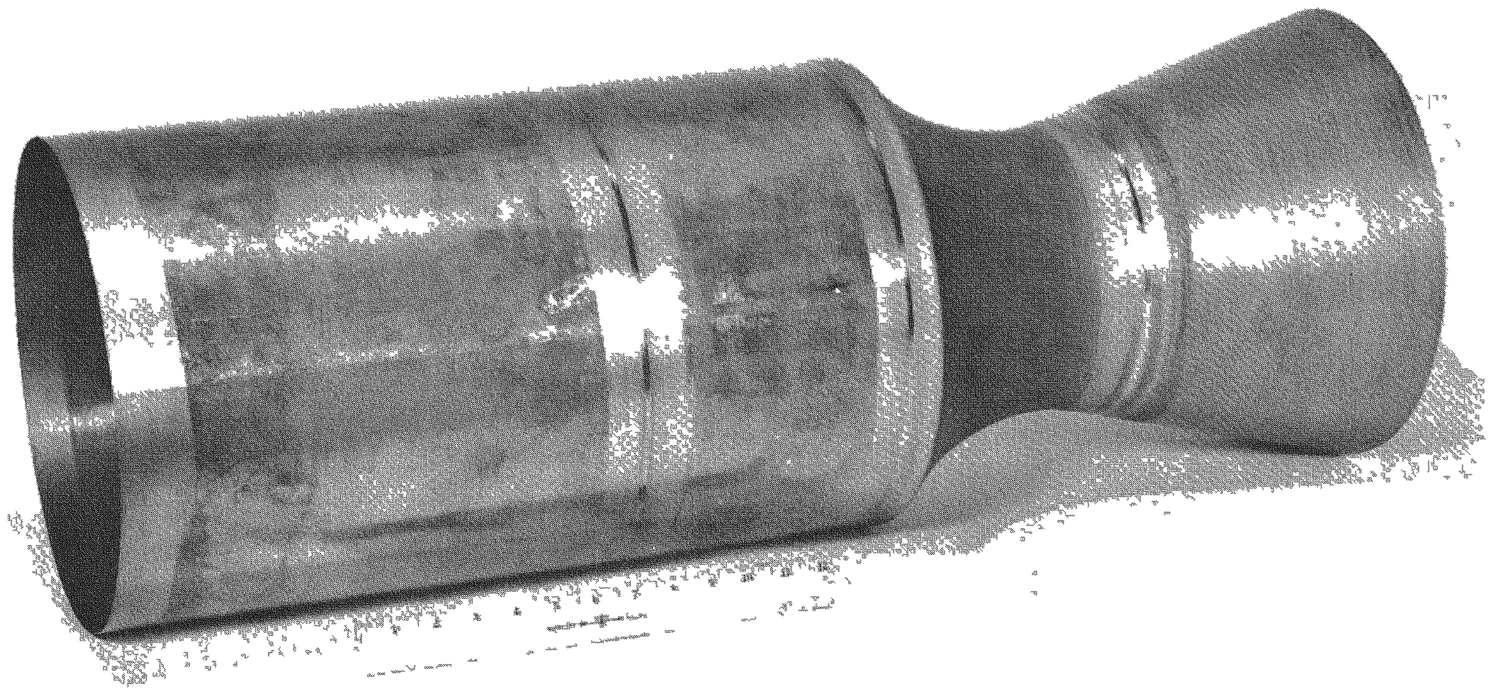


FUSION WELDED MOLYBDENUM CYLINDER
9-23-59 2653-1a



WELDED MOLYBDENUM NOZZLE SECTION
4-4-60 3103-1a

FIGURE 15



0.5% TITANIUM-MOLYBDENUM WELDED COMBUSTION CHAMBER AND EXIT NOZZLE
5-31-60 3198-1a

FIGURE 16

UNCLASSIFIED