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EXPLORATORY STUDY OF BEND DUCTILITY OF SELECTED REFRACTORY METAL WELDMENTS

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SUMMARY

Preliminary tests were made to determine the influence of welding method, purity of weld atmosphere, and subsequent heat treatment on the ductility of welded 0.030-inch-thick sheets of columbium and tantalum base alloys, Cb - 1 percent Zr, Cb-752, B-33, FS-85, D-43, T-111, and T-222, as measured by a room temperature bend test. Bead-on-plate welds were made on the materials by using the electron-beam process or by using the gas tungsten-arc (TIG) process in argon atmosphere containing either 2 to 4 ppm oxygen with the moisture level ranging from 5 to 19 ppm or approximately 100 ppm oxygen (moisture not monitored). Additional heat treatments and aging conditions similar to those that might be encountered during fabrication or use of these materials in test space-power systems were imposed on some of the specimens before bending.

INTRODUCTION

Columbium and tantalum alloys have been proposed for use in advanced space-power systems as containment materials for alkali metal fluids in the temperature range of 1400° to 2200° F. A requirement for containment material is weldability. Two likely methods for joining refractory metal alloys for use in advanced space-power systems are gas tungsten-arc (TIG) and electron-beam welding. The major factors affecting weld acceptability for high temperature liquid metal service are believed to be weld contamination and aging. Contamination of refractory metal alloy welds by oxygen, for instance, is known to have a marked effect on weld ductility and on corrosion resistance to liquid metals. Aging after welding also has an effect on weld ductility; however, its effect on corrosion resistance, if any, is not known.

In order to evaluate the effect of some of these parameters on the weldability of several commercially available columbium and tantalum alloys of interest for use in space-power systems simulation loops, a series of preliminary weld tests was carried out. All welds were bead-on-plate. In these tests only ductility was investigated. The criterion for judging ductility was a guided bend test made at room temperature by using a 1 T radius punch.

Specimens of Cb - 1 percent Zr, Cb-752, B-33, FS-85, D-43, and T-111 were TIG welded by using "clean" laboratory conditions (approximately 2 ppm oxygen in an argon atmosphere) and by using "field" conditions (approximately 100 ppm oxygen in argon atmosphere). In addition, these six alloys were also welded by the electron-beam process. Specimens so welded were bend tested both in the as-welded condition and after being aged for 10 hours at 1800° F in a vacuum.

Following this brief test, four promising alloys of columbium and tantalum (FS-85, D-43, T-111, and T-222) were subjected to bead-on-plate runs by using TIG welding with clean laboratory atmospheric conditions (argon atmosphere with 2 to 4 ppm oxygen). These welded specimens were bend-tested after receiving the following aging treatments (in vacuum):

(1) Aged 10 hours at 1800° F

(2) Aged 10 hours at 2000° F

(3) Heat-treated 1 hour at 2500° F, aged 10 hours at 1800° F

(4) Heat-treated 1 hour at 2500° F, aged 10 hours at 2000° F

Unwelded control specimens were solution-treated for 1 hour at 3000° F and bend-tested following each of the four heat treatments just listed.

Chemical analyses and metallographic studies were used to analyze the results of the bend tests.

MATERIALS

The alloys used in this investigation were purchased as stress relieved sheets nominally 0.030 inch thick. These materials represent several categories of commercially available columbium and tantalum base alloys. Chemical compositions are shown in tables I and II.

APPARATUS

Heat treatments were carried out in a vacuum furnace, with a platinum - platinum-13-percent-rhodium thermocouple being used for temperatures of 2500° F or below and a tungsten - tungsten-26-percent-rhenium thermocouple for temperature measurements above 2500° F.

Electron-beam welding was done with a high-voltage welding machine rated at 150 kilovolts and 13.5 milliamperes. The TIG welding using clean laboratory conditions was carried out in a vacuum dry box equipped with an oxygen trace analyzer and an electrolytic hygrometer for continuous monitoring of oxygen and moisture in the argon welding atmosphere. The argon supplied to the dry box from a manifold tank system contained approximately 1 ppm oxygen and 5 ppm moisture as measured from a sampling line in parallel with the box. A view of the welding equipment is presented in figure 1.

The TIG welding using field conditions was carried out in a polyvinyl chloride bag placed around the work. The bag was supported by wires and secured with adhesive-backed paper tape. An oxygen trace analyzer was used for continuous monitoring of oxygen in the welding atmosphere; the moisture level was not determined. The argon used was 99.995 percent high-purity bottled argon.

TABLE I. - CHEMICAL ANALYSES BY NASA ON 0.030-INCH-THICK SHEETS USED FOR

FIRST SCREENING TESTS ON ALLOYS WELDED BY THREE METHODS

[Electron beam, gas tungsten-arc clean conditions, and gas tungsten-arc field welding.]

Element	Cb - 1 percent Zr	Cb-752	B-33	FS-85	D-43	T-111
Oxygen	243 ppm	270 ppm	550 ppm	180 ppm	96 ppm	14 ppm
Nitrogen	45 ppm	53 ppm	86 ppm	42 ppm	31 ppm	20 ppm
Carbon	69 ppm	57 ppm	121 ppm	80 ppm	860 ppm	14 ppm
Hydrogen	2 ppm	5 ppm	4 ppm	2 ppm	1 ppm	1 ppm
Columbium	Balance	Balance	Balance	Balance	Balance	100 ppm
Tantalum	[a]< 500 ppm	-------	[a]< 500 ppm	27.76 weight percent	2000 ppm	Balance
Tungsten	[a]< 500 ppm	9.17 weight percent	[a]< 500 ppm	9.49 weight percent	9.05 weight percent	7.60 weight percent
Vanadium	----------	-------	4.45 weight percent	-------------------	------------------	------------------
Zirconium	1.01 weight percent	2.74 weight percent	900 ppm	0.80 weight percent	1.04 weight percent	200 ppm
Iron	20 ppm	-------	10 ppm	30 ppm	50 ppm	10 ppm
Hafnium	[a]< 300 ppm	-------	[a]< 100 ppm	[a]< 100 ppm	[a]< 100 ppm	2.36 weight percent
Molybdenum	10 ppm	-------	[a]< 10 ppm	300 ppm	20 ppm	20 ppm
Silicon	[a]< 10 ppm	-------	40 ppm	10 ppm	20 ppm	[a]< 10 ppm
Titanium	[a]< 10 ppm	-------	[a]< 10 ppm	[a]< 10 ppm	190 ppm	[a]< 10 ppm

[a]Not detected.

TABLE II. - CHEMICAL ANALYSES OF ALLOYS USED FOR AGING TESTS[a]

Alloys	FS-85		D-43		T-111		T-222	
	Manufacturer	NASA	Manufacturer	NASA	Manufacturer	NASA	Manufacturer	NASA
Oxygen	50 ppm	99 ppm	----	131 ppm	26 ppm	51 ppm	18 ppm	--
Nitrogen	42 ppm	63 ppm	--	39 ppm	21 ppm	22 ppm	41 ppm	--
Carbon	10 ppm	19 ppm	--	551, 926, 940	<10 ppm	20 ppm	125 ppm	--
Hydrogen	------	<1 ppm	--	2 ppm	------	<1 ppm	-------	--
Tantalum	27.4	27.62	--	-------------	Balance	Balance	Balance	--
Columbium	Balance	Balance	--	Balance	------	------	-------	--
Tungsten	9.8	9.92	--	9.44	8.37	7.97	9.2	--
Hafnium	------	-----	--	-------------	2.22	2.35	2.2	--
Zirconium	.95	.94	--	1.02	------	------	-------	--

[a]Weight percent unless otherwise indicated.

Specifications of the bend-test fixture are given in reference 1. Die span for the 0.030-inch sheet material was 1 inch. The punch radius was 0.030 inch, the same as the thickness of the sheet material (1 T). The environment for bend testing was the atmosphere at room temperature. Figure 2 presents a view of the bend-test fixture.

PROCEDURE

Flow charts showing the sequence of welding and heat treatments applied to the groups of specimens are presented in figures 3 to 5.

Specimen Preparation

Specimens were sheared from the 0.030-inch-thick sheet material. The long dimension of the $\frac{1}{2}$- by $2\frac{1}{2}$-inch specimens was parallel to the final rolling direction of the sheets. Prior to welding all specimens were vapor degreased in trichloroethylene. Columbium alloys were then chemically cleaned by being dipped in a solution consisting of 30 parts nitric acid, 7 parts hydrofluoric acid, and 63 parts water by volume. Tantalum alloys were cleaned by being dipped into 40 parts nitric acid, 40 parts sulfuric acid, and 20 parts hydrofluoric acid by volume. Both cleaning solutions were at ambient temperature.

Welding

Bead-on-plate welds were made down the center of each specimen. Welding parameters were adjusted to give full penetration in one pass. Electron beam welds were made on all columbium alloys at approximately 23 inches per minute by using approximately 95 kilovolts at 3 milliamperes. The tantalum alloy required approximately 120 kilovolts at 3 milliamperes to give complete penetration at a welding speed of 23 inches per minute. Electron-beam welding was performed in a vacuum of 1×10^{-4} torr or better.

Specimens to be TIG welded under clean laboratory conditions were placed in a vacuum dry box that was pumped down to 1.5×10^{-5} torr. The box was then backfilled with argon to approximately 1 atmosphere. The oxygen and moisture contents of the argon atmosphere in the backfilled chamber were 2 ppm and 17 ppm, respectively. Oxygen content of the chamber did not exceed 4 ppm at any time, and moisture level built up to a maximum of 19 ppm at the end of any run. Specimen welding speed averaged approximately 11 inches per minute, except for T-111 specimens, which were welded at an average speed of 8 inches per minute. The arc was started by using a high-frequency starter. The velocity of the manually controlled torch was varied as needed to produce the bead. Power requirements varied from 125 amperes at 26 volts to 190 amperes at 30 volts.

Specimens to be field welded were placed in a plastic bag that was then sealed with adhesive-backed paper tape. The bag was purged with 99.995-percent high-purity argon, which contained 15 ppm oxygen as it came from the bottle. The gas entering the bag passed through the welding torch with a flow rate sufficient to keep the polyvinyl chloride bag inflated. All argon leaving the bag passed through the oxygen trace analyzer. Welds were made with an atmospheric contamination level of approximately 95 ppm oxygen. A 2-inch-long bead was made on the specimens at a rate of approximately 12 inches per minute by manual torch operation. The work was done by using a 200-ampere arc welder. Power input was not measured. Typical specimens welded by the electron-beam and the TIG method are shown in figure 6.

Heat Treatments

Specimens to be heat-treated were wrapped in tantalum foil and placed in a vacuum-resistance furnace. Specimens were brought to the required temperature in approximately 2 hours and held at temperature for the required time. Power was then turned off, and the specimens were allowed to cool to room temperature. The vacuum maintained during heat treatments varied from 1×10^{-5} torr at the start to 1×10^{-7} torr at the end of the treatment.

Bend Testing

Contrary to the suggestion given in reference 1, surfaces of welded specimens were not ground. Specimens were placed on the die (fig. 2) so that weld faces would be in tension during the bend test. Testing was done at room temperature with a rate of punch descent of 1 inch per minute. Specimens were bent sufficiently so that the bend angles of the specimens after springback (after unloading) would fall between 90° and 105° as required by specifications given in reference 1. If audible cracking occurred, the test was terminated at that point. Bend angles under load were measured from an imprint of the loaded specimen in modeling clay. Bend angles of unloaded specimens (following spring-

TABLE III. - BEND-TEST RESULTS ON ALLOYS WELDED BY THREE METHODS

Alloy	Welding method	Condition of bend specimen	Bend angle, deg		Springback angle, deg
			Under load	After springback	
Cb - 1 percent Zr	Electron-beam	As-welded	112	102	10
		[a]Aged	114	101	13
	Gas tungsten-arc (dry box, clean conditions)	As-welded	122	110	12
		[a]Aged	119	98	21
	Gas tungsten-arc (field weld)	As-welded	[b]112	103	9
Cb-752	Electron-beam	As-welded	113	96	17
		[a]Aged	116	100	16
	Gas tungsten-arc (dry box, clean conditions)	As-welded	113	96	17
		[a]Aged	118	98	20
	Gas tungsten-arc (field weld)	As-welded	[c]35	---	--
B-33	Electron-beam	As-welded	118	93	25
		[a]Aged	121	97	24
	Gas tungsten-arc (dry box, clean conditions)	As-welded	112	94	18
		[a]Aged	124	100	24
	Gas tungsten-arc (field weld)	As-welded	[c]18	---	--

[a]Aged 10 hours at 1800° F in vacuum of 1.5×10^{-5} torr or better.

[b]Microscopic examination (×30) revealed cracking in fusion and heat-affected zones.

[c]Audible cracking at angle indicated.

back) were measured with a protractor. Some tested specimens are shown in figure 7. In addition to audible detection of cracking during bend testing, each bent specimen was examined under a binocular microscope at a magnification of 30 for any evidence of cracking.

Welded specimens of the four alloys used for aging studies that did not fail during bend testing (table IV) were straightened in a smooth-jawed vise, given a further heat treatment for 10 hours at 2200° F, and bend-tested again.

RESULTS AND DISCUSSION

Table III shows the results of the welding tests on Cb - 1 percent Zr,

TABLE III. - Concluded. BEND-TEST RESULTS ON ALLOYS WELDED BY THREE METHODS

Alloy	Welding method	Condition of bend specimen	Bend angle, deg		Springback angle, deg
			Under load	After springback	
FS-85	Electron-beam	As-welded	118	93	25
		[a]Aged	120	97	23
	Gas tungsten-arc (dry box, clean conditions)	As-welded	120	99	21
		[a]Aged	[c]91	76	--
	Gas tungsten-arc (field weld)	As-welded	[c]22	--	--
D-43	Electron-beam	As-welded	115	92	23
		[a]Aged	114	94	20
	Gas tungsten-arc (dry box, clean conditions)	As-welded	118	96	22
		[a]Aged	[d]~45	--	--
	Gas tungsten-arc (field weld)	As-welded	[c]20	--	--
T-111	Electron-beam	As-welded	116	93	23
		[a]Aged	118	98	20
	Gas tungsten-arc (dry box, clean conditions)	As-welded	115	102	13
		[a]Aged	120	105	15
	Gas tungsten-arc (field weld)	As-welded	[b]115	102	13

[a]Aged 10 hours at 1800° F in vacuum of 1.5×10^{-5} torr or better.

[b]Microscopic examination (×30) revealed cracking in fusion and heat-affected zones.

[c]Audible cracking at angle indicated.

[d]Broke into several pieces.

Cb-752, B-33, FS-85, D-43 and T-111 for electron-beam welds and TIG welds made under both clean and field conditions. Figure 3 gives the operations and sequence of operations performed on the specimens. The weld heat treatment used, 1800° F for 10 hours, was representative of a condition that was likely to be encountered during startup of liquid metal systems. Also, it has been reported that the aging of Cb - 1 percent Zr welds in the temperature range 1500° to 1800° F can result in loss of ductility (ref. 2).

All electron-beam welded specimens in the as-welded condition and those heat-treated for 10 hours at 1800° F were ductile. Photomicrographs of electron-beam welded specimens in the as-welded condition and after heat treatment are presented in figure 8.

All as-welded specimens that were TIG welded by using field welding conditions either cracked audibly during bend testing or the fully bent specimens were found to be cracked upon microscopic examination. Hence, no specimens from this group were aged at 1800° F.

Specimens of the six alloys TIG welded by using clean laboratory conditions all withstood bend testing in the as-welded condition without cracking. Following aging for 10 hours at 1800° F in vacuum, welded specimens of Cb - 1 percent Zr, Cb-752, B-33, and T-111 withstood the bend test without failure. The D-43 and FS-85 specimens cracked under load after approximately 45° and 91° of bending, respectively, as shown in table III. The angle of the FS-85 specimen was 76° after springback; the D-43 specimen shattered.

Oxygen analyses of the fusion zones of TIG welds were obtained in an effort to understand the reasons for the differences in ductility between the welds made in an argon atmosphere containing approximately 100 ppm oxygen and those made in an argon atmosphere containing 2 ppm oxygen. Results are shown in table IV.

TABLE IV. - OXYGEN ANALYSES OF FUSION ZONES OF WELDED SPECIMENS

Alloy	Oxygen in fusion zone of weld, ppm	
	Clean dry box welding[a]	Field welding[b]
Cb - 1 percent Zr	228, 226	280, 281
Cb-752	80, 85	162, 124
FS-85	190, 196	233, 239
B-33	212, 195	287, 289
D-43	116, 113	160, 155
T-111	25, 22	71, 71

[a]Argon with approximately 2 ppm oxygen.

[b]Argon with approximately 100 ppm oxygen.

These analyses indicate a small, but consistent, increase in oxygen content of the alloys welded by using field conditions as compared to the alloys welded by using clean laboratory conditions; however, the loss of ductility in the field-welded specimens was very pronounced. Probably only part of this loss of ductility may be attributed to the increase in oxygen content of the metal (refs. 3 and 4). The other contributing factor to the loss of ductility may well be nitrogen contamination. Nitrogen is reported to have a more adverse effect on the ductility of columbium alloys than oxygen (ref. 3). Facilities for monitoring the nitrogen content of welding atmospheres were not available at the time this work was done. But inasmuch as the argon gas atmosphere in the field-welding enclosure contained approximately 95 ppm oxygen and the argon coming from the cylinder contained only 15 ppm oxygen, it is reasonable to assume that air leakage accounted for the increase in oxygen. Such an air leakage would result in an atmosphere contaminated with about 400 ppm nitrogen.

Figures 4 and 5 present flow charts indicating heat treatments employed for the aging studies, while the bend-test results are summarized in table V. Figures 9 to 16 present microstructures of the alloys studied in the as-welded condition and following the various heat treatments. All welding for these aging studies was done in the welding dry box under clean laboratory conditions. The aging treatments of 10 hours at 1800° and at 2000° F were selected as typical conditions likely to be encountered when starting up a liquid metal loop for checkout with subsequent cooling prior to long-time steady-state operations. The heat treatment for 1 hour at 2500° F is an overaging treatment and represents approximately the upper limit in heat-treating actual loop components from a practical standpoint. Overaging has been found to improve weldment ductility and, in addition, to prevent loss of ductility when the columbium alloy was subsequently aged at 1800° F (ref. 5).

An anneal for 1 hour at 3000° F was used to put nonwelded control specimens in a solution-treated condition prior to subsequent heat treatments. Photomicrographs of heat-treated, nonwelded specimens are presented in figures 17 to 20.

Table V indicates that FS-85 was fully ductile in either the welded or the nonwelded condition following all of the heat treatments shown. There was no cracking during bend testing of the welded specimens that had been aged for 10 hours at 1800° F. This performance should be contrasted with the performance of the specimen in the earlier tests, prepared under the same conditions, that cracked on bending. The only significant difference between these two specimens is that they came from different heats of FS-85.

The bend-test results for the D-43 alloy, presented in table V, show that welded specimens subjected to aging for 10 hours at 1800° F failed to bend the required 90° to 105° as specified in reference 1. The two specimens tested cracked at 65° and 52°, respectively. Referring to table III, which presents bend-test results of the exploratory tests discussed earlier, it can be seen that D-43 also failed to pass the bend test following welding and aging for 10 hours at 1800° F; the specimen broke into several pieces after bending approximately 45°.

Welded D-43 specimens aged for 10 hours at 2000° F did not crack as a result of bend testing. Possibly the 2000° F heat treatment may have been high enough in temperature to result in overaging. This suggestion appears to be substantiated by the microstructures shown in figure 11. The TIG welded D-43 shows a large quantity of an acicular precipitate in both the as-welded condition (fig. 11(a)) and after aging for 10 hours at 1800° F (fig. 11(b)). Most of this precipitate appears to be dissolved after aging for 10 hours at 2000° F (fig. 11(c)). Gerken found a reduction in ductile-to-brittle transition temperature of TIG welded D-43 when aged for 8 hours at 2000° F and also a

TABLE V. - BEND-TEST RESULTS FOR AGING SERIES

Condition of specimen		Aged 10 hr at 1800° F			Aged 10 hr at 2000° F		
		Bend angle, deg		Spring-back angle, deg	Bend angle, deg		Spring-back angle, deg
		Under load	After springback		Under load	After springback	
FS-85							
Gas tungsten-arc welded (clean conditions)	As-welded	125 124	98 97	27 27	123 123	102 103	21 20
	Heat-treated 1 hr at 2500° F	118 119	103 102	15 17	123 122	103 102	20 20
Solution-treated 1 hr at 3000° F	As-solution-treated	120 119	102 102	18 17	122 124	103 104	19 20
	Heat-treated 1 hr at 2500° F	121 118	102 103	19 15	122 122	103 103	19 19
D-43							
Gas tungsten-arc welded (clean conditions)	As-welded	[a]65 [a]52	52 35	-- --	122 127	90 93	32 34
	Heat-treated 1 hr at 2500° F	123 126	96 94	27 32	128 128	100 101	28 27
Solution-treated 1 hr at 3000° F	As-solution-treated	127 122	102 102	25 20	128 126	102 103	26 23
	Heat-treated 1 hr at 2500° F	127 125	103 98	24 27	127 128	104 104	23 24
T-111							
Gas tungsten-arc welded (clean conditions)	As-welded	117 121	102 103	15 18	116 117	103 103	13 14
	Heat-treated 1 hr at 2500° F	117 118	103 103	14 15	117 129	102 111	15 18
Solution-treated 1 hr at 3000° F	As-solution-treated	115 118	103 103	12 15	117 118	103 103	14 15
	Heat-treated 1 hr at 2500° F	116 118	103 103	13 15	115 118	102 103	13 15
T-222							
Gas tungsten-arc welded (clean conditions)	As-welded	118 118	98 98	20 20	119 122	99 99	20 23
	Heat-treated 1 hr at 2500° F	116 117	98 97	18 20	121 122	100 100	21 22
Solution-treated 1 hr at 3000° F	As-solution-treated	116 120	98 98	18 22	119 119	99 100	20 19
	Heat-treated 1 hr at 2500° F	120 124	97 102	23 22	121 119	101 101	20 18

[a]Failed.

reduction in the amount of acicular phase present as compared to the welded structures (ref. 6). The remaining heat treatments for D-43 resulted in ductile specimens.

Table V indicates that both of the tantalum alloys tested (T-111 and T-222) exhibited full ductility following welding and all postwelding heat treatments.

Welded specimens from all conditions of heat treatment noted in table V that had withstood bend testing were straightened by pressing for further testing. During straightening, one of the two D-43 specimens that had given a full bend following welding and subsequent heat treatment for 10 hours at 2000° F cracked. Except for this, one specimen of each pair was given further heat treatment for 10 hours at 2200° F to uncover brittleness resulting from a duplex heat treatment. The other specimens were simply rebent. All rebent specimens gave full bends without cracking.

SUMMARY OF RESULTS

An investigation of room temperature bend ductility of selected columbium- and tantalum-base alloys in welded, aged, or welded-plus-aged condition yielded the following results:

1. All electron-beam welded specimens of Cb - 1 percent Zr, Cb-752, B-33, FS-85, D-43, and T-111 gave full bends (90° to 105° after springback when using a 1 T radius (0.030 in.) punch at room temperature, when bend tested in the as-welded condition (bead-on-plate), or following vacuum aging for 10 hours at 1800° F. No cracks were observed in any of the bent specimens when examined at a magnification of 30.

2. The Cb-752, B-33, FS-85, and D-43 specimens gas tungsten-arc (TIG) welded in an argon atmosphere containing 95 to 100 ppm oxygen cracked at bend angles of 35° or less, while Cb - 1 percent Zr and T-111 specimens so welded showed cracks in the fully bent specimens upon microscopic examination (×30).

3. These six alloys when TIG welded in an argon atmosphere containing approximately 2 ppm oxygen exhibited full 1 T radius bends, without cracking. After aging at 1800° F for 10 hours, however, the FS-85 specimen cracked after 76° of bending, and the D-43 specimen snapped into two pieces after bending approximately 45°.

4. A series of FS-85, D-43, T-111, and T-222 specimens were TIG welded in an argon atmosphere containing 2 to 4 ppm oxygen for further aging studies. Following aging at 1800° F for 10 hours in a vacuum, the FS-85, T-111, and T-222 alloys gave full bends, but the D-43 specimens cracked at 52° and 65° of bend. When aged at 2000° F for 10 hours in a vacuum, all specimens of the four alloys gave full bends without cracking.

5. All welded specimens of the four alloys from the group described under result 4 when heat-treated for 1 hour at 2500° F in a vacuum and subsequently aged for 10 hours in vacuum at either 1800° or 2000° F gave fully ductile bends.

6. Bend-tested specimens, described under results 4 and 5, were straightened by pressing. One specimen from each condition was given further heat treatment for 10 hours at 2200° F. These specimens, together with as-straightened specimens (controls) gave full bends without cracking.

7. Nonwelded specimens of the four alloys solution-treated at 3000° F for 1 hour and bend-tested following aging for 10 hours at either 1800° or 2000° F exhibited full bends.

8. Specimens of the four alloys (nonwelded) solution-treated at 3000° F for 1 hour, subsequently heat-treated at 2500° F for 1 hour, and bend-tested following aging treatments for 10 hours at either 1800° or 2000° F exhibited full bends.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, March 2, 1965.

REFERENCES

1. Marvel, Carl S.: Evaluation Test Methods for Refractory Metal Sheet Material. Rept. No. MAB-192-M. Materials Advisory Board, Apr. 22, 1963.

2. Franco-Ferreira, E. A.; and Slaughter, G. M.: Welding of Columbium-1% Zirconium. Welding J., vol. 42, no. 1, Jan. 1963, pp. 18s-24s.

3. Keller, D. L.; and Yount, R. E.: The Structural Stability of Welds in Columbium Alloys. Rept. No. ML-TDR-64-210, General Electric Co., June 1964.

4. Platte, W. N.: Welding Columbium and Columbium Alloys. Welding J., vol. 42, no. 2, Feb. 1963, pp. 69s-83s.

5. Gerken, J. M.; and Faulkner, J. M.: Welding Characteristics of Commercial Columbium Alloys. Welding J., vol. 42, no. 2, Feb. 1963, pp. 84s-96s.

6. Gerken, J. M.: A Study of Welds in Columbium Alloy D-43. Rept. No. TM 3865-67, Thompson Ramo Wooldridge, Inc., Mar. 25, 1964.

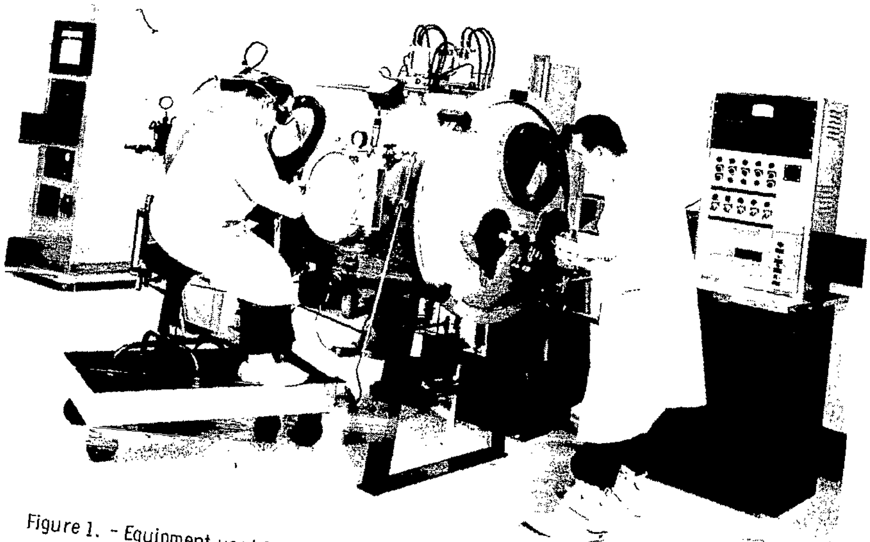


Figure 1. - Equipment used for gas tungsten-arc welding with clean laboratory conditions.

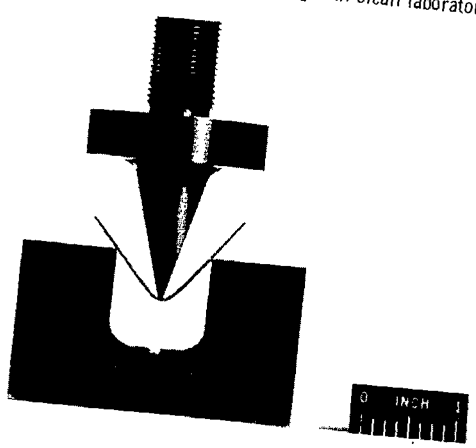


Figure 2. - Fixture used for bend testing.

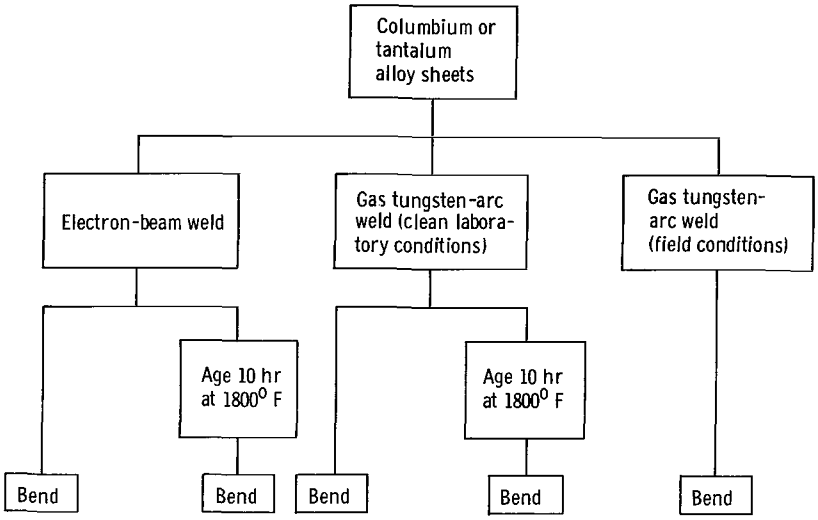


Figure 3. - Flow chart of operations performed on Cb-1 percent Zr, Cb-752, B-33, FS-85, D-43, and T-111 alloys.

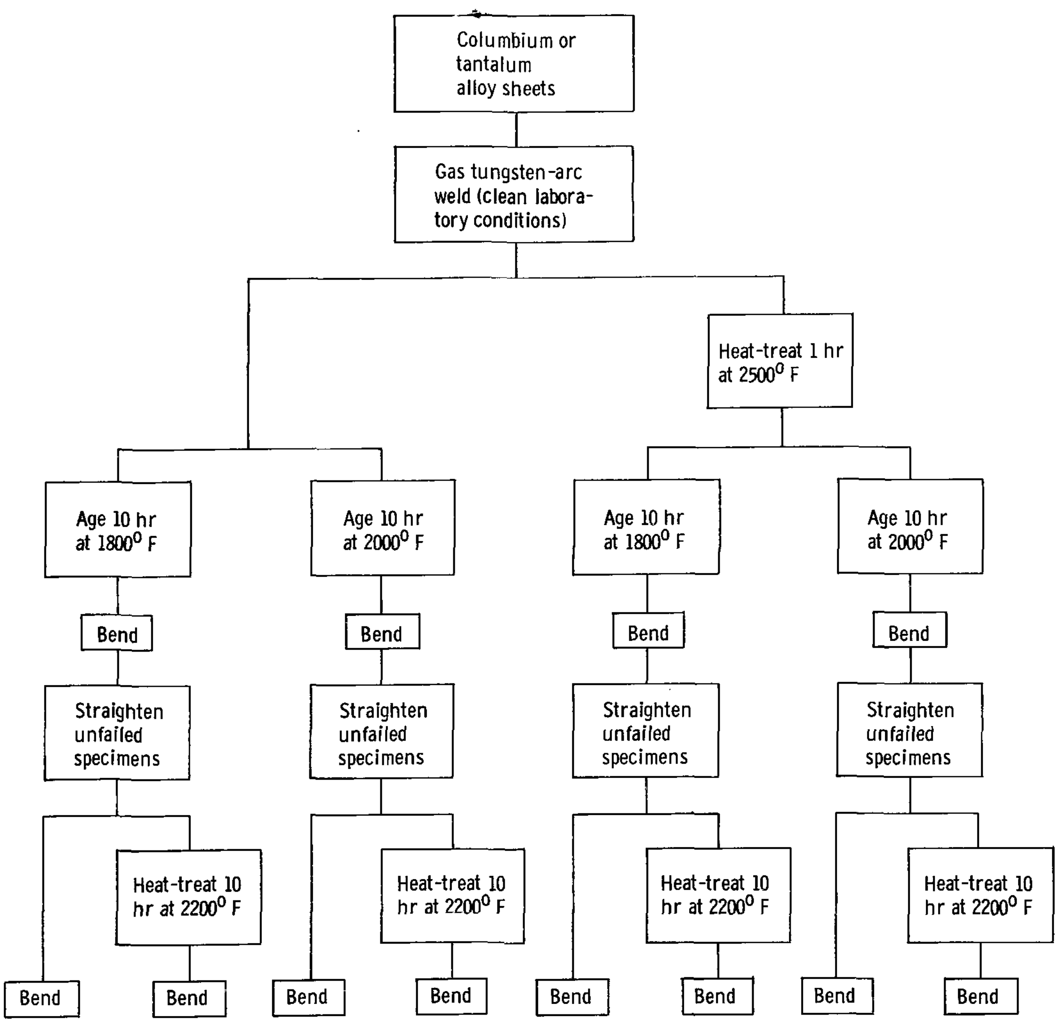


Figure 4. - Welding and subsequent operations performed on FS-85, D-43, T-111, and T-222 alloys.

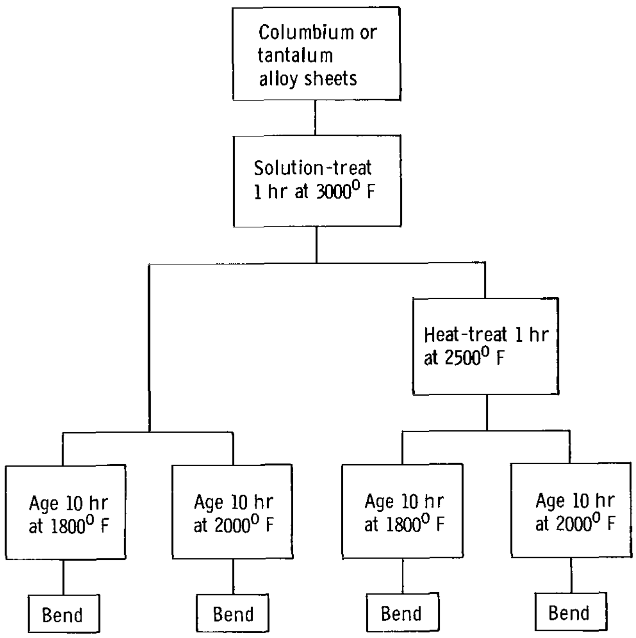


Figure 5. - Operations performed on nonwelded control specimens of FS-85, D-43, T-111, and T-222 alloys.

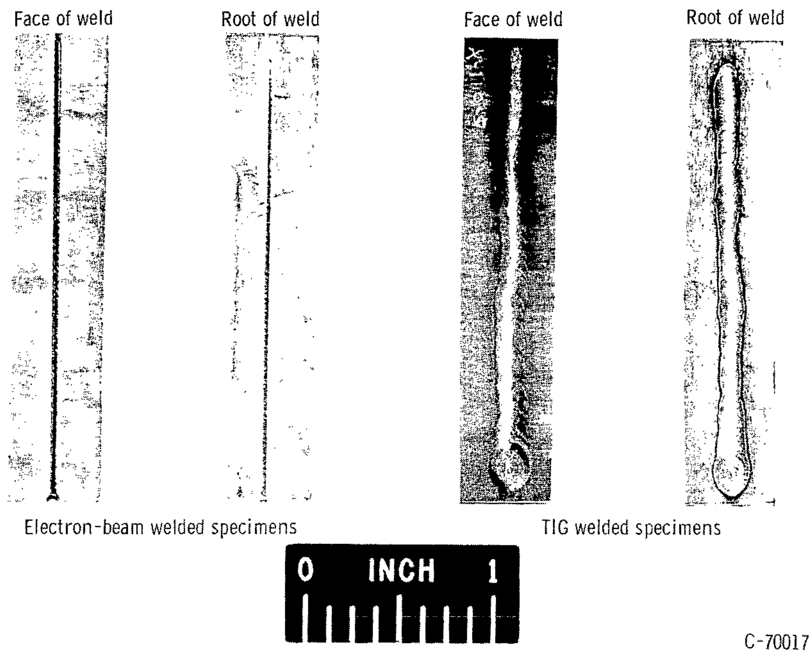


Figure 6. - Typical appearance of all electron-beam and gas tungsten-arc welded specimens.

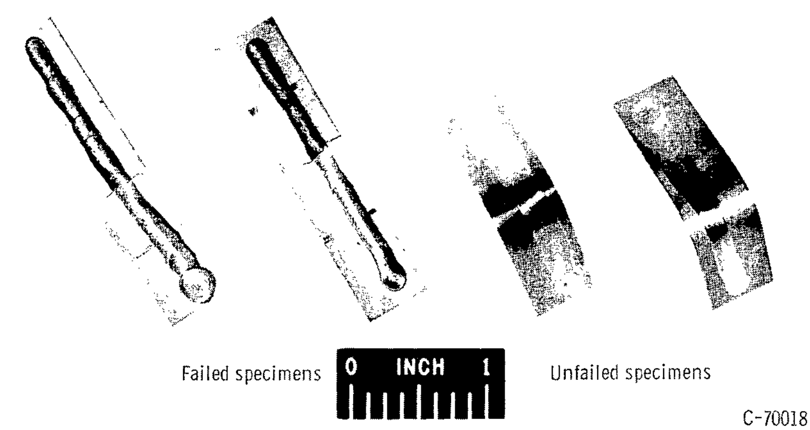


Figure 7. - Bend-tested specimens.

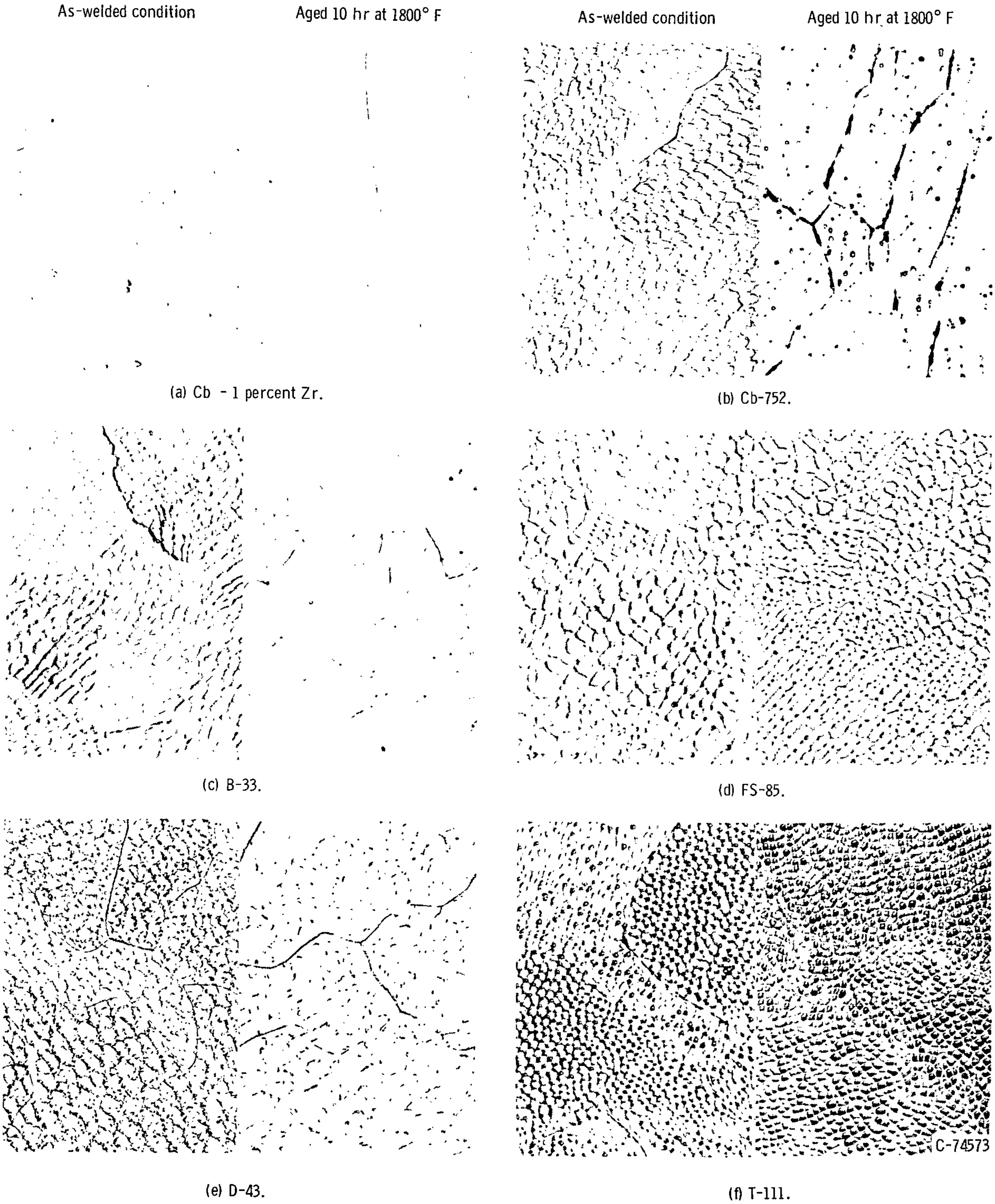
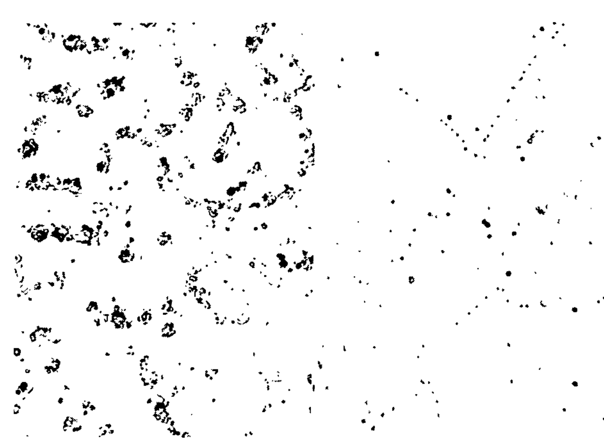


Figure 8. - Microstructures of fusion zones of electron-beam welded refractory metal sheets in the as-welded condition and after 10-hour aging at 1800° F. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.

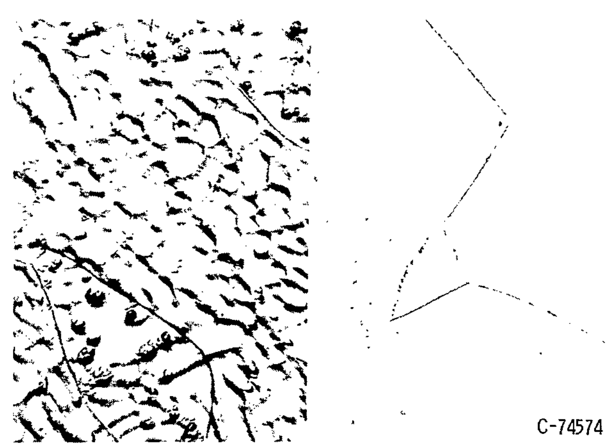
(a) As-welded condition.

(b) As-welded and then aged 10 hours at 1800° F.



(c) As-welded and then aged 10 hours at 2000° F.

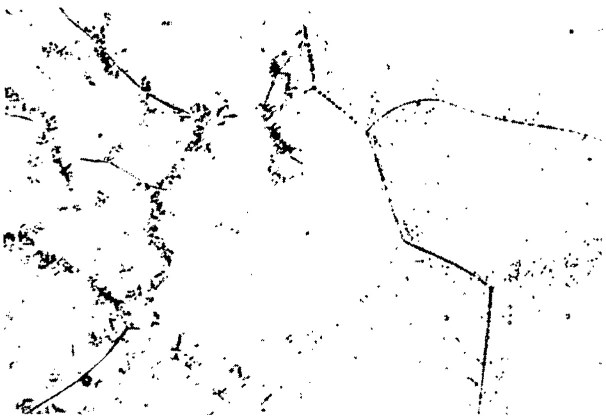
(d) As-welded and then aged 10 hours at 1800° F and 10 hours at 2200° F.



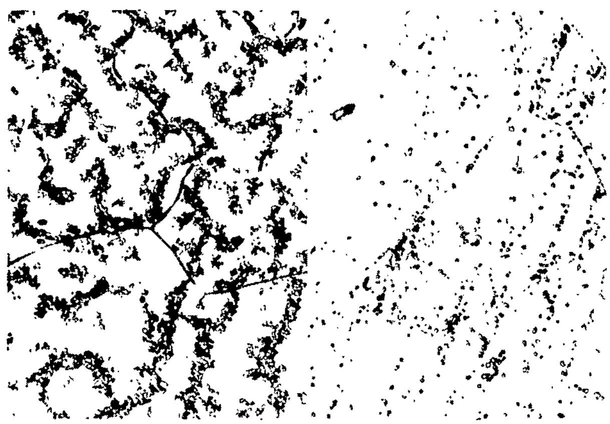
(e) As-welded and then aged 10 hours at 2000° F and 10 hours at 2200° F.

Figure 9. - Microstructures of gas tungsten-arc welded FS-85 sheet in as-welded condition and after 10-hour aging at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.

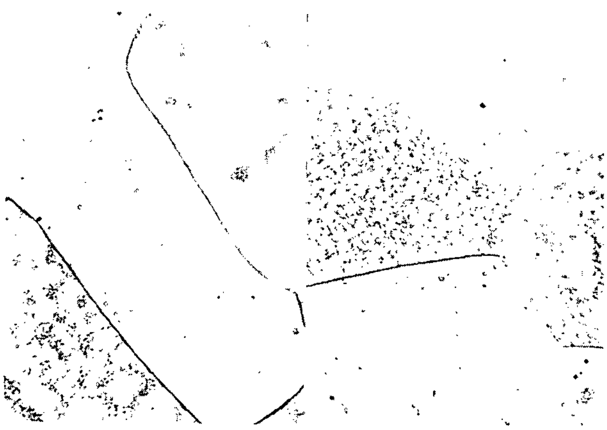
(a) Heat-treated 1 hour at 2500° F.



(b) Heat-treated and then aged 10 hours at 1800° F.



(c) Heat-treated and then aged 10 hours at 2000° F.



(d) Heat-treated and then aged 10 hours at 1800° F and 10 hours at 2200° F.

C-74575

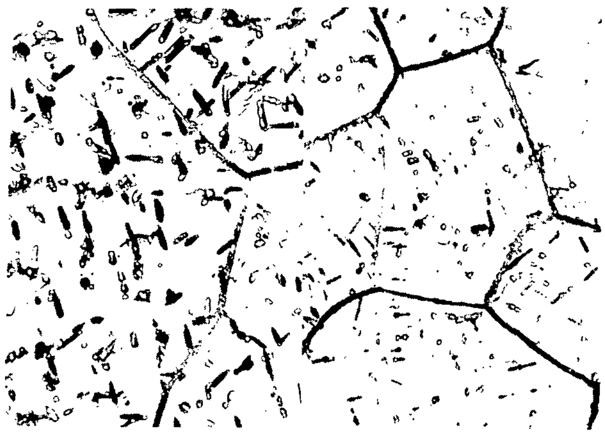
(e) Heat-treated and then aged 10 hours at 2000° F and 10 hours at 2200° F.

Figure 10. - Microstructures of gas tungsten-arc welded FS-85 sheet heat-treated for 1 hour at 2500° F and after 10-hour aging at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.

Fusion zone Heat-affected zone

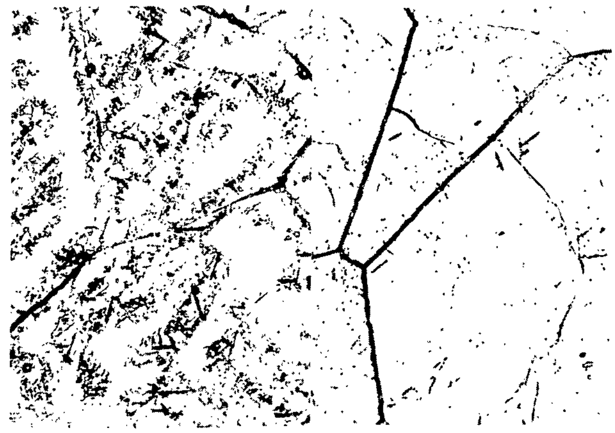
(a) As-welded condition.

Fusion zone Heat-affected zone



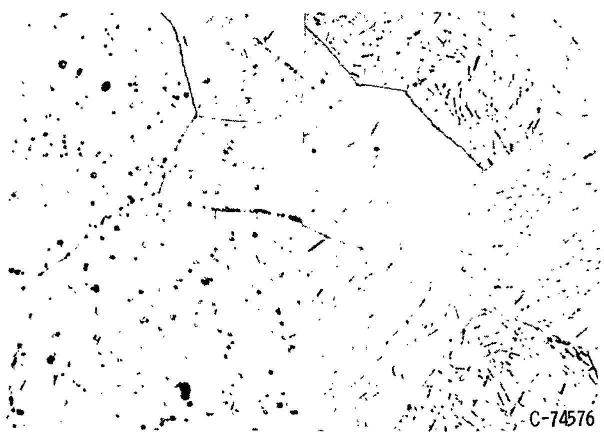
(b) As-welded and then aged 10 hours at 1800° F.

Fusion zone Heat-affected zone



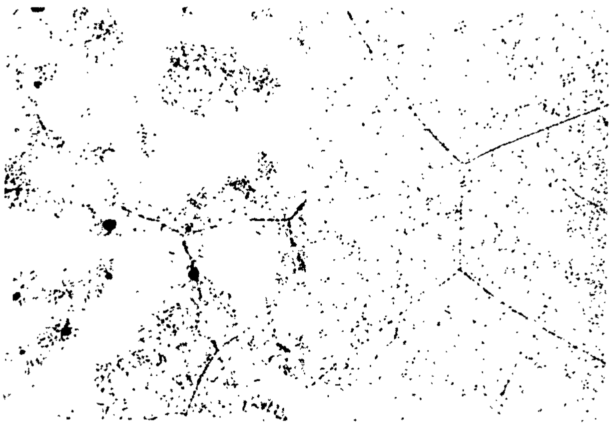
(c) As-welded and then aged 10 hours at 2000° F.

Fusion zone Heat-affected zone

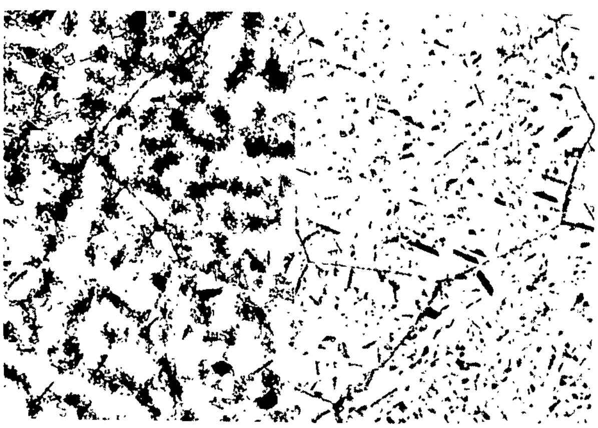


(d) As-welded and then aged 10 hours at 2000° F and 10 hours at 2200° F.

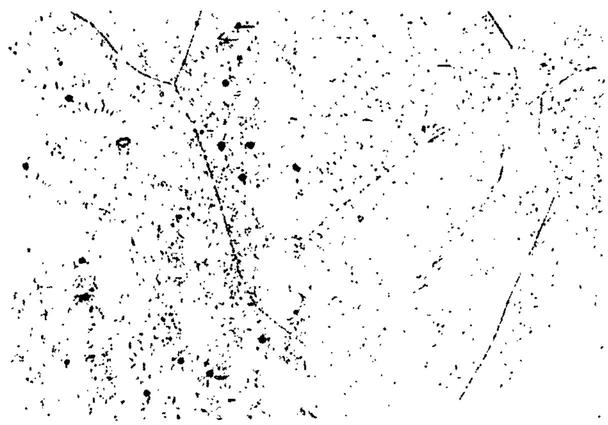
Figure 11. - Microstructures of gas tungsten-arc welded D-43 sheet in as-welded condition and after 10-hour aging at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.



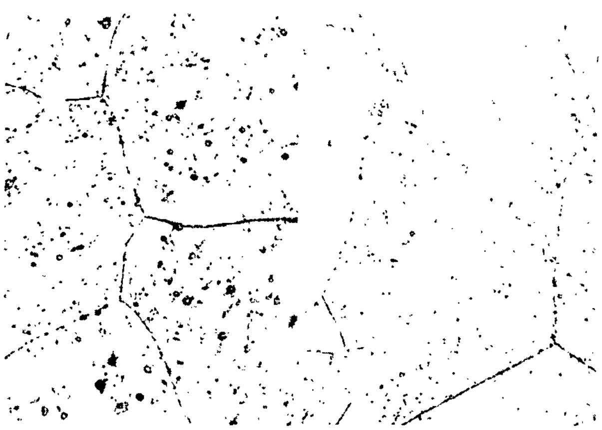
(a) As-welded and heat-treated 1 hour at 2500° F.



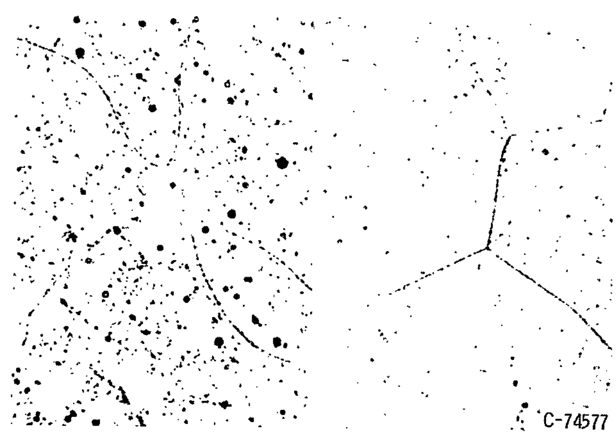
(b) Heat-treated and then aged 10 hours at 1800° F.



(c) Heat-treated and then aged 10 hours at 2000° F.



(d) Heat-treated and then aged 10 hours at 1800° F and 10 hours at 2200° F.



(e) Heat-treated and then aged 10 hours at 2000° F and 10 hours at 2200° F.

Figure 12. - Microstructures of gas tungsten-arc welded D-43 sheet heat-treated for 1 hour at 2500° F and after 10-hour aging at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.

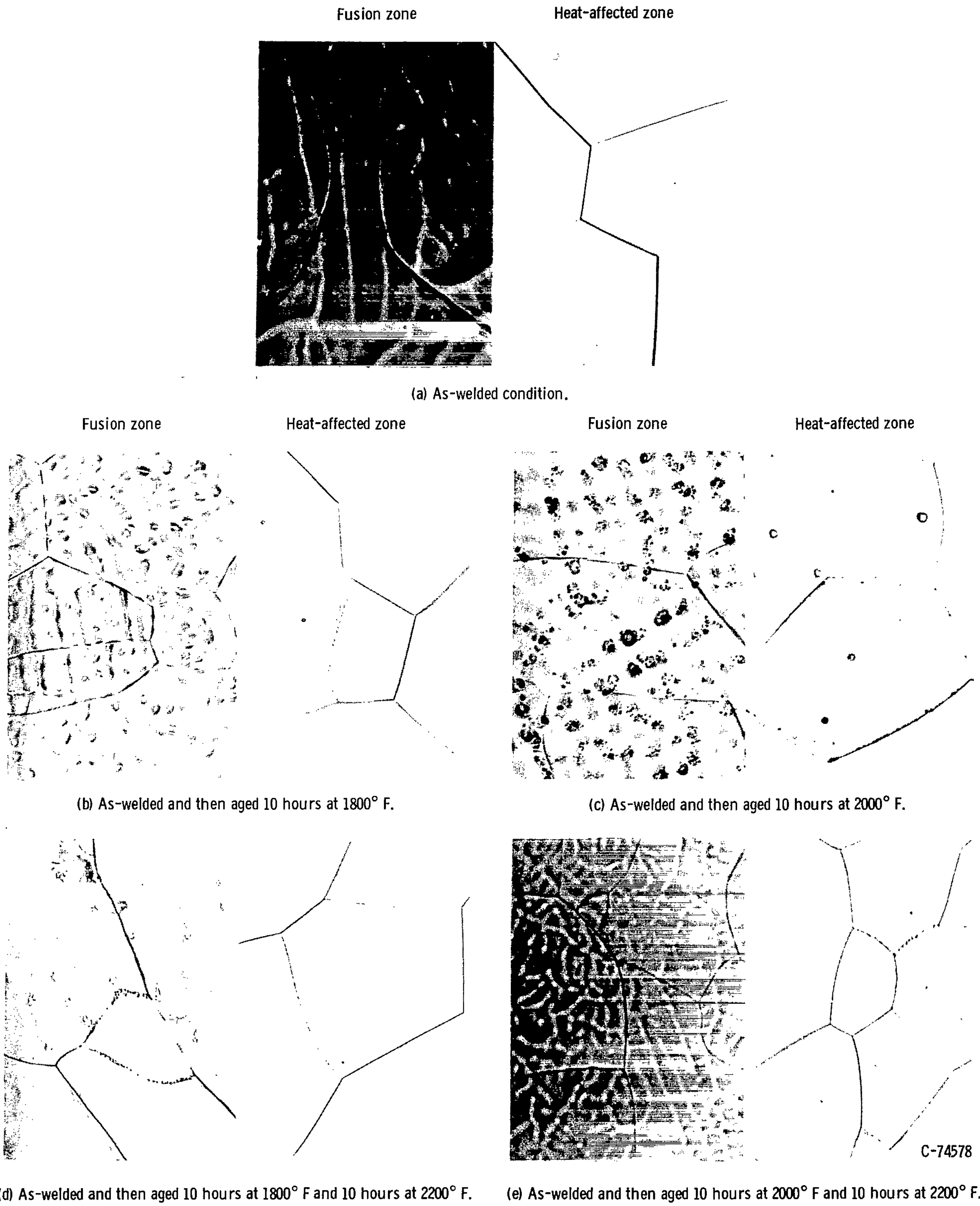
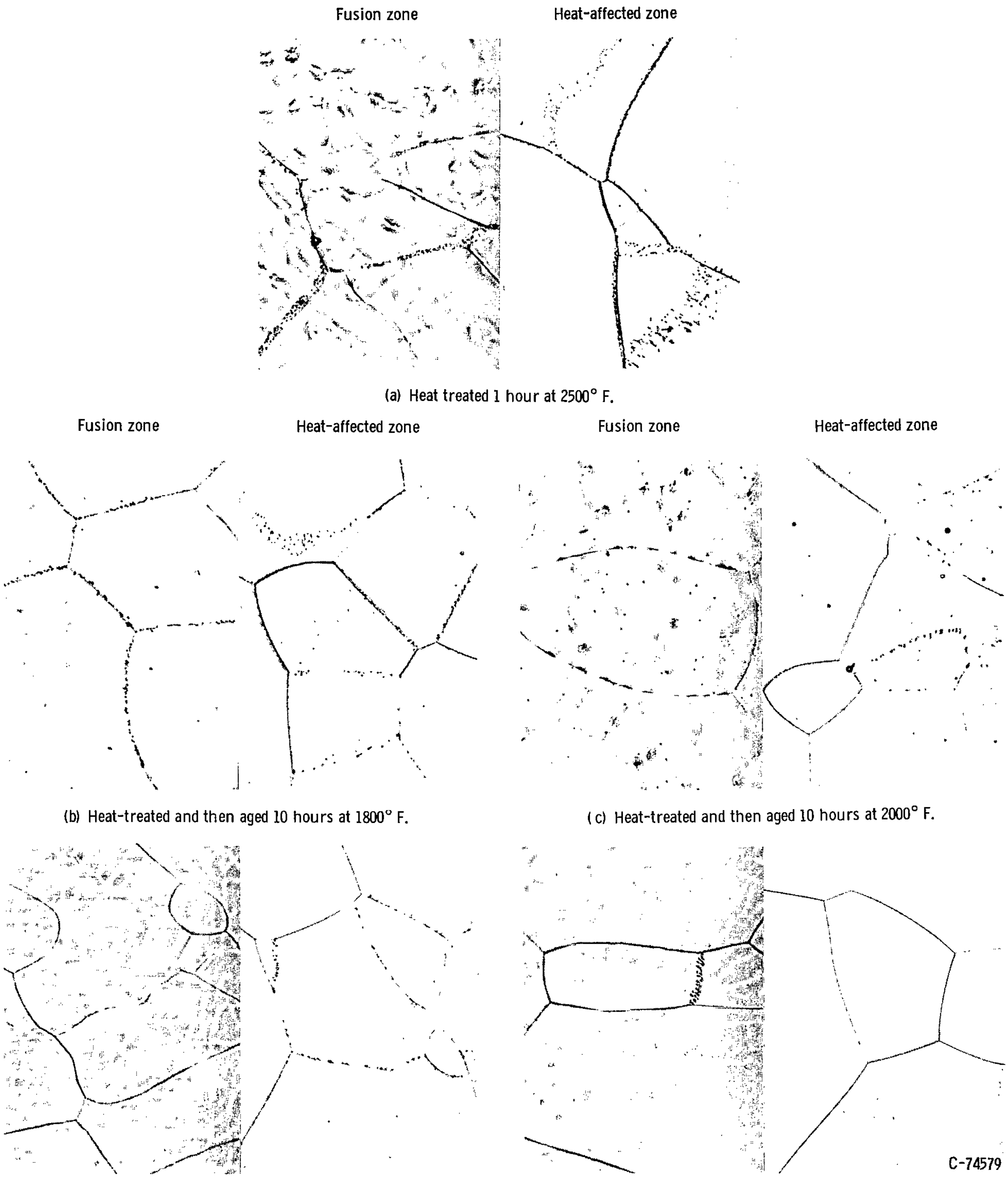


Figure 13. - Microstructures of gas tungsten-arc welded T-111 sheet in as-welded condition and after 10-hour aging at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.



(a) Heat treated 1 hour at 2500° F.

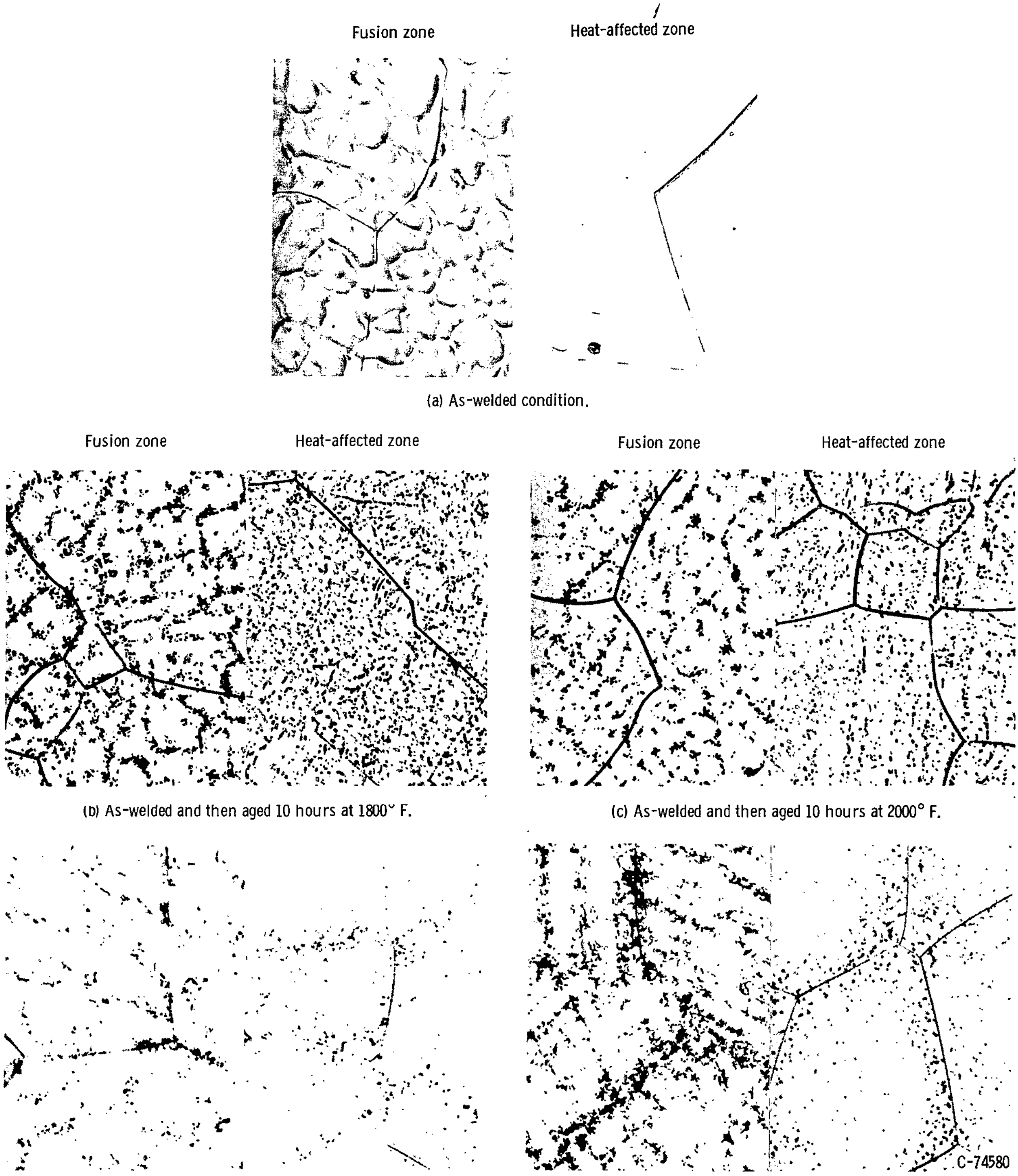
(b) Heat-treated and then aged 10 hours at 1800° F.

(c) Heat-treated and then aged 10 hours at 2000° F.

(d) Heat-treated and then aged 10 hours at 1800° F and 10 hours at 2200° F.

(e) Heat-treated and then aged 10 hours at 2000° F and 10 hours at 2200° F.

Figure 14. - Microstructures of gas tungsten-arc welded T-111 sheet heat-treated for 1 hour at 2500° F and after 10-hour aging at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.



(a) As-welded condition.

(b) As-welded and then aged 10 hours at 1800° F.

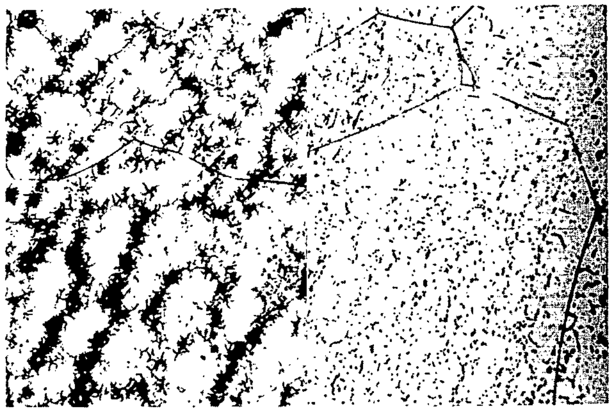
(c) As-welded and then aged 10 hours at 2000° F.

(d) As-welded and then aged 10 hours at 1800° F and 10 hours at 2200° F.

(e) As-welded and then aged 10 hours at 2000° F and 10 hours at 2200° F.

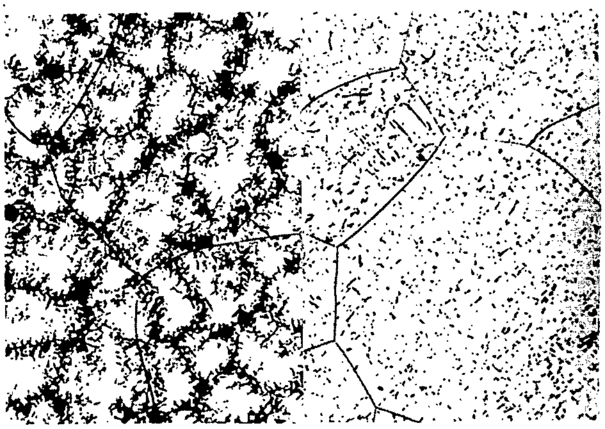
Figure 15. - Microstructures of gas tungsten-arc welded T-222 sheet in as-welded condition and after 10-hour aging at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.

Fusion zone　Heat-affected zone



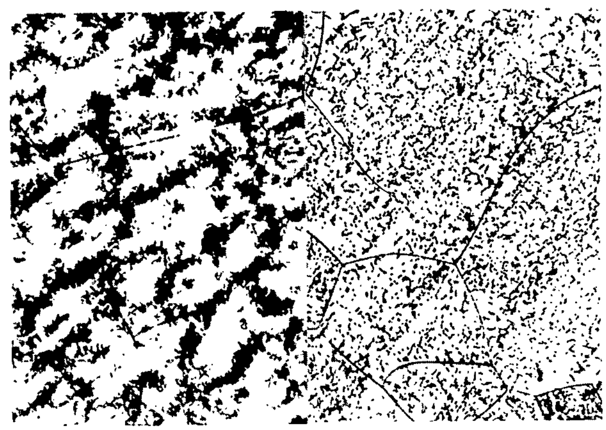
(a) Heat-treated 1 hour at 2500° F.

Fusion zone　Heat-affected zone

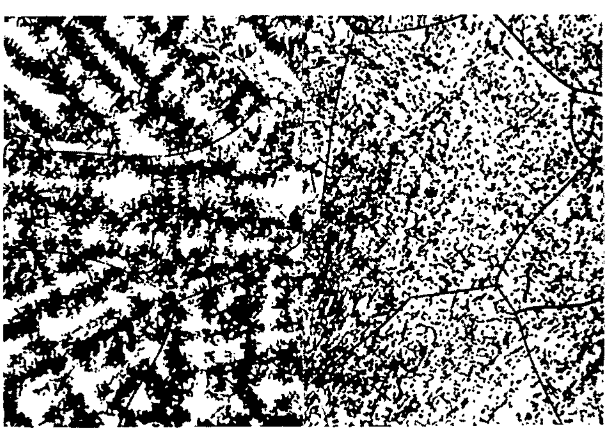


(b) Heat-treated and then aged 10 hours at 1800° F.

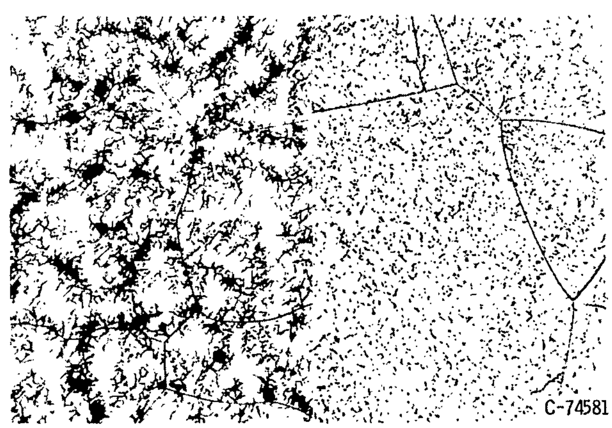
Fusion zone　Heat-affected zone



(c) Heat-treated and then aged 10 hours at 2000° F.

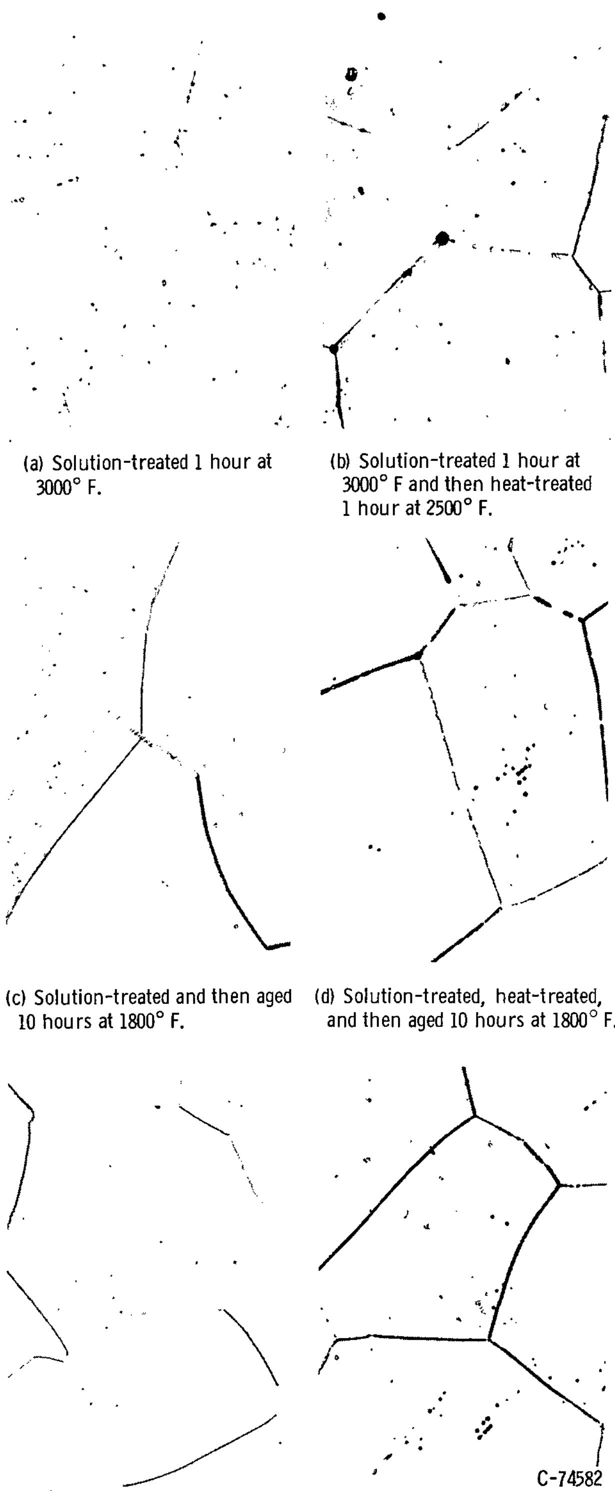


(d) Heat-treated and then aged 10 hours at 1800° F and 10 hours at 2200° F.



(e) Heat-treated and then aged 10 hours at 2000° F and 10 hours at 2200° F.

Figure 16. - Microstructures of gas tungsten-arc welded T-222 sheet heat-treated for 1 hour at 2500° F and after 10-hour aging at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.



(a) Solution-treated 1 hour at 3000° F.

(b) Solution-treated 1 hour at 3000° F and then heat-treated 1 hour at 2500° F.

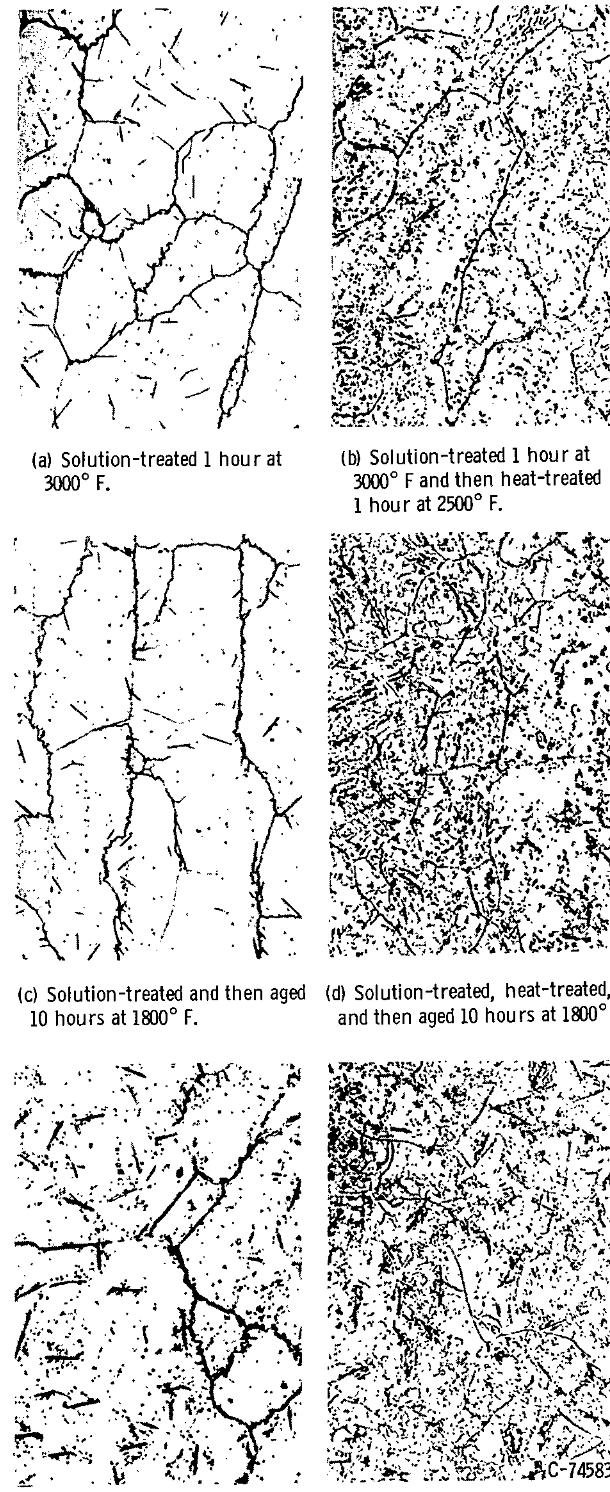
(c) Solution-treated and then aged 10 hours at 1800° F.

(d) Solution-treated, heat-treated, and then aged 10 hours at 1800° F.

(e) Solution-treated and then aged 10 hours at 2000° F.

(f) Solution-treated, heat-treated, and then aged 10 hours at 2000° F.

Figure 17. - Microstructures of FS-85 sheet solution-treated 1 hour at 3000° F, heat-treated 1 hour at 2500° F, and then aged 10 hours at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.



(a) Solution-treated 1 hour at 3000° F.

(b) Solution-treated 1 hour at 3000° F and then heat-treated 1 hour at 2500° F.

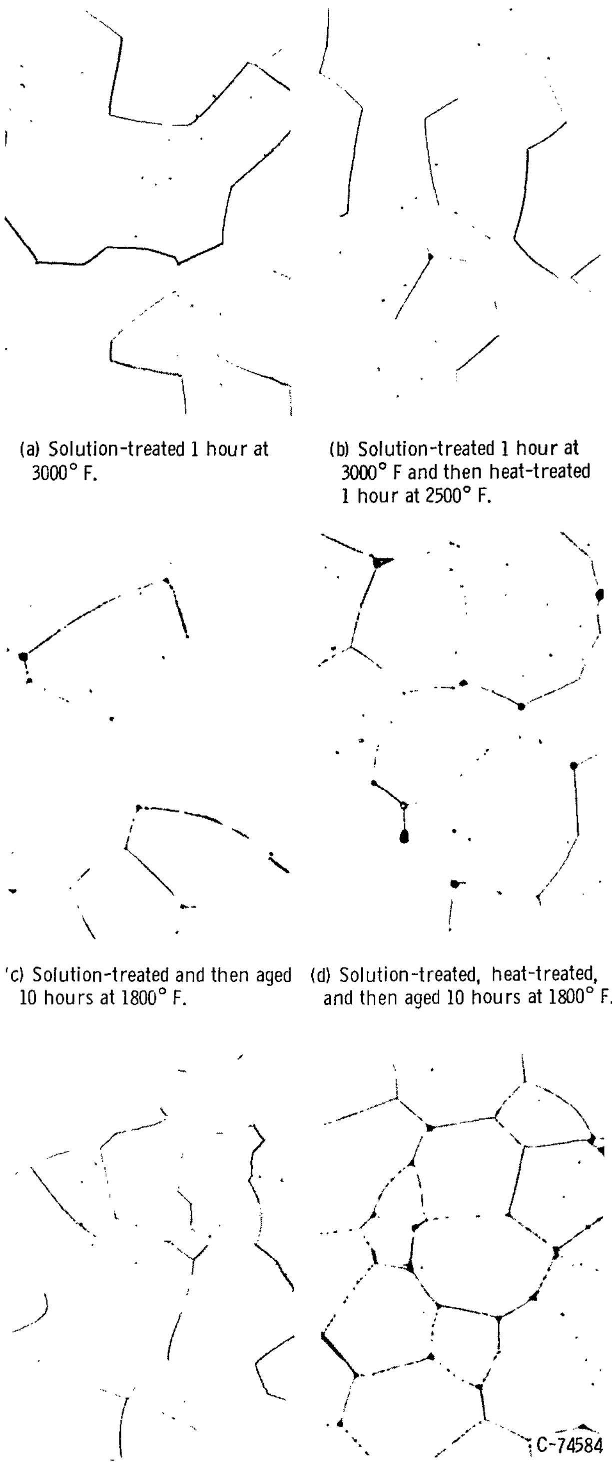
(c) Solution-treated and then aged 10 hours at 1800° F.

(d) Solution-treated, heat-treated, and then aged 10 hours at 1800° F.

(e) Solution-treated and then aged 10 hours at 2000° F.

(f) Solution-treated, heat-treated, and then aged 10 hours at 2000° F.

Figure 18. - Microstructures of D-43 sheet solution-treated 1 hour at 3000° F, heat-treated 1 hour at 2500° F, and then aged 10 hours at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.



(a) Solution-treated 1 hour at 3000° F.

(b) Solution-treated 1 hour at 3000° F and then heat-treated 1 hour at 2500° F.

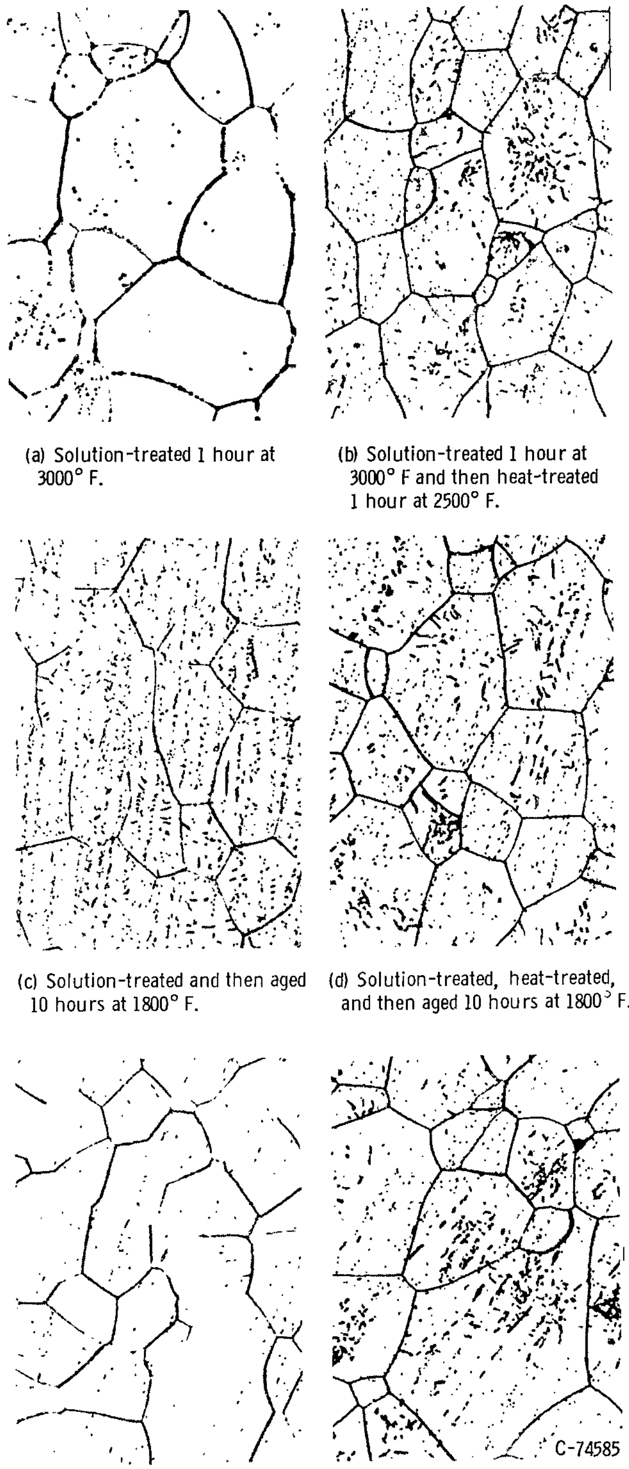
(c) Solution-treated and then aged 10 hours at 1800° F.

(d) Solution-treated, heat-treated, and then aged 10 hours at 1800° F.

(e) Solution-treated and then aged 10 hours at 2000° F.

(f) Solution-treated, heat-treated, and then aged 10 hours at 2000° F.

Figure 19. - Microstructures of T-111 sheet solution-treated 1 hour at 3000° F, heat-treated 1 hour at 2500° F, and then aged 10 hours at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.



(a) Solution-treated 1 hour at 3000° F.

(b) Solution-treated 1 hour at 3000° F and then heat-treated 1 hour at 2500° F.

(c) Solution-treated and then aged 10 hours at 1800° F.

(d) Solution-treated, heat-treated, and then aged 10 hours at 1800° F.

(e) Solution-treated and then aged 10 hours at 2000° F.

(f) Solution-treated, heat-treated, and then aged 10 hours at 2000° F.

Figure 20. - Microstructures of T-222 sheet solution-treated 1 hour at 3000° F, heat-treated 1 hour at 2500° F, and then aged 10 hours at various temperatures. Etchant, nitric acid and hydrofluoric acid. X1000. Reduced 50 percent in printing.

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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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