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BEND TRANSITION TEMPERATURE OF ARC-CAST MOLYBDENUM AND MOLYBDENUM — 0.5-PERCENT-TITANIUM SHEET IN WORKED, RECRYSTALLIZED, AND WELDED CONDITIONS

by John H. Sinclair Lewis Research Center Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • OCTOBER 1964



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SUMMARY

An investigation was conducted to determine the effects of welding on the ductile-to-brittle transition temperature of carbon-deoxidized arc-cast molybdenum and molybdenum - 0.5-percent-titanium sheet of two nominal thicknesses, 0.020 and 0.040 inch. Both materials, when annealed at 2000° or 2400° F to give recrystallized structures, gave fine grain structures (about 3500 grains/ sq mm). These materials had ductile-to-brittle bend transition temperatures (90°) bend over a 4 T radius punch descending at a rate of 1/2 in./min) of 0° to 40° F as compared with -110° to 5° F for the materials in the fibrous, asworked condition. When the specimens were arc welded in a helium atmosphere with a nonconsumable tungsten electrode, the transition temperatures of both materials varied from 140° to 350° F. The grain size in the fusion zone of the welded specimens was large (2 to 5 grains across a 40-mil (1.02-mm) specimen). Specimens annealed in a vacuum at 3500° F to yield grain sizes equivalent to those found in the fusion zones of welds had transition temperatures of 350° F and above. Chemical analyses indicated no pickup of interstitial impurities during either welding or vacuum annealing, and it was concluded that the loss of room-temperature ductility in the welds and in the annealed material was primarily associated with the massive grain structures. Metallographic study revealed fewer grain-boundary precipitates in the welded specimens than in those annealed at 3500° F, probably because the faster cooling rate of the welded metal had left less time for precipitation to occur.

INTRODUCTION

At the initiation of this investigation, commercial molybdenum sheet was available that retained some bend ductility in the welded state at room temperature (refs. 1 and 2). An experiment was planned to determine the extent of ductility loss resulting from the welding of good quality commercial sheet. Arc-cast, carbon-deoxidized molybdenum and molybdenum - 0.5-percent-titanium samples were selected as being representative of the molybdenum-based materials available. The materials were obtained in 0.020- and 0.040-inch nominal thicknesses. Welding was accomplished by means of the inert-gas tungsten arc process (TIG).

Bend transition temperatures were determined for the two materials in the worked, recrystallized, and welded conditions. Two different recrystallized grain sizes were included. Specimens were annealed at relatively low temperatures to give grain sizes similar to those found in the heat-affected zone of fusion welds and also at higher temperatures to give grain sizes comparable to those found in the fusion zone of the welds. Bend properties after annealing in a hydrogen atmosphere were compared with those obtained by annealing in vacuum. Some welded specimens were stress-relief annealed to determine if this would result in a lowering of the bend transition temperature. Microstructural studies and chemical analyses were utilized as aids to the analyses of bend test results.

MATERIALS

The materials selected for the program were arc-cast molybdenum and arccast molybdenum - 0.5-percent-titanium alloy. Both were carbon-deoxidized and were obtained in two nominal thicknesses, 0.020 and 0.040 inch. Both gages of each material came from a single heat produced by a commercial supplier. Chemical analyses for the sheet materials utilized are shown in table I. The final processing procedure was to polish the sheets parallel to the rolling direction. Profilometer measurements of the as-received sheets showed a root-meansquare surface roughness of approximately 50.

TABLE I. - CHEMICAL ANALYSES OF

SHEET MATERIALS

	Element	Sheet material		
		Molybdenum, parts per million	Molybdenum - 0.5-percent- titanium, parts per million	
	Carbon	390	310	
	Hydrogen	1.4	1.2	
	Nitrogen	55	28	
	Oxygen	106	22	
1	Sulfur	34	47	
	Cobalt	^a MD, <10	ND, <10	
	Chromium	10	ND, <10	
	Columbium	ND, <50	ND, <50	
	Iron	70	40	
J	Nickel	30	10	
	Silicon	50	50	
	Tantalum	ND, <500	ND, <500	
	Tungsten	ND, <500	ND, <500	
	Titanium	0	0.46 percent	

^aND, not detected.

APPARATUS

Annealing was carried out in a hydrogenatmosphere or a vacuum furnace. Temperature measurements were made by use of platinum platinum-13-percent-rhodium thermocouples in the hydrogen furnace and by tungsten tungsten-26-percent-rhenium thermocouples in the vacuum furnace.

Welding was done in a vacuum chamber incorporating an automatic welding head and a mechanical specimen-traversing device so that reproducible welding conditions could be maintained.

In inert-gas tungsten arc welding, arc voltage values increase or decrease with the length of the arc. The automatic welding head maintains a constant voltage and, hence, a constant arc length. If the arc length changes for any reason, such as the appearance of a contour in the surface of the work, the arc voltage changes. The voltage fluctuation is used to vary the amount of current to one of two drive motors in the automatic head that normally revolve at the same speed. This changes the speed of one of the motors and causes the electrode holder to move up or down until the preset voltage is again established. The welding fixture is shown in figure 1. It holds three bend specimens in line in addition to starting and runoff tabs. Fixture design was based on considerations of specimen geometry and the heat-transfer properties of molybdenum.

Specifications of the bend test fixtures are given in reference 3. Channel dies were used. Die spans were 0.50 and 0.25 inch for the 0.040- and the 0.020-inch sheet materials, respectively. Punch radii were four times the sheet material thicknesses. Punch travel was 1/2 inch per minute.

PROCEDURE

Specimen Fabrication

Specimens were cut from the sheet material with a water-cooled abrasive cutoff wheel. The lengths of the specimens were parallel to the final rolling direction of the sheets. All specimens were 2 inches in length. The 0.040inch-thick specimens were 1/2 inch in width for tests in the as-received and recrystallized conditions. To facilitate clamping for welding, it was necessary to use pieces 3/8 inch wide, which yielded welded specimens 3/4 inch wide. The 0.020-inch-thick sheet material was cut in 1/4-inch widths for asreceived and recrystallized bend specimens, while specimens to be welded were cut 0.3 inch wide to yield 0.6 inch-wide welded specimens. Edges of all bend specimens were hand lapped with 280-grit silicon-carbide paper and inspected for laminations under a binocular microscope at a magnification of approximately 15.

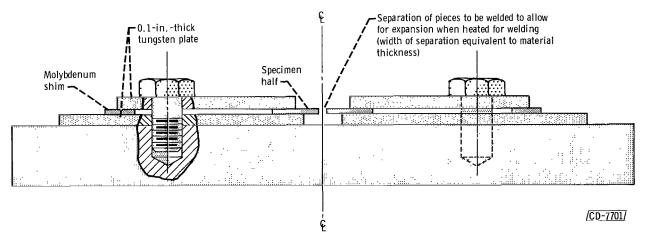


Figure 1. - End view of welding fixture (length, 9 in.).

Cleaning

Prior to welding, all specimens were vapor degreased and chemically cleaned. A cleaning method was selected that consisted of the following steps (ref. 4):

- (1) Five- to 10-minute dip in hot (150° to 180° F) deoxidizer consisting of 10 percent sodium hydroxide, 5 percent potassium permanganate, and 85 percent water (by weight)
- (2) Cold-water rinse
- (3) Approximately 5-minute dip in 15 percent sulfuric acid, 15 percent hydrochloric acid, 70 percent water (by volume) with 6 to 10 grams of chromic acid added per 100 milliliters
- (4) Hot-water rinse
- (5) Air drying

During cleaning, specimens were held in a basket fabricated from perforated molybdenum sheet. Thereafter, until welding was completed, specimens were handled only with tweezers.

Welding Techniques

The chamber was pumped down to a pressure of 1×10^{-4} millimeter of mercury or less, and then back filled with helium to approximately 2/3 atmosphere (as indicated by a Bourdon gage), in order to support a stable arc. Specimens were preheated to approximately 300° F prior to welding by infrared lamps within the welding chamber. Preheating temperature was measured with type K thermocouples (ref. 5) in contact with the welding fixtures.

Gettering was employed to improve the atmospheric purity in the weld box before the molybdenum strips were welded. Either bead-on-plate runs were made on titanium sheet prior to welding or titanium filaments located near weld specimens were heated to incandescence prior to and during welding.

A welding speed of 7 inches per minute was used for welding all of the bend-test specimens. A motor-operated screw moved the welding fixture and specimens past the stationary welding head at constant speed. A 1/16-inchdiameter, 2-percent-thoriated-tungsten electrode was selected for welding both the 0.020- and the 0.040-inch sheet material. Fusion of the sheet material was accomplished by use of direct-current straight-polarity arc welding; no filler metal was added. Power input to the arc for welding the 0.040-inch molybdenum at 7 inches per minute varied from 120 to 180 amperes at 15.5 volts. The 0.040inch molybdenum - 0.5-percent-titanium sheet required from 110 to 190 amperes at 15.5 volts. The welding fixture temperature went up as the welding progressed, and gradual reduction of the welding current to avoid burn-through was necessary as welding progressed. These reductions were based on visual estimation of the width of the fused zone. The 0.020-inch molybdenum - 0.5-percenttitanium sheet required 80 to 85 amperes at 15.5 volts for welding. Since these specimens warped too badly during welding to permit bend testing, 0.020inch molybdenum was not welded.

Stress-Relief Annealing

An experiment was conducted on two groups of 0.040-inch molybdenum - 0.5-percent-titanium specimens to determine whether stress-relief annealing after welding would lower the bend transition temperature. This procedure was based on common practice (refs. 6 and 7). One group was stress-relief annealed in a vacuum furnace at 1800° F for $1\frac{1}{2}$ hours and furnace cooled to approximately 200° F in about $2\frac{1}{2}$ hours.

A second group was stress-relief annealed at 1800° F for 1 hour in flowing hydrogen and cooled to approximately 200° F in about 1 hour before being removed from the furnace. All stress-relief annealing was done after the specimens were ground flat.

Recrystallization

Specimens annealed in the hydrogen atmosphere furnace were brought to temperature in approximately 1 hour if annealed at 2000° F (molybdenum) and in approximately $1\frac{1}{2}$ hours if annealed at 2400° F (molybdenum - 0.5-percent-titanium alloy). At the end of the annealing time the power was shut off and the furnace was cooled to nearly room temperature with hydrogen flow throughout the cooling period. The specimens annealed at 2400° F required approximately $1\frac{3}{4}$ hours for cooling; those annealed at 2000° F required about $1\frac{1}{2}$ hours. Specimens annealed in the vacuum furnace were brought to 2000° F in approximately 20 minutes (molybdenum), to 2400° F in approximately 30 minutes (molybdenum - 0.5-percent-titanium alloy), or to 3500° F in approximately 45 minutes (both materials). After the specimens were held at 2000° or 2400° F for 1 hour or at 3500° F for 66 minutes, the power was turned off and the specimens were allowed to cool to room temperature.

Bend Testing

Only the specimens that exhibited complete penetration were prepared for bend testing. Welded specimens were ground to eliminate warpage and projecting weld metal before bend testing. Otherwise, point contact of the punch and/or die could introduce uneven stresses on the specimens, which would result in erratic bend results. Lapping scratches ran in the longitudinal direction of the specimens. Some sunken areas in the fused zones of the welds were not re-

moved entirely since this would have resulted in extreme reduction of specimen thickness.

Welded specimens were centered face down on the die so that the weld faces would be in tension during the bend test. Bending above room temperature was done in heated mineral oil. The specimens were held at the desired temperature in the oil for at least 10 minutes prior to bending. The oil was kept in circulation by an air operated propeller stirrer to keep the temperature nearly uniform throughout. The temperature was measured by a thermometer placed on the die block within 1/2 inch of the specimen. Bending below room temperature was done in an acetone - dry-ice mixture. For temperatures lower than -108° F liquid nitrogen passed through copper tubes immersed in the acetone was used. Stable temperatures were not achieved during liquid-nitrogen use: hence. temperature measurements made during liquid-nitrogen use were not as accurate as those made when only acetone and dry ice were used. The environment was brought to a temperature approximately 5° F below that required by the addition of dry ice to the acetone and held there for at least 10 minutes; then the temperature was allowed to rise to the desired temperature, and the bending was initiated.

When specimen failure was indicated by audible cracking of the specimen, the test was terminated. Specimens that did not fail could be bent to a maximum of approximately 170° before the punch reached bottom. A microswitch was adjusted to shutoff punch travel just before the punch reached the bottom of the die.

RESULTS AND DISCUSSION

Bend Transition Properties

Transition temperatures of as-received (as-worked), recrystallized, and welded molybdenum and molybdenum - 0.5-percent-titanium specimens as determined by bend testing are shown in table II. Bend angles of fracture against test temperatures are plotted in figures 2 and 3. The transition temperature was arbitrarily defined as the temperature at which the bend angle of fracture was 90° .

Worked materials. - The bend transition temperatures for molybdenum specimens tested in the as-worked condition were approximately -90° F for the 0.020-inch-thick material and 5° F for the 0.040-inch-thick material (table II, figs. 2(a) and 3(a).

The bend transition temperatures for molybdenum - 0.5-percent-titanium specimens were approximately -110° F for the 0.020-inch-thick material and -100° F for the 0.040-inch-thick material (figs. 2(b) and 3(b)).

<u>Recrystallized materials.</u> - For the 0.040-inch-thick sheet materials, results are given for specimens annealed at two different temperatures (table II). Specimens annealed at relatively low temperatures (2000° and 2400° F) show grain sizes equivalent to those found in the zones of welds affected by heating

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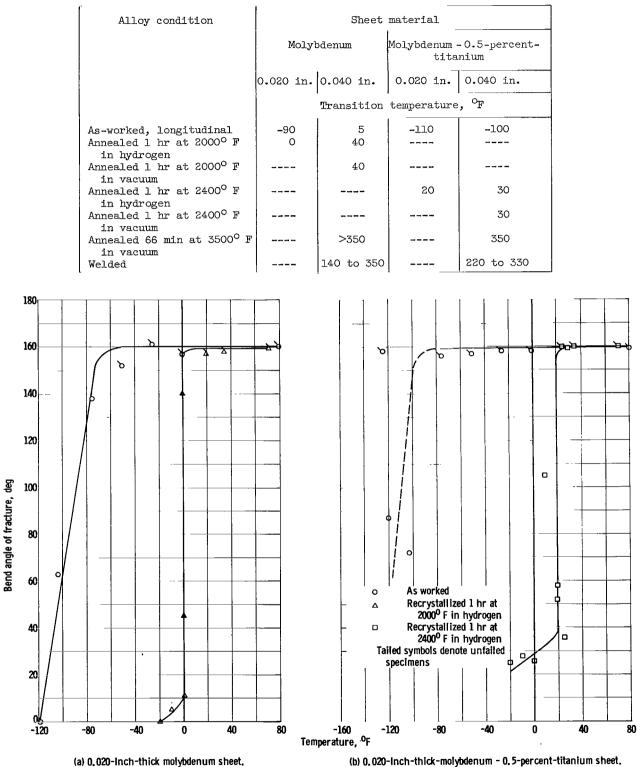
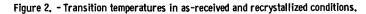
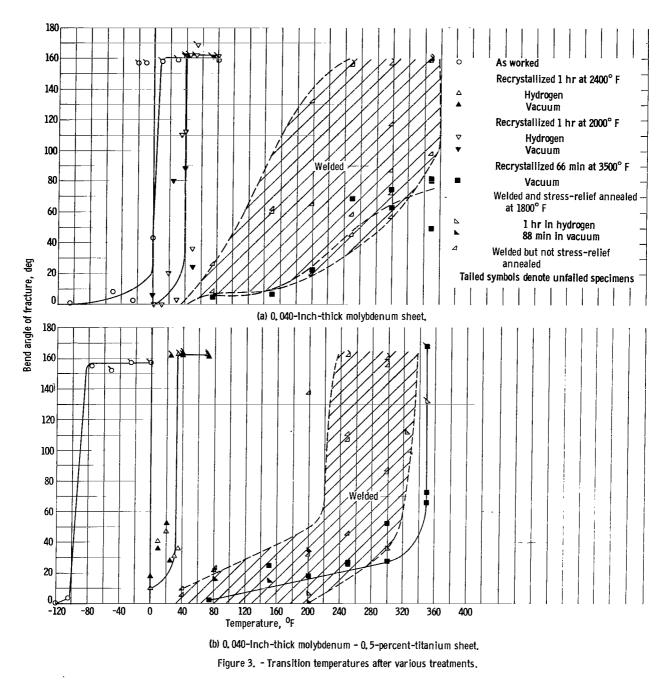


TABLE II. - APPROXIMATE TRANSITION TEMPERATURES



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I.



(fig. 4). Another group annealed at high temperatures (3500° F) shows grain sizes essentially the same as those obtained in the fusion zones of welds (figs. 5(a), (d), (f), and (i)); that is, the specimens have approximately 2 to 5 grains across the 0.040-inch thickness.

The specimens recrystallized at lower temperatures $(2000^{\circ} \text{ and } 2400^{\circ} \text{ F})$ are further divided into two groups, those recrystallized in vacuum $(3\times10^{-5} \text{ mm Hg})$, and those recrystallized in flowing hydrogen. Apparently no measurable difference in the transition temperatures of those specimens recrystallized in vacuum





(a) 0.040-Inch-thick molybdenum specimen annealed at 2000° F in vacuum.

(b) 0.040-Inch-thick molybdenum - 0.5-percent-titanium specimen annealed at 2400° F in vacuum.

Figure 4. - Specimens annealed at low temperatures; grain sizes similar to those in heat affected zones of welds. Etchant, potassium hydroxide and potassium ferricyanide. X100. (Reduced 25 percent in printing.)

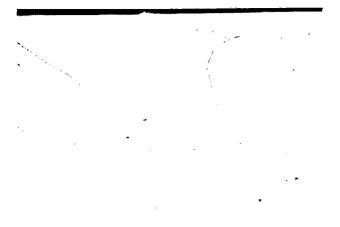
and those recrystallized in flowing hydrogen (fig. 3) exists, and they are well below room temperature.

The specimens annealed at the higher temperature (3500° F) to give a grain size comparable with that of the fusion zone of a weld show transition temperatures higher than 350° F for the 0.040-inch-thick molybdenum sheet and approximately 350° F for 0.040-inch-thick molybdenum - 0.5-percent-titanium sheet (fig. 3). Even though the recrystallization operation was carried out in a 3×10^{-5} -millimeter-of-mercury vacuum, the transition temperatures were as high or higher than those obtained for the welded specimens (table II).

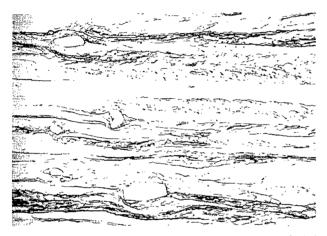
Welded materials. - Figure 6 (p. 12) presents an overall view of a typical welded specimen (0.040-in.-thick molybdenum - 0.5-percent-titanium alloy) that shows the variation in grain size after welding. At the extreme left is a portion of the fused zone of the weld where the total cross section of the metal is comprised of as few as 2 to 5 grains. Farther to the right is the heataffected zone, where recrystallization has occurred. The grain size decreases toward the right until the area containing fibrous, unaffected base metal is finally reached.

The transition temperatures for the welded materials are recorded in table II. Bend-test data from which transition temperatures of welded 0.040-inch molybdenum and 0.040-inch molybdenum - 0.5-percent-titanium sheet materials were determined are plotted in figure 3.

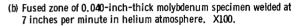
In the case of the welded materials, the points in figure 3 define a band rather than a curve; that is, the data points are considerably scattered. This may be partially due to variation in welding current and the resulting welds.



(a) 0.040-Inch-thick molybdenum specimen annealed 66 minutes at 3500° F in 3x10⁻⁵-millimeter-mercury vacuum. X100.



(c) Longitudinal section of 0.040-inch-thick molybdenum as received. X500.





(d) 0.040-Inch-thick molybdenum specimen annealed 66 minutes at 3500° F in 3x10⁻⁵-millimeter-mercury vacuum. X500.

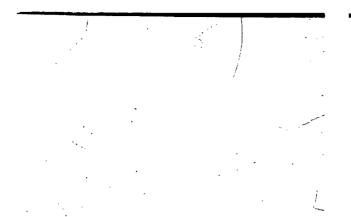


(e) Fused zone of 0.040-inch-thick molybdenum specimen welded at 7 inches per minute in helium atmosphere. X500.

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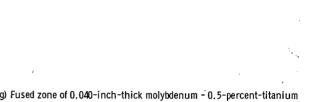
Figure 5. - Comparison of molybdenum and molybdenum - 0.5-percent-titanium specimens after various treatments. Etchant, potassium hydroxide and potassium ferricyanide. (Reduced 25 percent in printing.)



(f) 0.040-Inch-thick molybdenum - 0.5 percent-titanium specimen annealed 66 minutes at 3500° F in 3x10⁻⁵-millimeter-mercury vacuum. X100.



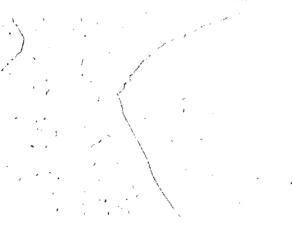
(h) Longitudinal section of 0.040-inch-thick molybdenum - 0.5-percenttitanium specimen as received. X500.



(g) Fused zone of 0.040-inch-thick molybdenum - 0.5-percent-titanium specimen welded at 7 inches per minute in helium atmosphere. X100.



(i) 0.040-Inch-thick molybdenum - 0.5-percent-titanium specimen annealed 66 minutes at 3500° F in 3x10⁻⁵-millimeter-mercury vaccum. X500.



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(j) Fused zone of 0.040-inch-thick molybdenum - 0.5-percent-titanium specimen welded at 7 inches per minute in helium atmosphere. X500.

Figure 5. - Concluded. Comparison of molybdenum and molybdenum - 0.5-percent-titanium specimens after various treatments. Etchant, potassium hydroxide and potassium ferricyanide. (Reduced 25 percent in printing.)

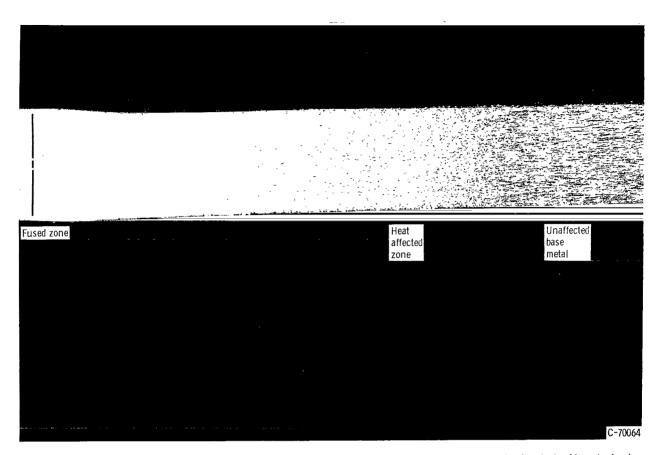


Figure 6. - Various zones in welded 0.040-inch-thick molybdenum - 0.5-percent-titanium specimen. Etchant, potassium hydroxide and potassium ferricyanide. X20. (Enlarged 20 percent in printing.)

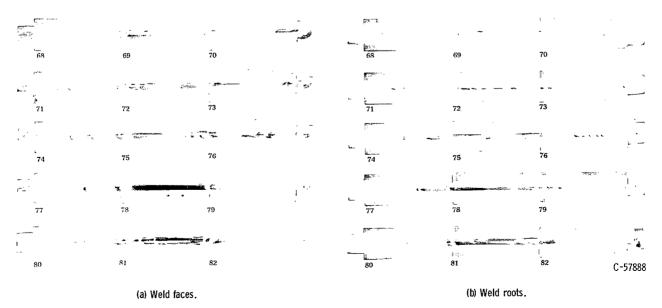


Figure 7. - Comparison of the weld faces and roots of typical welded specimens.

Welded molybdenum specimens (0.040-in. thick) showed transitiontemperature ranges of 140° to 350° F, and molybdenum - 0.5-percent-titanium specimens of the same thickness showed transition temperatures from approximately 220° to 330° F.

Photographs of typical welded specimens are presented in figure 7. Specimens 77 to 79 show large variations in the width of the fused zones on the weld-root sides (fig. 7(b)). Although welding voltage and rate of specimen traverse were constant for all three specimens, specimens 78 and 79 apparently received less heat than did specimen 77. This was a result of the necessary manual control of the amperage during welding. As welding progressed along specimen 77 and toward specimens 78 and 79, the temperature of the welding fix-tures began to increase. To avoid burn-through, gradual reduction of the amperage was necessary. This current adjustment was based on visual observation of the width of the weld bead on the top side of the weld specimens (fig. 7(a)). A uniform width of fused zone was maintained on the weld face side, and yet there was a great variation in fused zone width on the root side of the welds.

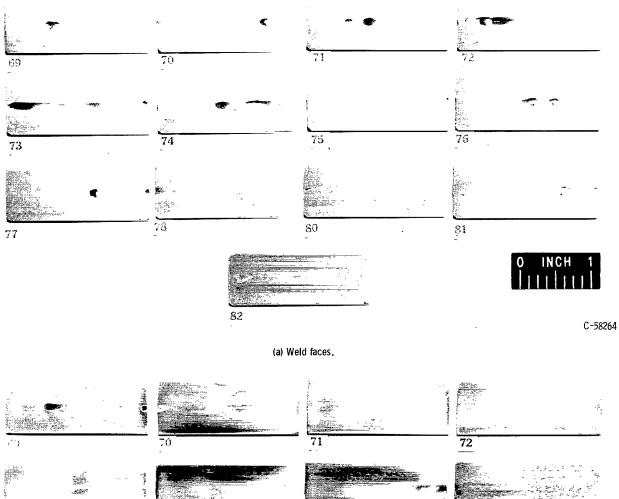
The data plotted in figure 3(b) (p. 8) indicate that stress-relief annealing of 0.040-inch-thick molybdenum - 0.5-percent-titanium bend specimens in vacuum or in hydrogen after welding was not beneficial. Neither group showed superior bend properties when compared with welded specimens that were bend tested with no stress-relief annealing (fig. 3(b)). Stress-relief annealing after welding was not applied to the remainder of the specimens.

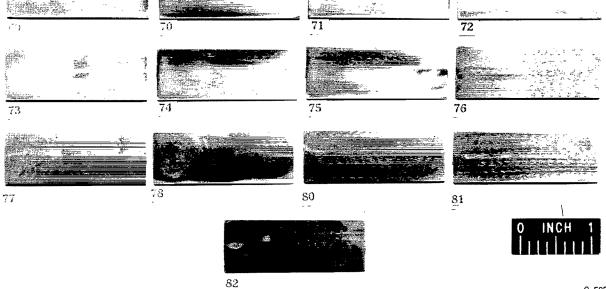
Typical welded specimens ground flat for bend testing are shown in figure 8. As was stated in the section PROCEDURE, sunken areas in the fused zones of welds could not be completely removed without making the specimens too thin to meet the thickness requirements described in reference 3. Bend testing indicated that these sunken areas (fig. 8) in themselves probably had no measurable detrimental effect on the ductility of the specimens. A series of three specimens was selected consisting of one with a great deal of sunken area in the bend zone, one with a moderate amount of sunken area in the bend zone, and one with no sunken area. All were bent at the same temperature. The worstappearing specimen had the greatest bend angle of fracture, and the bestappearing specimen had the smallest bend angle of fracture. The experiment was repeated with reverse results. Therefore specimens containing sunken areas apparently have bend transition temperatures within the scatter band of the data for all welded specimens. All bend specimens were between 0.037 and 0.040 inch in thickness following the grinding operations that flattened the warped specimens.

Microstructural Studies

Although specimens recrystallized at 3500° F in vacuum have the same grain size as the fused zones of welded specimens (figs. 5(a), (b), (f), and (g)), there is a difference in the metallurgical structure. Worked molybdenum - 0.5-percent-titanium specimens show small carbide inclusions (fig. 5(h)). Upon recrystallization the carbides redissolve, and upon slow cooling large carbide precipitates appear at the grain boundaries, as shown in figure 5(i). The







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(b) Weld roots. Figure 8. - Typical welded specimens ground for bend tests.

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welded specimens mainly show fine precipitates in the matrix (fig. 5(j), p. 10). This structure is the result of the redissolving of the precipitate upon fusion. The more rapid rate of cooling during the welding process allowed carbides to precipitate within the grains. Very little improvement in bend transition temperature resulted, however, from the apparently more desirable physical structure of the welded specimens as compared with the structure of the as-recrystallized specimens (figs. 5(j) and (i)). Worked molybdenum specimens show very large carbide particles (fig. 5(c)). The welded structure (fig. 5(e)) shows a discontinuous grain-boundary network and a fine-matrix precipitate. This structure is probably the result of the rapid fusion and cooling rates that accompany welding. Here, again, the welded specimens with the more desirable structure show very little improvement in bend transition temperature as compared with the recrystallized ones.

Chemical Analyses

To help determine whether impurities were being increased during recrystallization or welding operations, chemical analyses were made on the 0.040-inch

TABLE III. - CHEMICAL ANALYSES OF MOLYBDENUM -

0.5-PERCENT-	TITANIUM	ALLOY
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Alloy condition .	Concentration, parts per million		
	Oxygen	Nitrogen	Carbon
As received	22	28	310
Recrystallized 66 min at 3500 ⁰ F in vacuum	22	7	220
Recrystallized 60 min at 2400 ⁰ F in vacuum	22	3	357
Fusion zone of weld	22	6	353

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molybdenum - 0.5-percenttitanium alloy before and after these operations. The results obtained by a commercial assaver are shown in table III. This analysis indicated that no oxygen or nitrogen was acquired by the allov either during recrystallization in a vacuum or from the welding atmosphere. The vendor's analysis of the as-received molybdenum -0.5-percent-titanium alloy showed 220 parts carbon per million. while the commercial analysis given in table III indicates 310 parts carbon per million.

SUMMARY OF RESULTS

The following results were obtained from an investigation of bend transition temperatures of arc-cast molybdenum and molybdenum - 0.5-percent-titanium sheet in the worked condition and after recrystallization and welding:

1. The ductile-to-brittle transition temperatures for both molybdenum and molybdenum - 0.5-percent-titanium specimens were well below room temperature (approximately 5° to -100° F) when the metals were in the as-worked condition.

2. Inert-gas tungsten arc welding raised the transition temperature of both materials considerably above room temperature (140° to 350° F).

3. Bend transition temperatures for the two differently annealed materials with grain sizes equivalent to those occurring in heat-affected zones of welds (3000 to 4000 grains/sq mm) remained below room temperature (0° to 40° F). They were 35° to 130° F higher than bend transition temperatures, however, for the materials in the as-worked condition.

4. Bend transition temperatures for both types of annealed materials having grain sizes equivalent to those occurring in fusion zones of welds (2 to 5 grains across the 0.040-in.-thick specimen) were approximately equivalent to those of the welded specimens (350° F and higher).

5. While the welded specimens and specimens annealed at 3500° F exhibited similar recrystallized grain sizes, the microstructures differed. The recrystallized specimens had larger carbide particles, often concentrated in the grain boundaries, and the welded specimens apparently had many smaller carbide precipitations, scattered more uniformly along the grain boundaries and throughout the matrices.

6. Based on chemical and metallographic analyses this experiment indicated that welding raised the transition temperature because it created a large grain size and not because impurities from the atmosphere contaminated the welded metal.

7. In general, the bend transition temperatures for 0.020-inch-thick materials were lower than those for 0.040-inch-thick materials tested in the same condition.

Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio, July 24, 1964

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