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Welding Studies on Arc-Cast Molybdenum

Procedures developed for a nuclear application are pertinent to the entire field of refractory and reactive metal welding

BY A. J. MOORHEAD AND G. M. SLAUGHTER

ABSTRACT. Although molybdenum has several metallurgical characteristics that make its fabrication (especially by welding) difficult, it has some important properties that give it good potential for use in the next generation of nuclear reactors or in radioisotope thermoelectric generators. To increase our understanding of the characteristics that have limited the use of molybdenum, a program was undertaken in support of the Molten Salt Breeder Reactor in which very complex prototype components for a chemical processing test stand were fabricated from arc-cast molybdenum by extrusion, machining, welding, and brazing. This paper presents some of the results of the welding development portion of that program. Procedures were developed for cleaning and stress-relieving this material before welding and for making helium-leak-tight welds by both the gas tungsten-arc and electron beam processes. A commercial orbiting-arc weld head was modified so that molybdenum tubing could be field joined outside the space limitations of a glove box. Using a combination of many procedures and processes, several 3.875 in. diam (9.84 cm) cylindrical vessels were successfully fabricated, thus demonstrating the feasibility of constructing complex structures from molybdenum.

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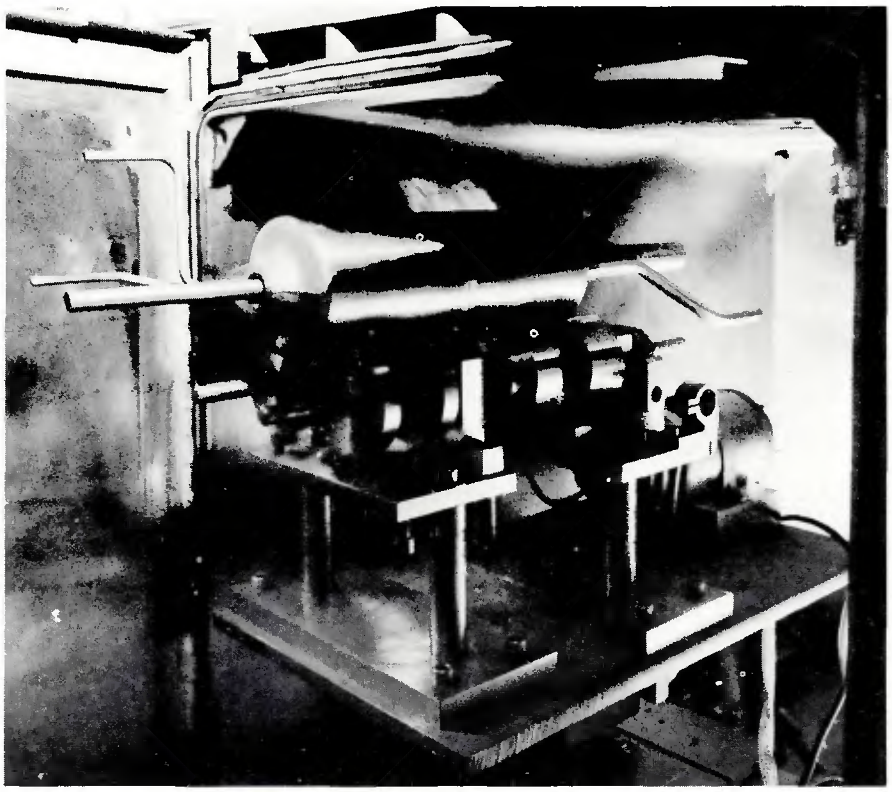

Molybdenum vessel produced by electron beam welding of two back-extruded half sections

Introduction

Because it has a high melting point (4730 F, 2610 C), good high temperature strength, and excellent corrosion resistance (especially to liquid metals), molybdenum is under investigation for use in some of the next generation of nuclear reactors, such as the Molten Salt Breeder Reactor or the Controlled Thermonuclear Reactor, and in radioisotope thermoelectric generators. However, molybdenum does have several shortcomings that make its fabrication, especially into complex welded structures such as shown in the lead photo, quite difficult. The metallurgical characteristics that hinder attempts to weld unalloyed molybdenum are: (1) a ductile-to-brittle transition temperature well above room temperature for recrystallized or cast (weld metal) structures, (2) hot-cracking, (3) porosity formation in weld fusion zones, and (4) abnormal grain growth.

This paper presents the results of a welding study that was undertaken to obtain more definitive information about these characteristics, particularly with regard to the arc-cast material. The ultimate goal was to fabricate a large pumped chemical test stand. However, the technology developed is pertinent to the entire field of refractory and reactive metal welding.

Base Metal

The base metal chosen for this study was "pure" molybdenum produced by an arc-casting process. This material is now being commercially cast with very low impurity element concentrations. For example, typical impurity levels for a wide range of products from ¼ in. diam (6.4 mm) tubes to 4 in. diam (10.2 cm) extrusion billets were, in ppm: C, 10-50; O, 5-10; and N, 1-2. Low impurity levels are important (especially O, N, and C) because they have adverse effects on the ductile-brittle transition temperature and weldability of arc-cast molybdenum (Refs. 1-3). Oxygen is especially detrimental because it forms low melting films with molybdenum, and the presence of these films at grain boundaries can cause hot cracking in the welds. Molybdenum produced by powder metallurgy techniques was not considered for this program because our welds (and those of others) (Ref. 4) in commercially available materials of this type have generally contained large amounts of porosity. The selection of low carbon, low oxygen arc-cast base metal helps to minimize two of the detrimental characteristics in molybdenum welding — hot cracking and porosity formation — both of which are influenced by impurities in the base metal. However, great care must also be taken in subsequent operations to ensure that these elements are not introduced to weldments through surface contamination or by an impure welding atmosphere.

Forms of Material Welded

Several different forms of molybdenum were successfully joined in this program. Half sections to form various 3.875 in. diam (9.84 cm) vessels were fabricated at ORNL by a closed die back-extrusion process (Ref. 5) in lengths up to 12 in. (30.5 cm). Tubing of the following sizes was commercially produced by extrusion, swaging, and drawing:

Outside diameter, in.	mm	Wall thickness, in.	mm
0.250	6.4	0.020	0.51
0.375	9.5	0.025	0.64
0.500	12.7	0.030	0.76
0.875	22.2	0.080	2.0
1.125	28.6	0.060	1.5

Additional components to be welded were machined from either extruded and rolled plate, extruded bar, or upset forged disks. Before welding, all the components were processed as described below.

Preweld Treatments

All parts were cleaned as follows using that portion of a complex cleaning procedure attributed to Ryan by Thompson (Ref. 6).

(1) Degrease with acetone.
(2) Immerse for 5 min in a 150-175 F (65—80 C) solution of 10 wt % sodium hydroxide, 5 wt % potassium permanganate, and 85 wt % distilled water.
(3) Rinse in flowing tap water, brushing to remove smut.
(4) Immerse for 5-10 min in a room-temperature solution of 15 vol % sulfuric acid (95-97% H_2SO_4), 15 vol % hydrochloric acid (37-38% HCl), 70 vol % distilled water plus 6-10 % chromium trioxide (CrO_3).
(5) Rinse with tap water.
(6) Rinse with distilled water.
(7) Dry with hot air.

Although in this study we did not find any correlation (as far as leak-tight welds were concerned) with the length of time between cleaning and welding, in some cases where parts had been excessively handled after the initial cleaning we did take the precaution of repeating steps (1 and 4-7) of the procedure immediately before welding.

After chemical cleaning, all components were given a vacuum stress relief treatment for 60 min at 1600-1650 F (870-900 C) at a pressure of 5×10^{-5} torr or less. The combination of the chemical and thermal treatments left the components with surfaces that were a lighter gray than those in the as-received condition with no noticeable grain boundary attack.

Results of Welding Studies

Tube-to-Header Joint

A typical joint found in many systems that might be constructed of molybdenum is the one required to attach a tube to a larger component such as a vessel. After careful consideration of several joint designs, we decided to emphasize the "corner-flange" geometry. This joint was formed by machining a groove or trepan on the inside of the vessel around a hole, as illustrated in the upper right inset of Fig. 1. The reasoning behind our decision to emphasize this particular joint design is contained in the discussion section.

Procedures were developed for electron beam welding tube-to-header joints in tubes of sizes listed above. The parameters for welding two sizes of tubing are given in Table 1, and an example of these two welds is shown in Fig. 2. The parameters for all of the welds are reported elsewhere (Ref. 5). The electron beam welding machine used was the high voltage, low current type (150 kV, 0.040 A max) with a chamber 36 in. wide × 23 in. deep × 24 in. high (91 × 58 × 61 cm).

In preliminary welds, in which flat plates simulated the vessel ends, we made welds both by the conventional method of rotating the work under the beam and by using a simple system for manually rotating the beam in a circle (up to 5/8 in. diam) (16 mm) on the workpiece. Although we made acceptable welds using both techniques, we had greater difficulties in getting reproducible results with the

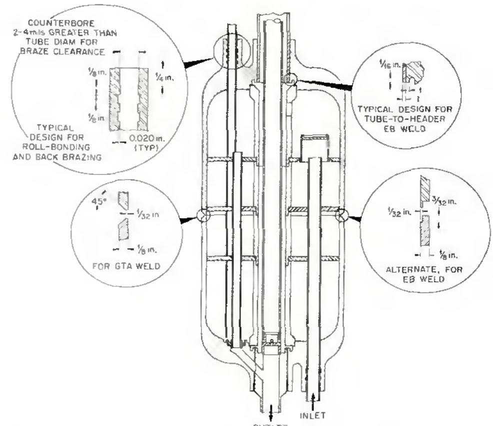

Fig. 1 — Joint designs used in fabricating molybdenum vessels

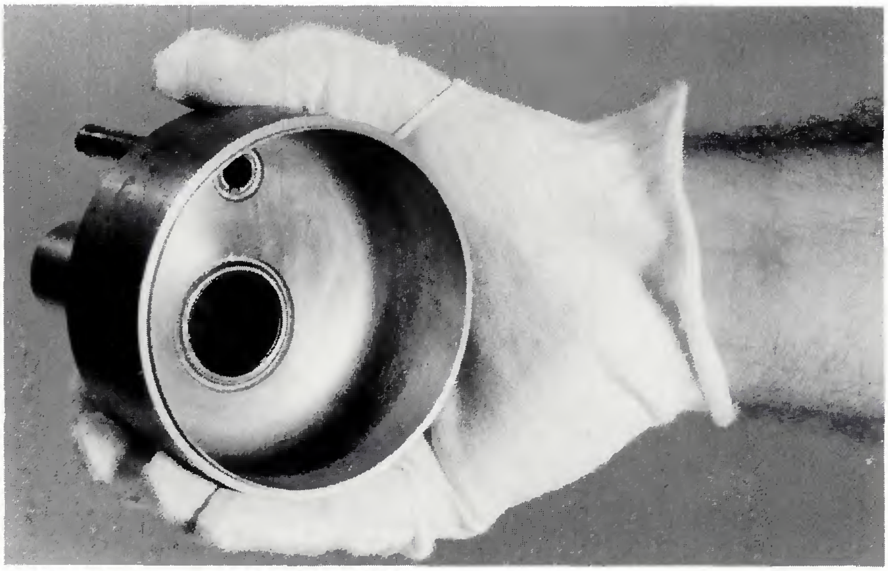

Fig. 2 — Back extruded half section with two tube-to-header welds made by the EB process

Fig. 3 — Closeup of a 0.375 in. diam tube-to-header weld showing the machined groove

beam rotation technique, so all subsequent prototype welds were made by rotating the joint rather than the beam. This latter technique was somewhat complicated in the case of tubes that were located away from the centerline of the vessel. However, this was compensated for by a simple fixture with cross slides for centering the weld under the beam. A closeup of a 0.375 in. diam (9.5 mm) tube-to-header weld is shown in Fig. 3, and a photomicrograph of a helium leak-tight weld joining a 1.125 in. OD × 0.060 in. wall (28.6 × 1.5 mm) tube to a back-extruded molybdenum half section is shown in Fig. 4.

Cylindrical Girth Joints

Both the gas tungsten-arc (GTA) and electron beam (EB) welding processes were investigated for the weld required to join two half sections to form a vessel such as the one in the lead photo. The joint design used for each process is illustrated in the lower left and right insets of Fig. 1. Our prototype back-extruded half sections had a wall thickness of 0.125 in. (3.2 mm).

The GTA welds were made manually in a vacuum purged argon-backfilled glove box. This chamber is 35 in. ID × 56 in. long (89 × 142 cm), but with available extensions the length can be increased to over 12 ft (3.7 m). The chamber was evacuated to a pressure of 5×10^{-5} torr or less before backfilling with argon having minimum purity of 99.997 vol %. Filler metal was 0.060 in. diam (1.5 mm) low carbon, low oxygen welding rod prepared commercially from cast stock. The vessel was tacked in three locations and then manually rotated during welding. The electrode was 2% thoriated tungsten, 0.125 in. diam (3.2 mm). No auxiliary preheating was required for the GTA welds because the high conductivity of molybdenum leads to a rapid distribution of heat through the parts (i.e., preheat) before the fusion temperature is reached at the joint. A photograph of one of these welds (which had a helium leak rate less than 1×10^{-8} atm cm^3 sec^{-1}) and a photomicrograph of a cross section through it are shown in Fig. 5.

Electron beam girth welds were made with the rotary fixture shown in the lead photo. A general requirement for welding with this process, if filler metal is not added, is that the joint must be held tightly together so that coalescence will occur and so that the resulting weld bead will not be underfilled. The harmful effects of an underfilled, concave weld bead are especially pronounced in crack sensitive materials such as molybdenum.

Unfortunately, the complex geometry of the vessel half-sections with their protruding tubes and internal baffle plates made the application of end loading by conventional means quite difficult. To overcome this problem, a technique was developed in which three 0.032 in. diam (0.81 mm) molybdenum pins through the step joint were used to hold the halves in intimate contact during welding. These pins were subsequently fused into the weld.

The parameters for electron beam welding the cylindrical girth joint are given in Table 1. Note that extensive preheating was carried out with the defocused beam before welding to

Table 1 — Parameters for Electron Beam Welding[a] Two Sizes of Molybdenum Tubing to a Back-Extruded Half Section and for Joining Two Cylindrical Half Sections

	Tube-to-header joints		
	0.375 in. OD[b]	1.125 in. OD[c]	Girth joint
Joint type	Corner flange[d]	Corner flange[d]	Step[e]
Joint thickness, in. (mm)	0.050 (1.27)	0.120 (3.05)	0.094 (2.39)
Accelerating potential, kV	120	120	150
Beam current, mA:			
preheat	—	11	12
weld	5	23	25
Number of revolutions:			
preheat	—	6	6[f]
weld	1½	1¼	1¼
Travel speed, ipm (mm/sec)	14 (5.9)	14 (5.9)	32 (14)
Focus, A defocus	0.015	0.015	0.015
Work distance,[g] in. (cm)	6 (15.2)	12 (30.5)	4 (10)

(a) All welds were made with a self-accelerating triode electron gun; manual beam current downslope was used to prevent crater formation.
(b) Tube wall and recess lip thickness each 0.025 in. (0.64 mm).
(c) Tube wall and recess lip thickness each 0.060 in. (1.5 mm).
(d) Illustrated in Fig. 1, upper right inset.
(e) Illustrated in Fig. 1, lower right inset.
(f) Two preheat passes each on the left and right sides of the joint and 1 in. (2.5 cm) away, and then two passes on the joint itself.
(g) Distance from gun heat shield to work surface.

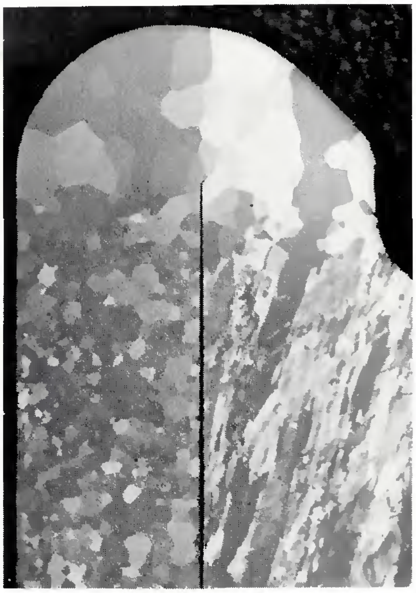

Fig. 4 — Cross section through a weld joining a 1.125 in. OD Tube to a vessel half section. Etchant: 20 vol % H_2O_2—10 vol % H_2SO_4 (96% H_2SO_4) — 70 vol % H_2O. X29, reduced 49%

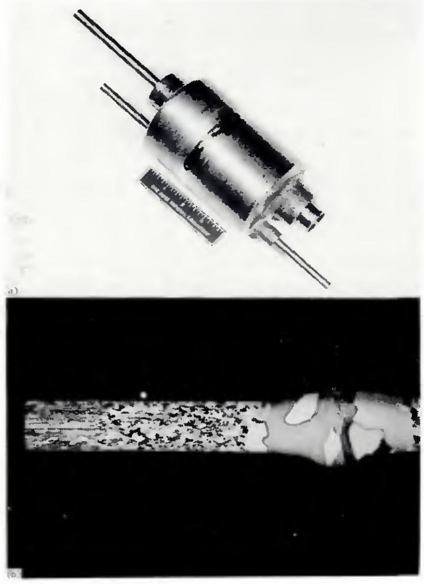

Fig. 5 — (a) Back-extruded molybdenum half sections joined by a manual gas tungsten-arc weld. (b) Cross section through this weld. Etchant: 20 vol % H_2O_2—10 vol % H_2SO_4 (96% H_2SO_4) — 70 vol % H_2O. X6, reduced 65 %

minimize the possibility of cracking. A photomicrograph of an early electron beam girth weld, in which penetration through the step was not achieved, is shown in Fig. 6.

Tube-to-Tube Butt Joint

Tube-to-tube butt welds, in five different sizes of molybdenum tubing were successfully made in an argon filled glove box by the gas tungsten-arc process, both manually and with an automatic orbiting-arc technique. Using either technique we were readily able to produce welds that were helium leak-tight and had little or no porosity as shown by radiographic and metallographic examination.

However, the orbiting-arc technique became of particular interest after we found that our welders had considerable difficulty in manually making butt welds in thin walled molybdenum tubing without excessive root reinforcement and misalignment. These problems were compounded by the necessity of making the welds in a glove box, which hampers the senses of sight and touch.

Three commercial orbiting-arc weld heads were evaluated, including the head shown in Fig. 7. In this unit, the electrode is carried around the joint (which remains stationary) by a copper ring, which also holds a set of boron nitride gas cups. Inert gas is fed through the handle to provide an atmosphere suitable for welding in the cavity formed by the cups and the body housing. The electrode carrier is rotated by a small dc motor (with tachometer feedback) in the handle. The tubes are held together and in alignment by a pair of clamshell clamps with removable inserts to adapt to the various tubing sizes.

To further reduce any stresses applied to the weld (such as by tube misalignment) we used several sizes of yoke-type fixtures outside the weld head clamps, such as the one shown in Fig. 8. Devices of this type are invaluable because molybdenum weldments are highly susceptible to cracking under the influence of even small stresses. Welds were made by use of a magnetic amplifier power supply with capabilities for programming variations in the welding current and in the rotational speed of the orbiting electrode carrier.

Figure 8 also shows a feature that we added to the orbiting-arc head to permit us to make molybdenum welds outside the confinement of a glove box — molded rubber sleeves to seal tightly around the head and the tubes. Without these sleeves, we were unable to make crack-free welds outside the inert atmosphere chamber (i.e. in the field) whereas with the sleeves we were able to make helium leak-tight welds in the 0.250, 0.375, and 0.500 in. diam (6.4, 9.5, and 12.7 mm) tubes. However, the results were not as reproducible with the "field" welding technique as they were with the orbiting-arc unit inside the glove box. Therefore, field welding should not be used unless dictated by size limitations of the glove box.

Weld inserts, such as the one shown in Fig. 9 between two tees, were used to control root reinforcement and joint misalignment when orbiting-arc welds were made in these tubing sizes. Because it protrudes slightly above the tube surface, it also provides a small amount of filler metal for the joint, thus producing convex weld beads, which we have found to be less prone to crack than concave beads.

The procedure for butt welding of 0.250 in. OD × 0.020 in. wall (6.4 × 0.51 mm) molybdenum tubing is given in Table 2. The important features of the welding cycle are the relatively long portion at a lower "initial current," which preheats the joint without fusion and the gradual increase in rotational speed to minimize "weld bead widening," which occurs in tube welds as the overlap portion is approached. A cross section through a typical tube-to-tube weld made with the orbiting-arc weld head is shown in Fig. 10.

Discussion of Results

All the components that were welded in this program were given

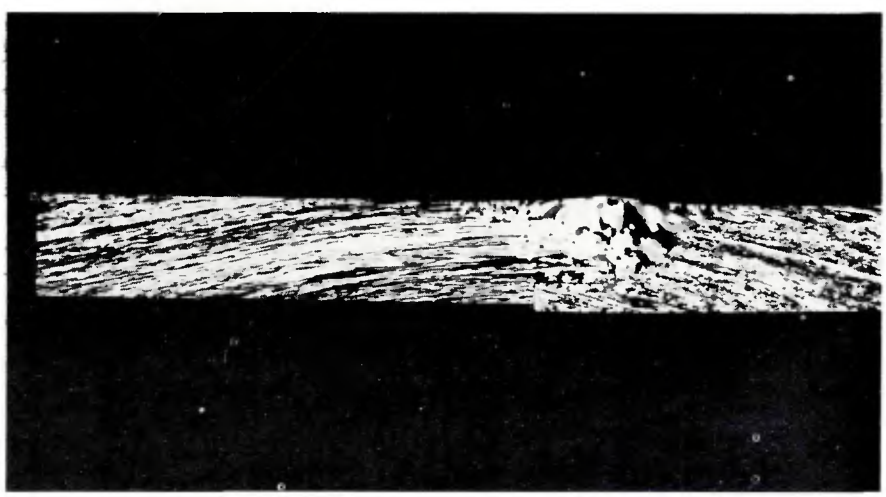

Fig. 6 — Cross section through electron beam girth weld shown in the lead photo. Etchant: 20 vol % H_2O_2 — 10 vol % H_2SO_4 (96% H_2SO_4) — 70 vol % H_2O. X6, reduced 25%

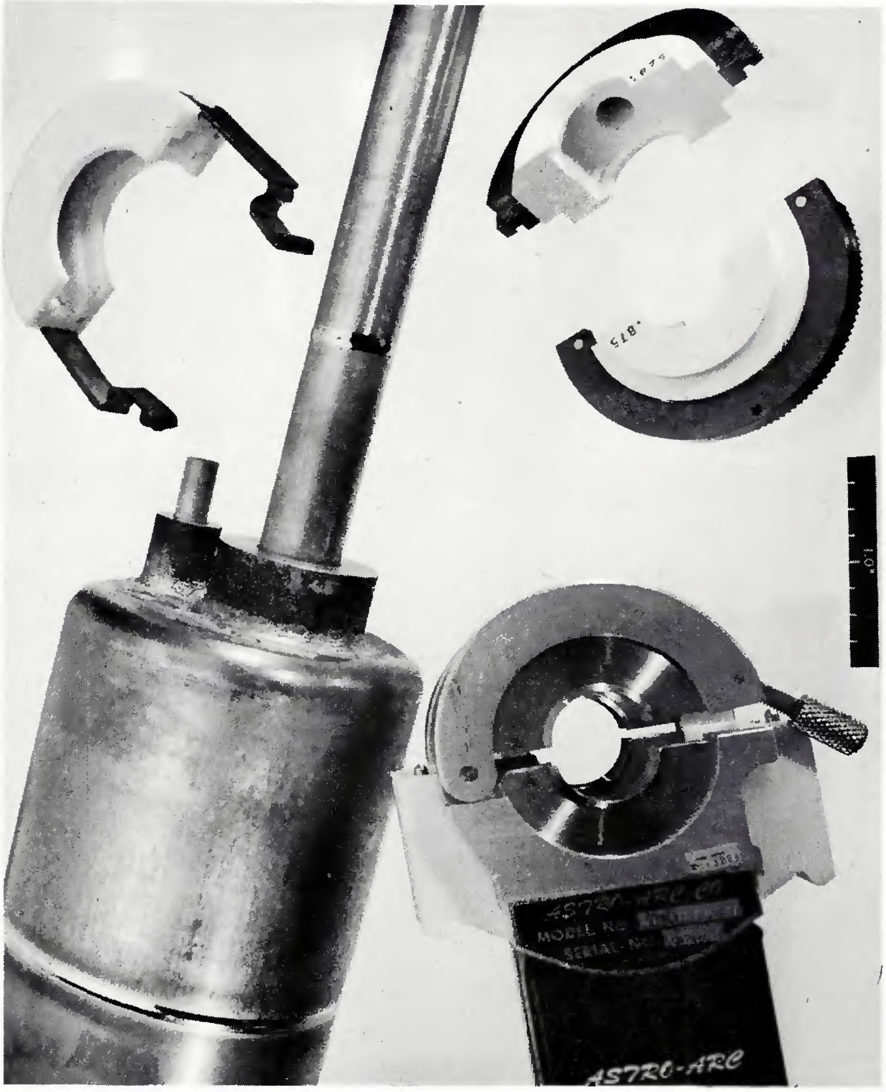

Fig. 7 — Orbiting-arc weld head and 0.875 in. diam tube-to-tube weld

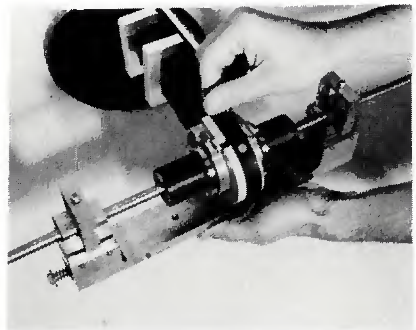

Fig. 8 — Orbiting-arc weld head with attached rubber sleeves for welding molybdenum tubing outside a glove box and with yoke-type fixture

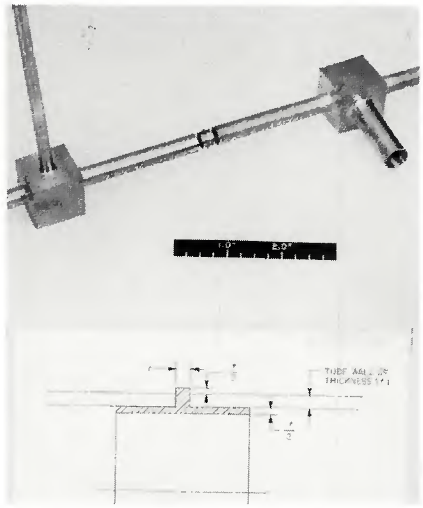

Fig. 9 — (Top) Molybdenum tees with attached tubes and insert in position before assembly for welding. (Bottom) Design of typical insert for 0.250, 0.275, and 0.500 in. OD tube-to-tube welds

chemical cleaning and stress relief treatments before welding, as described previously. Chemical cleaning ensured that the initially low impurity levels of the base metal were not increased by surface contamination. Several different cleaning procedures were evaluated in the preliminary phase of this study, and we found little difference as far as their relationship to weld cracking was concerned. However, it was beyond the scope of this program to carry out a detailed, extensive investigation into the effects of various cleaning techniques on weldability and mechanical properties. Therefore, we selected the procedure described because it resulted in satisfactory weldments and was capable of removing more visible surface contamination than the other techniques evaluated.

Our preliminary efforts showed that a stress relief heat treatment had a beneficial effect on the weldability of molybdenum. For example, one half of a sample length of 0.375 in. OD × 0.025 in. wall (9.5 × 0.64 mm) tubing was cleaned as previously described and then stress relieved in vacuum for 60 min at 1600 F (870 C). The other half of the tube was chemically cleaned only. In a series of bead-on-tube field welds on these two pieces using an orbiting-arc weld head, we found that the welds on the cleaned and stress-relieved tube were crack-free, while those on the cleaned only tube had extensive centerline cracking. Similar results occurred on other sizes of tubing and on back-extruded half sections. We tried several other stress relief temperatures to determine the lowest temperature that would produce the desired improvement in weldability and at the same time have the least effect on base metal ductility due to recrystallization. A temperature of 1600-1650 F (870-900 C) is apparently optimum from a weldability standpoint and has little effect on the base metal microstructure of the various components, as illustrated by Fig. 11.

Several different joint designs, each with advantages and disadvan-

Table 2 — Parameters for Welding Butt Joint Between 1/4 in. OD × 0.020 in. Wall Molybdenum Tubing Using An Orbiting-Arc Weld Head Inside An Argon Filled Glove Box

Joint type — square butt with insert[(a)]
Joint thickness — 0.020 in.[(b)] (0.64 mm)
Electrode:
  EW-Th2 — 0.062 in. diam (1.57 mm)
  30 deg conical tip
Arc length — 0.032 in. (0.81 mm)
Gas flow through head — 5 cfh Ar
Welding conditions:
  Initial:
    current — 30 A
    time — 6 sec
  Weld:
    current — 56 A
    time — 10 sec[(c)]
  Finish:
    current — 1 A
    Time — 5 sec[(d)]
Travel speed:
  Initial — 7.8 ipm (3.3 mm/sec)
  Weld — 9.4 ipm (4 mm/sec)
  Finish — 9.4 ipm (4 mm/sec)

(a) Insert design shown in Fig. 9(b).
(b) Plus slight penetration into insert.
(c) Includes 3 sec upslope from initial to weld current.
(d) Downslope time from weld current to finish current.

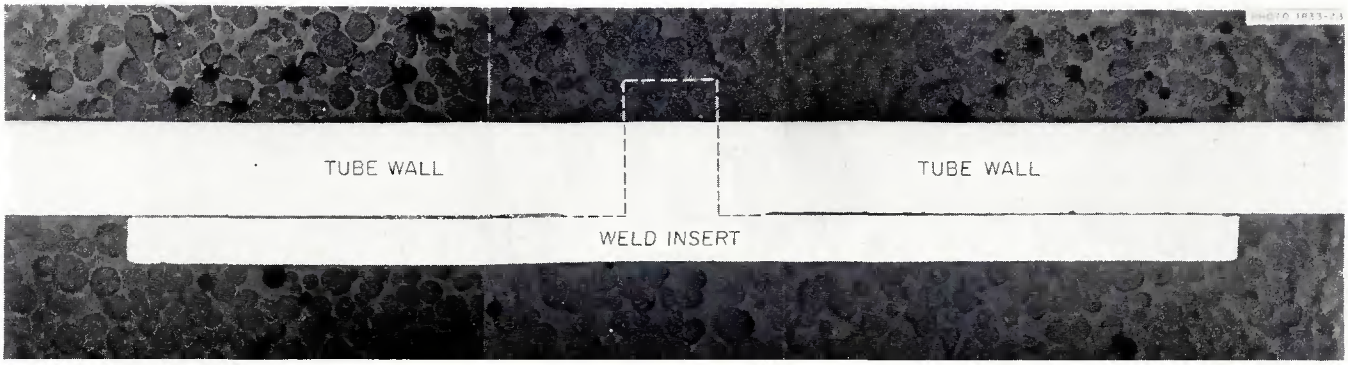

Fig. 10 — Composite of photomicrographs of a cross section through an orbiting-arc field weld joining two 0.250 in. OD × 0.020 in. wall tubes. Etchant: 50 vol % NH_4OH—50 Vol % H_2O X50, reduced 30%

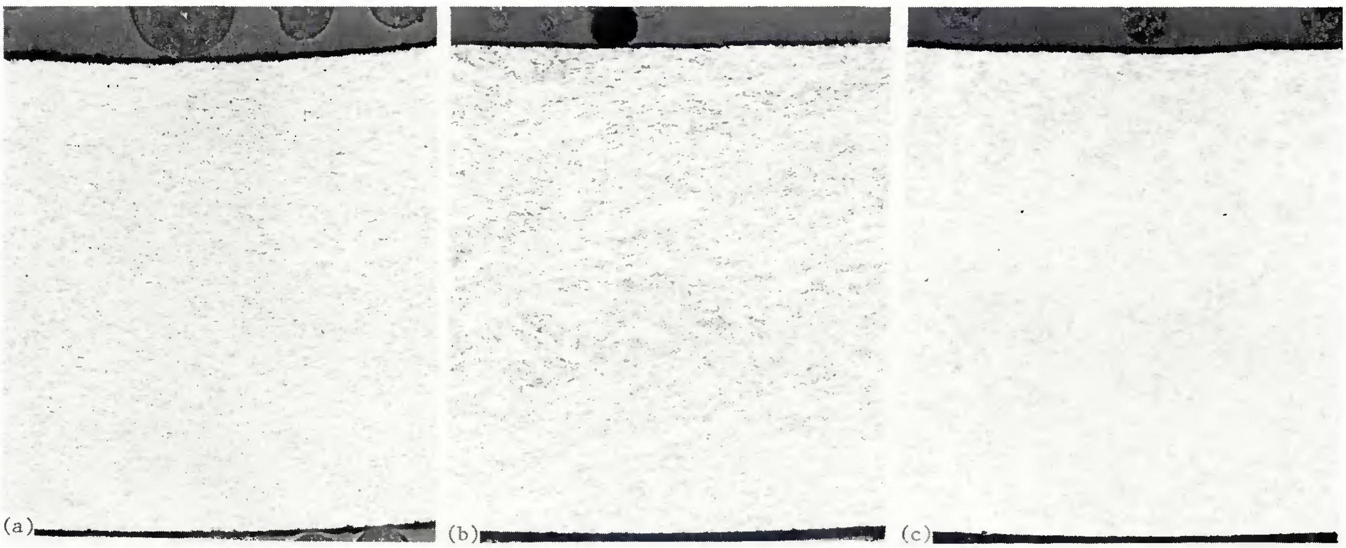

Fig. 11 — Effect of heat treatment (in vacuum) on the microstructure and hardness of 0.500 in. OD× 0.030 in. wall molybdenum tubing. Etchant: 50 vol % H_2O_2—50 vol % NH_4OH. X100, reduced 24%. (a) As-received, diamond pyramid hardness (DPH)241. (b) 60 min at 900 C, DPH 236. (c) 60 min at 925 C, DPH 206. Note that there is partial recrystallization (resulting in a decrease in hardness) in the sample heat treated at 925 C

tages, were considered for applications in which a tube had to be attached to a larger component, such as a vessel. A square groove butt joint is most desirable from the standpoint of economy and inspectability. However, it was not included in this program because some conceptual designs that were under consideration indicated that the close proximity of several tubes attached to a given vessel would make access for this type of joint (especially in automatic welding) quite difficult. Socket joints are somewhat more amenable for welding (manually) than are square groove joints, but the welds are difficult to inspect radiographically and have undesirable mechanical properties. Both the square groove and socket types also would be very prone to accidental damage during subsequent fabrication steps in a multiple tube assembly sequence, since in either case the geometry is such that the weldment is not supported mechanically.

The "corner-flange" joint, formed by a groove on the inside of the vessel, is favorable in that this joint provides adequate heat balance between components of unequal mass, is readily weldable, and results in a minimum amount of restraint. In addition, this joint has built in mechanical support for the weld. In our opinion these favorable factors outweigh the disadvantages of being difficult to inspect (other than by dye penetrant or leak detection) and being somewhat inaccessible for machining or welding. A critical feature is the tight fit required between the hole and the tube, and this is especially important for welds that have little ductility at room temperature. We found that a Class LC5 fit (Ref. 7) was tight enough to ensure good weldability, but not so tight that it caused galling during assembly of the molybdenum components before welding.

Two competitive factors played important roles in the procedures developed for welding complex molybdenum structures. Molybdenum tends to undergo abnormal grain growth during welding, and large grained material, in general, has a lower fracture stress than fine grained material (Ref. 8). Impurity atoms tend to concentrate at grain boundaries, and the reduced grain boundary surface area in large grained structures leads to a concentration of these impurities, thus intensifying problems of low ductility and hot cracking. Therefore, on the one hand it would seem that welds with steep thermal gradients (i.e., those made at high energy densities and low total energy input, such as occurs with a sharply focused electron beam) have fine grained structures that would be ideal.

However, the concentration of residual stresses over a smaller area in this type of weld often results in failure in brittle materials such as molybdenum. We found this to be particularly true for more highly restrained welds in large components such as the 0.875 and 1.125 in. diam (22.2 and 28.6 mm) tube-to-header welds, and the 3.875 in. diam (9.84 cm) cylindrical girth welds. Therefore, these welds were made only after the joint area was preheated with the defocused electron beam, and the beam was not sharply focused during welding. These welds had relatively large grains in the fusion zones, but they were not as large as those in equivalent GTA welds, as is evident in comparing Figs. 5(b) and 6.

Although cylindrical girth welds made by both GTA and EB welding were satisfactory from the standpoint of penetrant inspection and helium leak testing, we used only the EB process in our later prototype welds. This method not only produced a finer grained microstructure, but also the self aligning feature of the step joint design made this assembly much easier to set up and weld, and resulted in a better aligned final part. The GTA process was not applicable to these components because of the alignment requirements for the tightly fitting internal tubes, baffles, and baffle supports.

Summary and Conclusions

Based on our developmental work and the fabrication of relatively large, complex structures of this material, we can summarize the welding behavior of commercially available arc-cast molybdenum as follows:

1. Helium leak-tight welds can be made in various forms of this material by both the gas tungsten-arc and electron beam welding processes in inert gas and vacuum chambers, respectively.

2. Leak-tight welds can be made in the "field" on small diameter molybdenum tubing by use of a commercial orbiting-arc weld head and rubber sleeves to provide a locally pure inert gas environment. However, the quality of these welds was not as reproducible as those made with the same device in an inert atmosphere chamber.

3. Two procedures were adequate for preweld chemical cleaning of molybdenum components, and there was no correlation between weld quality and the time elapsed after cleaning.

4. Vacuum stress relief for 60 min at 1600-1650 F (870-900 C) reduced the likelihood of weld cracking.

5. Preheating (with the defocused beam) was necessary to avoid weld cracking when 3.875 in. diam vessel half sections were joined with an electron-beam girth weld.

6. A tight fit between mating parts was a critical parameter in attaching molybdenum tubes to vessels with a "corner-flange" weld joint geometry.

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