Wettability of Brazing Filler Metals on Molybdenum and TZM

**ABSTRACT.** Vacuum brazing studies have been performed on molybdenum and the alloy 0.5Ti-0.08Zr-Mo (TZM). Wettability tests have been conducted for nineteen brazing filler metals on molybdenum and thirty-two brazing filler metals on TZM over a wide range of temperatures. A wetting index, which is a function of contact angle and braze contact area, was determined for each brazing filler metal at each brazing temperature. The nature and extent of interaction between the brazing filler metals and the base metals were analyzed by conventional metallography, scanning electron microscopy, and electron microprobe analysis. A comparison is made between the behavior of brazing filler metals on molybdenum and TZM; the brazing filler metals consistently exhibited less wettability on TZM than on molybdenum. The lower wettability is believed to be due to a small amount of titanium in the surface oxide on TZM. Cracking was observed in the base metal under some of the high-temperature braze deposits. The cracking is shown to arise from liquid-metal embrittlement from nickel in the high-temperature braze alloys.

**Introduction**

Because of its excellent high-temperature mechanical properties and compatibility with certain environments, molybdenum or one of its alloys is often considered for various uses in the space and nuclear industry (Ref. 1). Molybdenum has a high modulus of elasticity and is about as strong as steel. It is half as dense as tungsten and has good thermal conductivity but poor high-temperature oxidation resistance (Ref. 2). The presence of minute quantities of oxygen, nitrogen and carbon lowers the ductility of molybdenum, and recrystallized molybdenum has reduced strength and ductility. Therefore, its use should be avoided. The addition of titanium and zirconium to molybdenum raises its recrystallization temperature, allowing use of higher-temperature brazing filler metals. Unalloyed molybdenum recrystallizes at about 1180°C (2150°F), while the Mo-Ti-Zr alloy recrystallizes at about 1460°C (2700°F).

Although some information on the brazing of molybdenum (Ref. 1) and TZM (Ref. 3) is available in the literature, a comprehensive, systematic study of the wettability of brazing filler metals on these base metals has not been reported. This study was conducted to provide information on a wide variety of commercial silver-based and gold-based filler metals.

A liquid wets a solid when the interfacial tension between the liquid and the solid is such that the contact angle is between zero and 90 deg (Ref. 4). Wettability is a measure of the degree of wetting. Since high wettability means that the ther-
mocapillary attraction that fills the braze joint is strong, wettability is an important component of braze performance. To condense the data, the wettability index (WI) as developed by Feduska (Ref. 5) was used. The WI is defined as the area covered by the brazing metal filler metal times the cosine of the contact angle. Therefore, the higher the WI, the better the brazing filler metal wets the base metal. It should be emphasized that the WI, as defined here, is an empirical number dependent on the volume of filler metal used.

Most metals will crack during brazing if they are stressed while in contact with various other molten metals. The cracking occurs virtually instantaneously during the brazing operation. The molten filler metal follows and fills in the cracks, making the cracks easily visible. This type of failure is known as liquid-metal embrittlement (LME). The LME is generally believed to be specific, i.e., a particular solid will be significantly embrittled only by certain liquid metals (Refs. 6, 7).

**Experimental Procedure**

Wettability coupons, 3.17 × 50.8 × 50.8 mm (0.125 × 2 × 2 in.) were fabricated from commercially pure molybdenum and TZM plate stock. A 1.58-mm (0.063-in.) diameter by 12.2-mm (0.5-in.) deep thermocouple hole was drilled at the edge of each specimen. The coupons were thoroughly degreased with acetone and polished on 400-grit metallographic paper and degreased again.

Brazing filler metal samples were also thoroughly degreased in acetone. A list of the brazing filler metals, their compositions and their liquidus and solidus temperatures are given in Table 1. All brazing filler metals were in wire form of 0.51-mm (0.020-in.) or 0.63-mm (0.025-in.) diameter. The wire was formed into coils of approximately 3.17-mm (0.125-in.) OD of varying weights so that the total volume of braze metal used was the same for each test (7.5 mm³/0.0005 in.³).

The coupons, with the brazing filler metal coils placed on them, were placed in a standard, cold wall vacuum furnace. Chromel-alumel thermocouples were inserted into the thermocouple hole in each coupon. The furnace was evacuated to 5 × 10⁻⁵ torr or less. The coupons were heated to the selected brazing temperatures and held for five minutes. The temperatures were selected based on preliminary studies that determined the initial melt and flow points. After heating, the coupons were cooled to at least 10°C (18°F) below the brazing filler metal solidus temperature in static argon gas and then cooled rapidly in flowing argon gas to 100°C (212°F) before removing them from the furnace. The area of brazing filler metal flow and the contact angle were then measured on each specimen, and the wettability index calculated.

Selected specimens were cross-sectioned for metallography and examined in optical and electron microscopes at several magnifications. Some specimens were chosen because they are commonly used brazing filler metals. Others were selected for microstructural characterization because of the level of wettability exhibited on them by the brazing filler metals used, or because the braze was an alloy, which is rarely used. Elemental maps of molybdenum and the major alloying elements in the filler metals were obtained across the cross-sections of selected specimens by x-ray microprobe analysis. This type of map permits qualitative analysis of the composition of phases present.

Surface analysis was employed to investigate the origin of the wettability difference between TZM and molybdenum, and to determine the origin of cracking observed in the base metal under some of the braze deposits. The technique used was x-ray photoelectron spectroscopy, also known as ESCA. In this technique, the sample is irradiated with a monochromatic Alkα x-ray beam in a vacuum chamber typically at 10⁻⁹ to 10⁻¹⁰ torr. The incident x-rays provide sufficient energy to eject some electrons from their orbits in atoms on or near the material surface. Since the ejected electrons are of low energy, only electrons originating from atoms in the first few atomic layers on the surface have a significant probability of escaping from the sample. The set of binding energies of electrons to the nucleus is unique for each element, so an energy analysis of the electrons ejected from a sample provides an identification of the element or elements from which they originated on or very near the surface.

To investigate the origin of the wettability difference between TZM and molybdenum, coupons of each with the surface preparation described above were run simultaneously through a 1000°C wettability cycle as described previously, but with no brazing filler metal present. After removal from the vacuum furnace, the coupons were immediately wrapped in clean aluminum foil and transported as quickly as practicable to the ESCA instrument, in order to minimize carbon contamination of the surfaces. This effort was quite successful; the samples had less than a monolayer of carbon contamination. The surfaces of the TZM and molybdenum samples were analyzed

Table 1—Alloys Examined

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(a) Compositions are given in wt%.
to determine any differences in composition that might be responsible for the wettability difference.

A similar approach was used to investigate the origin of cracking under some of the high-temperature braze deposits. Braze coupons were prepared using Nioro and Palniro 7 braze alloys (which had been shown previously to be associated with subsurface cracking) and pure gold. A small square with the top surface covered with braze deposit was then cut from the braze coupon with a hacksaw. This sectioning technique minimizes sample contamination since no cutting fluid or coolant is required, and the cutting chips are large, easily identified and easily removed. The samples either fell apart or were easily separated after sectioning, exposing the crack surfaces. The samples were promptly inserted into the ESCA instrument. Again, contamination of the fracture surfaces was analyzed to attempt to determine what was responsible for the cracking.

Results and Discussion

Wettability Indices

The wettability indices (WI) for each brazing metal filler metal at each temperature on molybdenum and on TZM are provided in the Appendices A and B, respectively. Wettability indices greater than 0.05 are indicative of good performance during brazing, and WI greater than 0.10 are indicative of excellent performance during brazing (Ref. 8).

Plots of WI versus temperature for selected alloys are given in Figs. 1-4. Figures 1 and 2 show the wettability of a series of Ag-Cu-Pd alloys, with the palladium content varying from 0 to 25%. Two effects are apparent in these plots: 1) the WI tends to increase with increasing temperature, and 2) the WI tends to increase with increasing palladium content. A correlation between wettability indices and temperature was expected because surface tension is a function of temperature. A correlation between palladium content and WI on molybdenum has previously been reported (Ref. 9). Figures 3 and 4 show the wettability of a series of Au-Ni-Pd alloys on molybdenum and TZM respectively, with the palladium content varying from 0 to 34%. Once again, wettability appears to increase with increasing palladium content and increasing temperature.

Origin of the Wettability Difference between Molybdenum and TZM

The only elements detected on the surface of the blank molybdenum braze coupon were molybdenum, carbon and oxygen. The same elements, with the addition of a small amount of titanium, were found on the TZM coupon (zirconium could not be detected on the TZM surface). Depth profiles on both coupons demonstrated that the carbon layer was less than one monolayer thick and the same thickness on both coupons. The carbon almost certainly arose from contamination during transport from the brazing furnace to the ESCA. The oxide thickness on both coupons was also the same. Thus, the only difference between the molybdenum and TZM surfaces is the presence of a small amount of titanium in the surface oxide on TZM. We propose that the titanium alters the wetting characteristics of the oxide and is responsible for the wettability difference between molybdenum and TZM.

Metallography

Optical Metallography: Optical metallography of cross-sections showing deposit morphology dramatically illustrates differences in wettability indices. For example, in Figs. 5-7, three deposit morphologies are shown. The deposit in Fig. 5 is Braze 580 on TZM heated to 800°C (1472°F). This deposit has a somewhat spherical morphology and a low wettability index of 0.007. The deposit in Fig. 6 is also Braze 580 on TZM, but in this
The morphology is typical of deposits metal interactions. These features include interesting features of the brazing filler with good wettability indices. The deposit in Fig. 4 has a wettability index of 0.07. This deposit is nearly flat, with a temperature is illustrated by the less spherical, more oval deposit shown in Fig. 6. Neither of these deposits exhibited acceptable wettability. The deposit in Fig. 7 is Ticsoil on TZM heated to 950°C (1742°F). This deposit is nearly flat, with a corresponding wettability index of 0.07. This morphology is typical of deposits with good wettability indices.

Scanning Electron Microscopy: Scanning electron microscopy revealed several interesting features of the brazing filler metals, base metals and filler metal/base metal interactions. These features include the occurrence of cracking and the formation of intermetallic compounds. The formation of brittle intermetallics are of engineering importance but will not be discussed here as no work was done to determine the brittleness of the observed intermetallics. Only the occurrence of cracking in the base metal of several coupons will be discussed herein.

Table 2 contains information concerning which coupons exhibited cracking. All of the braze alloys involved in the occurrence of cracking contained nickel.

Origin of Cracking

An ESCA spectrum from the crack surface under the Nioro braze deposit on TZM is shown in Fig. 8. The crack surface is covered with a thin layer of braze alloy — gold and nickel. The crack surface under the Palino 7 braze deposit on both molybdenum and TZM was also covered with a layer of braze alloy, i.e., gold, nickel and palladium. Micrographs of polished cross-sections through the cracked regions of the molybdenum and TZM coupons are shown in Figs. 9 and 10, respectively. Depth profiles demonstrated that the braze layer was only a few atomic layers thick, roughly 50 Å.

Coverage of the crack surface by braze metal indicates that cracking arises from liquid-metal embrittlement. Braze metal penetrates grain boundaries normal to the surface (as shown in Fig. 11), probably by a simple corrosion mechanism. About 100 microns (0.004 in.) below the surface, a suitable stress state — and perhaps a plane of relative weakness originating from casting and rolling the plate — exists and the liquid-metal embrittlement crack is formed.

It is tempting to ascribe the liquid-metal embrittlement to the gold, but no cracking was found under the pure gold braze deposits. The embrittlement is therefore likely due to the nickel dissolved in the gold-base braze, possibly assisted by the palladium in the Palino 7 braze. Nickel is also found in several of the silver-base brazes studied. No cracking was found under any of these braze deposits. The nickel content of these braze alloys is much lower (Table 1) than the gold-base braze alloys, and they wet the surface much more poorly than the gold-nickel alloys. Thus, the lack of cracking under the nickel-containing silver-base brazes is probably due to the low-nickel content, the lack of grain boundary penetration to transport the braze alloy below the surface, or both.

Finally, it should be noted that the stresses at 1000°C (1832°F) in a small, unrestrained coupon heated in a resistance vacuum furnace must be very low. It is quite remarkable that liquid-metal embrittlement would occur under these conditions. Thus, molybdenum and TZM must be extraordinarily sensitive to liquid-metal embrittlement by liquid brazes con-
Fig. 5 – Morphology of deposit of Braze 580 on TZM coupon heated to 800°C

Fig. 6 – Morphology of deposit of Braze 580 on TZM coupon heated to 875°C

Fig. 7 – Morphology of deposit of Ticusil on TZM coupon heated to 950°C

Fig. 8 – ESCA spectrum from the cracked surface under the Nioro braze deposit on TZM

Fig. 9 – Scanning electron micrograph of cracked region of molybdenum coupon

Fig. 10 – Scanning electron micrograph of cracked region of TZM coupon
taining nickel. This fact should be considered in braze alloy selection and fix-
turing so that external stresses applied to the joint during brazing are as low as possible.

Conclusions

1) Brazing filler metals generally wet molybdenum significantly better than they wet TZM.

2) The decrease in wettability of the brazing filler metals on TZM versus molybdenum is believed to be due to the presence of titanium in the oxide on the surface of TZM.

3) Some nickel-containing brazing filler metals have been shown to embrittle both molybdenum and TZM. Liquid-metal embrittlement is believed to be the mechanism by which this embrittlement occurs.

Appendix A

Wettability Index for Commercial Brazing Alloys on TZM

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Acknowledgment

This work was supported by the Department of Energy, Albuquerque Operations Office. Its support is gratefully acknowledged.
### Appendix B

#### Wettability Index for Commercial Brazing Alloys on Molybdenum

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**WRC Bulletin 336
September 1988**

**Interpretive Report on Dynamic Analysis of Pressure Components—Fourth Edition**

This fourth edition represents a major revision of WRC Bulletin 303 issued in 1985. It retains the three sections on pressure transients, fluid structure interaction and seismic analysis. Significant revisions were made to make them current. A new section has been included on Dynamic Stress Criteria which emphasizes the importance of this technology. A new section has also been included on Dynamic Restraints that primarily addresses snubbers, but also discusses alternatives to snubbers, such as limit stop devices and flexible steel plate energy absorbers.

Publication of this report was sponsored by the Subcommittee on Dynamic Analysis of Pressure Components of the Pressure Vessel Research Committee of the Welding Research Council. The price of WRC Bulletin 336 is $20.00 per copy, plus $5.00 for postage and handling. Orders should be sent with payment to the Welding Research Council, Suite 1301, 345 E. 47th St., New York, NY 10017.

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**WRC Bulletin 339
December 1988**

**Development of Tightness Test Procedures for Gaskets in Elevated Temperature Service**

By A. Bazergui and L. Marchand

In this report, different elevated temperature gasket tightness test procedures are compared. A two-tier test approach, involving aging of the preloaded gasket in a kiln followed by a short duration tightness test was evaluated. The procedures were evaluated using spiral-wound gaskets with two different fillers: a mica-graphite filler and an asbestos filler.

Publication of this report was sponsored by the Subcommittee on Bolted Flanged Connections of the Pressure Vessel Research Committee of the Welding Research Council. The price of WRC Bulletin 339 is $16.00 per copy, plus $5.00 for postage and handling. Orders should be sent with payment to the Welding Research Council, 345 E. 47th St., Suite 1301, New York, NY 10017.