Properties of TZM and Nuclear Behavior of TZM Brazements

Various properties of TZM and of Zr-5Be and Au-18Ni brazing filler metals are determined for brazing TZM to Zr and to steel

BY A. SEMENIUK AND G. R. BRADY

ABSTRACT. On the basis of its low rate of hydrogen permeation, TZM (0.5Ti-0.08Zr-Mo) was considered for incorporation as a hydrogen barrier in transition joints between steel and zirconium alloys in 400 C organic-cooled nuclear reactors. To aid with the joint design an investigation was conducted to determine the following properties of TZM: (1) tensile and yield strength at 20 and 400 C, (2) impact strength from 20 to 500 C, (3) coefficient of thermal expansion, (4) corrosion behavior in organic coolants (HB-40) and CO_2 atmospheres, (5) hydrogen permeation rate.

Since brazing was selected for joining TZM to either steel or zirconium alloys, tests were conducted to select suitable brazing filler metals. Initially, 16 filler metals were screened using wettability tests. Filler metals having sufficient wettability were then used to fabricate corrosion specimens for testing in organic coolant, and in wet and dry CO_2. Only two alloys, Zr-5Be for joining TZM to Zr, and Au-18Ni for joining TZM to steel, showed promise for further testing. Corrosion test results for Zr-5Be are presented graphically for various material and environment combinations. Corrosion of Au-18Ni was negligible in the organic coolant and in the CO_2 atmospheres.

The tensile strength of butt-brazed specimens of TZM/Zr and TZM/steel were compared in the unirradiated and irradiated conditions at ambient temperature and at 400 C. Results show that Zr-5Be was not damaged by irradiation but Au-18Ni was severely damaged and would require shielding even in moderate irradiation fields.

On the basis of the various small specimen tests it was concluded that TZM could be used as a hydrogen barrier in transition joints, however further research and development is necessary on prototype joints before they are accepted for nuclear applications.

The authors are with the Whiteshell Nuclear Research Establishment, Atomic Energy of Canada, Limited, Pinawa, Manitoba, Canada.

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Introduction

In CANDU (CANada Deuterium Uranium) reactors, the in-core pressure tubes are made from zirconium alloys and the out-of-core piping from steel. One method of forming a leak-tight connection between these two metallurgically incompatible materials is to use rolled joints. The disadvantage of rolled joints is that steel is in direct contact with the zirconium, permitting hydrogen to pass directly through the steel and embrittle the zirconium. In pressurized-heavy-water (PHW) CANDU reactors, the operating temperature of ~300 C is sufficiently low to limit the hydriding rate to an acceptable level. However, for an organic cooled reactor (OCR) with an operating temperature of ~400 C the zirconium in the rolled joints of the design in use at the time this investigation started, would hydride at a rate which would limit their life to about five years. Economically, it is desirable to have a joint with a 20 to 30 year life. (Methods have since been devised to protect rolled joints from hydriding and increase their life significantly).

A program was initiated to develop and test the feasibility of using a hydrogen barrier between the carbon steel and zirconium, thus eliminating a direct steel to zirconium contact. The type of joint considered would have a hydrogen barrier of TZM* incorporated into areas where steel is in direct contact with the zirconium. The barrier ring would be brazed for leak tightness and an outer collar assembled to transmit the axial load between the pressure tube and the steel piping. Development of the brazed transition joints for use in an OCR is documented in a report by Semeniuk and Brady (Ref. 1).

Before a transition joint with a TZM hydrogen barrier ring could be designed, it was necessary to have a knowledge of the various properties of TZM. Since some of the necessary information could not be located in the literature, a program was initiated

**Alloy composed of 0.5Ti-0.08Zr-Mo expressed in percent by weight.*

at the Whiteshell Nuclear Research Establishment to obtain it. The results of this preliminary investigation are presented.

Test Results for TZM

Material Description

Several types and grades of molybdenum alloys are available commercially. TZM wrought bar was selected as the most suitable material on the basis of its superior high temperature tensile strength and a high recrystallization temperature (between 1400 and 1600 C). A high recrystallization temperature is particularly important as this eliminates the possibility of recrystallization during braze welding causing a loss in ductility and strength.

Test specimens were fabricated from 4.125 in. (105 mm) solid bar stock, which was stress relieved for 2½ h at 1315 C. The nominal chemical composition of the material is:

Element	Weight %
Titanium	0.43
Zirconium	0.081
Carbon	0.081
Oxygen	0.0007
Hydrogen	<0.0001
Nitrogen	0.0001
Iron	0.003
Nickel	<0.001
Silicon	0.002
Molybdenum	Balance

Corrosion Rates in HB-40, Wet and Dry CO_2

In an organic cooled reactor, the pressure tubes are exposed to CO_2 gas on the outside and HB-40** on the inside. The corrosion rate of TZM in these environments was determined by exposing coupons approximately 1.6 cm square by 0.15 cm thick to HB-40 and wet and dry CO_2. Weight measurements were taken at various time intervals until a sufficient number of operating hours had accumulated to establish a corrosion rate.

The test apparatus for corrosion testing in CO_2 consisted of a tube furnace 60 cm long containing 1.9 cm diam Vycor tubes with either wet or dry CO_2 flowing through them. The wet CO_2, obtained by bubbling the gas through water, was found to contain 0.6 to 1.4% by volume water vapor. The dry CO_2 was obtained by passing the gas through a drying agent, magnesium perchlorate, before entering the furnace. The water content for dry CO_2 did not exceed 0.25%. Analyses show that the water content variation in the CO_2 annulus in the WR-1 reactor is 0.19 ± 0.16% under reactor operating conditions, hence, results obtained from the dry CO_2 test are most representative of reactor conditions. The flow rate for wet CO_2 was 133 ± 35 cm^3 min^{-1} and for dry CO_2 it was 200 ± 35 cm^3 min^{-1}. The temperature for both test conditions was 440 C.

Corrosion specimens in HB-40 were fastened to a corrosion holder and installed in a sample-coupon vessel in the WR-1 reactor where the temperature was approximately 400 C.

All samples were washed in xylene before weighing to remove any fouling film which formed during testing. The corrosion test results are shown in Fig. 1. The curves representing corrosion rates in CO_2 are the average values obtained from three or more samples. To check whether the heating of the TZM during brazing would alter its corrosion behavior in HB-40, a comparison was made between the corrosion rate of specimens heated at 1000 C for five minutes and "as-fabricated" specimens. The difference in the results between the two sets of samples was insignificant, hence the values from both tests were averaged before plotting.

Visual examination of the samples exposed to HB-40 showed very slight attack. The only change noted was the presence of a thin blotchy-brown film which had the appearance of a stain.

Specimens exposed to wet CO_2 gained weight for the initial 1750 h followed by a weight loss. Visual inspection showed an initial formation of a shiny, violet-colored film which became progressively more porous with increased exposure. The porous film could easily be scraped off which would explain the eventual weight loss.

The behavior observed in dry CO_2 consisted of periods of rapid and slow weight gain. Similar behavior was observed by O'Driscall, Tyzack and Raine (Ref. 2) for molybdenum exposed to dry CO_2 at 500 C under eight atmospheres pressure. For the first 2500 h, the specimens were covered with a very dark-violet film. After this initial period, the film turned black and was very adherent. Attack was uniform with no pitting, although periodically the surface would become covered with minute amounts of loose black powder. The weight gain recorded by O'Driscall et al for molybdenum exposed to dry CO_2 gas for 2500 h is within 10% of the value observed for TZM, although the temperature and pressure conditions were somewhat different.

Tensile Strength

Longitudinal and transverse Instron tensile specimens, having the orientations shown in Fig. 2, were machined from solid bar. Tensile tests were done at ambient temperature and 400 C using a strain rate of 0.020 min^{-1}. To evaluate whether the heating of TZM during brazing would alter its tensile strength, tests were

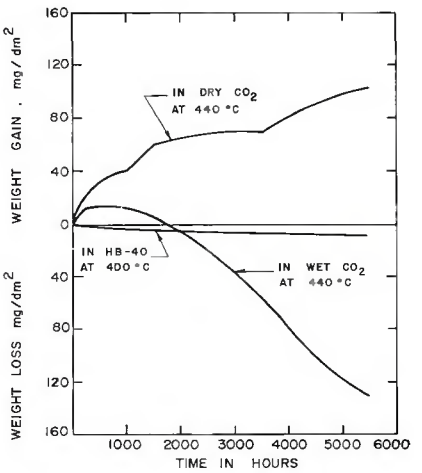

Fig. 1 — Corrosion of TZM

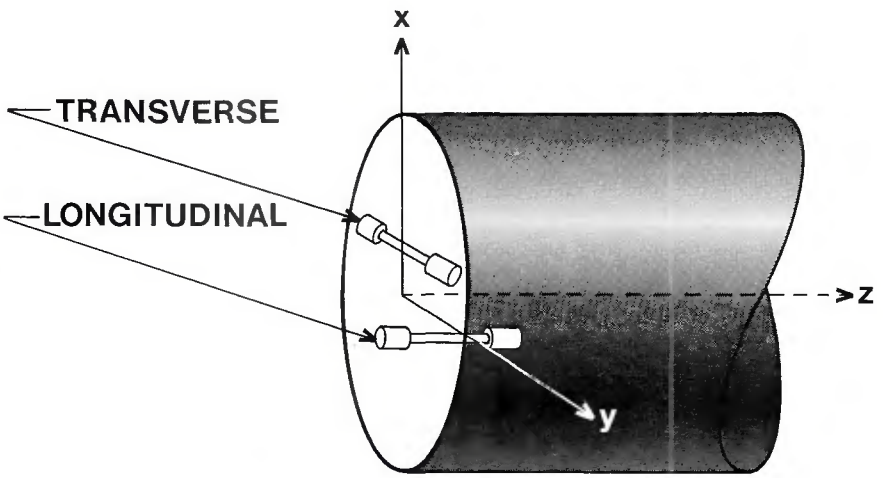

Fig. 2 — Orientation of Instron tensile specimens

***Trade name of Monsanto Corporation for the organic coolant used in WR-1 reactor. (The designation has since been changed to OS-84).*

conducted on specimens heated in a vacuum to 1000 C for 30 min to simulate the braze cycle. The results, given in Table 1, indicate that the brazing cycle does not alter the tensile strength of TZM. The transverse specimens tested at ambient temperature did not exhibit a yield point, however, the lowest fracture stress recorded was 71.1 ksi (490 MN/m²).

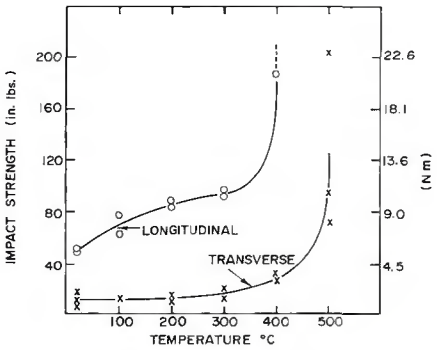

Fig. 3 — Impact test results for TZM

Impact Properties and Hydrogen Permeability

The impact property of TZM was determined by testing longitudinal and transverse Charpy V-notch specimens from room temperature to 500 C. The results are shown in Fig. 3. Since the impact strength of TZM is low at ambient temperature, designs which eliminate the use of TZM as a structural member are necessary to prevent brittle failures from occurring.

The permeation to hydrogen was determined at 600 and 800 C. No permeability was detected at 600 C, however, at 800 C, the permeability constant is 4.0×10^{-5} cm³ (NTP)mm cm^{-2} sec^{-1} atm$^{-½}$ indicating that TZM is an excellent barrier to hydrogen permeation. The 800 C permeability constant for TZM compares favorably with 2.0×10^{-5} found for molybdenum by Webb (Ref. 3).

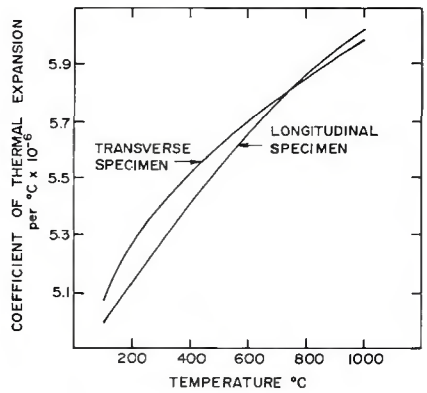

Fig. 4 — Average coefficient of thermal expansion for TZM

Coefficient of Thermal Expansion

Failure to design properly for thermal expansion can have detrimental effects on the joint assembly. To aid with the joint design, the coefficient of thermal expansion was determined for temperatures of 100 to 1000 C. The results shown in Fig. 4 are the average values between room temperature and the particular temperature considered.

Table 1 — Tensile Strength of TZM

Specimen	Material condition	Test temp., C	0.2% yield strength ksi	0.2% yield strength MN/m²	Tensile strength ksi	Tensile strength MN/m²	Elong., %
L-1[a]	As rec'd	RT[e]	89.1	614	94.0	648	16.0
L-2	As rec'd	RT	88.6	611	96.5	665	17.0
T-1[b]	As rec'd	RT	—	—	90.5[d]	624	—
T-2	As rec'd	RT	—	—	81.5[d]	562	—
L-3	As rec'd	400	58.7	405	66.4	458	19.5
L-4	As rec'd	400	58.7	405	65.9	454	18.9
T-3	As rec'd	400	56.5	390	63.7	439	15.6
T-4	As rec'd	400	56.5	390	64.2	443	15.6
L-5	Braze sim.[c]	RT	93.5	645	97.8	674	13.2
L-6	Braze sim.	RT	91.0	627	95.3	657	10.4
T-5	Braze sim.	RT	—	—	77.6[d]	535	—
T-6	Braze sim.	RT	—	—	71.1[d]	490	—
L-7	Braze sim.	400	56.2	387	63.2	436	18.9
L-8	Braze sim.	400	57.7	398	64.9	447	18.9
T-7	Braze sim.	400	57.7	398	65.2	450	13.2
T-8	Braze sim.	400	59.2	408	66.7	460	13.2

(a) Longitudinal specimen
(b) Transverse specimen
(c) Heated in vacuum for 30 min at 1000 C to simulate brazing cycle
(d) Fractured before exhibiting a yield point
(e) RT = room temperature

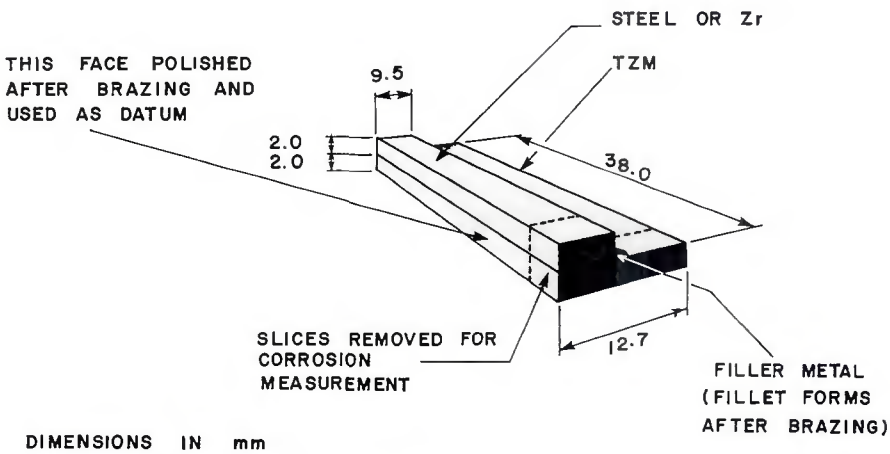

Fig. 5 — Brazed specimen for corrosion testing

Brazing Filler Metal Selection

Filler Metal Requirements

When brazing is used for joining dissimilar metals, the brazing filler metal must satisfy three major requirements:

1. the filler metal must accommodate the thermal stresses developed due to the difference in the coefficient of thermal expansion between the base metals,
2. it must be compatible with both base metals, and
3. it must be compatible with the environment.

A total of 16 filler metals were screened using wettability tests. Where possible, an alloy having an eutectic composition was chosen to retard intergranular penetration by minimizing total time above the solidus temperature. Filler metals having sufficient wettability were then subjected to corrosion tests in the organic coolant (HB-40) and wet and dry carbon dioxide. To assess irradiation damage in the brazing filler metals, tensile test results were compared between irradiated and unirradiated tensile specimens.

Test Procedure

Wettability tests were performed using a high-frequency induction heating furnace. The tests were done by placing small quantities of the filler metal on base metal samples, heating in a vacuum of 10^{-4} mm Hg,

and noting the wetting behavior. An optical pyrometer was used for temperature measurements. Alloys for further testing were selected on the basis of acceptable wetting characteristics.

Corrosion specimens consisting of TZM/Zr and TZM/steel (ASTM A/105) were brazed to form lap-type joints as shown in Fig. 5. The test specimens were made by placing the brazing filler metal on the TZM component adjacent to the joint and allowing it to flow by capillary action. After the specimens were brazed, the edge without the filler was polished and used as a datum for depth of corrosion measurements. The area with the fillet was left in the "as-brazed" condition. The corrosion specimens were exposed to the organic coolant or to either wet or dry carbon dioxide in the same manner and to identical test conditions as described earlier for TZM corrosion specimens. At various intervals of exposure, small slices were removed from each specimen (as shown in Fig. 5) and examined metallographically for depth of corrosion penetration into the braze filler metal.

Butt-brazed tensile specimens were used to test the effect of irradiation on tensile strength of brazing filler metals.

Filler Metal Selection

The results of the wettability tests are summarized in Table 2. Following these tests, two alloys were chosen as being suitable for braze welding TZM to zirconium. One of these, 48Zr-48Ti-4Be, was dropped early from the program because of poor corrosion properties. Further testing was performed only on Zr-5Be. The results of these tests are discussed later.

Based .on the wettability tests, three potential brazing filler metals for joining TZM to steel are: (1) 82Au-18Ni, (2) Zr-5Be and (3) 49Cu-49Ti-2Be. Testing of alloys (2) and (3) was terminated early in the program since these alloys were unable to accommodate the differential in the thermal expansion of the base metals and cracking of the braze occurred. Further testing was therefore restricted to 82Au-18Ni. The results of these tests are also discussed later.

Test Results for Zr-5Be

Corrosion resistance of the lap-type specimens, where the fillet had been left intact, was excellent. In no samples were there indications of excessive corrosion. However, on the polished face where the base/filler metal interface was directly exposed, corrosion resistance was poor, especially in the wet CO_2 atmosphere. The depth of corrosion into the filler metal in wet and dry CO_2 and in HB-

Table 2 — Wettability Test Results

Filler metal	Brazing temp, C	Type of wetting on[a] Zr	TZM	Steel
77Ni-13Cr-10P	980	Reactive	Fair	Fair
60Au-20Cu-20Ag	850	Good	N.T.	N.T.
68Ag-27Cu-5Pd	825	Reactive	N.T.	Good
58Ag-32Cu-10Pd	860	Reactive	N.T.	N.T.
72Ag-28Cu	780	Reactive	N.T.	N.T.
71.15Ag-28.10Cu-0.75Ni	795	Reactive	N.T.	N.T.
87.75Cu-12.00Ge-0.25Ni	965	Reactive	Poor	Good
70Ti-15Cu-15Ni	960	Fair	Good	Good
9Ga-9Pd-82Ag	920	Good	N.T.	Good
68.8Ag-26.7Cu-4.5Ti	850	Reactive	Good	Good
82Au-18Ni	1000	Reactive	Good	Good
48Zr-48Ti-4Be	975	Good	Good	Good
49Cu-49Ti-2Be	980	Reactive	Good	Good
95Zr-5Be	1000	Good	Good	Good
93Cu-4.5Ge-2.5Si	1000	Reactive	Poor	N.T.
99.99Ag	980	Good	Poor	Fair

(a) Good: Continuous filleting, extensive spreading
Fair: Intermittent filleting, little spreading
Poor: No flow, wetting only at contact points
Reactive: Formed intermetallics or eroded material
N.T.: Not tested

Table 3 — Tensile Test Results for TZM/Zr Specimens Butt-Brazed with Zr-5Be

Specimen condition	Test temp., C	Fracture location	Ultimate tensile strength ksi	MN/m²
As Brazed[a]	20	Braze	53.7	370
As Brazed	20	Braze	47.8	329
As Brazed	440	Zr	18.2	129
As Brazed	440	Zr	18.3	126
Brazed then annealed[b]	20	Braze	45.3	312
Brazed then annealed	20	Braze	47.8	329
Brazed then annealed	20	Braze	47.8	329
Brazed then annealed	440	Zr	18.2	125
Brazed then annealed	440	Zr	18.7	127
Brazed then irradiated[c]	20	Braze	42.8	295
Brazed then irradiated	20	Braze	51.0	352
Brazed then irradiated	20	Zr	38.8	268
Brazed then irradiated	440	Zr	16.4	113
Brazed then irradiated	440	Zr	15.7	108

(a) Samples were brazed for 5 min at 1010 C
(b) Annealing period was 100 h at 440 C
(c) Samples were irradiated to a fluence of 6×10^{20} n cm^{-2} (>1.0 MeV)

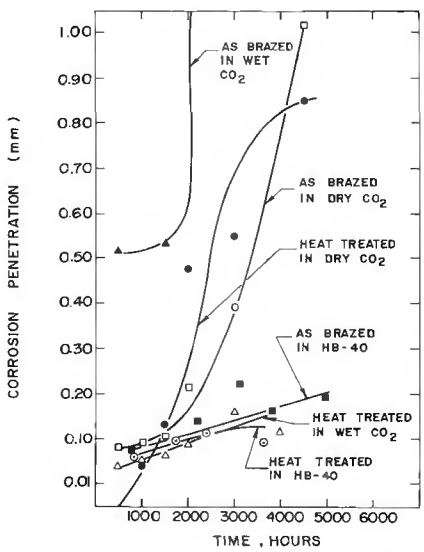

Fig. 6 — Depth of corrosion of braze for TZM/Zr specimen brazed with Zr-5Be

Fig. 7 — Corrosion of TZM/Zr-5Be/Zr bond exposed to HB-40 for 7179 h. X100, reduced 46%

Fig. 8 — Corrosion of TZM/Zr-5Be/Zr bond exposed to dry CO_2 for 3000 h. X100, reduced 47%

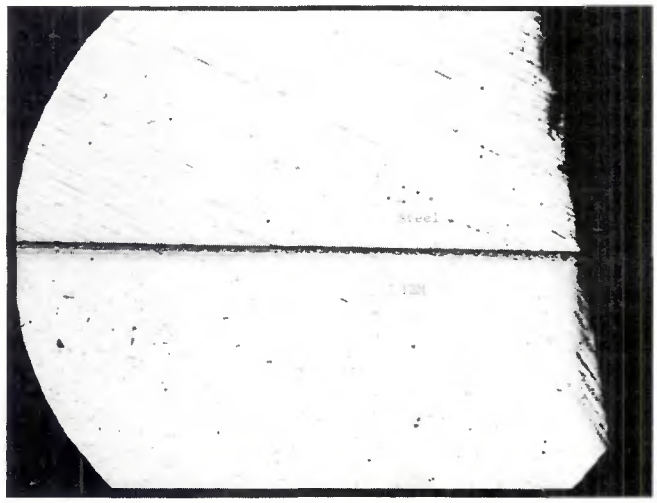

Fig. 9 — Corrosion of TZM/Au-18Ni/steel bond exposed to wet CO_2 for 1900 h. X100, reduced 47%

Table 4 — Tensile Test Results for TZM/Steel Specimens Butt-Brazed with Au-18Ni

Specimen condition	Test temp, C	Tensile strength ksi	Tensile strength MN/m^2
As brazed	20	49.7 (b)	343
As brazed	20	54.2 (b)	374
As brazed	440	27.9	192
As brazed	440	30.5	210

(a) All samples were brazed 5 min at 1000 C
(b) Fractured in TZM

40 is shown in Fig 6. Figures 7 and 8 show a cross-section through two of the corroded specimens. These photomicrographs show that accelerated attack occurred along the TZM interface, possibly due to the formation of poor corrosion resistant intermetallics during brazing. In hopes of improving the corrosion resistance, additional specimens were heated in vacuum for 5 h at 800 C. Corrosion was reduced very significantly in wet CO_2, however, the heat treatment had little effect on corrosion in dry CO_2 or in HB-40.

Although such brazing would normally have a fillet and would therefore behave satisfactorily in-reactor, it is apparent that there is a significant risk of failure resulting from any damage to the fillet and exposure of the base/filler metal interface to either CO_2 or HB-40. Hence, Zr-5Be braze filler metal for joining TZM/Zr for long term service in a CO_2 environment is not recommended.

Room temperature tensile tests shown in Table 3, on as-made or annealed (100 h at 440 C) butt-brazed specimens indicated an acceptable minimum strength of 45.3 ksi (312 MN/m^{-2}). The base zirconium metal fractured, rather than the braze, in all specimens tested at 440 C.

Irradiation does not appear to alter the strength of the joints significantly as indicated by the fracture stress of specimens irradiated to 6×10^{20} n cm^{-2} (>1.0 MeV).

Test Results for 82Au-18Ni

The brazing filler metal flows and wets excellently on both TZM and steel, and it has sufficient ductility to accommodate the differential in thermal expansion of the base metals. Corrosion of the filler metal was negligible, when exposed to HB-40, wet or dry CO_2. Figure 9 shows the wet CO_2 test specimens.

The tensile test results on butt-brazed specimens are summarized in Table 4. At room temperature both specimens fractured in the TZM. However, specimens tested at 440 C fractured in the braze. Specimens irradiated to 6×10^{20} n cm^{-2} (>1.0 MeV) all fractured in the braze during handling, indicating severe damage to the filler metal by irradiation. Shielding of the braze is obviously necessary if such a joint is to be used in an environment of high irradiation fields.

Conclusions

With the possibility of using TZM as a hydrogen barrier in transition joints in an OCR, tests were performed to determine various properties and in-reactor behavior of TZM. Very little corrosion was observed on specimens exposed to HB-40 and it will take ~13 years for corrosion to penetrate 0.025 mm (0.001 in.) into the material in either a wet or dry CO_2 atmosphere. Since TZM gains weight in dry CO_2 and loses weight in a wet CO_2 atmosphere, it can be concluded that the rate of corrosion is sensitive to the water content of the CO_2. The good agreement between corrosion rates of Mo at a pressure of eight atmospheres as obtained by O'Driscall et al (Ref. 2), and the results presented in this paper for corrosion of TZM at atmospheric pressure, would indicate that CO_2 pressure is not a factor on corrosion of TZM. The impact strength of TZM is low at ambient temperature, however it starts to increase very rapidly between 400 and 500 C. The possibility of a brittle fracture occurring can be eliminated by proper joint design. The high temperature strength exhibited by TZM is excellent. The rate of hydrogen permeation is very low making TZM a good barrier to hydrogen flow.

Based on wettability tests, corrosion studies and tensile strength, Zr-5Be braze filler metal can be used to braze TZM to zirconium alloys. The fillets on the corrosion specimens did not appear to corrode excessively in either HB-40, wet or dry CO_2. However, where the fillet was removed and the base/filler metal interface exposed directly, the filler metal along the TZM interface was corroded severely in wet CO_2. The corrosion rate was reduced signif-

icantly in wet CO_2 by heating the specimens in a vacuum at 800 C for 5 h before corrosion testing. Excellent tensile strength was exhibited by both the irradiated and unirradiated butt-brazed specimens, indicating that irradiation does not reduce the tensile strength of Zr-5Be braze metal.

82Au-18Ni was selected as a filler metal for brazing TZM to steel. Corrosion in HB-40, wet or dry CO_2 is negligible. The tensile strength of unirradiated butt-brazed specimens is excellent, however the braze metal is severely damaged by irradiation. This is not unexpected since Au has a very large neutron capture cross-section. Shielding of the braze metal, is therefore, necessary if it is used in an environment of high irradiation fields.

Acknowledgements

The authors wish to express their appreciation to A. Sawatzky for evaluating the rate of hydrogen permeation through TZM.

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3. Webb, R. W., "Hydrogen Permeation Through Metals," NAA-SR-9844, 1964.

WRC Bulletin No. 195
June 1974

"A Review of Bounding Techniques in Shakedown and Ratcheting at Elevated Temperatures"
by F. A. Leckie

"A Review of Creep Instability in High-Temperature Piping and Pressure Vessels"
by J. C. Gerdeen and V. K. Sazawal

"Upper Bounds for Accumulated Strains due to Creep Ratcheting"
by W. J. O'Donnell and J. Porowski

"Cyclic Creep — An Interpretive Literature Survey"
by Erhard Krempl

In recent years considerable effort has been devoted to developing a methodology based on detailed analysis to design structures which will operate under conditions of high temperature and periodic large thermal transients such that there exists a high level of confidence in their structural integrity. This methodology encompasses analytical methods, material behavior and design criteria. There has been excellent progress in all of these areas; however, it has become obvious that simplified procedures are needed, since the costs associated with performing a rigorous time-history analysis of a structure which is subjected to significant transient loadings while operating in the creep regime are very high, particularly if three-dimensional representation is required.

The Pressure Vessel Research Committee believes that progress in further developing this methodology will be assisted by the creation and wide distribution of a series of topical reports. This report series will serve to inform both by making available techniques and data which are relatively unknown in this country and by summarizing the current state of the art. In this manner the PVRC believes that technical progress can be stimulated and focused. However, the technology is in the developmental state and a full description of ancilliary information is often not available (e.g., a complete description of the creep and plasticity response of a candidate material). Also, sufficient confirmatory experimental data on structures of similar geometries, materials and operating conditions does not exist for many of the proposed design methods such as those contained in the following report. Experimental programs such as those sponsored by the USAEC are expected to provide such confirmation and define the range of applicability of proposed methods. Thus the topical reports published in *WRC Bulletin 195* are not recommendations by the PVRC to industry on the appropriate technique for pressure-vessel design at this time, but rather are topical reports of the status of an aspect of elevated temperature design at a point in time to aid the current development work in this field.

For structures other than semi-infinite right circular cylinders of uniform thickness subjected to continuous internal pressure and cyclic radial thermal gradients, no closed form analytical methods of demonstrated conservatism exist. The use of finite element time-history analysis has proven to be, on occasion, extremely expensive. Thus a clear and urgent need exists for the development of simplified analytical techniques to permit the economic evaluation of potential ratcheting configurations.

The concepts discussed in these reports are expected to have significant value in reducing the analytical efforts for the design of elevated temperature structures. At the current time insufficient experimental data are available to permit the PVRC to endorse the techniques for bounding the response of potential ratcheting problems. Further experimental data on the basic response of candidate materials as well as ratcheting experiments on typical structures are required. These reports are recommended to the industry as a source of potentially valuable techniques. It is believed that these proposals deserve detailed examination and should be tested against the body of experimental data as it becomes available.

The price of *WRC Bulletin 195* is $11.00. Orders should be sent to the Welding Research Council, United Engineering Center, 345 East 47th St., New York, N.Y. 10017.