

Journal of Nuclear Materials 283-287 (2000) 1224-1228



www.elsevier.nl/locate/jnucmat

Furnace brazing type 304 stainless steel to vanadium alloy (V-5Cr-5Ti)

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Abstract

In this investigation, pure copper was joined to type 304 stainless steel and V–5Cr–5Ti by brazing in a high vacuum furnace. Microstructural changes in the brazed region and surrounding substrates were examined as a function of holding time at temperatures of 20°C, 40°C and 60°C above the melting point of copper. Reaction layers, which were extremely brittle, formed between the Cu and V–5Cr–5Ti substrates. The formation of intermetallic phases at the filler metal/substrate interfaces was evaluated. Additionally, precipitates (FeCu₂ and FeCu₁₈) formed in the Cu rich filler region. For temperatures $\geq 60^{\circ}$ C above the melting point of Cu, extensive transverse cracking was observed. Hardness tests substantiated the hypothesis that the Cu/V–5Cr–5Ti reaction layer was extremely brittle, since micro-cracks propagated from the tips of the diamond-shaped indentations. Results of mechanical properties tests of the brazed material are also presented. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Vanadium-based alloys in the range of V–(3-6)Cr–(3-6)Ti are candidates for fusion reactor materials in the first wall and blanket assemblies [1–3]. Low-activation and neutron absorption are intrinsic characteristics of these alloys. The V-based alloys possess excellent mechanical properties, e.g. tensile strength and ductility, at elevated temperatures, have high thermal conductivity, and they are corrosion resistant in the anticipated liquid alkali metal environments [4–6]. Consequently, vanadium alloys are considered excellent materials for fusion reactor applications.

Producing components for fusion reactor applications mandates employing some type of joining technique for dissimilar metals such as welding or brazing. Of the aforementioned, brazing is the most feasible because it involves melting of the filler material only, thus eliminating problems that occur when dissimilar metals are fused. To avoid the intrusion of detrimental impurities such as oxygen, it is mandatory that vanadiumbased alloys are joined in an inert environment. Dissimilar metal joints of V alloy to stainless steel are anticipated, and copper has been identified as an initial filler material because it provides good chemical compatibility in fusion reaction applications and it is inexpensive.

In this paper, preliminary wetting conditions are determined for pure Cu on type 304 stainless steel (SS) and V–5Cr–5Ti substrates. V–5Cr–5Ti is joined to type 304 stainless steel by means of a filler metal, pure copper (99.98%), in a high-vacuum furnace $(10^{-4}-10^{-5} \text{ Pa})$. Data evaluating the integrity of the resulting braze joints are presented.

2. Experimental procedure

V-5Cr-5Ti and type 304 stainless steel were supplied in plate form (Wah Chang heat 832394). Both parent materials were machined by electric discharge machining (EDM). After mechanical preparation, the specimens were ground to a 600-grit finish and degreased with

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methanol. Following material preparation, rudimentary characterization was performed on the as-received base materials. An etchant consisting of 20% HNO₃ + 20% HF + 60% H₂ O was used to reveal the microstructure of V–5Cr–5Ti. Nital (98% ethanol + 2% HCl) was used to reveal the microstructure of the austenitic stainless steel. The average ASTM grain size numbers for the V–5Cr–5Ti along the longitudinal and transverse directions were 8.0 and 6.0, respectively. For stainless steel, the ASTM grain size numbers were 8.0 and 7.0, respectively.

Copper shot, 3 mm in diameter, was used in the wetting experiments. To obtain a clean surface, the Cu was degreased in an aqueous solution of 50% HCl for 5 min. The wetting experiments were conducted in an ultra-high vacuum chamber, heated by an induction generator. An optical pyrometer was used to measure the temperature of the Cu shot. On the verge of melting, the emissivity was adjusted to read wavelength energies comparable with those expected [7,8]. The properly calibrated emissivity value was used to determine the temperature. At various times, frames were captured using a Canon AE-8 video recorder. From the frames, hard copies were produced to calculate the receding wetting angles.

A JEOL 733 Super probe with energy dispersive spectrum (EDS) and wave dispersive spectrum (WDS) capabilities was used to view the microstructure at higher resolutions and to perform qualitative analysis. A beam energy of 20 keV and a beam current of 18.50 nA was employed.

Hardness values were obtained for the joints using a Micromet 2001 Microhardness tester. The hardness values were measured using a Vicker's hardness (HV) scale with a 100 g load. Static tensile tests were performed using a materials testing system MTS 810 equipped with a 22 kip load cell and two type 647.10A-01 hydraulic wedge grips. The extension velocity was 2.54×10^{-5} m/s and the gage length was 20 mm, resulting in a strain rate of 0.0013/s. All the tests were conducted at ambient temperature.

Brazing was performed in a horizontal tube furnace with a maximum temperature rating of 1700° C. The joint assembly was brazed according to a prescribed brazing matrix. The brazing temperatures were chosen in 20°C increments above the filler metal's melting temperature. Brazing was conducted at 1100° C, 1120° C and 1140° C. After rapidly heating the furnace to 500° C, the heating rate to reach the braze temperature was 10° C/min. A constant holding time of 300 s was used in each experiment. Upon reaching the brazing temperature and holding for the time allotted, the specimens were cooled 10° C/min until the furnace temperature reached 500° C. Afterwards, the specimens were allowed to cool to room temperature in the vacuum.

3. Results

After heating to approximately 10° C above the melting temperature of Cu and holding for a short span, Cu exhibited a receding angle of 61° on the V–5Cr–5Ti substrate. Most of the angle contour was attributed to dominating adhesive forces which cause the Cu bead to spread. Conversely, a receding angle of 43° was measured for Cu/304 stainless steel. Unlike the wetting behavior of Cu/V–5Cr–5Ti, the final angle observed was a result of Cu penetration (interaction with the 304 stainless steel surface) as opposed to spreading. Thus, chemical equilibrium was not maintained. Overall, pure copper exhibited good wetting angles on both substrates. Fig. 1 shows the wetting bead for Cu on type 304 stainless steel.

After brazing, an acceptable braze joint (Fig. 2) was cross-sectioned and mounted for optical and electron microscopy evaluation. Optical micrographs are shown in Fig. 3 for specimens held for 300 s at 1120°C and



Fig. 1. Cu wetting type 304 stainless steel.



Fig. 2. Completed braze joint of V–5Cr–5Ti (top) and type 304 stainless steel (bottom) using pure Cu braze material.



Fig. 3. (a) Cross Section through a braze joint of type 304 stainless steel Cu-(V-5Cr-5Ti) brazed at 1120°C. (b) Cross section through a braze joint of type 304 stainless steel Cu-(V-5Cr-5Ti) brazed at 1140°C.

1140°C. After holding for 300 s at 1120°C and cooling, reaction layers formed at the parent interfaces. Within the bulk brazing metal, second phase precipitates formed (Fig. 3(a)). Increasing the braze temperature to 1140°C promoted the growth of these precipitates and the reaction zones (Fig. 3(b)). Micro-hardness measurements taken for each sample at room temperature were made along the transverse direction of the joint and averaged. Joint evaluation using microscopy and mechanical testing are discussed under the following subheadings.

3.1. Optical and electron microscopy evaluation

The reaction layer extends approximately 50 μ m into the stainless steel substrate. In this region, Cu appears to have penetrated the grain boundaries and concentrations of 0–2.6 wt% were found in the Fe-rich substrate. At 40°C above the melting point of copper, penetration of Cu into the grain boundaries was more extensive. However, variation of the Cu concentration within the substrate was negligible. According to the binary Cu–Fe phase diagram [9], Cu is miscible in Fe up to approximately 3.5 wt% at temperatures of 1120°C and 1140°C. For this reason, small concentrations of Cu in Fe are not uncommon.

The bulk copper filler metal was no longer homogenous. Second phase precipitates proliferated throughout the filler metal region. The darker precipitates were mostly $FeCu_2$ and the lighter precipitates were predominantly $FeCu_{18}$ as determined by WDS.

In this study, Fe migrated across the Cu-rich filler material and into the V reaction layer. For temperatures



Layer

Fig. 4. High Fe concentration adjacent to Cu/V–5Cr–5Ti layer in a braze joint of (V–5Cr–5Ti)–Cu 304SS.



Fig. 5. Fe compositional profile of the braze zone a measured by wave dispersive analysis.

between 800°C and 1200°C, the binary Fe–V phase diagram [9] manifests the formation of an extremely brittle σ -phase for concentrations ranging from 30 to 65 wt%. Moderate concentrations of Fe atoms in the Cu/V–5Cr– 5Ti reaction layer produced severe lattice strain which induced high stresses in the reaction layer. A region of high Fe concentration also formed in a thin layer less



Fig. 6. Average hardness profile for stainless steel/Cu/V.



Fig. 7. Fracture morphology of: (a) Cu/stainless steel, (b) Cu/V-5Cr-5Ti (500 ×). (a) Represents the macrograph showing a sort of continent shape of Cu (outlined in white).

than 2 µm at the Cu/V–5Cr–5Ti interface, as shown in Fig. 4. Fig. 5 shows a compositional profile of Fe atoms in the braze. Such behavior may have developed from the rejection of Fe atoms from the Cu/V–5Cr–5Ti layer during solidification. As a result, an extremely brittle σ phase (FeCr₃) developed in the Cu/V–5Cr–5Ti reaction layer. Wave dispersive spectroscopy revealed the formation of FeCr₃ in the thin layer at the Cu/V–5Cr–5Ti interface.

3.2. Mechanical properties

High hardness values were recorded for the braze, as shown in Fig. 6. In addition, micro-cracks were observed at the indentation corners while obtaining micro-hardness data at the Cu/V–5Cr–5Ti interface, which support the hypothesis that FeCr₃ formed in this layer. The ultimate tensile strength observed for the braze was 85 Mpa, and the total elongation was 0.17%. This poor ductility compared to the ductility (8%) of 304 stainless steel [10] plus residual stresses in the interlayer may be the reason for failure initiation at this site. The fracture morphologies are shown in Fig. 7.

4. Conclusions

Copper possesses good wetting characteristics on 304 stainless steel and V–5Cr–5Ti. At temperatures above the melting point of pure Cu and less than 1120°C, suitable brazes may be accomplished if the formation of FeCr₃ σ -phase can be suppressed. At 1120°C and higher, FeCr₃, σ -phase, forms which causes severe embrittlement in the Cu/V–5Cr–Ti region. Consequently, the formation of micro-cracks leads to premature failure of the joint, which results in low strength and poor ductility.

Acknowledgements

The author wishes to thank Larry Walker of the Oak Ridge National Laboratories for performing the electron micro-probe and WDS analysis. Special thanks is extended to Dr Stan David and the Materials Joining and Non-Destructive Testing Group at the Oak Ridge National Laboratories for their helpful discussions and use of their facilities. This research was sponsored by the Department of Energy, Office of Fusion Energy.

References

- [1] B.A. Loomis, D.L. Smith, J. Nucl. 191-194 (1992) 84.
- [2] B.A. Loomis, R.H. Lee, D.C. Smith, J.R. Peterson, J. Nucl. Mater. 155–157 (1988) 631.

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- [3] D.L. Smith, B.A. Loomis, A.B. Hull, J. Nucl. Mater. 179– 181 (199) 148.
- [4] O.K. Chopra, D.L. Smith, J. Nucl. Mater. 115–157 (1988) 631.
- [5] S. Vano, M. Tada, H. Matsui, J. Nucl. Mater. 179–181 (1991) 779.
- [6] M.L. Grossbeck, J.A. Horak, ASTM-STP 956 (1986) 291.
- [7] K. Nagata, T. Nagane, M. Susa, ISIJ Int. 37 (1997) 399.
- [8] C.C. Bidwell, Phys. Rev. 3 (1914) 439.
- [9] T.B. Massalski, Binary Alloy Phase Diagrams, ASM International, Metals Park, OH, 1986.
- [10] D.R. Askeland, The Science and Engineering of Materials, 3rd Ed., PWS Publishing, Boston, MA, 1994.