



JOINING OF FERRITIC OXIDE DISPERSION STRENGTHENED ALLOYS

Venu G. Krishnardula, Nofrijon I. Sofyan, William F. Gale and Jeffrey W. Fergus
Materials Research and Education Center, Auburn University, Alabama 36849, U.S.A

ABSTRACT

Oxide dispersion strengthened (ODS) alloys exhibit superior high temperature properties compared to conventional alloys. Joining of these ODS alloys poses a challenge and limits their application in the industry. The transient liquid phase (TLP) bonding technique was employed in the present study to join two ferritic ODS alloys, MA956 and PM2000, using physical vapor deposited thin-film boron as an interlayer. Successful bonds with minimal disruption of the substrate microstructure and limited second phase precipitation at the bond-line were achieved when substrates were joined in the fine grained condition, under an applied bonding stress, with substrate orientation parallel to the extrusion direction.

Keywords Oxide dispersion strengthened (ODS) alloy; Transient liquid phase (TLP) bonding; Mechanical alloying; Scanning electron microscopy (SEM); Microstructure.

1. INTRODUCTION

High-temperature materials increase the efficiency of equipment in the aircraft gas turbine and power-generation industries. Oxide dispersion strengthened (ODS) alloys are high temperature alloys which possess superior oxidation resistance, corrosion resistance and strength at elevated temperatures¹.

ODS alloys are produced by the mechanical alloying (MA) process in which elemental powders undergo extensive cold welding, work hardening and fracture by impact in high-energy dry ball mills². The resulting powder is consolidated by hot isostatic pressing (HIP), extruded and subjected to an annealing heat treatment to result in coarse grains with nanosize oxide dispersoids aligned in the direction of extrusion. No melting occurs at any stage of the process. Yttria (Y_2O_3), a thermodynamically very stable oxide, dispersion in the metal matrix inhibits the dislocation motion, which helps in retaining high temperature properties. Ferritic ODS alloys have better radiation induced swelling resistance and strength-to-weight ratio when compared to austenitic ODS alloys. Hence, ferritic ODS alloys are well suited for high temperature applications in nuclear industry.

The application of ODS alloy in the nuclear industry requires joining of ODS alloys to similar or dissimilar alloys. Joining techniques like arc welding, electron beam welding, and laser welding require melting of the ODS alloy and lead to the disruption of the dispersoid distribution. This can lead to premature failure of the joints³⁻⁶. Hence, the joining technique limits the application of ODS alloys.

The transient liquid phase (TLP) bonding technique is employed in the present investigation to join two ferritic ODS alloys, MA956 (Fe-20Cr-4.5Al-0.5Ti-0.5Y₂O₃ wt %) and PM2000 (Fe-20Cr-5.5Al-0.5Ti-0.5Y₂O₃ wt %), to attain a microstructure with uniform composition, yttria dispersion and grain growth across the bond-line. The advantages of TLP bonding over other joining techniques are the absence of a requirement of a high applied pressure (as in solid-state bonding processes), low joining temperature and tolerance for faying surface oxide layers. TLP

bonding is performed by inserting an interlayer between two substrates to be joined and heating the sandwich-like structure at a temperature above the liquidus of the interlayer and below the solidus of the substrate material. The interlayer forms an eutectic with a substrate component and substrate dissolution occurs at the joining temperature. When held at the joining temperature, interdiffusion of the elements from the solid and liquid leads to isothermal solidification. Homogenization of the bond is attained by continued holding at the bonding temperature to attain a microstructure and chemical composition similar to those of the substrate material⁷.

2. EXPERIMENTAL TECHNIQUES

TLP bonding was conducted in a Gleeble 1500, a thermo-mechanical processing system. Materials used in the experiments were MA956, received as 11 mm square billets and 20 mm round bars in the fine and coarse grain conditions, respectively, and PM2000, received as 25 mm rounds bars in the fine grain condition. Substrates were EDM (electrical discharge machine) cut into 2 mm thick samples normal (transverse) and parallel (longitudinal) to the direction of extrusion. The substrates were then surface ground and cleaned in an acetone bath. Physical vapor deposited 1 μm , 500 nm and 250 nm thick boron thin-films were used as interlayers. Bonding was performed in a vacuum of less than 2×10^{-3} Pa at 1250 °C for up to 1 hour, under a compressive stress of 1 – 5 MPa to extrude out excess liquid formed at the bond-line. Bonds were conducted with substrates in the longitudinal to longitudinal orientation (L-L), transverse to transverse orientation (T-T), and longitudinal to transverse (L-T) orientation. Post bond heat treatments (PBHT) were performed in a radiantly heated vacuum furnace at 1300 °C and 1385 °C for MA956 and PM2000 bonds, respectively. A solution consisting of 2 grams CuCl_2 , 40ml HCl and 40 - 80 ml methanol for 3 - 30 s was used for etching. Light microscopy and scanning electron microscopy (SEM), the latter employing a JEOL JSM-840 instrument, operated at 20 kV, were used to investigate the microstructural features of the material. EDS analysis was also performed to observe the compositional profile across the bond-line.

3. RESULTS AND DISCUSSION

3.1 MICROSTRUCTURE OF THE BONDS

Successful bonds were achieved with minimal microstructural disruption at the bond-line. The use of thin-film boron interlayers causes minimal dissolution of the substrates, which limits the microstructural disruption to very small thicknesses in the μm range.

TLP bonds conducted using MA956 fine grain substrate and 1 μm boron interlayer at 1250 °C for bonding times from 20 seconds to 1 hour formed borides at the bond-line to different extents. It was observed that the amount of boride formation decreased with increase in the bond time. The centerline eutectic was removed for the 1 hour bonds, but boride formation in the adjacent substrates was still seen. The bonds were subjected to PBHT in vacuum at 1300 °C up to 8 hours. The boron at the bond-line diffused into the substrates and the composition of the material across the bond became more uniform, see Figure 1.

The above mentioned post bond heat treatment (1300 °C up to 8 hours) leads to the partial recrystallization of the bonded samples when compared to the unbonded bulk material which underwent heat treatment similar to the PBHT. The grain size of the unbonded bulk substrate was larger than that of the grain size of the bonded substrate, see Figure 2. This difference in the recrystallization phenomena could be attributed to the thermal cycle undergone by the material during bonding (in contrast the boron induced premature recrystallization has been documented in the literature⁸).

The extent of recrystallized grains, see Figure 3, and boride formation in the MA956 bonds also decreased as the thickness of the boron interlayer. The formation of fine grains and agglomerates, at the bond-line/substrate interface, is reduced in the bonds using 500 nm boron interlayer when compared to the bonds using 1 μm boron interlayer. This can be related directly to the amount of boron available at the bond-line.

Initial bonding trials were conducted on PM2000 fine grain material using a 250 nm boron interlayer at 1250 °C for short times (< 500 seconds) and stresses higher than 5 MPa. The bonds were subjected to PBHT at 1385 °C for 2 hours. Decreasing the interlayer thickness and increasing the bonding stress for short bonding times, proved to be helpful. Successful bonds were achieved with PM2000 with a minimal amount of boride formation and once recrystallized, grain growth occurred across the bond-line, see Figure 4. Bonds conducted without applied load resulted in poor bonds with unbonded regions, as compared to the bonds when load was applied, which resulted in bonding across the length of the bond.

3.2 EFFECT OF SUBSTRATE GRAIN CONDITION

Joining was performed using MA956 in both the fine grain and coarse grain condition using 1 μm boron interlayer at 1250 °C. Bonding occurred when joining a fine grain (i.e. as cold worked) condition substrate to another fine grain condition substrate and when joining a fine grain condition substrate to a coarse grain (i.e. recrystallization annealed) condition substrate. However, unbonded regions were seen in the bonds when a coarse grain condition substrate was bonded to another coarse grain condition substrate. This difference in the bonding phenomenon could be attributed to the condition of the ODS alloy. Note that there are large amounts of energy stored in the cold worked, fine grained condition, as compared to the annealed, coarse grained condition of ODS alloys. The thickness of the region containing the recrystallized grains, in the fine grain material, was almost three times larger than that in the coarse grain material, when a fine grain condition substrate was joined to a coarse grain condition substrate. This suggests that there is an effect of the substrate grain condition on the bonding phenomena, but the mechanisms involved are not yet clear.

3.3 EFFECT OF BOND ORIENTATION

The bond-line is almost invisible, and similar to the substrate, in the L-L bonds, see Figure 1(d), when compared to L-T bonds and T-T bonds, see Figures 1(c) and 3(b), respectively. The thickness of the layer of fine grain formation ranges up to 100 μm in 'L-T' bonds compared to 'L-L' and 'T-T' bonds which range up to 20 and 30 μm respectively. Hence, there is an effect of substrate direction on the bonding phenomena.

The yttria dispersoids in the ferritic ODS alloys are aligned in the direction of extrusion and have columnar grains with a large grain aspect ratio (GAR)⁹. Hence, the orientation of the substrate determines the number of dispersoid stringers and grains cut. Substrates machined transverse to the direction of extrusion cut more yttria stringers and grains as compared to the substrates machined in the longitudinal direction. Equiaxed fine grain formation was sometimes observed in the longitudinal substrate in L-T bonds, see Figure 1(c) conducted at 1250 °C for 5 min. The formation of equiaxed fine grains in the 'L-T' orientation bonds can be result of two factors; (1) driving force for grains to grow in the working direction and (2) Influence of boron induced recrystallization from the interlayer into the substrate¹⁰.

4. CONCLUSIONS

In the present investigation it is concluded that

- (1) Physical vapor deposited thin-film boron can be used successfully as an interlayer for bonding ferritic ODS alloys.

- (2) The substrate grain size and the diffusion of boron into the substrate can influence the recrystallization behavior of ODS alloys.
- (3) There is significant effect of the orientation of the substrates when joining ferritic ODS alloys.

ACKNOWLEDGEMENTS

This research was supported by the Idaho National Engineering and Environmental Laboratory (INEEL). The authors would like to thank the INEEL program manager, Dr. Terry C. Totemeier, for his valuable guidance. The authors would also like to express their gratitude to Mr. Charles Ellis of Auburn University's Department of Electrical and Computer Engineering for providing the EBPVD thin-film coatings.

REFERENCES

1. deBarbadillo J and Fisher J J, ASM Handbook, vol. 2, 1990, p 943; Materials Park, OH, ASM International.
2. Suryanarayana C, ASM Handbook, vol. 7, 1998, p 80; Materials Park, OH, ASM International.
3. McKimpson M G and O'Donnell D, JOM 46 (July 1994) 49.
4. Shoemaker L E, Advances in Welding Science and Technology, 1986, p 371, ed. David SA, Metals Park, OH, ASM International.
5. Kelly T J, Trends in Welding Research in the United States, 1982, p 471, ed. David S A, Metals Park, OH, ASM International.
6. O'Donnell D, ASM Handbook, vol. 6, 1993, p 1037. Materials Park, OH, ASM International.
7. Gale W F and Butts D A, Sci. Technol. Weld. Join., In Press, (2004).
8. Khan T I and Wallach E R, J. Mater. Sci. 31 (1996) 2937.
9. Hupalo M F, Terada M, Kliauga A M and Padilha A F, Mat. Wiss. U. Werkstofftech., 34 (2003) 505.
10. Markham A J, PhD Thesis, University of Cambridge, UK, 1988.

FIGURES

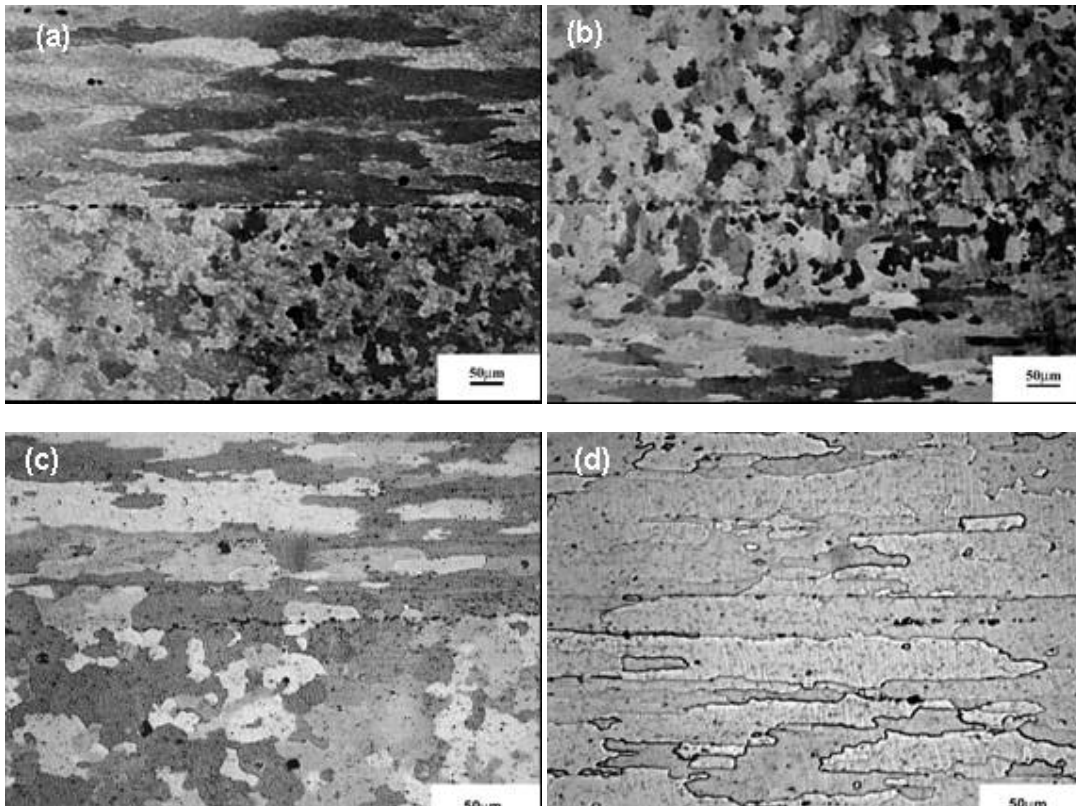


Figure 1. MA956 Fine grain bonds using 1 μm boron interlayer conducted at 1250 $^{\circ}\text{C}$ (PBHT 8 h, 1300 $^{\circ}\text{C}$) (a) L-T bond 30 s, (b) L- T bond 5 min., (c) L-T bond 30 min. (d) L-L bond 60 min.

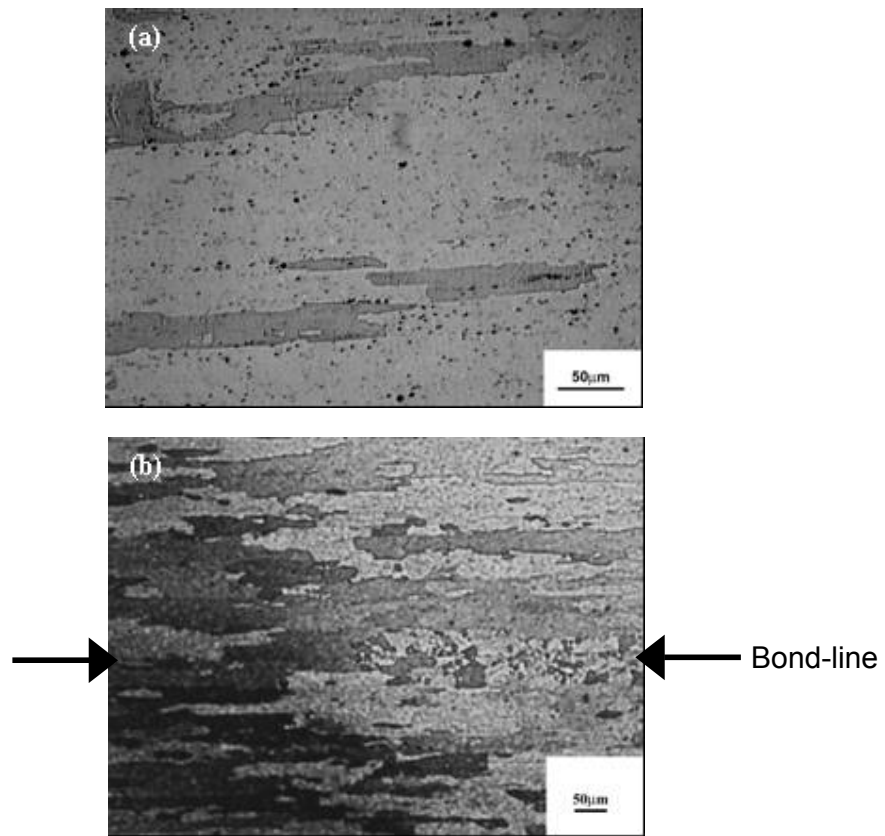


Figure 2. Bond using 1 μm (a) MA956 fine grain bulk material recrystallized (8 h, 1300 $^{\circ}\text{C}$) (b) MA956 fine grain L-L orientation bond 1 h, 1250 $^{\circ}\text{C}$ (PBHT 8 h, 1300 $^{\circ}\text{C}$).

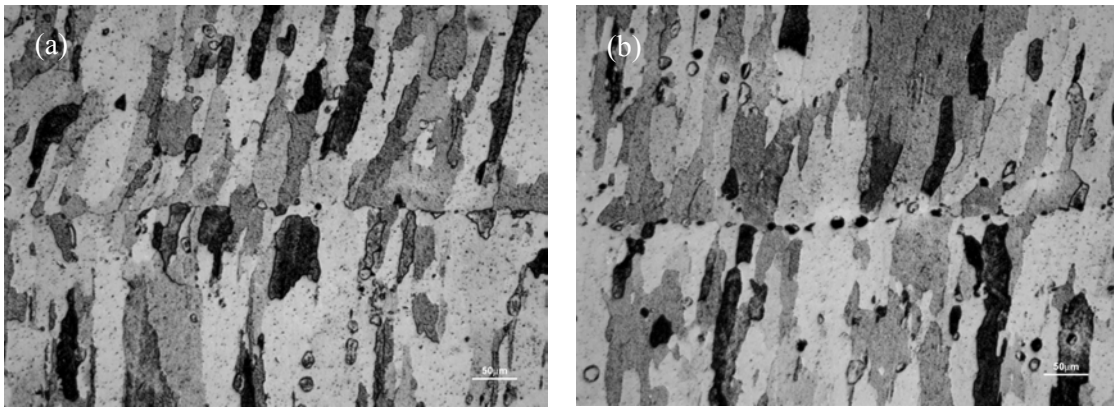


Figure 3. (a) MA956 fine grain T-T orientation bond with 500 nm boron interlayer 343 s, 1250 °C (PBHT 1 h, 1250 °C + 1 h, 1300 °C). (b) MA956 fine grain T-T orientation bond with 1 μm boron interlayer 256 s, 1250 °C (PBHT 1 h, 1250 °C + 1 h, 1300 °C).

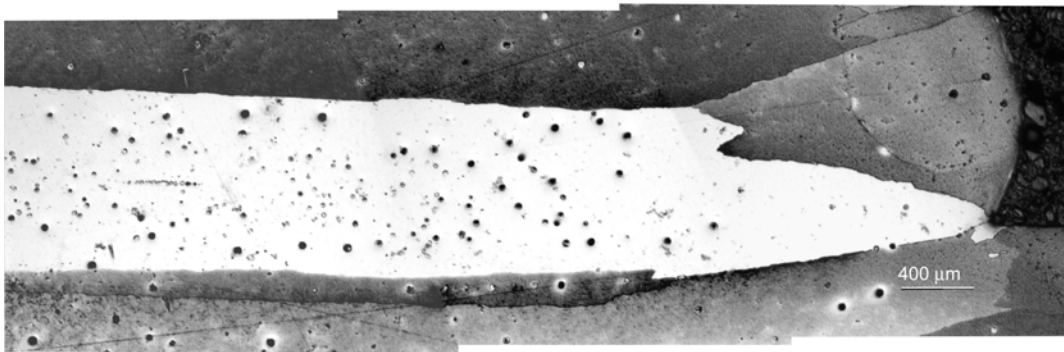


Figure 4. PM2000 fine grain L-L orientation bond 250 nm boron 30 s, 1250 °C (PBHT 2 h, 1385 °C). A large recrystallized grain spanning the bond-line is observed.