

Strength and microstructure of diffusion bonded titanium using silver and copper interlayers

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ABSTRACT

Commercially pure Ti has been diffusion bonded using silver and copper interlayers and without any interlayer. The microstructure of the bonded zone is affected by the bonding temperature, bonding time and interlayer type. With a silver (Ag) interlayer at 980 °C for 10 h, the diffusion zone consisted of a solid solution of Ag at the center and a zone of AgTi intermetallics on both sides of it. As the temperature and time increased (1030 °C, 30 h), AgTi with small amount of dispersed AgTi₂ formed at the center of the diffusion zone and next to it a eutectoid mixture of Ti solid solution and AgTi appeared. When Cu was used as an interlayer at 900 °C for 10 h, Cu–Ti solid solution, a zone of different intermetallics, and Ti–Cu solid solution formed in the bonded zone. However, at 1000 °C or higher temperatures, no continuous zone of intermetallics was found in the bonded region, only eutectic mixtures and Ti–Cu solid solutions appeared. The maximum tensile strengths achieved were 160 MPa, 502 MPa, and 382 MPa when Ag, Cu and no interlayers were used, respectively.

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1. Introduction

Titanium (Ti) and its alloys are considered technically superior and cost-effective engineering materials for use in industrial applications. They have been widely used in aerospace and chemical industries because of their high specific strength, good erosion resistance and favorable high temperature properties [1–3]. With the increased use of Ti and its alloys, the joining of Ti and its alloys is of great interest. Unfortunately, welding of Ti and Ti alloys is difficult as they are highly chemically reactive at high temperatures and tend to oxidize at very low partial pressures of oxygen. During the welding process, Ti alloys pick up oxygen and nitrogen from the atmosphere very easily [4]. As a result, diffusion bonding is a preferred joining method for Ti and Ti alloys.

Many reports on diffusion bonding and transient liquid phase bonding of Ti and Ti alloys have been published. Elrefaey and Tillmann [5] reported diffusion bonding of Ti to low carbon steel using a copper-based interlayer (Cu–12Mn–2Ni, wt.%). The optimum parameters were found to be 850 °C for 90 min. However, Cu–Ti intermetallics were formed in the bond line. The optimum bonding strength (tensile-shear) also occurred at 850 °C for 90 min when an Ag–Cu–Zn interlayer was used [6]. Kundu et al. [7] con-

ducted diffusion bonding of titanium to 304 stainless steel using copper interlayers. The strength of the bond reached that of base Ti (319 MPa) for the sample bonded at 900 °C for 1.5 h and the strength decreased with increasing bonding temperature due to the higher volume fraction of intermetallics. Dezellus et al. [8] performed transient liquid phase bonding of titanium to aluminum nitride using a CuSi (28Cu–Ag) interlayer. In this bonding process, Ti formed different intermetallics with copper. As a result, the copper content of the interlayer alloy decreased but the layer still remained in the liquid state. They observed that the solidification occurred faster at 795 °C than at 850 °C due to fast formation of the Cu-rich compound TiCu₄.

Sheng et al. [1] reported the bonding of a Ti–22Al–25Nb alloy using a Ti–15Cu–15Ni interlayer (melting point, 932 °C). The authors found the optimum parameters in terms of strength were 970 °C for 90 min using slow cooling. The strength reached 93% of the base alloy. Sheng et al. [9] reported on phase transformation superplastic diffusion bonding of titanium alloy to stainless steel without using any interlayer. When a material is repeatedly heated and cooled in a transformation temperature range, it can be greatly deformed. This phenomenon is known as dynamic superplasticity. Using the optimum parameters (maximum and minimum temperature, number of heating and cooling cycles, specific pressure and heating rate) a tensile strength of 307 MPa (96.8% of the strength of Ti) was obtained.

In the present study, pure Ti was diffusion bonded using Ag, Cu interlayers and without any interlayer. The microstructure of the bonded zone has been investigated after bonding at different

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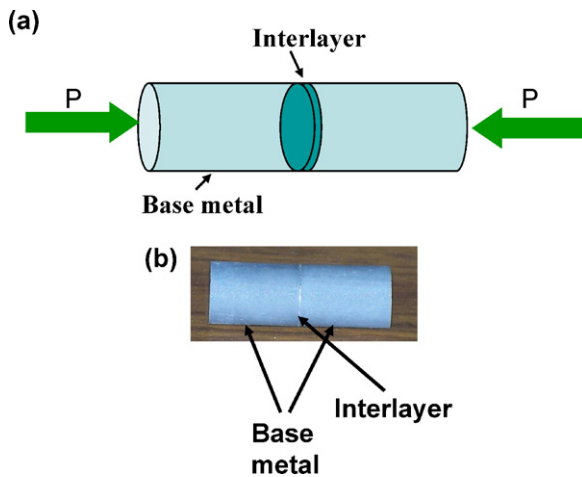


Fig. 1. (a) Schematic representation of diffusion bond geometry, and (b) diffusion bonded sample.

temperatures and exposure times. Tensile strengths of the diffusion bonded samples were also investigated.

2. Experimental procedure

Cylindrical grade 2 Ti rods (Online Metals) 6.35 in mm diameter and ~8 mm in length were used for diffusion bonding and subsequent microstructural examination. The samples were polished to a 600 grit finish and the surfaces were cleaned in an ultrasonic bath using isopropyl alcohol and stored in alcohol before bonding. The samples for tensile testing had a gauge length of 59 ± 0.5 mm and a diameter of 9.0 ± 0.1 mm in the gauge section. The bonding surface of each sample was polished to a 120 grit finish before bonding. The diffusion bonding was done with commercially pure copper (99.999% pure, Alfa Aesar, USA), or silver (99.998% pure, Alfa Aesar, USA) interlayers and without any interlayers. The thickness of both interlayers was 100 μm . The samples to be bonded were held in a jig made of Ti (to eliminate the thermal expansion mismatch) under static pressure and placed in a tube furnace. Schematics of the diffusion bond geometry and diffusion bonded samples are shown in Fig. 1. The heating chamber was repeatedly evacuated and filled with argon gas 10 times to make it oxygen-free and then filled

with argon gas to have inert environment for bonding. An oxygen trap was used in the line of argon flow into the heating furnace. Ti joints with Ag interlayers were bonded at 1030°C for 10–30 h and with Cu interlayers were bonded at $900\text{--}1100^\circ\text{C}$ for 10 h. Ti bonds without any interlayers were bonded at $980\text{--}1030^\circ\text{C}$ for 10–30 h. A longitudinal section of each bonded sample was polished down and prepared for microstructural observation. Scanning electron microscopy (SEM) observations of these samples were carried out using a Hitachi S-3400N scanning electron microscope (SEM) and the compositions and different phases in the bonded zone were analyzed by energy dispersive spectrometry (EDS).

To determine the mechanical properties of the bonded specimen, uniaxial tensile tests were performed using a screw-driven AG-IS 50 kN universal testing machine (Shimadzu) machine with a cross head speed of 1 mm/min. Cylindrical tensile specimens were prepared according to the ASTM standard E 8M-99.

3. Results and discussion

3.1. Microstructures

Fig. 2 shows the SEM images of diffusion bonded titanium (Ag interlayer) at 1030°C for 10 h. The bond centerline consisted of AgTi matrix with small particles (submicron to $\sim 2.5 \mu\text{m}$) of AgTi_2 (intermetallic, Fig. 2(a)). A eutectic mixture of $\alpha\text{-Ti}$ and AgTi_2 formed just outside to this central region. Closer to the base material, a mixture of Ag–Ti intermetallics and a solid solution of Ti and Ag was found (Fig. 2(b)). The bond region farthest from the centerline consisted of only a solid solution of Ti and Ag (Fig. 2(c)). As the bonding time increased to 30 h at 1030°C , the same microstructures appeared in the bond region but the dimensions of each region increased somewhat. Also, some regions of Ti–Ag solid solution were found in the AgTi matrix at the bond centerline (Fig. 3). According to the equilibrium diagram of Ti–Ag, Ti can dissolve more than 29.2 wt.% Ag at 1030°C . But as the temperature decreases, the solubility of Ag into Ti also decreases and at room temperature it is about 1 wt.%. Our aim was to obtain Ti–Ag solid solution or at least to reduce the intermetallics in the bond centerline because the intermetallics are brittle and thereby reduce the joint strength. However, a continuous layer of AgTi intermetallics was found under all conditions.

Although Ag and Cu both form intermetallics with Ti, the microstructure of the bonded zone was different when Cu was

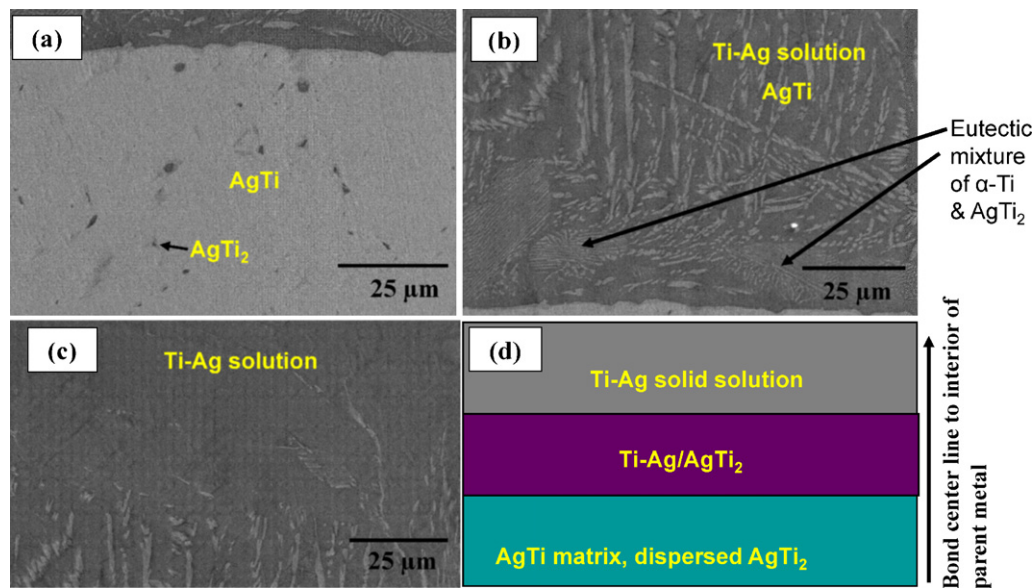


Fig. 2. SEM images of diffusion bonded Ti using Ag interlayer at 1030°C for 10 h: (a) bond centerline, (b) $\sim 80 \mu\text{m}$ away from the bond centerline, (c) $\sim 160 \mu\text{m}$ away from the bond centerline and (d) the schematic representation of different regions from the bond centerline to the interior of base material.

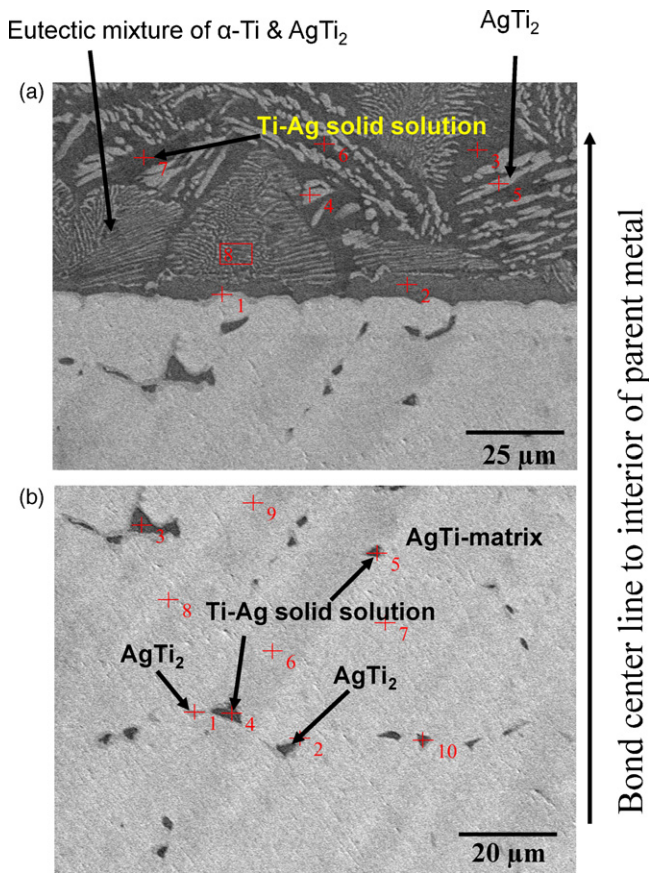


Fig. 3. SEM images of diffusion bonded titanium using Ag interlayer at 1030 °C for 30 h.

used as an interlayer than when an interlayer of Ag was used. At 900 °C for 10 h, the bonding zone consisted of a region of Cu–Ti solid solution, a region of different intermetallics (TiCu_4 , Ti_2Cu_3 , TiCu), a region of eutectic mixture of Ti and intermetallics and a region of Ti–Cu solution (Fig. 4). The composition of Cu was discontinuous in the intermetallic region depending on the composition of intermetallics. As the bonding temperature increased to 1000 °C, the continuous region of different intermetallics disappeared from the bond centerline. The centerline consisted of a eutectic mixture of Ti and Cu. A cross-section of the joint centerline is shown in Fig. 5. The similar microstructures were obtained at the centerline at 1020 °C for 10 h (Fig. 6). Although the melting point of Cu is 1085 °C, we did not find any residual Cu in the bond region at 1000 °C or above. The reason could be the formation of β -Ti above 880 °C which dissolves the maximum ~17 wt.% of Cu at 1000 °C

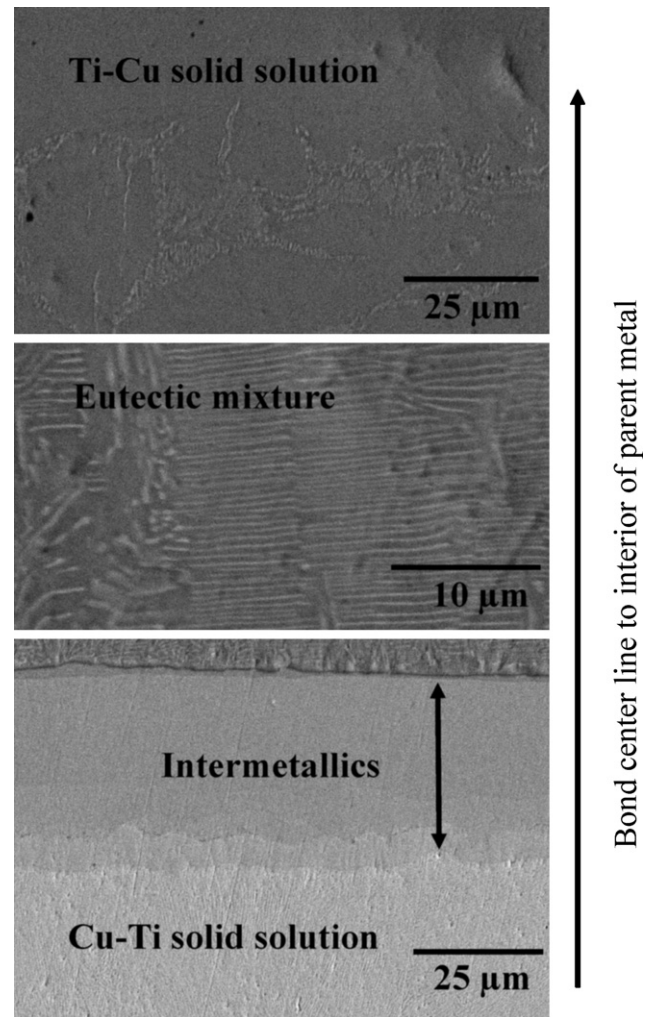


Fig. 4. SEM images of diffusion bonded titanium using Cu interlayer at 900 °C for 10 h.

(refer to the Ti–Cu phase diagram). However, at 1050 °C for 10 h some pores appeared in the bond region and the pore size increased at higher temperatures. The bond centerline consisted of Ti–Cu solid solutions, eutectic mixtures and some small areas of intermetallics, possibly Ti_3Cu . A cross-section of this joint is shown in Fig. 7.

Ti was also diffusion bonded without any interlayers. Fig. 8 shows the SEM images of diffusion bonded titanium without interlayer at 980 °C for 20 h (etched with 8% HF and 4% HNO_3). The bond

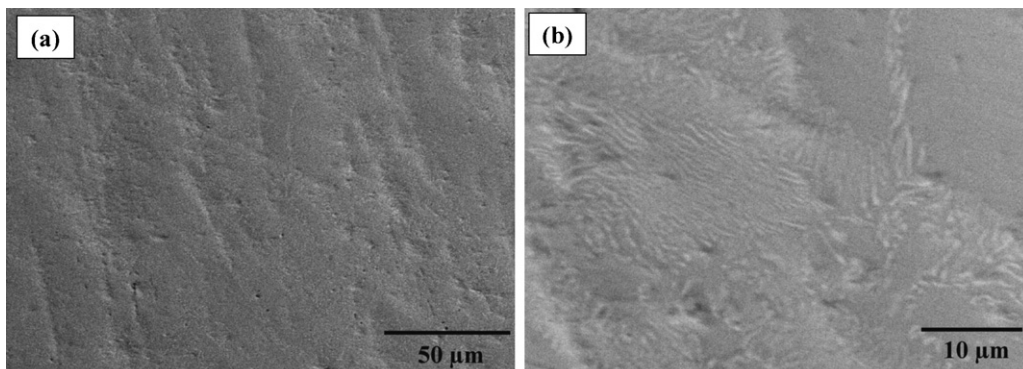


Fig. 5. SEM images (joint centerline) of diffusion bonded titanium using Cu interlayer (a) at 1000 °C for 10 h and (b) enlarged image of (a).

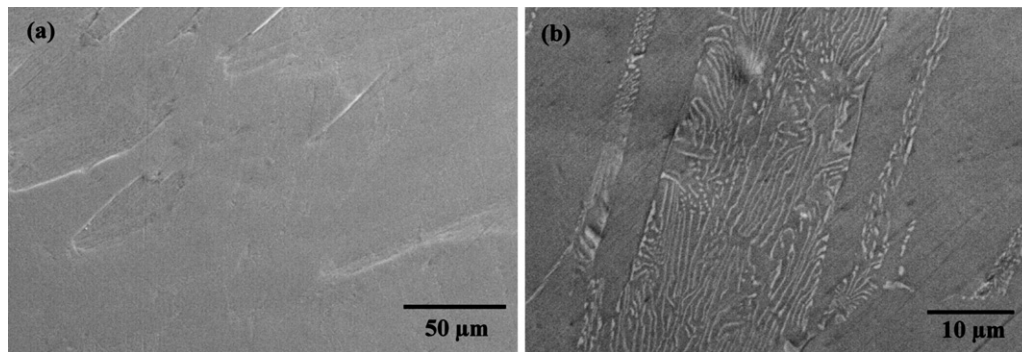


Fig. 6. SEM images (joint centerline) of diffusion bonded titanium using Cu interlayer (a) at 1020 °C for 10 h and (b) enlarged image of (a).

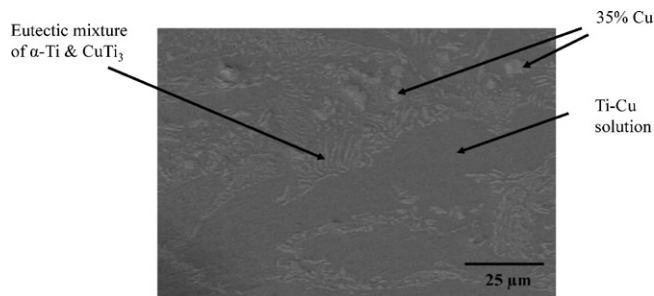


Fig. 7. SEM images (joint centerline) of diffusion bonded titanium using Cu interlayer at 1100 °C for 10 h.

line was not visible under SEM until etched and it was not continuous, some regions were completely bonded while voids were present elsewhere. The bonding temperature (980 °C) was ~65% of the melting point of Ti.

3.2. Mechanical properties

To check the mechanical properties of the bonded samples, uniaxial tension tests were performed. Table 1 shows the tensile properties of the bonded samples with Ag and Cu interlayers and without any interlayer. The strengths of the sample bonded using Ag interlayer were very low due to the formation of continuous layer of fragile intermetallics in the bonded region. The maximum strength achieved for this interlayer was 160 ± 25 MPa at 1030 °C for 30 h. The maximum strength increased to 502 ± 7 MPa when Cu was used as an interlayer at 1030 °C for 10 h which is ~98% of the base metal strength (511 MPa). This might be due to the absence of a continuous region of intermetallics as discussed earlier. The ultimate tensile strength was also determined for bonding at 980 °C ($0.65T_m$, T_m is the melting point of Ti) and 1030 °C

Table 1

Ultimate tensile strengths of diffusion bonded samples with Ag, Cu interlayers and without any interlayer.

Interlayer	Temperature (C)	Time (h)	UTS (MPa)
Commercially pure Ti (Grade 2)			511 ± 3
Ag	1030	10	72 ± 17
Ag	1030	30	160 ± 25
Cu	980	10	460 ± 14
Cu	1000	10	468 ± 10
Cu	1030	10	502 ± 7
None	980	10	296 ± 9
None	980	20	380 ± 11
None	980	30	352 ± 8
None	1030	10	343 ± 5
None	1030	20	382 ± 13
None	1030	30	358 ± 8

($0.67T_m$) for 10–30 h without any interlayer. The strength increased with temperature and 10–20 h bonding time, but decreased at 30 h bonding time. The maximum ultimate tensile strength of 382 MPa was achieved at 1030 °C for 20 h.

In addition to bonding time and interlayer composition, both the flatness of the bonding surfaces and surface roughness were found to influence the strength of the diffusion bonded joints. Initially, tensile specimens were polished by hand on a polishing wheel to a 600 grit finish. However, the bonding surface of these specimens was found to be somewhat curved, especially near the edges, and the outer surfaces did not bond well. The effect was not as pronounced on the smaller samples for microscopic analysis. Fig. 9 shows the SEM images of fracture surface of Ti–Ti bonded at 980 °C for 10 h; the bonding surface was polished by (a) hand to 600 grit and (b) grinding wheel to 120 grit before bonding. Due to the coarser grit, the wheel-polished specimen shows many unbonded regions compared to specimen polished by hand. However, the wheel-polished specimen surface is much flatter, resulting in a larger overall area of contact between specimen faces. This leads

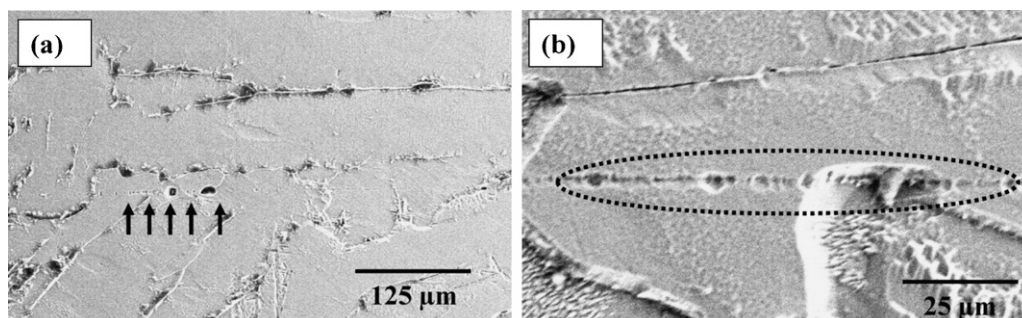


Fig. 8. (a) SEM images of diffusion bonded titanium without interlayer (a) at 980 °C for 20 h (etched with 8% HF and 4% HNO₃), and (b) magnified image of (a).

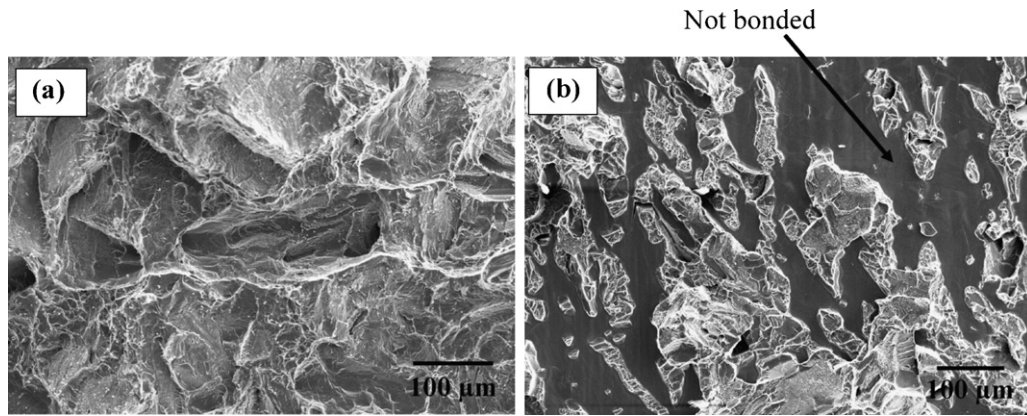


Fig. 9. SEM images of fracture surface of Ti–Ti bonded at 980 °C for 10 h, the bonding surface was polished by (a) hand and (b) wheel before bonding.

to higher joint strengths for the wheel-polished specimens. Only one grinding wheel grit was available for the current work. Additional study is needed to quantify the effect of surface roughness on the final joint strength.

4. Conclusions

This study investigated the diffusion bonding of commercially pure Ti using Ag, Cu and without any interlayer. The formation of brittle intermetallics could not be avoided for an Ag interlayer even for longer periods of bonding time. Hence, the strength of the joints was very low. On the other hand, formation of a continuous band of intermetallics was avoided using a Cu interlayer. The maximum strength obtained was 502 MPa using Cu interlayer and the joints reached ~98% of the strength of the base metal. The strength reached ~75% of the parent metal when no interlayer was used. Further investigations are necessary to check the effects of surface finish and surface flatness on the tensile strengths of the diffusion bonded Ti.

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