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Scanning Electron Microscopy (SEM) Analysis and Hardness of Diffusion Bonded Titanium-Titanium and Titanium-Copper Plates with Static Force and without Interlayers

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Abstract

In the present research, commercially pure Ti (grade-2) has been diffusion bonded with Ti and Cu plate under static force without any interlayers. The diffusion bonded samples were tested for micro hardness and micro structural analysis through optical microscopy and SEM. It is found from the present investigation that the bonded zone is affected by the processing variables such as bonding time (1 - 2 h), bonding force (250 N), bonding temperature (973 - 1073 K) and surface roughness. Results of the investigation revealed that temperature range of −973 - 1073 K along with time duration of 1 - 2 hours in vacuum has resulted in a joint having high hardness with minimum pores. Hardness of the bond depends on the grain boundary diffusion at the interface and maximum hardness was achieved in the case of Ti-Cu joints. When Ti-Cu plates were used for bonding at 973 K for 2 hours, Cu-Ti solid solution along with a zone of different intermetallics was formed in the bonded zone. However, at higher temperatures, no continuous zone of intermetallics was found in the bonded region but instead Ti-Cu solid solution appeared.

Keywords

Diffusion Bonding, Titanium, Micro Hardness, Surface Roughness, Microstructure

1. Introduction

Diffusion Bonding (DB) technique is based on the atomic diffusion of elements at the joining interface and the process actually is the transport of mass in the form of atomic movement or diffusion through the lattice of a crystalline solid. Diffusion bonding is the solid state metal joining process comprising of similar and dissimilar metals or non-metals. Diffusion of atom occurs by many mechanisms such as exchange of places between adjacent atoms, motion of interstitial atoms or motion of vacancies (unoccupied site) in a crystalline lattice structure. Macroscopic deformation in a diffusion controlled process is induced by applying definite heat and pressure for a finite interval of time and the preferable mechanism involved here is the low activation energy required for atomic movement [1] [2]. Diffusion of atoms is a thermodynamic process where temperature and infusibility of the material are considerable parameters. In general, the diffusion rate [3], in terms of diffusion coefficient D is given by the well known Fick's law of diffusion.

The aerospace industry is a larger market for titanium products primarily due to the exceptional specific strength, corrosion resistance and high resistance and elevated temperature properties [4] [5]. With increased use of Ti and its alloys, the joining of Ti and its alloys is of great interest. Unfortunately welding of Ti and its alloys is very difficult as they are highly reactive at high temperatures and tend to oxidize with oxygen [6] [7] [8]. Brazing is an effective method for dissimilar materials joining, but the brazing joint of Ti is difficult to guarantee access to the entire surface of the joint continuity [9]. Therefore, diffusion bonding becomes a popular and preferred solid-state joining process for Ti-Ti and Ti-Cu because the coalescence of contacting surfaces occurs with minimum along with high joining quality.

The quantitative treatment of unsteady state diffusion processes is also formulated and one can consider it to examine some relevant solutions quantitatively. Oxidation rate constant follows an Arrhenius relationship and the effective activation energy for oxidation was obtained from the Arrhenius plot. The activation energy for the titanium material is improved by heating the specimen almost near the recrystallization temperature. At higher temperature, creep problems can be eliminated by creating the inert atmosphere through the use of rotary and diffusion pumps. **Figure 1** shows the diagram indicating steady state and unsteady state diffusion bonding process.

The activation energy for atomic diffusion at the surface, interface and grain boundaries is relatively low compared to the bulk diffusion due to a looser bond of the atoms and higher oscillation frequency of the diffusing atom. This enhances

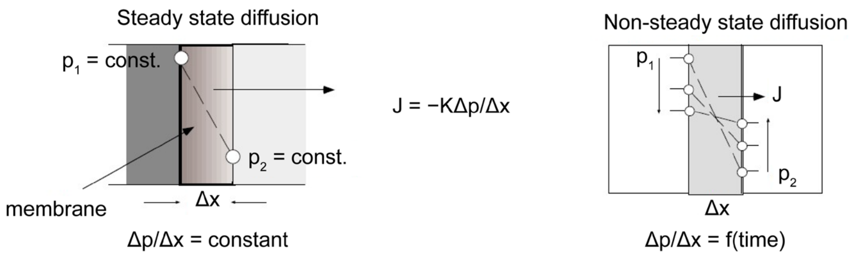

Figure 1. Steady state and unsteady state diffusion bonding.

the atomic diffusion, and thus eases the diffusion bonding of two metal pieces assuming that a perfect interface contact exists. The interface contact can be optimized by a treatment of the surface to be bonded through a number of processes, such as mechanical machining, polishing, etching, cleaning and coating. Creep mechanism allows a material flow to produce full intimate contact at the joint interface as required for diffusion bonding. Therefore, surface treatment and selection of bonding temperature and loading are basically important factors for the diffusion bonding process. Other factors such as thermal conductivity, thermal expansion, and bonding environment also affect the bonding process, particularly at high bonding temperature.

Many reports on diffusion bonding and transient liquid phase bonding of Ti and its alloys have been published [10].

The optimum bonding strength occurred at 850˚C for 90 min when an Ag-Cu-Zn interlayer was used along with the formation of intermetallics in the bond line [11]. Kundu *et al*. [12] conducted diffusion bonding of Ti to 304 stainless steel using Cu interlayers. Dezellus *et al*. [13] performed transient liquid phase bonding of Ti to Al nitride using a Cusil (28Cu-Ag) interlayer. In this bonding process, Ti formed different intermetallics with Cu. Cu alloy is widely used in aviation, navigation and automobile industries because of its good elasticity, high strength and good abrasive resistance. In some locations, the performance requirements for titanium and Cu alloy components are special. So the bonding of titanium with Cu form a compound structure which reduces the mass of structure. Therefore, study on the technology for bonding of titanium with Cu is of great importance. However, the properties of Ti and Cu have great differences in crystal structure, melting point, heat conductivity, and linear expansion coefficient. Thus, the traditional fusion method is hard to realize the joining process.

In the present research the joining of Ti-Ti and Ti-Cu alloy is realized by diffusion bonding to fabricate composite structure without any interlayer has open up a new way to broaden the application of titanium and copper alloys. Hence the focus of the present investigation is aimed at vacuum diffusion bonding of Ti-Ti and Ti-Cu alloy to realize the resulting hardness and microstructural characteristics at the interfacial zone of specimens tested after bonding at different temperatures and exposure times.

2. Experimental Procedure

Diffusion bonding process of commercially pure Ti and Cu plates without any interlayers was conducted in a vacuum thermal-imitation machine and the working chamber was evacuated to a pressure of 1.1×10^{-3} Pa. **Figure 2** and **Figure 3** shows the diffusion bonding process diagram as well as the experimental setup. This set up includes a vacuum chamber to minimize the chemical reaction during the bonding process by creating the necessary vacuum using the rotary pump and a diffusion pump along with a furnace as the heating system. Diffusion pump and vacuum chambers are continuously cooled through water

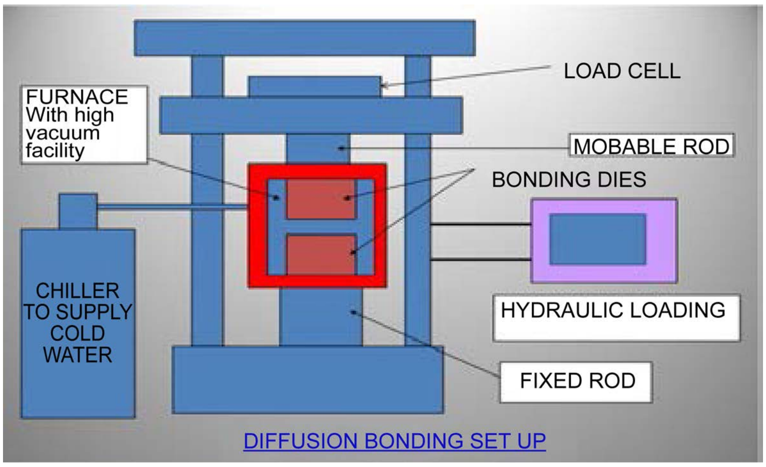

Figure 2. Diffusion bonding processes diagram.

Figure 3. Diffusion bonding experimental set-up.

circulation system with a pump and chillers. The required force on the specimens during bonding is applied using hydraulic loading mechanism. Ti-Ti joints were bonded at 1073 K for 1 - 2 h and Ti-Cu were bonded at 973 K for 1 - 2 h both under a static force of 250 N. Plate type of specimens of size 20 × 10 mm and of thickness 3 mm were used for diffusion bonding and subsequent hardness and microstructural examination. The mating surfaces of the specimens were ground with SiC (emery) polished using diamond paste and finally cleaned in an ultrasonic bath using acetone reagent acetone for 30 seconds before bonding. The surface roughness of the prepared specimens was finally measured using a standard electronic surface roughness profile meter. The samples to be bonded were held in a jig made of Ti (to avoid the thermal expansion mismatch) under static force and placed in a diffusion bonding furnace.

A longitudinal section of each bonded sample was polished for microstructural observation. Neophot −21 metallurgical microscope was used for optical microscopy of the polished sepcimens. Scanning electron microscopy (SEM) observations were carried out using JEOL K-8140B scanning electron microscope and the composition of different phases in the bonded zone were analyzed by EDS method. To determine the mechanical property of the bonded specimens micro hardness tests were performed on the right and left side of the bonded area using a Hitachi make micro hardness tester.

3. Results and Discussion

3.1. Surface Roughness and SEM Fracture Analysis

In addition to bonding temperature, bonding time, bonding force, flatness and roughness of the bonding surfaces were found to influence the hardness of the diffusion bonded joints. Figure 4 and Figure 5 shows the surface roughness profile of ground and polished Ti and Cu plates. The surface roughness value in terms of Ra was measured using the standard procedure. This was done using with different grades of emery papers of which emery papers of grade above 2000 along with polishing (using diamond paste) gave very smooth surface having no surface irregularities and aspirates. This is very important for atomic diffusion and hence the bond hardness. Figure 6 and Figure 7 shows the SEM images of the fractured surfaces (under tension) of Ti-Ti bonded at 1073 K and

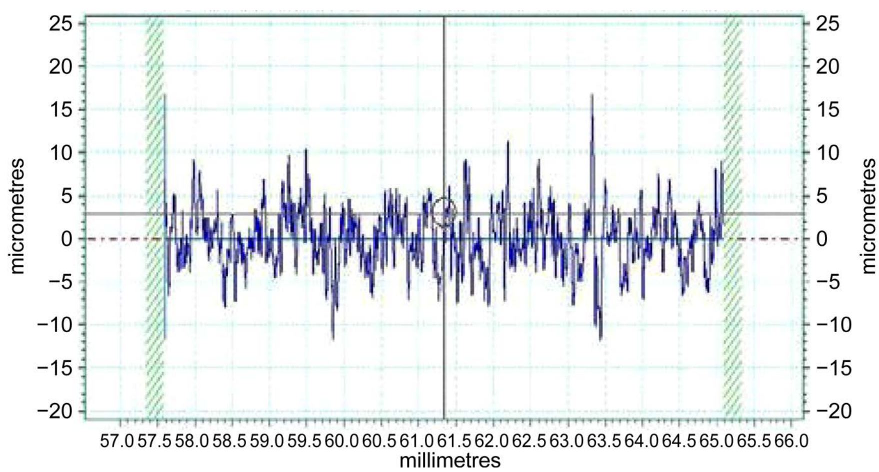

Figure 4. Surface roughness profile of Ti plate before bonding.

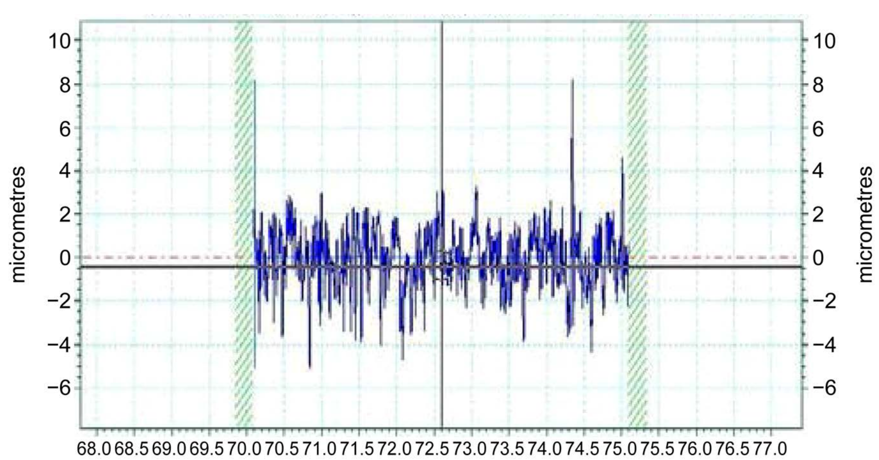

Figure 5. Surface roughness profile of Cu plate before bonding.

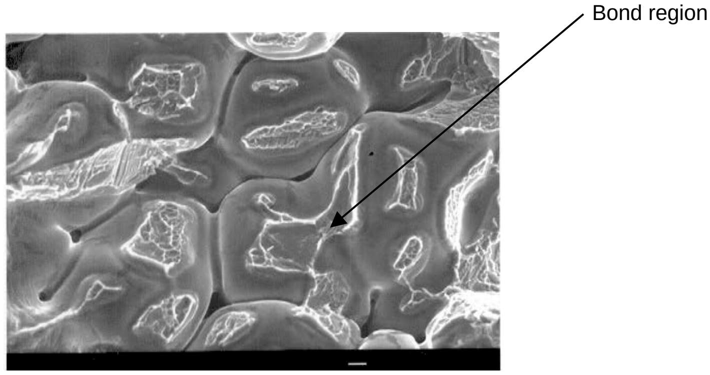

Figure 6. SEM fractograph of surface (failed under tension) of Ti-Ti bonded (at 1073 K for 2 h).

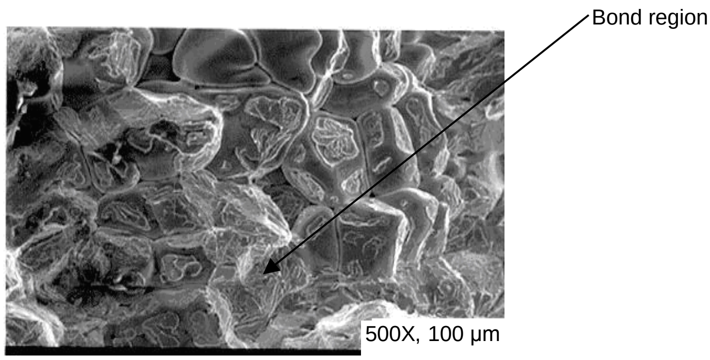

Figure 7. SEM fractograph of surface (failed under tension) of Ti-Cu bonded (at 973 K for 2 h).

Ti-Cu bonded at 973 K for 2 h. It is observed from the fractured surfaces that the mode of fracture of fracture is cleavage having sound metallurgical joint with minimum un-bonded areas.

3.2. Optical Microstructure of Base Metals, SEM Analysis of Bonded Joints and EDX

The microstructures of the base material and the interfaces resulting from joining were characterized by optical microscopy (OM) at 100 X magnification as well as using the scanning electron microscopy (SEM). Figure 8 and Figure 9 shows the optical microstructure of commercially pure Ti and Cu plates. Figure 10 shows the SEM image of diffusion bonded Ti-Ti plates at 1073 K for 2 hours under a static force of 250 N indicating the concentration profile across the interfaces resulting from bonding. In the case of Ti-Ti plates diffused at 1073 K at 1 hour, the bond centerline consisted of Ti-Ti matrix with small particles of Ti-Ti solid solution just outside the central region. The bond region farthest from the center line consisted of only a solid solution of Ti-Ti. As the bond time is increased to 2 hours at 1073 K, the same microstructure appeared in the bond region but with an increase in the bond width. Note that as the temperature increases the solid solubility also increases. In the case of Ti-Ti diffusion bonding, the formation of intermetallics in the bonding area was totally absent. When Ti

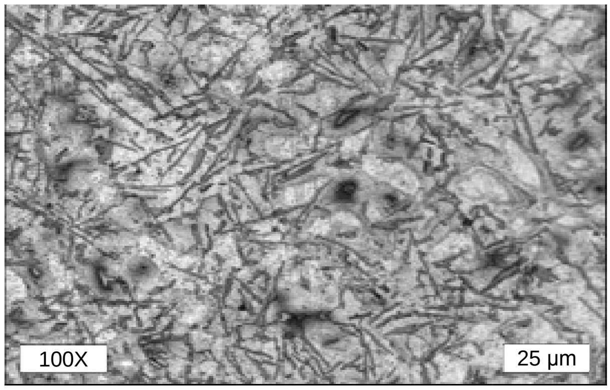

Figure 8. Microstructure of commercially pure Ti plate (grade-2).

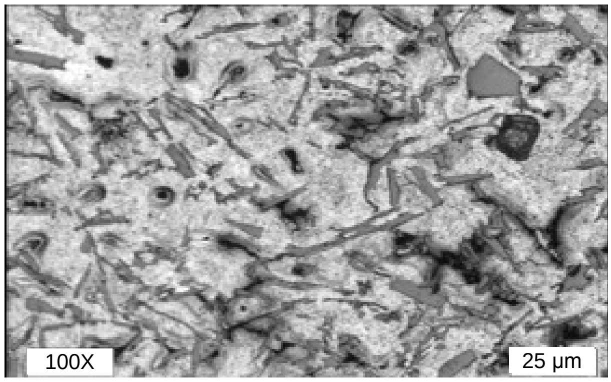

Figure 9. Microstructure of commercially pure Cu plate.

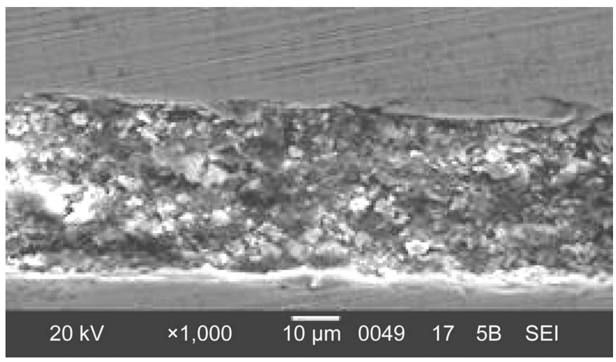

Figure 10. SEM photograph of diffusion bonded Ti-Ti plates (at 1073 K, 2 hours at 250 N).

is bonded with Ti, the bond line was not clearly visible until it was etched (8% HF and 4% HNO_3) and it was not continuous (see **Figure 10**) having some regions completely bonded while few voids were present elsewhere.

Figure 11 shows the cross section of the diffusion bonded Ti-Cu joint at 973˚C for 2 hours under a static force of 250 N. Although Cu form intermetallics with Ti, microstructure of the bonded zone was different when Ti-Cu was bonded. It is to be remembered here that intermetallics are brittle and they increase hardness by reducing the bond strength a little. At a temperature of 973 K for 2 hours, the bonding zone consisted of a region of Ti-Cu solid solution, a

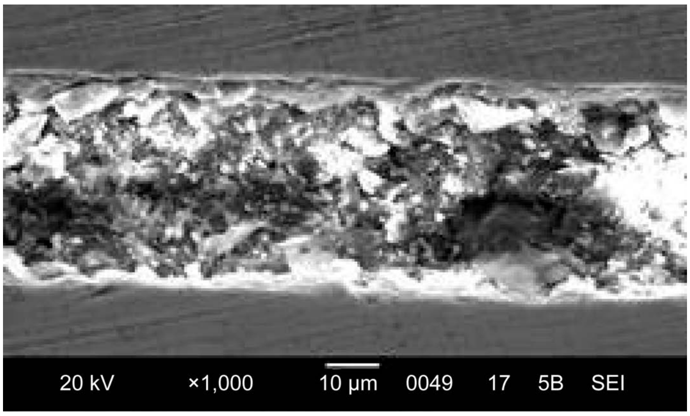

Figure 11. SEM photograph of diffusion bonded Ti-Cu plates (at 973 K, 2 hours at 250 N).

region of different intermetallics and a region of eutectic mixture of Ti (Figure 11). Here the composition of Cu was discontinuous in the intermetallics region depending on the composition of the intermetallics. When the bonding time is further increased beyond 2 hours (at 973 K), the continuous region of different intermetallics disappeared from the bond centerline and the centerline consisted of only the eutectic mixture of Ti and Cu. Although the melting temperature of Cu is 680˚C (or 953 K), no evidence was found the existence of residual Cu in the bond region at 973 K or little above. Finally, the bond centerline consisted of Ti-Cu solid solutions, eutectic mixtures and some small areas of intermetallics, possibly Ti_3Cu. However at 973 K at 1 hour some pores appeared in the bond region and the pore size decreased a little at higher temperatures.

In both the cases (Ti-Ti and Ti-Cu) the concentration profile indicate that there is a diffusion flux of one atom towards the other in opposite direction and is controlled by grain boundary diffusion. Figure 12 and Figure 13 shows EDX analysis photographs of diffusion bonded Ti-Ti and Ti Cu at 1073 K and 973 K for 2 hours.

3.3. Hardness of Diffusion Bonded Joints

To check the mechanical property of the bonded samples, micro hardness tests were performed. Tables 1-4 show the micro hardness of the bonded samples of Ti-Ti and Ti-Cu bonded plates at different temperatures and time duration. It is observed that the maximum hardness was obtained when Cu was bonded with Ti at 973 K for 2 hours was 367.15 HV. This might be due to the presence of intermetallics (not continuous) that are brittle has increased the hardness.

Hardness test results of Ti-Ti plates bonded at 1073 for 1 - 2 hours are shown in Table 1 and Table 2. The hardness increased with temperature up to 2 hours of bonding time but decreased when the bonding time was increased beyond 2 hours. The maximum hardness of Ti-Ti achieved was 244.14 HV at 1073 K for 2 hours. Figure 14 and Figure 15 shows micro hardness test indentation marks for Ti-Ti and Ti-Cu bonded samples near the bond region at a temperature of 1073 K and 973 K for 2 hours respectively.

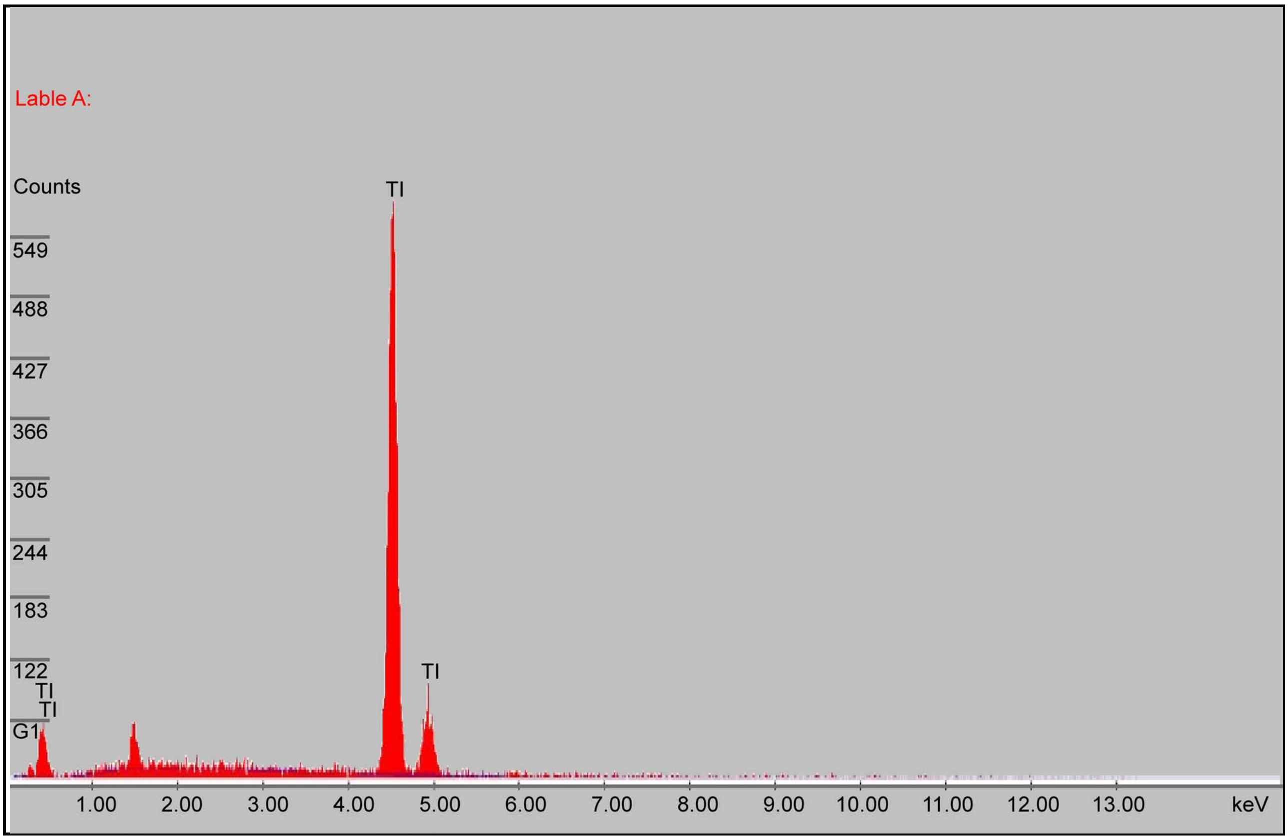

Figure 12. EDS photograph of diffusion bonded Ti-Ti and (at 1073 K for 2 hours).

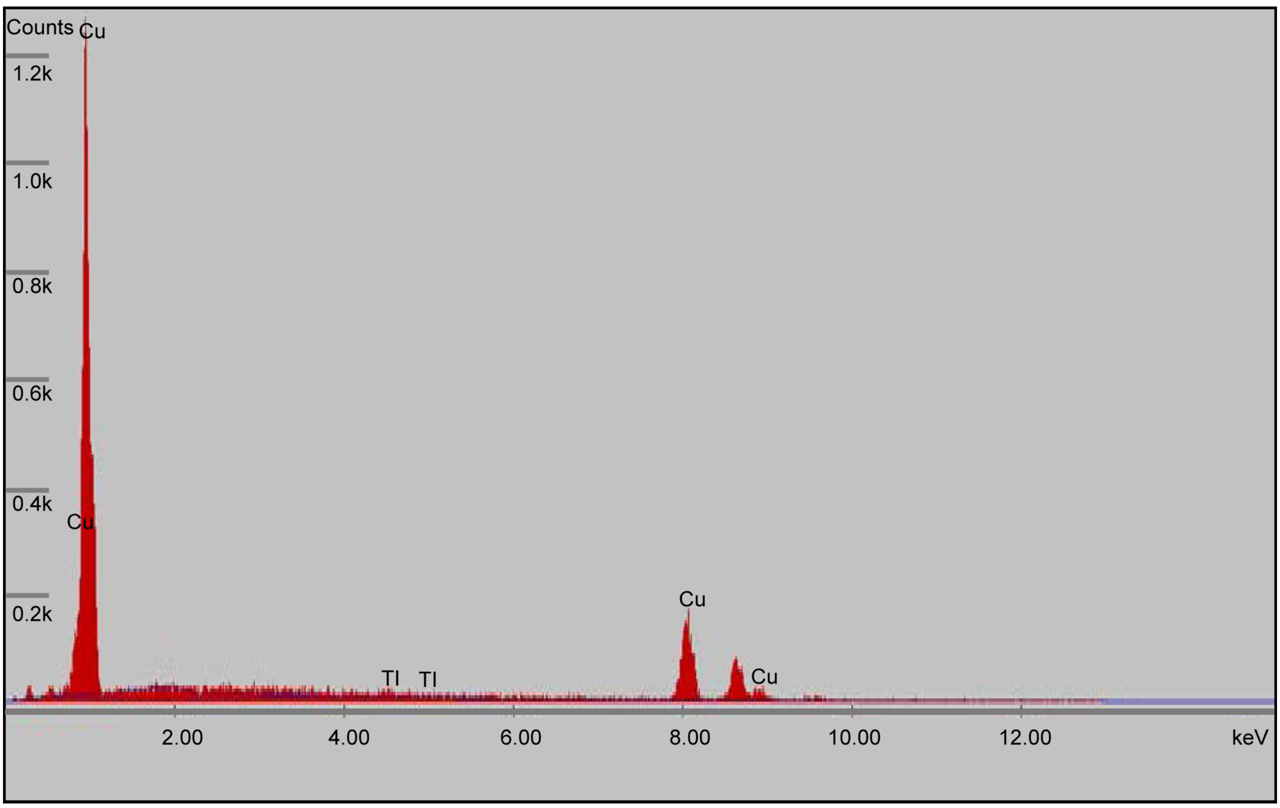

Figure 13. EDS photograph of diffusion bonded Ti-Cu and at (973 K for 2 hours).

Table 1. Micro hardness test results of Ti-Ti bonded samples (at 973 K for 1 hour).

location	Right side	Left side
50 μm	209.39 HV0.3	209.39 HV0.3
150 μm	197.36 HV0.3	201.86 HV0.3
250 μm	204.64 HV0.3	206.52 HV0.3

Table 2. Micro hardness test results of Ti-Ti bonded samples (at 973 K for 2 hours).

location	Right side	Left side
At 50 μm	244.14 HV0.3	216.33 HV0.3
At 150 μm	198.25 HV0.3	207.47 HV0.3
At 250 μm	218.37 HV0.3	209.39 HV0.3

Table 3. Micro hardness test results of Ti-Cu bonded samples (at 1073 K for 1 hour).

location	Right side	Left side
50 μm	273.31 HV0.3	297.93 HV0.3
150 μm	231.28 HV0.3	309.77 HV0.3
250 μm	222.55 HV0.3	229.05 HV0.3

Table 4. Micro hardness test results of Ti-Cu bonded samples (at 1073 K for 2 hours).

location	Right side	Left side
50 μm	331.75 HV0.3	367.15 HV0.3
150 μm	339.61 HV0.3	367.15 HV0.3
250 μm	315.05 HV0.3	364.91 HV0.3

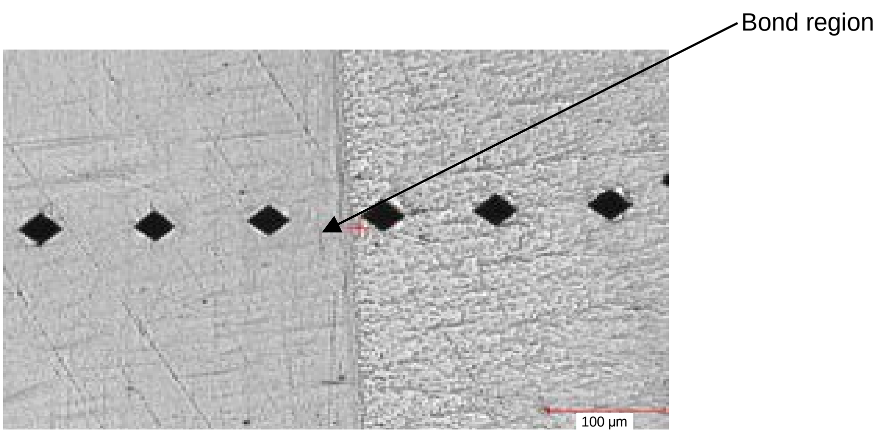

Figure 14. Micro hardness test indentation marks for Ti-Ti bonded sample (at 1073 K for 2 hours).

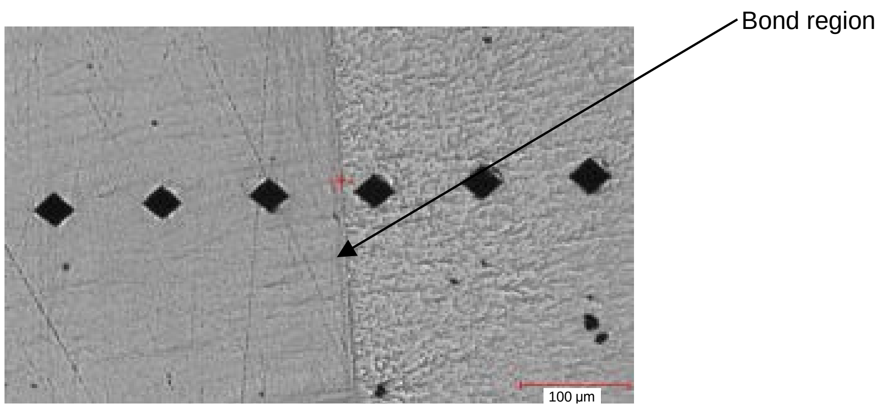

Figure 15. Micro hardness test indentation marks for Ti-Cu bonded sample (at 973 K for 2 hours).

4. Conclusion

The present research indicated the diffusion bonding of commercially pure Ti and Cu plates under a static force without any interlayers. Bond hardness with minimum pores mainly depends on the bond temperature (1073 - 973 K), the bond time (1 - 2 h) and static force applied (250 N) applied during bonding. Use of vacuum and diffusion pump avoids reaction due to oxidation during bonding. The maximum hardness (367.15 HV) was obtained in the case of Ti-Cu bonded plates and the joint reached 53% of the hardness of the base metal. This is mainly because of formation of continuous band of hard intermetallics. Hardness was low in the case of Ti-Ti bonded plates only solid solution of Ti-Ti was appeared but no intermetallics was found in the bonded region. In addition to bonding temperature, bonding time, static force, surface preparation was found to be very important for atomic diffusion and hence the bond hardness.

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