



## Effects of ion implantation on the brazing properties of high purity alumina

Haiyang Xia, Aiping Wu<sup>\*</sup>, Yinglong Fan, Guisheng Zou, Jialie Ren

Key Laboratory for Advanced Materials Processing Technology, Ministry of Education, Department of Mechanical Engineering, Tsinghua University, Beijing 100084, PR China

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### ABSTRACT

In this study, ion implantation was used as a surface modification method for active and inactive brazing of alumina ceramics to metals. Alumina was implanted with Ti ions at acceleration voltages of 35 kV and 55 kV at doses ranging between  $2 \times 10^{17}$  and  $1 \times 10^{18}$  ions/cm<sup>2</sup>, with Ni ions at an acceleration voltage of 55 kV at doses ranging between  $2 \times 10^{16}$  and  $6 \times 10^{17}$  ions/cm<sup>2</sup>, and with Al ions at 55 kV with a dose of  $2 \times 10^{17}$  ions/cm<sup>2</sup>. After implantation, the brazing of alumina to Nb was performed using the active brazing metal Ag<sub>70</sub>Cu<sub>27</sub>Ti<sub>3</sub> (wt.%) at 850 °C/870 °C and the inactive brazing metal Ag<sub>72</sub>Cu<sub>28</sub> (wt.%) at 830 °C in a vacuum respectively. The surface properties of implanted alumina, e.g., implanted ions depth and concentration distribution, newly formed phases, sheet resistance etc., were studied by Rutherford backscattering (RBS), Glancing X-ray diffraction (GXR) and four-probe method. Shear strengths of the active and inactive brazing joints were measured. Microstructures of the joints were analyzed by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). There is no obvious evidence that Ti and Ni ion implantation can enhance the quality of active brazing joints. However, it has been found that Al ion implantation can significantly increase the shear strengths of active brazing joints to average value 139 MPa, which is 30% greater than in non-ion implantation (107 MPa). It has also been found that ion implantation can improve the shear strengths of alumina–Nb inactive brazing joints. Maximum average shear strength of the inactive brazing joints can reach 43 MPa when Ni ions were used at an acceleration voltage of 55 kV in a dose of  $2 \times 10^{17}$  ions/cm<sup>2</sup>. Finally, the effects of ion implantation on alumina–Nb active and inactive brazing were discussed.

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### 1. Introduction

Alumina ceramics have high mechanical strength with low electrical conductivity and excellent resistance to corrosion and wear when exposed to high temperatures or corrosive environments [1]. Alumina ceramics have wide applications in electrical and electronic industries, vacuum fields and metallurgical and wear resistance areas, such as internal combustion engines, industrial heat exchangers and wear resistance materials for metal processing [2]. In some particular applications, for example, the components of ultra-high vacuum require high purity and fine grained alumina ceramics [1]. Alumina ceramics must often be bonded to other ceramics or metals for their applications. Mo–Mn method and direct active brazing are two widely used techniques for the joining of alumina ceramics to itself or to metals. The Mo–Mn method requires two processing steps, namely, metallization of the ceramics which is conducted at a high temperature (about 1500 °C) and the subsequent inactive brazing. Mo–Mn method is often used for the joining of normal purity alumina ceramics to metals and is usually readily available [3–6]. However,

high purity and fine grained alumina ceramics are hard to be Mo–Mn metalized due to lack of glass phase.

Direct active brazing is a particularly simple and cost-effective ceramics joining technique and is often used for the joining of high purity and fine grained alumina [7]. A popular active brazing alloy is based on Ag–Cu eutectic alloy with a few Ti atoms addition. The active element Ti can react with O in alumina to form titanium oxides which are wettable for brazing metals. However, addition of the active element Ti slightly raises the melting point, reduces the fluidity and embrittles the alloy [8]. Furthermore, the active element is difficult to disperse uniformly throughout the brazing filler metal. The non-uniformity of its distribution can lower the quality of the active brazing joints. Most importantly, the brittle reaction layer which is formed between the brazing filler metal and alumina is the key to bonding. The thickness, constituents and phase composition of this layer have a strong impact on the properties of active brazing joints. The formation of the reaction layer may also induce residual stresses in the near surface region of alumina, so the active brazing joints are mostly fractured at the interface between reaction layer and alumina or in the ceramic subsurface during shear strength test. Therefore, controlling the thickness, constituents and phase composition of the reaction layer and reducing residual stresses at the subsurface of alumina is the key to enhancing the active brazing joints of alumina to metals. Ion implantation into the surface region

<sup>\*</sup> Corresponding author. Tel./fax: +86 10 62773859 2.  
E-mail address: [wuaip@tsinghua.edu.cn](mailto:wuaip@tsinghua.edu.cn) (A. Wu).

**Table 1**  
The ion implantation parameters.

Ion species	Acceleration voltage (kV)	Beam current density (mA/mm <sup>2</sup> )	Ion dose (ions/cm <sup>2</sup> )				
Ti	35	3	–	–	–	6 × 10 <sup>17</sup>	–
	55	3	–	–	2 × 10 <sup>17</sup>	6 × 10 <sup>17</sup>	1 × 10 <sup>18</sup>
Ni	55	3	2 × 10 <sup>16</sup>	6 × 10 <sup>16</sup>	2 × 10 <sup>17</sup>	6 × 10 <sup>17</sup>	–
Al	55	3	–	–	2 × 10 <sup>17</sup>	–	–

of alumina prior to active brazing may be a potential method to accomplish this.

Ion implantation into alumina can affect its mechanical properties, such as, hardness, elasticity, strength and fracture toughness [9,10]. The implanted ions could result in the temporary or permanent distortions in alumina lattices. These results depend on the ion species and implantation conditions (energy, dose and temperature). Several effects of ion implantation have been observed, such as formation of new phases from the initial lattice, local or global amorphization and precipitation of the implanted species. Moreover, ion implantation leads to an increase in the volume of alumina and to residual compressive stresses in the implanted layers [11,12]. The increase of fracture toughness, the formation of new phases and the generation of residual compressive stresses may have positive effects on the active brazing of alumina to metal.

Only several studies on the effects of ion implantation on the inactive brazing of ceramics to metal have been reported until now. M. Barlak et al. used ion implantation as a pre-treatment method for inactive bonding of aluminum nitride with copper and found that the joints formed on ion implanted pre-treated aluminum nitride exhibited superior quality compared with conventionally treated material [13,14]. B.R. Zhao et al. studied the influence of Ni ion implantation on the inactive brazing of alumina ceramics with AISI321 and concluded that the strength of joints increased with ion dose, peaking at the dose of  $5 \times 10^{16}$  ions/cm<sup>2</sup>. After this, the strength of the joints decreased upon further increasing the dose [15]. M. Samandi et al. investigated the effect of titanium ion implantation on inactive brazing of alumina ceramics to metal [16]. However, previous references reveal that the effects of ion implantation on inactive brazing of alumina have been disputed and are still somewhat unclear. Moreover, the research on the influence of ion implantation in active brazing has not yet been reported.

The main objective of this study is to improve the surface properties of alumina by ion implantation to affect the interface reaction during brazing and eventually enhance the properties of the active brazing joints. The other aim is to modify the alumina surface via ion implantation metallization so as to realize the inactive brazing potential. In both cases, the influences of ion implantation parameters (ion species, energy and dose) on the joint properties were investigated.

## 2. Experimental procedure

The substrate material used was a commercial alumina of 99.5% purity (polycrystalline  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>). The dimensions of rectangular alumina specimens were  $5 \times 5 \times 10$  mm<sup>3</sup>. The size of the implantation brazing surface was  $5 \text{ mm} \times 10 \text{ mm}$ . The active brazing filler metal used in this study was Ag<sub>70</sub>Cu<sub>27</sub>Ti<sub>3</sub> (wt.%) alloy foil with a thickness of 0.1 mm. Ag<sub>72</sub>Cu<sub>28</sub> (wt.%) eutectic alloy foil with a thickness of 0.1 mm was used in inactive brazing. Nb was chosen to be joined to alumina in both active and inactive brazing because its thermal expansion coefficient is similar with the alumina. The dimensions of Nb were the same as those of alumina.

A metal vapor vacuum arc (MEVVA) ion source was used for ion implantation. Ti, Ni and Al ions were chosen to implant into alumina. The ion implantation parameters, including the ion species,

acceleration voltage, beam current density and dose, are shown in Table 1. The temperature of the alumina was about 300 °C during ion implantation. The base pressure of the implanter was below  $1 \times 10^{-3}$  Pa.

The alumina was bonded to Nb after ion implantation by respective active and inactive brazing. Active brazing used Ag<sub>70</sub>Cu<sub>27</sub>Ti<sub>3</sub> (wt.%) alloy at 850 °C or 870 °C, while inactive brazing used Ag<sub>72</sub>Cu<sub>28</sub> (wt.%) alloy at 830 °C. Both the active and inactive brazing was conducted in a vacuum furnace evacuated to an initial pressure of  $1.5 \times 10^{-3}$  Pa. The holding time for the active and inactive brazing was 20 min each. A small pressure of 0.04 MPa was applied to maintain good contact with the interface.

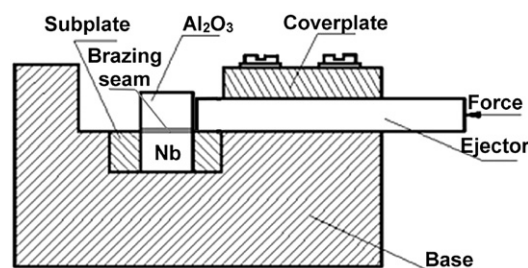
The implanted ions depth and concentration distributions were characterized by Rutherford backscattering (RBS) using a 2.011 MeV He<sup>2+</sup> beam with a backscattering angle of 165°. Glancing X-ray diffraction (GXR) was performed to identify the phases of the implanted layers. A four-point probe method was adopted to monitor the change of sheet resistance on the implanted alumina.

The microstructures of the active and inactive brazing joints were studied by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). Shear strengths of the specimens were tested by Gleeble 1500D. The schematic illustration of shear strength test is shown in Fig. 1. The average value of five specimens (having the same ion implantation and brazing parameters), was used as the average shear strength for each parameter condition.

## 3. Results

### 3.1. The effects of ion implantation on alumina ceramics

The concentration profiles of implanted Ti and Ni, derived from the RBS spectra, are presented in Figs. 2 and 3 respectively. Fig. 2 shows how depth of the peak concentration increases with increasing acceleration voltage. When the same acceleration voltage of 55 kV was used, the Ti concentration profiles are approximately the same at doses of  $2 \times 10^{17}$ ,  $6 \times 10^{17}$  and  $1 \times 10^{18}$  ions/cm<sup>2</sup>. In contrast, the maximum concentration of Ni rises as the implantation dose increases from  $2 \times 10^{16}$  ions/cm<sup>2</sup> to  $2 \times 10^{17}$  ions/cm<sup>2</sup>. One possible explanation is that the implanted Ti ions are saturated at the dose of  $2 \times 10^{17}$  ions/cm<sup>2</sup>, which means more ions can't be implanted into the substrate at the acceleration voltage of 55 kV. The maximum concentrations of Ti and Ni implanted at a dose of  $2 \times 10^{17}$  ions/cm<sup>2</sup> are about 25% at a depth of 25–35 nm.



**Fig. 1.** Schematic illustration of the shear test used to assess shear strengths of active and inactive brazing joints.

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