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Joining of sapphire and hot pressed Al_2O_3 using $Ag_{70.5}Cu_{27.5}Ti_2$ brazing filler metal

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Abstract

Sapphire and hot-pressed 99% Al_2O_3 ceramic were joined using $Ag_{70.5}Cu_{27.5}Ti_2$ bracing filler metal in a vacuum electric furnace. The interface reaction between $Ag_{70.5}Cu_{27.5}Ti_2$ alloy and sapphire, hot-pressed Al_2O_3 ceramic during brazing is reported. The joining strength and the airtight of the specimen are influenced by the surface condition of Al_2O_3 ceramic and the factor of active brazing condition.

Keywords: A. Joining; B. Interfaces; D. Al_2O_3; Sapphire; $Ag_{70.5}Cu_{27.5}Ti_2$ alloy

1. Introduction

In recent years, the sapphire plays an important role in electronic industry because of its superior physical, chemical, mechanical and optical properties. The joining of the sapphire to ceramics is of importance to practical application of the ceramic. There are two principle difficulties in joining two different material: the poor wettability of most convenient brazing filler metal on ceramics, and a very high residual stress around the ceramic–metal interface. Ti as an active metal component of brazing alloys has been used on most occasions [1–9], in this paper, we use $Ag_{70.5}Cu_{27.5}Ti_2$ alloy to brazing the sapphire and alumina ceramic.

The maim aim of this investigation is to study the wetting behavior of $Ag_{70.5}Cu_{27.5}Ti_2$ alloy on different surface profile ceramic and the relationship between the annealing and the joining strength. It is also discussed what the interface reaction have happened between the brazing filler metal and alumina ceramic.

2. Experimental

2.1. Materials

The hot-pressed sintered 99% alumina used in this study was obtained from Tsinghua University, the sapphire was obtained from the Tianjin Institute of Semiconductor. The size of 99% alumina pieces was showed in Fig. 1, the size of sapphire pieces was Φ 10.0 mm×500 μm. The alumina joining face was polished with sand paper with abrasive number 150, 400, 600, 800, 1200, and diamond paste, respectively. $Ag_{70.5}Cu_{27.5}Ti_2$ brazing filler metal was rolled into 500 μm strip and cut into an appropriate size for brazing. The brazing filler metal, 99% alumina ceramic and sapphire were carefully cleaned with acetone and baked before joining.

2.2. Joining experiments

The test sample for a butt joint, consisting of two pieces of alumina separated by a layer of sapphire (the materials between them is the brazing filler metal), was fixed in an iron jig. The jig was heated in a vacuum furnace to brazing temperature and then held some time. The heating rate below 750 °C was 20 °C/min, and then 10 °C/min to the brazing temperature. The cooling rate after brazing was 5 °C/min above 600 °C and then held some time, then about 2 °C/min to room temperature. The pressure of the furnace chamber was kept between 1×10^{-4} and 1×10^{-5} par during the heating and the cooling.

2.3. Mechanical tests and microscopy

The joining strength of the butt joint was determined by tensile tests (Fig. 2) with a crosshead speed of 0.1 mm/

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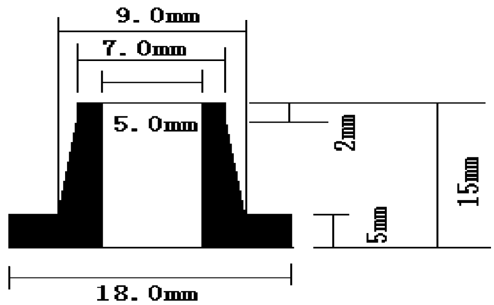

Fig. 1. Size of alumina ceramic.

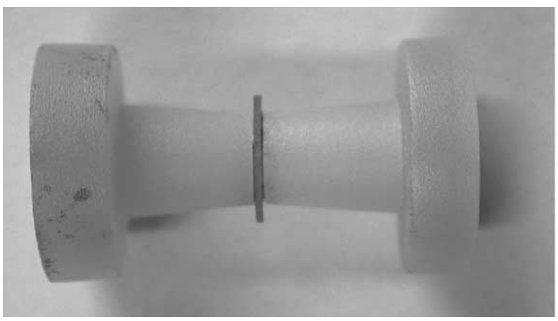

Fig. 2. Photograph of tensile testing sample.

min at room temperature. The supersonic microscopy was used to detect the macroscopic defection. Helium Leak Detector QualyTest HLT 270 tested the leakage rate of joint.

The distribution of the elements in the unbroken joint was examined by scanning electron microscopy (SEM). The faying surface was analyzed by X-ray diffraction (XRD) to identify the phases.

3. Results and discussion

3.1. Effect of the ceramic surface profile

Many alumina parts are polished by sand paper, so they have different surface profile, it would influence the adhesion of alumina–sapphire joint. Fig. 3 shows the sand paper number polishing alumina dependence of the joining strength of alumina–sapphire joints using $Ag_{70.5}Cu_{27.5}Ti_2$ brazing filler metal. Fig. 4 shows the relationship between the sand paper number polishing alumina and the leakage rate of alumina-sapphire joint. The maximum joining strength was 62.59 MPa, the minimum leakage rate of alumina-sapphire joint is lower than 5.00×10^{-13}Pa m^3/S.

From Figs. 3 and 4, we observe that the fine polishing material can improve the joining strength and decrease the leakage rate obviously. As the polishing material is changed from the abrasive number 150 to diamond paste, the ceramic surface is becoming more fine (Ra from 4.4057 to 0.1620), but the sapphire surface profile is fixed (surface roughness is 0.25 nm), so the contact angle between brazing filler metal and alumina ceramic is the key factor to influence the joining of sapphire–alumina. When we use the No. 150 sand paper polishing the alumina joining surface, the brazing filler metal can not wet it well; but if we use diamond paste, the brazing filler metal can wet alumina surface and fill the convex-concave of alumina surface well, so we can gain the high joining strength and low leakage rate by polishing ceramic surface (62.59 MPa, 5.00×10^{-13}Pa m^3/S).

We measure the contact angle between brazing filler metal and alumina polished by diamond paste just 7.13926°. Fig. 5 is a photo of supersonic microscopy of joining interface (scan speed is 8.0 in/s); we can see there are some holes in the interface (if there are some holes, the sound wave will be echoed, 1, 2 and 3 are the hole's echo), the brazing filler metal wets the ceramic surface not very well, it decreases the joining strength of sapphire–alumina, and it also increases the leakage rate of them.

3.2. Effect of the welding condition on joining strength

Fig. 6 shows the relationship between the joining strength and leakage rate of joining temperature, polished by diamond paste, and the holding time is 6 min, no annealing. Fig. 7 shows the relationship between the annealing time and joining strength, joining temperature is at 855 °C. From Fig. 6, we observe that the joint parts can gain the good joining strength and low leakage rate when the joining temperature is between 855 and 857 °C. The maximum joining strength can reach 62.5 MPa, and the minimum leakage rate can reach 5.00×10^{-13} Pa m^3/S. From Fig. 7, we know that the annealing can improve the joining strength, and the higher the annealing temperature is, the higher strength it can reach. When the annealing time is longer than 12 h, the effect of improving strength will decrease, the main reason is that the residual stress can decrease obviously at the beginning of annealing, and as we prolong the annealing time, the residual stress will not decrease but the thickness of intermetallic compound layer will increase, so the joining strength will decrease slightly. Fig. 8 is the photo of joint after annealing (polished, no etched), there are two thick intermetallic compound layers between brazing filler metal and ceramic, we analyze the element of them by scanning electron microscopy (wt: 34.14%O, 44.02%Al, 10.54%Ti, 7.11%Cu, 4.18%Ag), it is different from the brazing filler metal.

3.3. Interface between ceramic and brazing filler metal

Fig. 9 shows the transverse cross-section photo of sapphire/$Ag_{70.5}Cu_{27.5}Ti_2$/alumina ceramic (SEM). From Fig. 9, we observer that there is no obviously change on alumina ceramic, but there is an interface layer between brazing filler metal and ceramic.

Fig. 10 shows the element line distribution images of titanium and aluminum (SEM), and Fig. 11 shows element

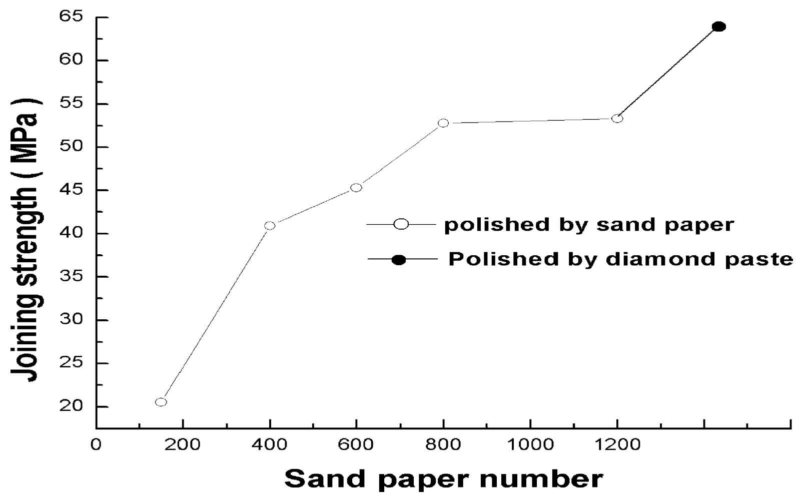

Fig. 3. Sand paper number vs joining strength.

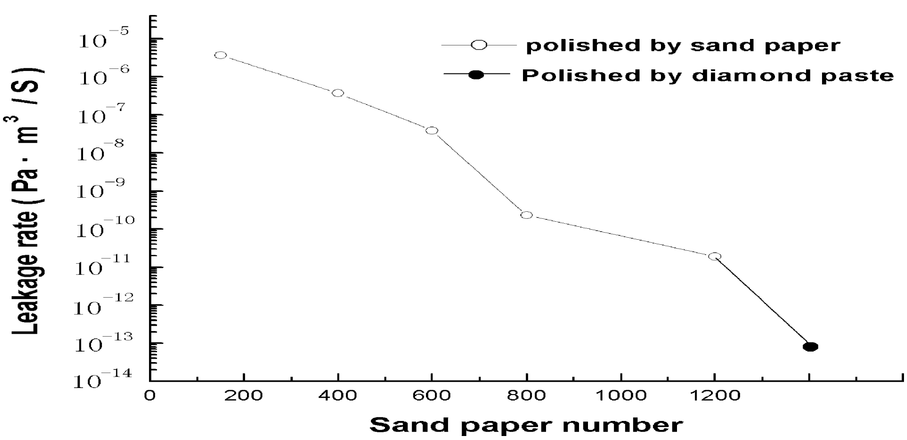

Fig. 4. Sand paper number vs leakage rate.

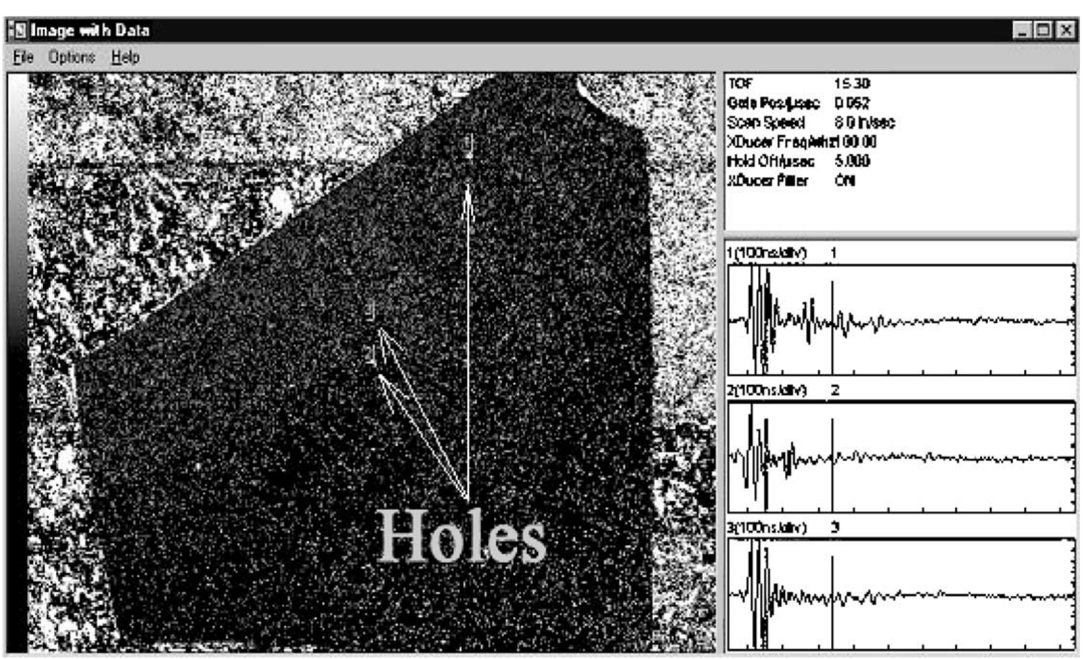

Fig. 5. Supersonic microscopy photograph of interface.

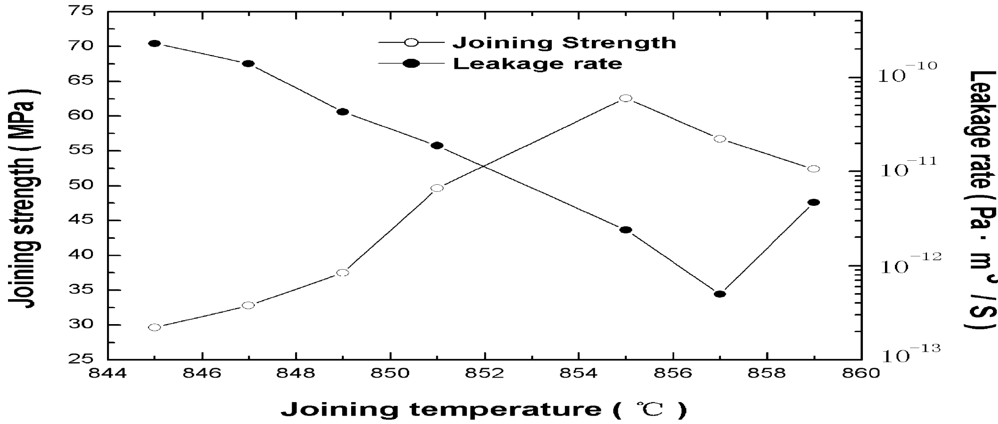

Fig. 6. Relationship between the joining strength and leakage rate of joining temperature.

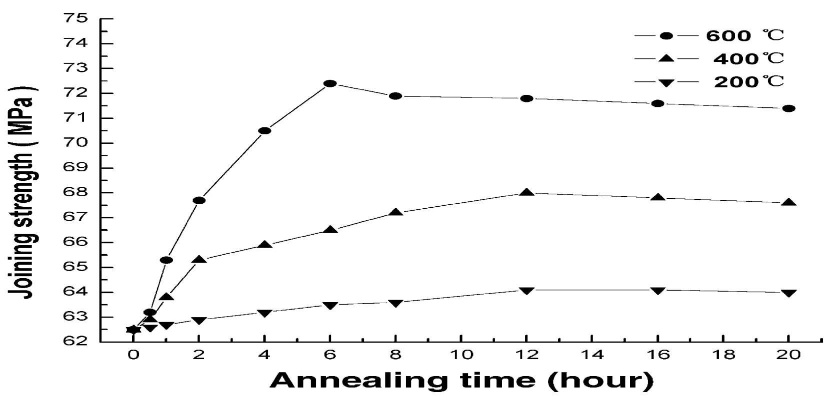

Fig. 7. Relationship between the annealing time and joining strength.

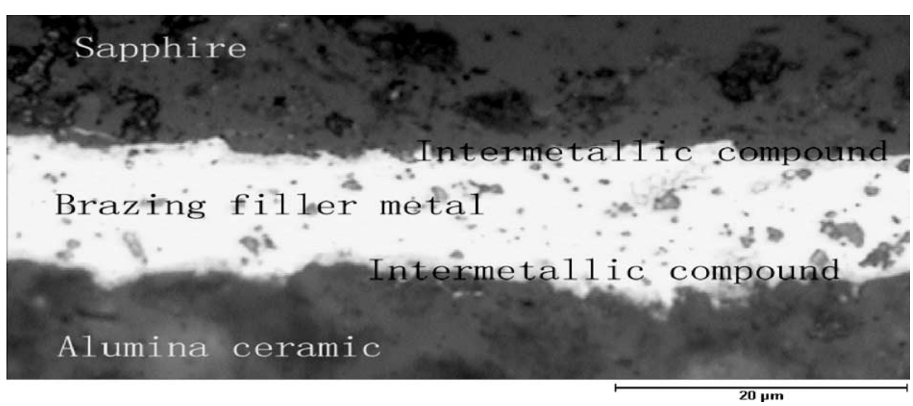

Fig. 8. Photograph of sapphire/brazing filler metal/alumina ceramic interface.

spot analysis of the braze metal of the joint (from alumina to sapphire). Fig. 12 is the X-ray diffraction pattern of the reaction interface.

From Figs. 10 and 11, we observe that the titanium element concentrates in the interface of metal and alumina, and the concentration of titanium in the middle of brazing filler metal is lower than nominal concentration (2%), there must be some reaction happened between the brazing filler metal and alumina, and titanium element in $Ag_{70.5}Cu_{27.5}Ti_2$ alloy diffuses to the reaction interface obviously in welding process, the thickness of reaction layer is about 2 μm. And we also can observer that the reaction layer thickness of sapphire side is thinner than the alumina side, so the fracture often occurred rigorously at the sapphire side when we do tension testing. From Fig. 12, we observe that there are some new phases different from the α-Al_2O_3 and AgCuTi alloy, they must be the product of reaction. The main titanium element shows as TiO, Cu_2Ti_4O and Ti, and the aluminum element exists as

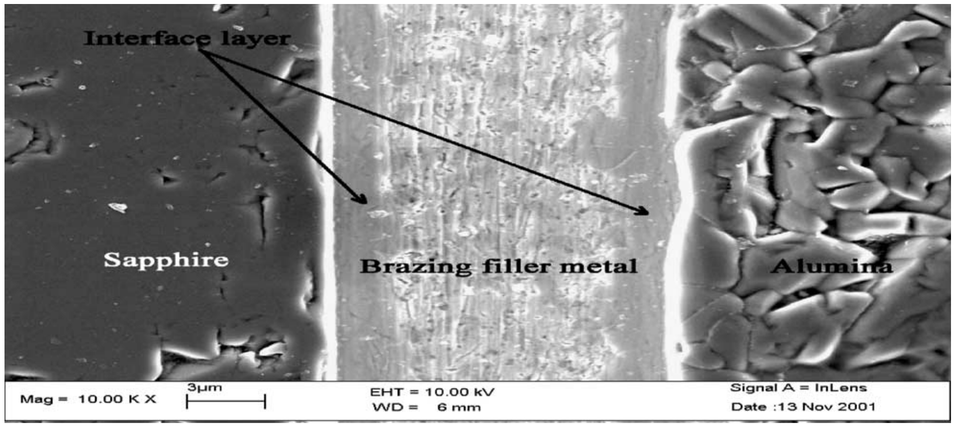

Fig. 9. SEM photograph of sapphire/AgCuTi/alumina ceramic (SEM).

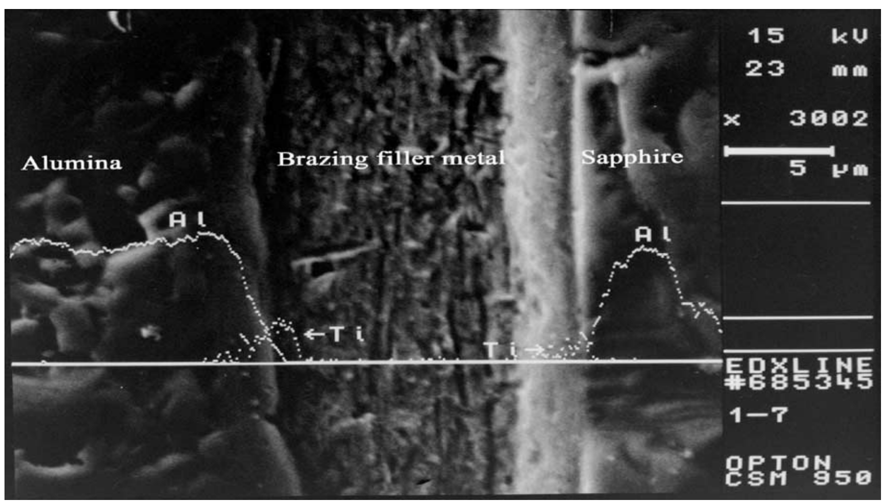

Fig. 10. Ti and Al line scanning of sapphire/AgCuTi/alumina ceramic (SEM).

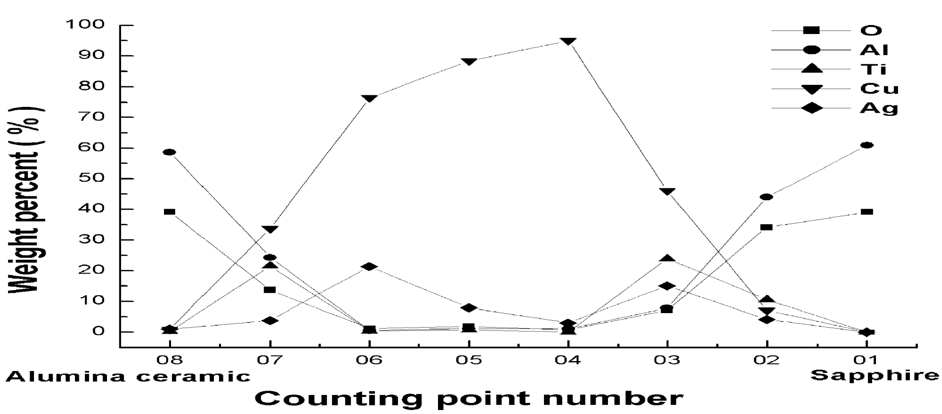

Fig. 11. Element spot analyses of sapphire/AgCuTi/alumina ceramic.

Al_2O_3 and $AlAg_3$. According to a hypothetical reaction [10]:

$$\mathrm{Ti} + x\mathrm{Al_2O_3} \rightarrow \mathrm{Ti(O)}_{3x} + 2x\mathrm{Al} \quad (1)$$

We can deduce the value of x is 1/3 in this reaction, and the original Ti/Al_2O_3 interface is replaced gradually by the titanium oxide/Al_2O_3 interface. The reaction product (TiO, Al) dissolves into Ag–Cu alloy, so we can find Cu_2Ti_4O and $AlAg_3$ phases on the reaction interface. Because there are titanium element existing on the reaction interface, we can deduce the main reason of fracture is due to insufficient interfacial reaction (1). When we prolong the holding time, the joining strength of parts can be improved because of .sufficient interfacial reaction (1).

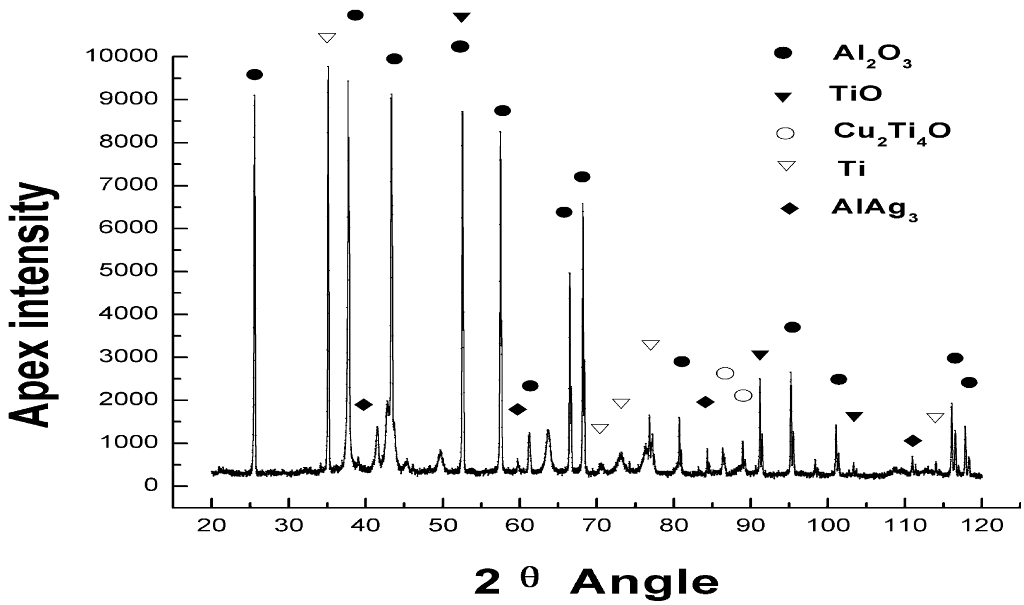

Fig. 12. XRD pattern of interface.

4. Conclusions

Sapphire was joined using $Ag_{70.5}Cu_{27.5}Ti_2$ brazing filler metal in a vacuum. The maximum joining strength of joint measured by tensile tests method was about 72.5 MPa when brazing at 855? for 6 min. The main reaction product between brazing filler metal and sapphire is TiO, Cu_2Ti_4O and $AlAg_3$.

From the above results, in order to obtain a high and airtight joint, the following points are suggested when one designs a brazing filler metal for joining sapphire to alumina ceramic:

1. The roughness of alumina ceramic is a key to the joining strength and leakage rate of sapphire–alumina joint.
2. The brazing temperature and time should be sufficient to ensure interface reaction of brazing filler metal with ceramic.
3. In some degree, the annealing can improve the adhesion strength by reducing the thermal stress.

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