Effect of Furnace Vacuum Pressure on the Joining Strength of Alumina Bonded Kovar

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Abstract

Ceramic to metal joining technique, which was used in this investigation includes the use of active filler alloy as a sandwich between the alumina and kovar alloy for brazing. High purity powdered metals of silver, copper, and additives of titanium were used to prepare the active filler alloy, through compacting the mixed powders and alloying in a furnace with argon atmosphere at the temperature of 800°C for 10 minutes. To use it as an active filler metal, it has been modified to a proper thickness. Two groups of alumina were prepared with different sintering temperatures (1450°C and 1650°C) and each group was tested under atmospheric pressure, vacuum furnace pressure of $2*10^{-4}$ torr and vacuum furnace pressure of $2*10^{-6}$ torr. All the process was at a same brazing temperature of 850C for 20 minutes. A reliable joint between the different types of alumina and kovar alloy was achieved by using vacuum furnace. On the other hand, weak bond was obtained by using atmospheric furnace. A maximum value of joint strength between alumina and kovar was obtained (130 MPa) under condition of $2*10^{-6}$ torr of vacuum furnace.

Keywords

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تأثير ضغط الفرن الفراغي على متانة ربط الالومينا الملتحمة بسبيكة الكوفار

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الخلاصه

استخدمت تقنية ربط السيراميك إلى المعدن في هذه الدراسة من خلال استخدام مالئة معدنية توضع بين السيراميك والمعدن لغرض لحامهما. استخدمت مساحيق عالية النقاوة من الفضة والنحاس وإضافات من التيتانيوم لتصنيع سبيكة المالئة بعد كبسهما وتسبكيهما في فرن يغذى بغاز الاركون عند درجة حرارة 800م ولمدة عشرة دقائق. أجريت عملية تهذيبا للسبيكة للحصول على سمك مناسب للاستخدام. تم تحضير مجموعتي ألومينا نقية أحداهما عند درجة حرارة تلبيد 1450م وأخرى عند 1650م على التوالي وذلك لربطهما مع سبيكة الكوفار باستخدام فرن يعمل تحت ظروف الضغط الجوي وآخر يعمل تحت ظروف الضغط الفراغي عند قيم $2*10^{-4}$ تور و$2*10^{-6}$ تور على التوالي بدرجة حرارة لحام 850م لمدة 20 دقيقة. تم الحصول على متانة ربط جيدة عند استخدام الفرن الفراغي بينما حصلنا على متانة ربط ضعيفة عند استخدام الفرن الاعتيادي. من خلال النتائج تبين ان أعلى قيمة ربط تم الحصول عليها بين السيراميك والمعدن هي 130 ميكا باسكال باستخدام الفرن الفراغي وتحت ضغط $2*10^{-6}$ تور.

Introduction

The mechanism of joining ceramic to metal is not fully understood. This is because some of the experimental factors which are employed affected the mechanism of the process. These factors are material matching, wettability of active filler for both ceramic and metal, furnace vacuum condition and so on [1]

Filler composed of (Ag – Cu – Ti) is one of the extensively used materials in electro-vacuum technology, because of its possession of the required properties ; low vapor pressure, satisfactory ductility, fluidity, and minimum gas generation. A vacuum furnace enables filler against resistance of oxidation and chemically inert [2]. M.A. Pavalova et al, used condition of $1.33*10^{-3}$ pa ----- $1.33*10^{-4}$ pa and reducing gas medium of $H_{\circ}$ and $H_2 + N_2$ to resist oxidation[3]. In his research, the nature of the dependence of strength of the bonded joints on pressure remains unchanged. In brazing the liquid brazing filler alloy diffuses along grain boundaries of the solid metal since the boundary atoms in the metal are characterized by a higher free energy than the mean energy of the surface [4]. However, when ceramic is brazed to metal, residual stresses may develop while the material is cooled from the brazing temperature to room temperature. These stresses are primarily due to the mismatch between coefficients of thermal expansion of the individual materials, and to a lesser degree to the mismatch of elastic properties of the ceramic and metal and the plastic flow of the filler metal[5].

Experimental

Two sets of pure alumina as a rectangular bars with dimension of (5×1.3×0.6) Cm^3 were fabricated for the joining purpose. One set was sintered at 1450 ̊C and the second set was sintered at 1650 ̊C using an atmospheric furnace. In order to obtain clean alumina surfaces, chemical cleaning was conducted by immersing the alumina pieces in a suitable chemical solution of dilute nitric acid and subsequent in distilled water. Finally, fired in an atmospheric furnace at the temperature 1000C for about one hour [6]. Kovar alloy sheets were cutted as a rectangular shape of dimension $(6*1*0.08)Cm^3$. It was necessary to remove any contaminated materials involved oxides layer by washing the kovar sheets in acetone and then by immersing in a suitable solution of ($0.125\%H_2SO_4$ + 0.15% HCl)with 50 gm ferric ammonium sulfate then in chromic acid solution. Finally rinsed in a hot distilled water and drying by air [7]. High purity powdered metals of Ag (63wt %), Cu(35 wt%) and Ti(2 wt%) were used to prepare the active filler alloy, through compacting the mixed powders and alloying in a furnace with argon atmosphere of 800C for 10 minutes. This method has been used previously in [8].To use it as an active filler metal, it has been modified to a proper thickness of 1mm. Active filler alloy was in between kovar strip and alumina as a sandwich form. A steel fixture was used to hold the assembly. Honeywell, vacuum furnace operated under two vacuum conditions, $2*10^{-4}$ torr and $2*10^{-6}$ torr respectively with the same brazing temperature of 850 ̊C for 20 minutes, was used to achieve the joining process. A reliable joint between kovar alloy and different types of alumina was inspected by using shear stress technique. Fractional surfaces were analyzed by using a non-destructive testing of both optical microscope (OM) and scanning electron microscope (SEM) techniques.

Results and Discussion

The average fracture stresses obtained for all fabricated joints are listed in table (1)

Table (1) Represents Fracture Modes and Fracture Strength Against Furnace Vacuum.

Alumina Sintering Temp. ̊C	Volume Porosity%	Furnace Vacuum	Fracture Mode	Fracture Strength M Pa
Group I Sintered at 1450 ̊C	7.49	Atmospheric Pressure	0	NILL
		$2*10^{-4}$ torr	MII	90±3
		$2*10^{-6}$ torr	MII	130±3
Group II Sintered at 1650 ̊C	4.03	Atmospheric Pressure	MI	12±3
		$2*10^{-4}$ torr	MI	93±3
		$2*10^{-6}$ torr	MII	120±3

Note:
Mode 0: Brazing joint was failed.
Mode I: Fracture propagated through filler interface with alumina or with kovar alloy.
Mode II: Fracture propagated through alumina.

Three modes of fracture are noticed (M0, MI and MII) in table (1). Due to oxidation layer forming at the contact area then no reliable joint is existed such as the results in the atmosphere furnace. The shear failure did not occur across the brazed joint; the specimen broke partially across the joints or broke across the alumina section as obviously noticed in modes (MI, MII). Shear test failures at loads in excess of up 90Mpa nearly always broke across the alumina rather than the brazed joint, indicating that the brazed joint was stronger than the alumina.

The modified shear test was developed to evaluate the active brazing filler metal brazed alumina to kovar alloy joint. The test results listed in table (1) showed that in order to obtain high reliabilibity brazed joints using active brazing filler alloy. Three influence variables are:

1- The alumina surface must be free of surface fractures.
2- Sufficient active element in the alloy to wet both kovar alloy and especially alumina.
3- The principle obstacle to be overcome in brazing ceramic is the fact that most metals do not easily wet the surface of most ceramics, these principles is agreement with many researchers.

The contact surfaces of both kovar and alumina must be free of any contaminated materials and then should be smooth without any obstacles. Because of molten filler alloy does not the perfect contact the actual alumina, these processes would be expected to be suitable for joining alumina to kovar.

During the brazing cycle, one can expected the element titanium segregates the alumina surface where it reacts to form a wettable surface. Depending on this process fact, the effective element titanium should be reacting with aluminum oxide to form compound by strong bonds.

Because of free oxygen is exist in the ambient furnace especially under low pressure (less than 10^{-4} MPa), then the oxygen can easily react with titanium forming a phases of TiOx at the interface of the braze filler. These TiOx have weakly bonds especially with alumina member. Considerably that the TiOx could not enable to wet the alumina surface.

Therefore the bonding becomes a weak as noticed previousely. These expected real could be clearly noticed on a bonding results between alumina and kovar alloy under the conditions of low pressure and atmosphere pressure as shown in the fig.(1).

Ti element can act as a getter for the vapor, N2 or O2 and developing on the degree of Ti oxidation, can alter the properties of the brazed joint especially bond strength. Under poor vacuum pressure, the greater part of the titanium can be converted to a brittle (high oxide) constituent, leaving of the metallic element for active brazing. On the other hand, the good adherence between the partners (alumina and kovar) is strongly dependent on the good wettability of the active filler to both kovar and alumina as clearly shown in fig.(2). According to the results, vacuum furnace with high vacuum pressure should be selected for a number of benefits are realized; one of these benefits is to reduce of oxide formation.

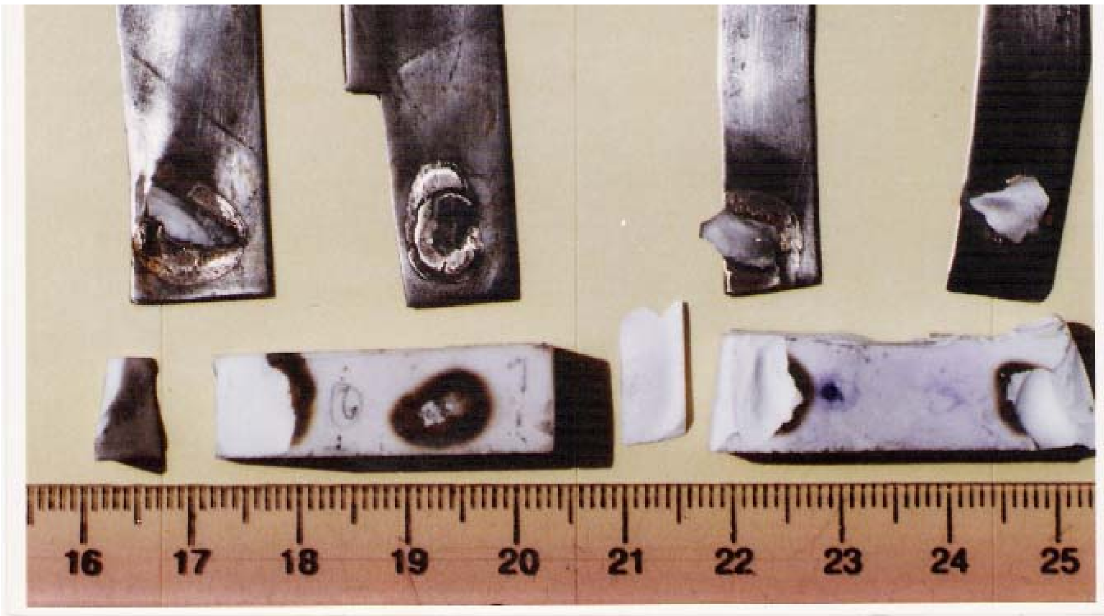

Fig.(1): Showed fracture Modes of Joints Between Alumina to Kovar.

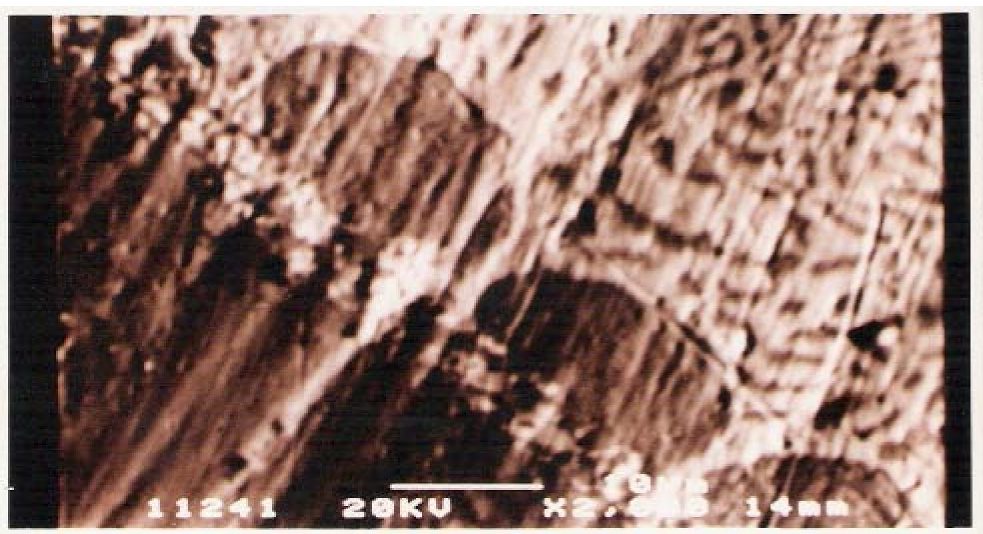

Fig.(2) Scanning Electron Microscopy of Interface Between Alumina to Kovar

Conclusions

Titanium, which has an affinity for oxygen, will react chemically with alumina, forming a boundary layer of Ti and TiO. A brazing metal can then be selected which will bored to the titanium and form the reliable joint.

Furnaces of high vacuum pressure is selected to reduce of oxygen formation and then to obtain a reliable joint.

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