Characterization of the ceramic–metal brazed interface using ultrasonic technique

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Abstract

Sintered Al2O3 is brazed with 304 stainless steel (SS) using 97(Ag28Cu)3Ti active filler alloy at 1000 °C. Electron Probe Micro Analysis (EPMA) studies of the interfaces are carried out. The thickness of the interfaces are measured from the concentration penetration profile. Shear strength of the joint is evaluated. The qualities of the joints are characterized using ultrasonic technique. Mechanical properties of the joints are correlated with the obtained ultrasonic signals during the ultrasonic scanning of the sample.

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1. Introduction

Ceramics has a very high potential to be used as structural materials and biomaterials in combination with high strength metals [1–11]. Making of sound metal ceramic brazed couple is still a challenge in terms of its direct application in the industry. It is observed that the microchemistry of the interface plays a vital role in determining the joint quality. Identification of the reaction products at the interface helps to understand the nature of the interface where as structure property correlation may lead to conclude the quality of the brazing. Kar and Ray have characterized the alumina–stainless steel (SS) brazed interface by Electron Probe Micro Analysis (EPMA) and Transmission Electron Microscopy (TEM) [12]. Reaction products at both the interface have been identified and the mechanical property (shear strength) of the interfaces has also been evaluated. It is realized that the evaluation of mechanical properties of the joint by nondestructive methods will serve the purpose of utilizing the joined component without affecting the physical nature of the joined sample. Ultrasonic technique has already been employed to characterize the bond quality of adhesive or metal–metal brazed components [13]. This paper aims to characterize the SS–Al2O3 metal–ceramic brazed interfaces using nondestructive ultrasonic technique and correlates the reflected ultrasonic signals with the shear strength of the joint.

2. Experimental

The starting materials used for the preparation of active filler alloy were Ag, Cu, and Ti with 99.9%, 99.9% and 99.5% purity respectively (all in wt.%). The alloy, 97(Ag28Cu)3Ti was prepared by melting and casting route. Conventional chemical analysis shows the concentration of Ag, Cu, and Ti in the filler alloy is 71.8 wt.%, 25.1 wt.% and 3.1 wt.% respectively. Alumina substrate was made by sintering Al16SG (Alcoa, USA) alumina powder, without any sintering aid, at 1600 °C using electric resistance furnace. The holding time for sintering was 30 min at the highest temperature. The density of the sintered alumina was 3.78 × 10⁻³ kg/m³. The sintered alumina was brazed to 304 stainless steel (Fe18Cr8Ni2Mn). The substrates and the filler alloy were cut by precision cut-off machine (MECATOME P100, Presi, France) using a diamond wafer blade. The dimensions and surface roughness of the materials used for joining are as follows: Al2O3 — 10(l)×8(b)×5(t) mm; 304 SS — 10(l)×8(b)×5(t) mm; filler alloy — 9(l)×7(b)×0.4(t) mm; roughness (Ra) of the Al2O3 is 0.46 μm and that of 304SS is 0.16 μm.

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Cut substrates and alloys were polished and cleaned ultrasonically using ethanol and acetone. The roughness of the cleaned substrates was measured by profilometer (Taylor Hobson precision, Taylsurf Series 2) having resolution of 16 nm. The filler alloy was sandwiched between the alumina and the stainless steel substrates. The assembly was placed in a graphite resistance furnace (ASTRO, Thermal Inc., USA). A fixed load of \( \sim 1 \) kg was kept over the sample to keep the assembly aligned during brazing. This enables a proper contact between the surface of the substrates and the filler alloy \([5,12–14]\). This load also facilitates the interfacial reaction \([15]\). The furnace was heated at the rate of 6 °C/min till it reached 1000 °C and kept for 15 min at that temperature. The cooling rate was maintained at 3 °C/min till it reached to 200 °C and thereafter furnace cooled. Before heating, the furnace was purged with argon gas (IOLAR I), and through out the brazing cycle \( \sim 50\)-milliTorr pressure was maintained by rotary pump.

The brazed samples were cut in order to get a fresh surface for characterization purpose. The cut samples were metallographically polished with 0.5 \( \mu \)m diamond paste and subjected to carbon coating for EPMA (JXA — 8600 M, Jeol, Japan). Line profile quantitative elemental analyses were carried out by EPMA across the brazed interface. A specially designed shear fixture has been used for shear test \([12]\).

Bonding quality at the interfaces was assessed by ultrasonic technique using a 200 MHz Pulser-receiver, Panametrics make. A 20 MHz longitudinal contact probe was used in pulse-echo mode for ultrasonic measurements. Measurements were carried out at different positions of the brazed specimen from one end to the other end at an interval of 0.25 mm. Signals were recorded placing the probe on both Al2O3 side and SS side. A constant load was applied to the probe during each measurement to avoid the error while measuring the amplitude of first reflected echo from the back wall, have been analyzed using indigenously developed software to determine the brazing quality of the interface. In order to carry out the shear test an Ultimate Tensile Testing Machine (UTM) (H10K-S, Hounsfield, 10KN capacity) was used. A cross head speed of 0.1 mm/min was applied during shear. Strength was determined by dividing the maximum load applied at which fracture takes place by the cross section area of the joint on which load is applied.

3. Results and discussion

3.1. Electron Probe Microanalysis (EPMA) of the braze joint

Fig. 1a and b exhibits the concentration penetration profile of the Al2O3–SS brazed interface. It has been reported elsewhere \([16]\) that the diffusivity of Ti controls the kinetics of the formation of the reaction products at the interface. Quantitative electron probe microanalyses (EPMA) of the Al2O3–SS brazed at 1000 °C suggests that the joining process is due to the interdiffusion of Ti, Ag and Cu towards the SS substrate and Fe, Ni, Cr towards the filler alloy leads to the formation of the SS interface. Similarly, the diffusion of Ti, Ag and Cu from the filler alloy towards Al2O3 and Al from Al2O3 towards the filler alloy forms the Al2O3 interface. From Fig. 1b it is observed that major amount of the titanium diffused towards the SS substrate leads to the formation of the SS interface, the diffusion zone observed from the EPMA (Fig. 1b).
is $\sim 15 \, \mu m$. This indicates that as a reactive species Ti reacts with the SS substrate, forms some intermetallic phases [12]. The diffusion zone at the Al$_2$O$_3$ side is observed to be $\sim 11 \, \mu m$ (Fig. 1a). Ti reacts and reduces the Al$_2$O$_3$ hence, Ti bearing phases observed [12] at the interface leads to the formation of alumina interface [12,17].

3.2. Ultrasonic testing

Acoustic impedance ($Z = \rho c$), i.e. the product of the ultrasonic velocity ($c$) through the material and the density of the material ($\rho$), has been determined by measuring ($c$) and ($\rho$) of each material, separately [18]. The acoustic impedances of Al$_2$O$_3$, filler material i.e. (Ag–Cu–Ti) and 304SS are determined as follows in Table 1.

Bonding quality at the interface of the Al$_2$O$_3$–(Ag–Cu–Ti)–SS brazed system is investigated by ultrasonic technique. After the brazing of alumina with SS using 97(Ag$_{28}$Cu$_3$Ti) active filler alloy, two different interfaces are developed adjacent to the alumina and SS substrates. When the ultrasonic wave is passed placing the probe on either of the substrate of the brazed couple, the incident wave will experience the presence of two interfaces (1 and 2) between three different materials with different acoustic impedances, which is schematically depicted in Figs. 2 and 3. During the transmission of ultrasonic wave through any material it will produce a reflected signal, only when it will experience any substantial impedance mismatch between the materials through which it is transmitting [19,20]. The bonding quality between SS and the filler alloy is assessed comparing the amplitude of the 1st reflected echo from the back wall placing the probe on SS substrate (inset Fig. 4). Similarly the bonding quality between alumina and the filler alloy is assessed comparing the amplitude of the 1st reflected echo from the back wall placing the probe on alumina substrate (inset Fig. 5). The amplitude of the reflected signal at different positions has been measured and plotted in a graph. Fig. 4 shows the amplitude plot as measured placing the probe on alumina substrate to assess the SS interface. The shear strength corresponding to five different positions as marked in Fig. 4, have been determined and found to be $479$, $480$, $483$, $484$, $484$ MPa for SS interface. Whereas the intensity plot, as measured placing the probe on SS substrate to assess the alumina interface and the corresponding shear strength values for alumina interface are found to be $90$, $90$, $92$, $95$ and $93$ MPa are depicted in Fig. 5, to correlate the strength with ultrasonic results. It has been observed that with the increase on the amplitude of the reflected signal from the backwall, interfaces exhibit the better shear strength values i.e. well bonded regions. The maximum amplitude of the reflected signal which corresponds to the position exhibits maximum shear strength.

4. Conclusions

Sintered alumina has been successfully brazed to 304SS at 1000 °C, using 97(Ag$_{28}$Cu$_3$Ti) active filler alloy. Two different interfaces formed i.e. alumina and SS are found to be of thickness of $\sim 11 \, \mu m$ and $\sim 15 \, \mu m$ respectively. During shear test, shear strength of the alumina interface and the SS interface have been evaluated. The shear strength results of the different positions of the brazed sample have been correlated with the ultrasonic reflected echo obtained from the corresponding positions. Well-bonded positions at both the interfaces produces higher amplitudes of the 1st reflected echo from the respective back walls. Hence it is concluded that the bonding strength of the ceramic metal brazed interfaces can be qualitatively assessed by nondestructive ultrasonic technique.

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