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Characterization of the ceramic–metal brazed interface using ultrasonic technique

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Abstract

Sintered Al_2O_3 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.

Keywords: Interface characterization; Brazing; Shear strength; Ultrasonics; Joining

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–Al_2O_3 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 A16SG (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/m^3. 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: Al_2O_3 — 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 Al_2O_3 is 0.46 μm and that of 304SS is 0.16 μm.

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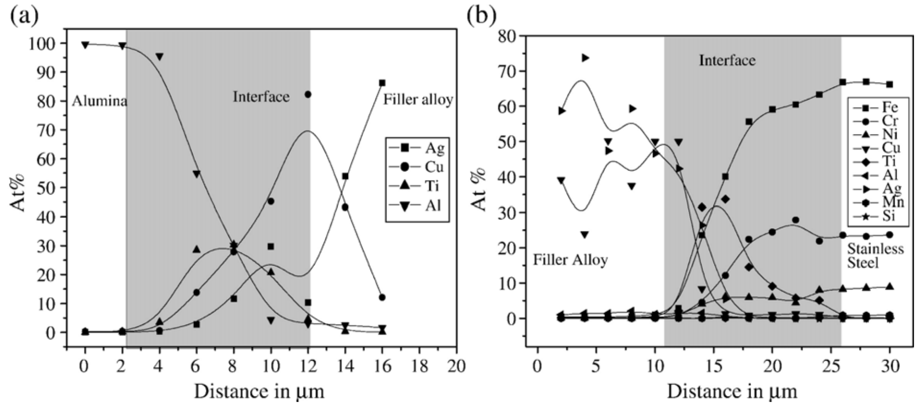

Fig. 1. EPMA line profile analyses of the Al_2O_3–304 SS brazed interface (a) Al_2O_3 interface and (b) SS interface. In both cases interface thickness has been considered with respect to the diffusion of Ti.

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 ~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 ~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 µm 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 Al_2O_3 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.

Table 1
Properties of the materials used as measured by ultrasonic technique

Material	Density (ρ), kg/m^3	Velocity (c), m/s	Impedance ($Z=\rho c$), $\times 10^6$ N s/m^3
Alumina	3780	10,046	~38
Filler material	8800	4107	~36
Stainless steel	7900	5826	~46

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 Al_2O_3 and SS 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 Al_2O_3–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 Al_2O_3 and Al from Al_2O_3 towards the filler alloy forms the Al_2O_3 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)

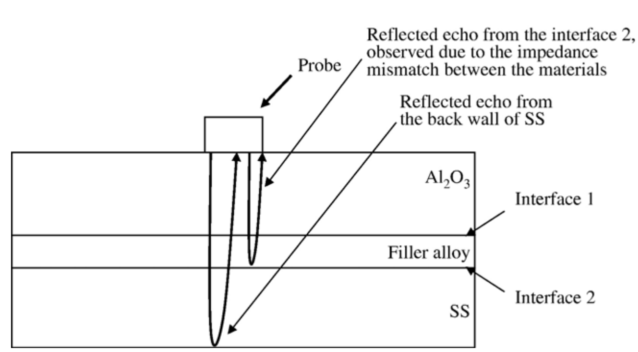

Fig. 2. Schematic representation of the ultrasonic measurement of the sample, while assessing the SS interface, amplitude of the back wall echo has been considered for the analysis.