

Diffusion Bonding of Alumina Ceramic and Austenitic Stainless steel

By

Tapan Kumar Pal

*Coordinator, Welding Technology Centre
Professor, Metallurgical Engg. Department
Jadavpur University, Kolkata*

INTRODUCTION

Ceramics are drawing more attention as materials for engineering devices having better heat resistance, abrasion resistance and corrosion resistance than metals. In many structural applications, specific characteristics of the ceramics have led to their selection for critical components forming parts of a total system that is largely metallic, and therefore depend for their success on the fabricator being able to form ceramic/steel joints of adequate quality [1]. Among the many techniques employed to join ceramics to steel, ranging from fusion welding to mechanical attachment, active metal brazing and diffusion bonding are generally preferred [2]. Applications of diffusion bonding have been increased since materials difficult to be fusion-welded, including combination of different kinds of material, are possible to be joined and precise fabrications of complex shapes of components are possible. Diffusion bonding involves the creation of a solid-state bond by interdiffusion across the interface between the two materials. However, the bonding parameters such as temperature, time and pressure have to be controlled for the formation of a reliable diffusion bonded joint [3].

When the joints are bonded at elevated temperatures, thermal expansion mismatch produces a

high stress concentration and leads to fatal damage in the joints. Hence compensation for mismatch is needed to get high quality joints [4]. Several methods, some using interlayers have been developed for this purpose [5-7]. Nicholas and Crispin [5] developed a soft metal method using Aluminium as an interlayer for the bonding of Alumina to Austenitic stainless steel. They achieved joints with a tensile strength of 42 Mpa after bonding at 625°C / 50MPa / 30 min in an evacuated chamber. Failure occurred through an intermediate layer at 19 microns thickness formed at the Al/steel interface. Bonding of Alumina to Austenitic steel was performed via a double Ti/Mo interlayer of 0.5 mm thickness at 1000°C / 12 MPa / 3 h in a vacuum [6]. They achieved joints with strength exceeding 60 MPa. Other authors [7] also tried to bond alumina to ferritic steel at 1000°C / 100 MPa / 30 min using Nb interlayer of 1 mm thickness and a double Nb/Mo interlayer of 0.5mm thickness each. The tensile strength of the steel/Nb/alumina joint was only 5.6 MPA whereas that of the steel/Mo/Nb/alumina joint was 63 MPa. From these previous investigations, it is apparent that bonding conditions including interlayer are not consistent. Furthermore, there is little information concerning the effect of interlayer on the formation of bond

between Alumina ceramic to steel. Nevertheless, a better understanding of the bonding process at the interface will increase the confidence of designers in specifying ceramics for advanced engineering systems.

In the present investigation a study has been made on diffusion bonding of Alumina to 316L type stainless steel using different interlayers.

EXPERIMENTAL PROCEDURE

Materials

Alumina (Alcoa chemicals A-16 SG grade reactive alumina) used in this work was of 99.8% purity. The alumina powder of particle size 0.3 to 0.6 μm was mixed with PVA binder and then pressed in cylindrical moulds of size 10 mm in diameter. The green compact was first dried in oven at 110°C for 24 hours and then, sintered at 1600°C for 3 hours. The cylindrical samples of alumina having density 3.93 gm/cc were ground both surfaces by diamond impregnated double disc lapping machine to optical flatness. The roughness of the cylindrical surface was about 0.3 μm . Stainless steel used was AISI type 316L austenitic stainless steel. These were purchased in the shape of 12 mm diameter single rod. Cylindrical samples were cut off from the rod to the size of 10mm (dia) X 10 mm. Steel surfaces were also ground and polished to surface roughness of about 0.5 μm . All the cylindrical

samples were cleaned with acetone just before bonding. The chemical compositions of the materials used in this study are given in Table I.

Bonding Treatment

Fig.1 shows the apparatus for bonding treatments. The cylindrical samples of alumina and 316L austenitic stainless steel with the desired interlayers in between were first assembled as a joint specimen set (Fig.2) by adhesive tape and then placed gently on the center position of the bottom Zirconia plate. Next, the top punch was slowly pushed downwards by the hydraulic jack of the pressure device so as to touch the samples with the top Zirconia plate. On further pushing, the pressure developed on the samples was indicated in the pressure gauge. An initial pressure was kept at 4 MPa and the samples were heated to a bonding temperature of 800°C – 1000°C for various soaking time in argon atmosphere. After the bonding treatment, the joints were cooled slowly to room temperature and were unloaded by releasing the pressure, which allowed the thrust to come completely. It is to be mentioned here that no reaction between the

Table - I Chemical compositions of the alumina, stainless steel and interlayers used:

(a) Alumina (Al₂O₃)

Al ₂ O ₃	Na ₂ O	CaO	SiO ₂	Fe ₂ O ₃	MgO
99.8%	0.06%	0.01%	0.03%	0.02%	0.03%

(b) Stainless Steel

C	Si	Mn	S	P	Ni	Cr	Mo	Fe
0.025	0.3	1.7	0.012	0.03	12.15	17.95	2.4	Rest

(c) Interlayer foils

Interlayer	Ni	Ti	Nb	Mo	Thickness (im)
Ni	99.9	-	-	-	300, 400 & 500
Ti	-	99.9	-	-	500
Nb	-	-	99.9	-	600
Mo	-	-	-	99.9	600

cylindrical samples and stabilized Zirconia plates was recognized.

Characterization

The interface structures were examined in a section perpendicular to the bonding interface using Optical and Scanning Electron Microscopy (SEM). The reaction products were identified by an X-ray diffraction (XRD) method. The shear strength of the bonded samples was

evaluated with the help of a special jig in an Instron with a crosshead speed of 0.01 mm/sec. The shear-testing jig is shown schematically in Fig.3.

RESULTS AND DISCUSSION

It is observed from Table II that bonding could be achieved only in case of Ni as interlayer for the bonding parameters used such as temperature, time and pressure. For

Table - II Different bonding experimental results using various interlayers :

Sl No	Interlayer	Temp (°C)	Time (Min)	Press (MPa)	Remarks
1	Ni	1000	60	45	Bonding occurred. 316L softening
2	Ni	800	180	70	Bonding occurred
3	Ni	900	180	45	Bonding occurred
4	Nb	900	60	45	No bond between Nb-Al ₂ O ₃
5	Nb	800	180	70	No bond between Nb-Al ₂ O ₃
6	Ti	800	30	45	Ti melts. No bond
7	Ti	775	120	70	No bond
8	Mo	800	180	70	No bond
9	Mo	900	120	45	No bond

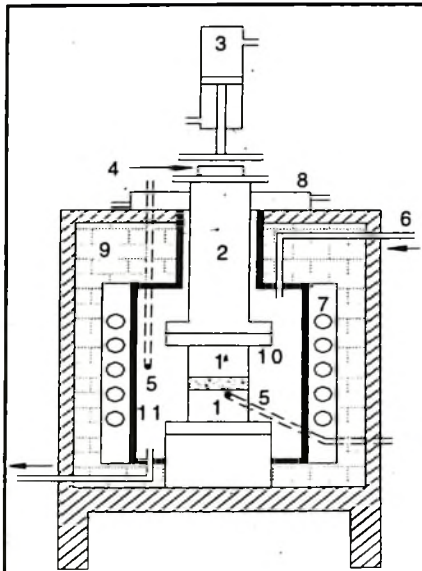


Fig.1: Diffusion bonding set up along with the assembled specimen: 1= sample, 2= Silicon carbide rod, 3= Hydraulic piston cylinder arrangement, 4= Load cell, 5=Thermocouple, 6=Shielding gas (argon) arrangement 7=Heating coils, 8=Gasket with water cooling arrangement, 9=Refractory brick, 10=Zirconia plate, 11=Nichrome tube

the solid state diffusion bonding, the interfacial contact is achieved by four distinguishable mechanisms, i.e plastic flow, creep deformation, interface diffusion and volume diffusion [8]. The effect of bonding pressure and bonding temperature on present bond area is shown schematically in Fig.4. The diagrams of bonding mechanisms allow the bonding process to be visualized by identifying the dominant mechanisms, resulting in the solid state bonding systematically divided into two processes of "deformation" and "diffusion". The beneficial effect of Ni interlayer which enable bonding at the bonding parameters studied (Fig.5a), is thought to be due to an intimate interfacial contact between 316L stainless steel and Alumina Ceramic achieved by plastic deformation of the interlayer.

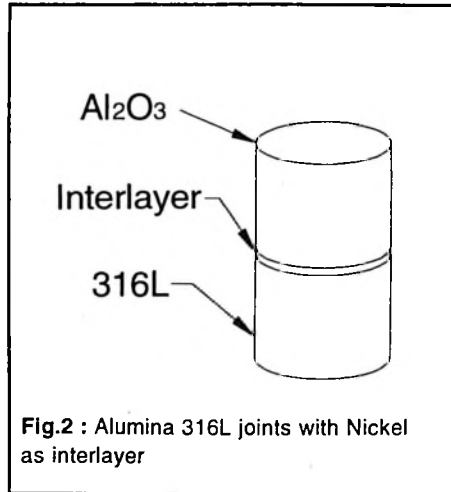


Fig.2 : Alumina 316L joints with Nickel as interlayer

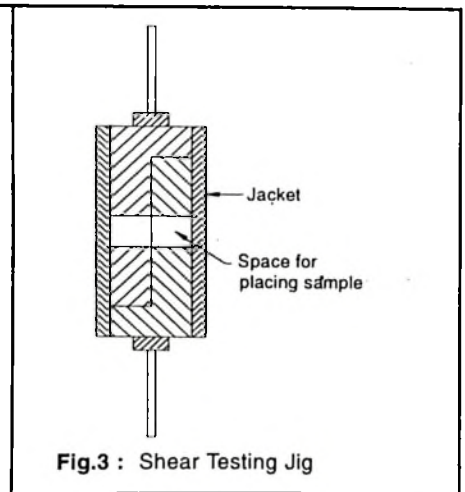


Fig.3 : Shear Testing Jig

It is interesting to note in Fig.5b that bonding has formed on the stainless steel side, but not with the Alumina side when Nb is used as an interlayer. The probable reason of such behaviour of Niobium with alumina are: the formation of a layer of (1) NbOx with x upto 0.15 at the interface [9], (2) CaO. 6 Al₂O₃ due to presence of certain impurity element such as Calcium [10] and the precipitation of δ - Al₂O₃ in the grain boundary of niobium due to decrease in solid solubility of aluminium and oxygen in niobium with decreasing the temperature [10]. Incomplete bonding between Nb and alumina suggests that the bonding conditions are inadequate. However, earlier investigation on diffusion bonding between alumina and Nb in vacuum observed the optimal joining temperature and pressure as 1700°C and 10 Mpa [8]. It is worth to mention that the temperature of 900°C was chosen as an experimentally convenient upper limit as the 316L stainless steel is deformed plastically.

When Ti is used as an interlayer, it gets soften at 800°C and at a pressure of 4.3 MPa and finally it squeezed out at the periphery of the Alumina sample as shown in Fig.6. Pure titanium is soft enough at the

bonding conditions to deform plastically and to get touch with alumina surface. In spite of good contact insufficient bonding of the alumina/titanium/316L stainless steel is probably due to the thermal expansion mismatch. Furthermore, it is also probable for the formation of reaction layer of TiO, TiAl etc because of the high diffusibility of oxygen in titanium.

In case of Mo interlayer, no bonding has been observed under the present experimental conditions. It is likely that the bonding of the molybdenum / steel interface is influenced by the surface flatness of molybdenum and 316L before bonding because the metals did not plastically deform enough to bring them into contact with each other at this temperature. Again it suggests higher temperature along with higher pressure for increasing the plastic deformation indicating the improper bonding conditions available in the present experimental conditions for molybdenum as an interlayer.

The optical micrograph of Al₂O₃/Ni/ 316L joint is shown in Fig.7. Since the chemical reactions between alumina ceramic and austenitic stainless steel during bonding have a dominant influence on the joint performance, bond surface has been

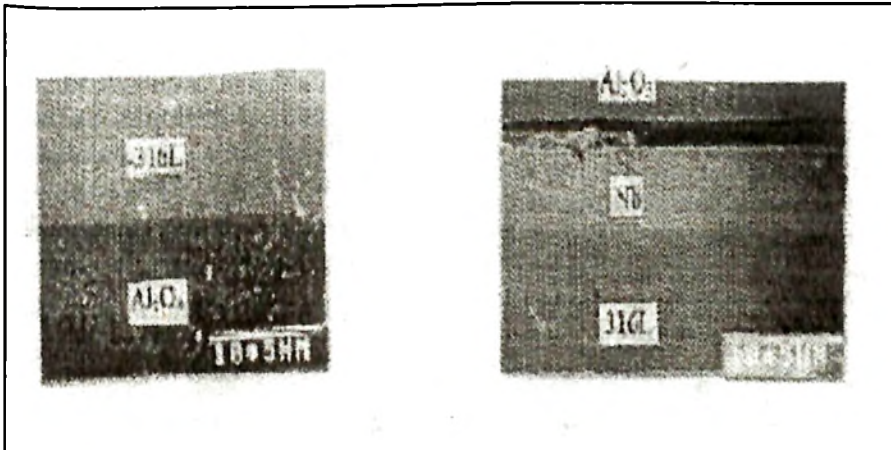
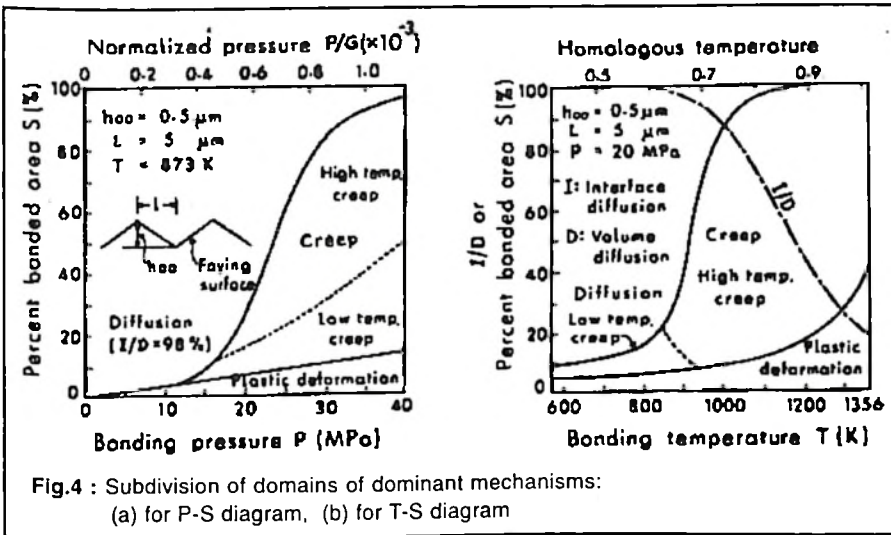


Fig.5 : Cross sectional appearance of Al₂O₃/316L joints: (a) good bond with Nickel interlayer (b) lack of bond between Nb and Al₂O₃

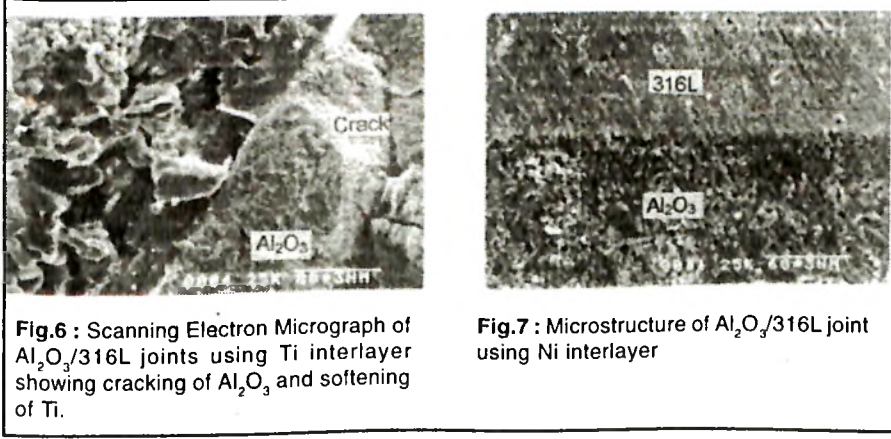


Fig.6 : Scanning Electron Micrograph of Al₂O₃/316L joints using Ti interlayer showing cracking of Al₂O₃ and softening of Ti.
 Fig.7 : Microstructure of Al₂O₃/316L joint using Ni interlayer

characterized by XRD. Fig.8 shows X-ray diffraction patterns taken at the interface of alumina/316L stainless steel diffusion bonded sample. The characteristics of the X-ray

diffraction pattern indicate that NiAl₂O₄ phase has formed at the interface. Experimental and thermochemical studies showed that the formation of nickel aluminate spinel

by solid state reaction between Ni and Al₂O₃ requires a certain oxygen activity which is less than that of the Ni-NiO equilibrium. Furthermore, the thermal expansion mismatch between the spinel phase and alumina is very low [2]. Fig.9 shows the effect of thickness of nickel interlayer on the shear strength of the joints. Shear strength decreases with increasing nickel thickness. Interlayers are usually used in diffusion bonding to reduce the thermal expansion mismatch, which produces a high stress concentration in the joints leading to fatal damage.

The magnitude of residual stress depends on the shape and dimension of the interface [11]. With the increase in thickness of the interlayer, the residual stress will increase. Furthermore, the distribution of residual stress in a ceramic/metal joint is not uniform even along the interface. Concentration of residual stress becomes severe as closing to the interface and to the free surface. Therefore, decrease in strength with increase in thickness of interlayer is not unexpected considering higher stress concentration in the joint.

Concluding Remarks

In the present investigation, diffusion bonding of Alumina and 316L using different interlayers was examined at low temperature and moderate pressure in argon atmosphere. Complete bonding was found with Nickel as an interlayer and the shear strength of the joint was decreased with increasing thickness of Nickel. Inadequate bond with interlayers such as Nb, Ti and Mo is considered to be due to unsuitable bonding parameters. Therefore, the selection of bonding parameters including the interlayer to give an operative domain of bonding mechanism is prerequisite.

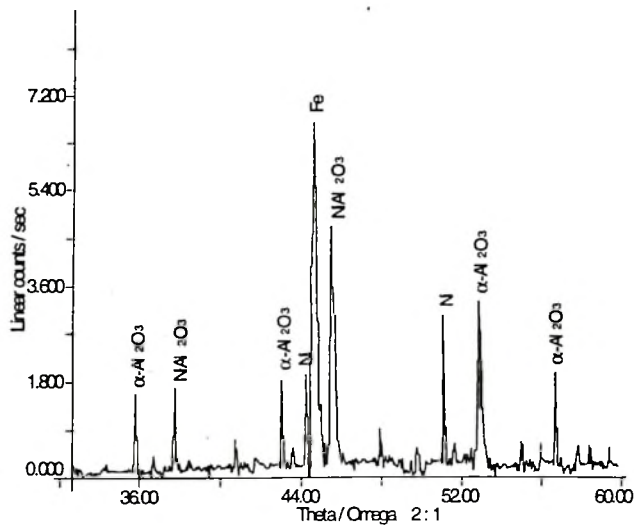


Fig.8 - X-ray diffraction pattern of the interface of Al₂O₃/Ni/316L joint

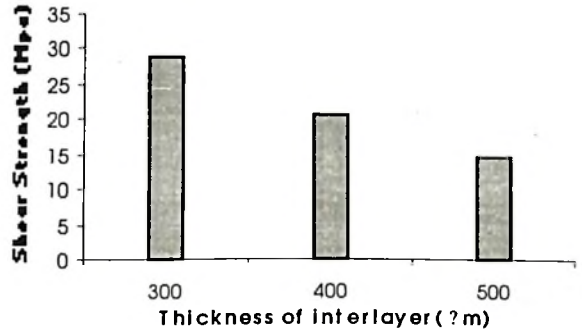


Fig.9 - Effect of Nickel interlayer thickness on shear strength of the joint

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Phone : 2243-1254, 2210-3849
 GRAM : BUTELBOW
 FAX NO. : 033-2242-6091
 E-mail : n_l_hazra@hotmail.com

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