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Diffusion bonding behaviour of austenitic stainless steel containing titanium and alumina

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A study has been conducted to identify the function of titanium in 1Cr18Ni9Ti steel and the effects of fabrication temperature, pressure, time and other variables on the strengths of diffusion-bonded alumina-1Cr18Ni9Ti. At temperatures of 750 to 1200 °C, 1Cr18Ni9Ti steel was successfully bonded to alumina, with a maximum tensile strength of 19 MPa. By EPMA titanium segregated to the interface of the joint, in contrast to the failure of bonding of 1Cr18Ni9 steel and alumina under the same conditions. Titanium can reduce alumina with the reactants of TiO and compounds of titanium and aluminium thermochemical calculation. Thus it is indicated that titanium is an important go-between element for bonding metal to alumina.

1. Introduction

Ceramics are very desirable for structural applications, as they are readily available, inexpensive, light-weight, very hard, and can withstand high temperatures. Oxides especially, with their extremely negative free energy of formation, have been identified as appropriate materials for high-temperature applications [1, 2]. This has provided a new impetus for research on the joining of ceramic and metal [3–6]. However, development of joining for fabricating oxide components is difficult; the basic reason is the very different bonding characteristics of ceramic and metals.

Among all the factors which influence the joining of metals and ceramics, the metal should have a thermal expansion coefficient and a modulus of elasticity close to those of the oxide, and should have a high strength. Above all, the metal can react with the ceramic to form an interlayer to join the metal and ceramic. The number of possible elements which can react with alumina is very limited. Titanium is an important element which falls into this category: it easily reacts with alumina above 650 °C [7], making it possible to join the metal and alumina at a low temperature. This is important to reduce the thermal contraction mismatch stresses.

In the present work the joining conditions of alumina and steel 1Cr18Ni9Ti were investigated, and at the same time a thin titanium sheet as a filler and 1Cr18Ni9 steel were used comparatively. Furthermore, the joining strength was measured.

2. Experimental procedure

Pressureless sintered alumina containing a few per cent of other oxides was used in the form of bar with a diameter of 10 mm and a length of 50 mm. One of these test pieces was bonded to a 50 mm length and 10 mm diameter sample of 1Cr18Ni9Ti steel, whose nominal composition is given in Table I. Samples were prepared with and without a titanium filler, which is 0.2 mm thick and cut from commercial purity foil.

Care was taken not to touch the bonding surface of alumina, steel and titanium test piece, which were polished and ultrasonically degreased in alcohol for 10 min.

The samples could be easily assembled in the hot-pressing facility sketched in Fig. 1. The hot-pressing chamber was evacuated to a vacuum before the samples were heated to the bonding temperature, which ranged from 750 to 1200 °C. The heat-up time was 1 to 2 h, after which the samples were subjected to a compressive stress of 7 to 15 MPa (load divided by the bonding surface area of the sample test piece) and held at temperatures for 10 to 60 min. At the end of this time, the power supply to the chamber heater was decreased slowly and the pressure released. The samples were allowed to cool slowly, not being unloaded until 3 h had elapsed.

The quality of bonded samples was assessed primarily by tensile tests conducted at an extension rate of 1 mm min^{-1} using an Autograph IS-10T machine. The simple parameter of failure load divided by the test piece bonding surface area was used to characterize tensile strengths, as the load–extension curves generated for each sample showed that fracture preceded yielding. For some samples, this simple evaluation of quality was supplemented by optical microscopy, electron probe microanalysis and the Vickers'

TABLE I Compositions of the alloys used in this work

Alloy	Composition
1Cr18Ni9Ti	17.92Cr, 8.45Ni, 0.06C, 0.5Si, 1.28 Mn, 0.026P, 0.015S, 0.92Ti, remainder Fe
1Cr18Ni9	18.25Cr, 8.335Ni, 0.055C, 0.59Si, 1.39Mn, 0.026P, 0.017S, remainder Fe

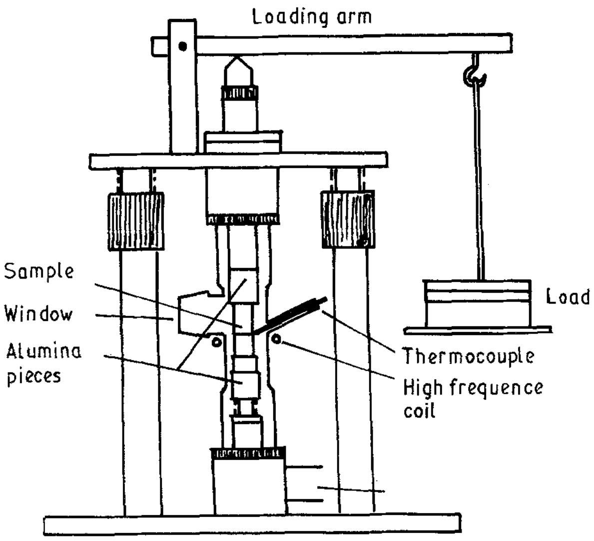

Figure 1 Diffusion bonding unit.

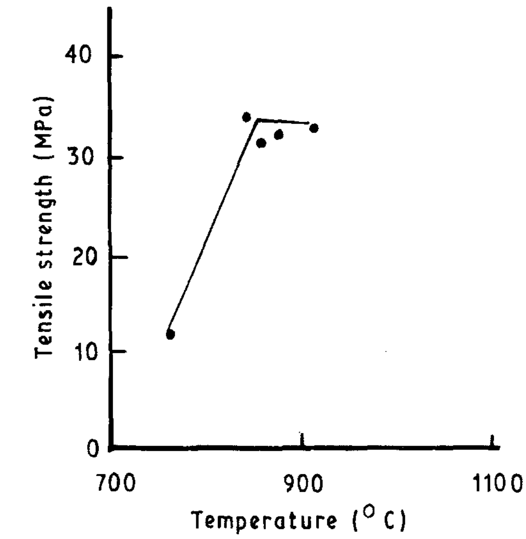

Figure 2 Tensile strengths of the samples with titanium filler bonded at various temperatures; pressure, 15 MPa; vacuum, 10^{-4} mm Hg; bonding time 0.5 h.

TABLE II Experimental data

Temperature (°C)	Pressure (MPa)	Time (min)	Vacuum (mm Hg)	Filler	Strength (MPa)
780	7	60	10	No	3.9
790	7	60	10	No	8.1
845	7	60	10	No	13.4
914	7	60	10	No	15.5
965	7	60	10	No	19.0
1040	7	60	10	No	18.5
1100	7	60	10	No	19.0
845	7	10	10	No	3.9
845	7	30	10	No	12.4
845	7	60	10	No	13.2
845	3.5	60	10	No	17.0
760	15	30	10	Ti	12.0
834	15	30	10	Ti	33.0
858	15	30	10	Ti	30.8
910	15	30	10	Ti	32.6

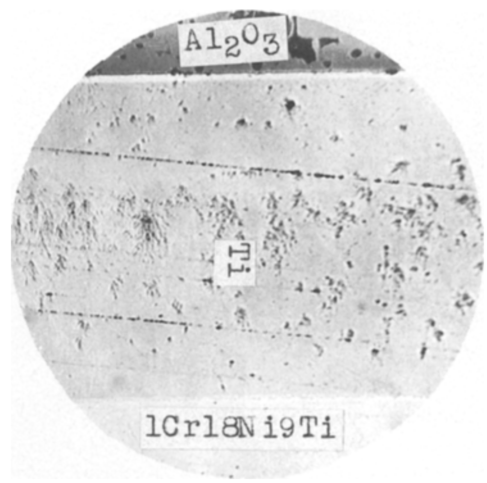

Figure 3 Micrograph of a cross-section of the alumina–1Cr18Ni9Ti joint with titanium filler (×250); bonding temperature 875 °C; tensile strength 32.0 MPa.

hardness test of polished cross-sections. At the same time, the thermochemical calculation was carried out to evacuate the possible products at the bonded interface between metal and ceramic. All experimental results were summarized in Table II.

3. Results

3.1. Diffusion bonding alumina to steel with titanium filler

Commercial pure titanium film, 0.2 mm thick, was used as a filler for diffusion bonding between steel and alumina. After a few experiments, the normal bonding conditions were defined as a pressure of 15 MPa applied for 30 min to a sample held in a vacuum of 10^{-4} mm Hg at 760 to 910 °C, with the bonding strengths as shown in Fig. 2. In the subsequent series of experiments, fabrication parameters were not knowingly allowed to deviate from the norm.

Normal bonding conditions did not obviously deform the titanium film and produced samples that could withstand tensile stresses of 33 MPa before fracturing at the alumina sample. Above the bonding temperature 865 °C, the bonding strength basically kept at 32 MPa, larger than the 13 MPa found by Hatakeyama *et al.* [4], which may be the result of different bonding conditions and the thickness of the filler. All the samples broke at the alumina, which was due to the very different thermal expansion coefficient between metal and alumina. Figure 3 is an optical micrograph of a polished cross-section of a sample bonded under normal conditions, and shows a definite boundary between alumina and titanium, and steel and titanium.

3.2. Diffusion bonding alumina to steel directly

Alumina was bonded directly to 1Cr18Ni9Ti steel under the bonding pressure of 7 MPa applied for 60 min in a vacuum of 10^{-4} mm Hg at 780 to 1100 °C. The tensile strengths of the bonded samples are shown

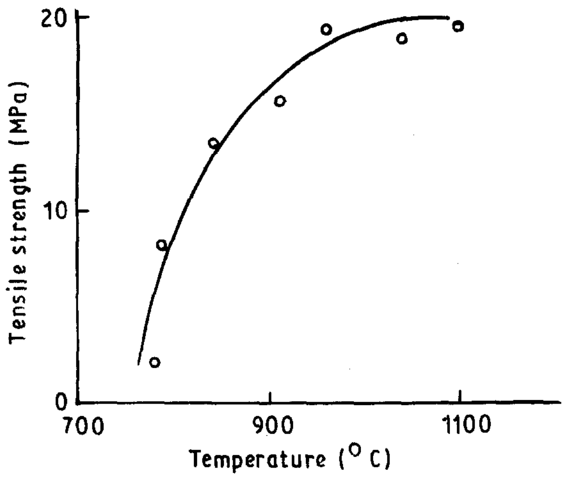

Figure 4 Tensile strengths of alumina–1Cr18Ni9Ti joints bonded at various bonding temperatures; pressure, 7 MPa; vacuum, 10^{-4} mm Hg; bonding time 1 h.

Figure 5 Bonded alumina–1Cr18Ni9Ti fractograph.

in Fig. 4. Above 950 °C, the tensile strengths kept at about 18 MPa. Figure 4 also shows that, above the bonding temperature, alumina successfully bonded directly to 1Cr18Ni9Ti steel, which is similar to the case with titanium filler. The sample also fractured at the side of alumina, as shown in Fig. 5, which is also due to the very different expansion coefficient of steel and alumina. Figure 6 shows the Vickers' hardness of the steel side as a function of distances from the interface between the alumina and steel under the experimental conditions of load 200 g, 15 sec. The Vickers' hardness decreased rapidly to normal values with distance, which may show the stress distribution in the steel. The influence of bonding time on tensile strength is shown in Fig. 7 under a bonding pressure of 7 MPa and bonding temperature of 845 °C in a vacuum, 10^{-4} mm Hg; when the bonding time is larger than 30 min, tensile strength varied less. Furthermore, halving the pressure or vacuum has no obvious influence on the tensile strength, as shown in Table II. Figure 8 is an optical micrograph of a polished cross-section of a sample bonded under pressure of 7 MPa, bonding temperature 914 °C, bonding time 60 min and vacuum 10^{-4} mm Hg, and shows a tightly bonded interface.

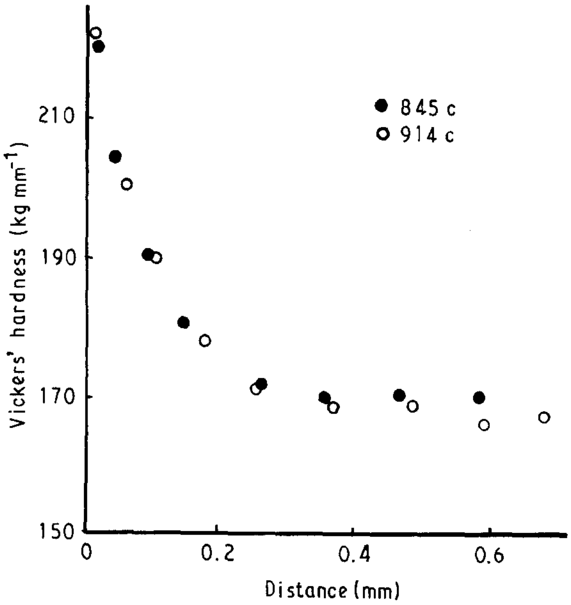

Figure 6 Vickers' hardness of the 1Cr18Ni9Ti side, as a function of distances from the interface of alumina–1Cr18Ni9Ti; load, 200 g; 15 sec.

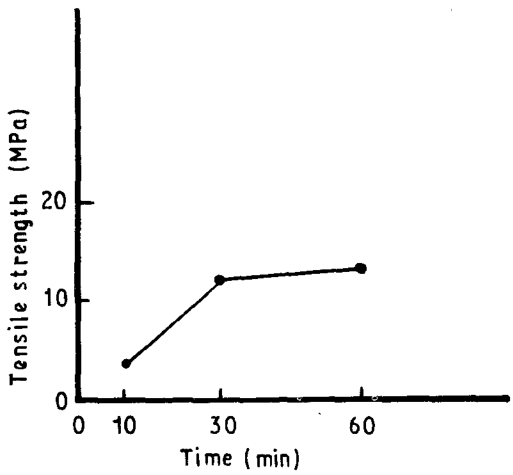

Figure 7 Tensile strengths of the samples bonded for various times. Temperature, 845 °C; pressure, 15 MPa; vacuum, 10^{-4} mm Hg.

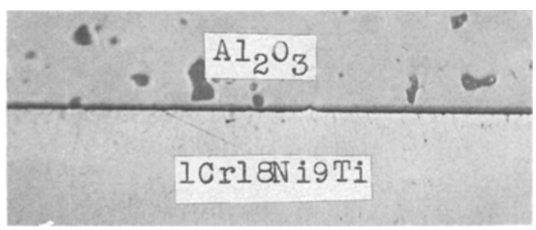

Figure 8 Interface (× 320) of alumina–1Cr18Ni9Ti joint, bonded at temperature 914 °C; pressure, 7 MPa; vacuum, 10^{-4} mm Hg; time, 1 h.

Alumina failed to bond directly to 1Cr18Ni9 steel under the same conditions as those for bonding alumina to 1Cr18Ni9Ti steel, which shows that titanium is an important element in bonding of alumina and 1Cr18Ni9Ti steel. With the aid of electron probe microanalysis of polished cross-section, it was shown

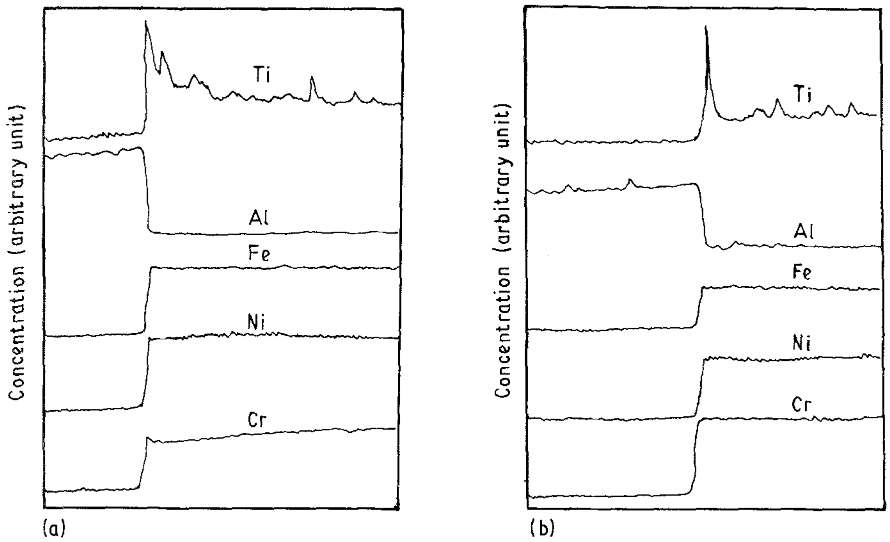

Figure 9 Concentration profiles of alumina–1Cr18Ni9Ti joints, bonded at (a) 845 °C, 0.5 h; (b) 950 °C, 4 h; pressure 7 MPa; vacuum 10^{-4} mm Hg.

that titanium obviously segregated to the interface between alumina and 1Cr18Ni9Ti steel; titanium segregated more the longer the time and higher the bonding temperature, as shown in Fig. 9.

4. Discussions

The primary objective of this work was the identification of titanium's function in the process of diffusion bonding alumina to steel, and of the conditions that could be used to fabricate stainless steel–alumina joints. For the present purpose, a strong joint is one that can withstand a stress of 19 MPa produced by a pressure of 7 MPa applied for 60 min in vacuum, 10^{-4} mm Hg, at 965 to 1100 °C. Titanium plays an important role in the diffusion bonding of 1Cr18Ni9Ti steel and alumina. The bonding behaviour of 1Cr18Ni9Ti steel and alumina, with or without titanium filler, shows similarities. All the samples with or without titanium filler failed at the side of alumina under tensile stress, which was resulted by the very different thermal expansion coefficient between steel and alumina. When the sample is cooled from bonding temperature to room temperature, there is a large thermal stress in the sample. Because of the poor plasticity of alumina, there is a stress concentration in the alumina, so that the alumina side broke first under the tensile stress. The present work suggests that the very different thermal expansion coefficient plays a vital role in getting a strong metal–ceramic joint.

Electron probe cross-section line analysis shows a definite titanium segregation, and no other element segregates in the interface. This kind of up-hill diffusion shows that, within the bonding condition, the interface has a strong affinity to the titanium in the steel; it is that titanium diffusing to the interface to react with the alumina and to form a reactant layer that joins the steel and alumina.

Tressler *et al.* [7] studied the reactivity of titanium–alumina composites, and showed that there are reactant TiO and Ti_3Al on the interface of alumina and titanium at 650 ° to 905 °C. For alumina to bond to metal, it is important that a chemical reaction occurs on the interface, which links metal and alumina strongly. When titanium reacts with alumina, there may be the following chemical reactions:

$$Al_2O_3 + 3Ti = 2Al + 3TiO \quad (1)$$

$$Al_2O_3 + \tfrac{3}{2}Ti = 2Al + \tfrac{3}{2}TiO_2 \quad (2)$$

$$Al_2O_3 + \tfrac{3}{5}Ti = 2Al + \tfrac{3}{5}Ti_3O_5 \quad (3)$$

$$Al_2O_3 + 2Ti = 2Al + Ti_2O_3 \quad (4)$$

According to the thermochemical data [9], the chemical reactions for free energy are shown in Fig. 10. At the isobaric and isothermic conditions, the chemical reaction can occur only when the free energy, G, is negative. It is obvious that the chemical reactions (2) to (4) could not happen at the above-mentioned bonding temperatures; but reaction (1) does happen above a temperature of about 677 °C. Therefore, below the 1100 °C, titanium reacting with alumina only produces the reactant TiO, as shown in [7]. Kubaschewski and Dence [10] point out that, above a temperature of 660 °C, titanium can react with aluminium to form compounds. As a result, the solid state

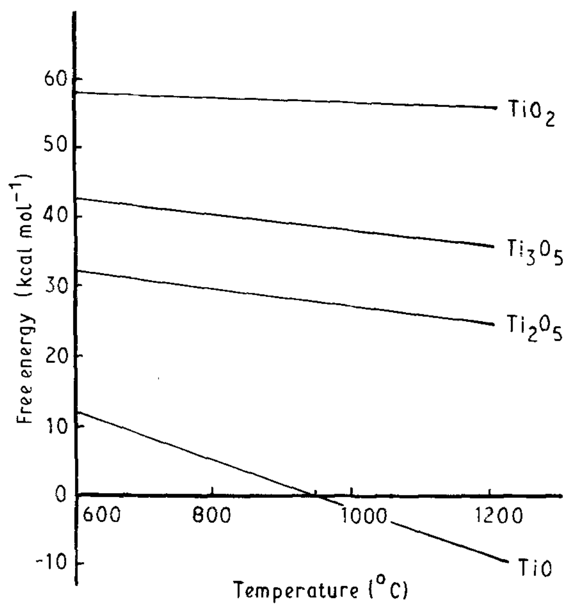

Figure 10 Free energy, G, and temperature diagrams of the various reactions between alumina and titanium.

reaction

$$Al_2O_3 + (3 + 2x)Ti = 2AlTix + 3TiO$$

above 677 °C, can happen from the thermodynamic view point. This result confirms that 1Cr18Ni9Ti steel, above 750 °C, can bond successfully to alumina. Thus it is felt that the technique of using titanium element is a good candidate for optimization and development as a practical fabrication process for alumina-steel joints that do or do not have to withstand high temperatures.

5. Conclusions

1. The diffusion-bonding behaviours of 1Cr18Ni9Ti steel and alumina, with or without titanium filler, are similar. The titanium in steel diffusing to the interface and reacting with the alumina causes 1Cr18Ni9Ti steel to bond tightly to alumina, with a tensile strength of 19 MPa.

2. Chemical–thermodynamic calculations show that, at the above-mentioned bonding conditions, titanium can react with alumina to produce TiO and compounds of titanium and aluminium. Therefore titanium is a good go-between in the bonding of metal and alumina at lower temperatures.

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