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Characterisation of the Join of Electric Discharge Pulse Welded Ceramics

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Abstract

A series of sialon, silicon carbide (SiC) and lead zirconium titanate (PZT) ceramics joined by an electric discharge pulse-welding process have been examined to provide information on joint characteristics. Techniques used include X-ray diffraction, scanning electron microscopy and energy dispersive X-ray analysis. Results indicate that joints with variable degrees of interfacial contact, interlayer porosity and thermal stress have been produced, depending on the precise conditions used. Titanium was found to provide better wetting characteristics than aluminium when used as an interlayer material.

Introduction

Advanced ceramics have developed to become major contenders for a number of high temperature, high strength applications.[1] The versatility of these materials is becoming more and more extensive as research programmes reduce the problems of brittleness and insufficient reproducibility of properties. Currently of high interest are the family of ceramic materials based on silicon nitride, including reaction bonded, pressureless sintered and hot pressed silicon nitride with a variety of sintering aids and the sialon group of ceramics based on solid solution between silicon nitride and alumina. Silicon carbide ceramics also possess the required characteristics for many structural engineering applications and are available in the same forms as silicon nitride.[2]

As the demand for larger and more complex shaped components increases, it becomes more difficult to fabricate ceramics using a single step shaping process and economically unfavourable to machine simple blocks to complex shapes. Thus, commercial use becomes dependent upon the availability of reliable techniques for ceramic–ceramic and ceramic–metal joining.[2-3] Various techniques for chemically joining ceramics have been investigated, the most common are active metal brazing and diffusion bonding.[1-3] Both processes can be carried out with or without intermediary layers. The purpose of the intermediary layer is to absorb the effects of the mismatch in thermal expansion between the components to be joined. Other processes which may offer a potential route for ceramic bonding include ceramic adhesives and electric discharge welding.[4] Of these the former offers high temperature stability but little mechanical strength, the latter involves a high energy pulse passing through a conductive interlayer, converting it into a plasma. A ceramic–metal interaction occurs and a bond is formed. The process is exceptional for its speed, bonding typically occurring in <60 μs, compared to many minutes for conventional brazing and diffusion bonding.

A schematic diagram of the process is shown in Fig. 1(a), and an example of a typical electric discharge pulse welded ceramic piece is shown in Fig. 1(b). A metallic foil approximately 5 μm in thickness is clamped between the ceramic specimens. No special surface condition is necessary in terms of polishing, but the surface must be clean. When the capacitor is discharged, the interlayer is vaporized and bonds to the ceramic. The process is currently under investigation at the E.O Paton Electric Welding Institute (E.O.P.E.W.I) in Kiev, Ukraine.[4]

The technique has been used principally to examine the bonding of silicon nitride based ceramics using various metallic interlayers. The properties of a good interlayer can be listed as:

(a) low electrical resistance;
(b) must wet the ceramic;
(c) must be clean and free from inclusions, porosity and gases.

Within the discharge zone the energy density is

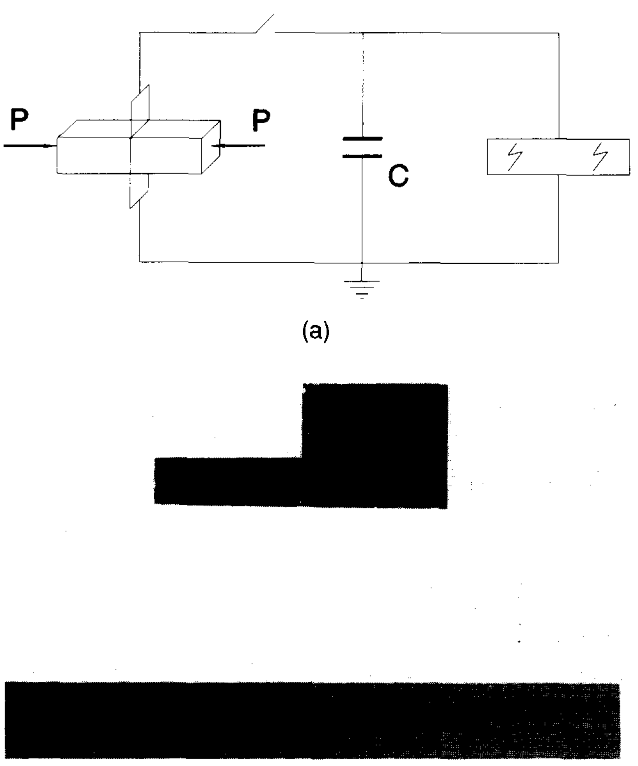

Fig. 1. (a) A schematic diagram of the electric discharge pulse welding process. P = pressure (< 20 MPa), C = capacitance (1–100 μF). Adapted from Ref. 4. (b) An example of a typical electric discharge pulse welded sialon ceramic joined using a 1·5 μm thick sputtered aluminium interlayer. Shown on a scale where each division is equal to 1 mm.

1–100 kJ/m^3, the plasma temperature approaches 10^4–10^5 K and pressures of 10^3–10^5 MPa are induced. All other conditions being equal, experiments have shown that the quality of a joint decreases with a decrease in charge energy below a minimum level. However, excess energy can result in either:

(a) fracture due to the high pressure pulse which accompanies the discharge, especially if the ceramic possesses low strength; or

(b) metallisation of the surfaces of the ceramic.

In the present study, sialon, silicon carbide and lead zirconium titanate ceramics joined by such a process, in which either an aluminium or titanium foil or sputtered interlayer was used, have been investigated.

Experimental

Ten joined ceramic–ceramic specimens were provided by the E.O.P.E.W.I. together with details corresponding to the materials involved and their respective processing parameters. This information is given in Table 1. The cross-sectional areas of all ten bonded ceramics were 5 × 5 mm and the length of each ceramic piece prior to joining was typically 6 mm.

Table 1. Composition and processing characteristics of electric discharge pulse welded specimens.

Specimen	*Material*	*Potential difference applied (V) (capacitance 12 μF, Time 40 μs)*	*Interlayer form*
1	Sialon/Al/Sialon	2100	5 μm foil
2	Sialon/Al/Sialon	2500	5 μm foil
3	Sialon/Ti/Sialon	3100	5 μm foil
4	SiC/Al/SiC	2500	5 μm foil
5	SiC/Ti/SiC	3200	5 μm foil
6	Sialon/Al/Sialon	1100	1·5 μm sputtered
7	Sialon/Al/Sialon	1500	1·5 μm sputtered
8	SiC/Al/Sic	2000	1·5 μm sputtered
9	SiC/Ti/SiC	1100	1·5 μm sputtered
10	PZT/Al/PZT	1100	5 μm foil

The specimens numbered 1–9 were sectioned perpendicular to the join, then one half mounted in a conductive resin and polished. Specimen 10, incorporated into the investigation at a later date, had previously been polished and was ready for use in the scanning electron microscope.

Three characterisation techniques were used to investigate the specimens, namely X-ray diffraction, scanning electron microscopy and energy dispersive X-ray analysis. The former was performed on both the dense, sintered samples provided and also on portions of the same samples ground into fine powder. Samples were carbon coated prior to examination on the SEM and both bulk analysis and spot analysis across the join were carried out using EDX. The latter permitted the degree of interlayer material diffusion into the bulk ceramics to be estimated. No analysis was undertaken to measure the bond strength of the joined ceramics as the number of specimens provided was too small to have any statistical merit.

Results and Discussion

X-ray diffraction analysis on both the monolithic, sintered samples and subsequently powdered material indicated that the sialon samples were composed of the X-phase ($Si_{12}Al_{18}O_{39}N_8$) as the primary phase with small amounts of (Si_3N_4)14H as a secondary phase. The primary phase present in the silicon carbide samples was (SiC)33R, however the secondary phase was too small to identify. Specimen 10 was identified as being the single phase $Pb(Z_{0{\cdot}52}Ti_{0{\cdot}48})O_3$. Table 2 indicates the principal impurities found in the samples.

The scanning electron microscope investigations

Table 2. Quantitative analysis of the impurities found in the samples.

Material	*Impurities*	*Relative proportions of impurities*
Sialon	Iron, calcium	Si:Al:Fe:Ca = 1:0·6:0·08:0·06
Silicon carbide	Aluminium	Si:Al = 1:0·20
Aluminium	Copper, nickel	Al:Cu:Ni = 1:0·2:0·05
Titanium	Vanadium	Ti:V = 1:0·2
Lead zirconium titanate	—	—

carried out have indicated that all of the specimens display a degree of poor contact at the ceramic–metal interface. This suggests that the optimum conditions for joining have not been met for any of the specimens examined. However, a positive feature of the process is that at distances greater than 20 μm from the join there is no indication in any of the specimens that the process has effected the properties of the bulk ceramic.

The aluminium foil on melting appears to wet the surface of the sialon better than the silicon carbide, resulting in improved adherence. The process has produced a highly porous interlayer material, with the pressure generated at the join pushing the Al into the pores at the interface of the ceramic, hence enlarging the surface area of contact (Fig. 2). Spot analysis* across the join for both the sialon/Al/sialon and SiC/Al/SiC specimens suggests, by the sharp transition of elemental intensities shown in Figs 3 and 4, that diffusion in either direction across the interface has not occurred as would be expected for such a short joining time.

The adherence of the titanium foil interlayer at the silicon carbide interface is far superior to that at the sialon interface and for both ceramics the joint characteristics are a significant improvement on those achieved when an aluminium interlayer is used. For the SiC/Ti/SiC specimens, bonding at the ceramic–metal interface is evident from the presence of cracks running through the titanium perpendicular to the join as in Fig. 5. These cracks are thought to be caused by the differences in coefficients of thermal expansion between the two materials creating stresses at the interface on cooling. Spot analysis across the join indicates a more gradual transition of elemental intensities (Fig. 6), suggesting the existence of reaction products at the interface. For the sialon/Ti/sialon specimen, spot analysis across the join does not distinguish either way whether reaction products exist or not.

In agreement with Ref. 4, at too low a charge energy no join is formed. The limit has not been

Fig. 2. Sialon ceramic joined using a 5 μm thick aluminium foil.

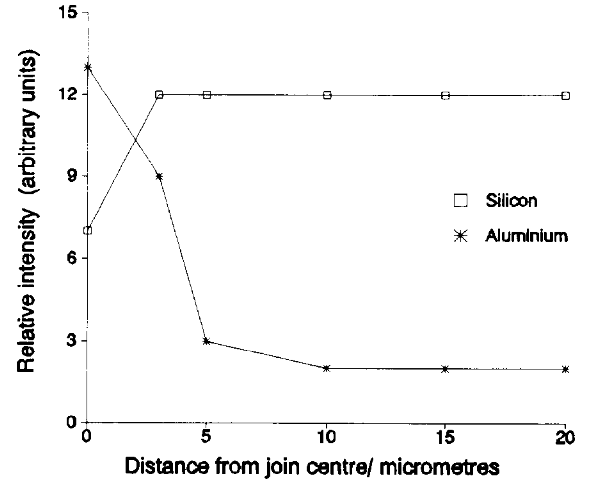

Fig. 3. Composition variation across the join for sialon ceramics joined using an aluminium interlayer.

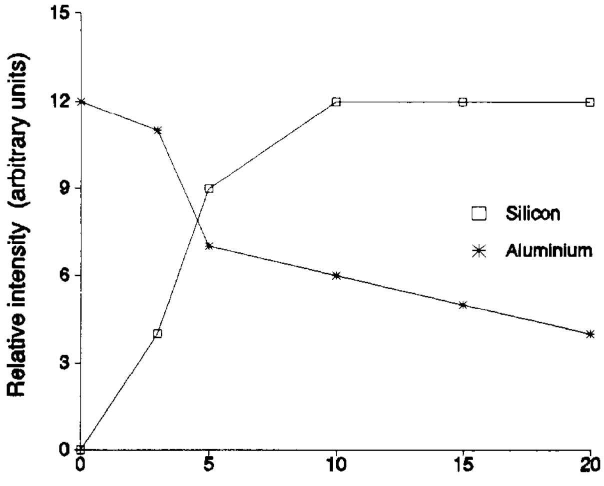

Fig. 4. Composition variation across the join for silicon carbide ceramics joined using an aluminium interlayer.

*The spot analysis, carried out at 25 keV under a magnification of × 1000 for 100 s, can only be considered as an estimate of the element distribution. The graphs which indicate the composition variation across the join for each specimen possess errors of ±1·0 in terms of intensity. The accuracy in positioning of the spot lies within ±2 μm, which when considering the total width of the interlayer is only 5 μm, then this error is considerable.

Fig. 5. Silicon carbide ceramic joined using a 5 μm thick titanium foil.

Fig. 7. Lead zirconium titanate ceramic joined using a 5 μm thick aluminium foil.

determined in the current work, however 1100 V appears to be too low for a sputtered interlayer 1·5 μm thick. The interlayer has melted but not bonded with the ceramic. Exceeding an applied voltage of 2500 V to join both sialon–sialon and SiC–SiC ceramics using an aluminium interlayer resulted in the fracture of the ceramic parallel to, and at distances of typically 10 μm from, the join. Using a titanium interlayer meant that the upper limit to the applied voltage was raised; fracture of the ceramic adjacent to the join was not observed until 3100 V was applied. At this voltage the behaviour of the ceramic is similar to that observed for aluminium at 2500 V.

The sialon and SiC ceramics have similar thermal expansion coefficients of approximately 5 MK^{-1}. Typical values for aluminium and titanium are 24 and 9 MK^{-1} respectively.[5] The variation in level of applied voltage required for damage to occur corresponds to the greater thermal expansion mismatch at the aluminium–ceramic interface compared to that at the titanium–ceramic interface. It is this mismatch which in turn results in the fracture of the ceramic. Further work is clearly required using a multilayer system, with the intention of reducing the thermal stresses developed due to the difference in materials used.

No direct comparison can be made between the two interlayer thicknesses used as the charge energy had also been varied in the samples analyzed. However the results suggest that the sputtered interlayer yields improved joins. This could be because the layer is thinner or because the sputtering process will have increased the initial ceramic–metal contact prior to joining.

The lead zirconium titanate specimen displays a similar degree of joining as the engineering ceramics (Fig. 7). The edges of the aluminium interlayer are very uniform and no evidence exists of mechanical keying by the aluminium in the pores at the ceramic interface. The electrical pulse has not weakened the ceramic adjacent to the join and thus no cracking is apparent. Spot analysis across the join (Fig. 8) shows a variation in intensity for

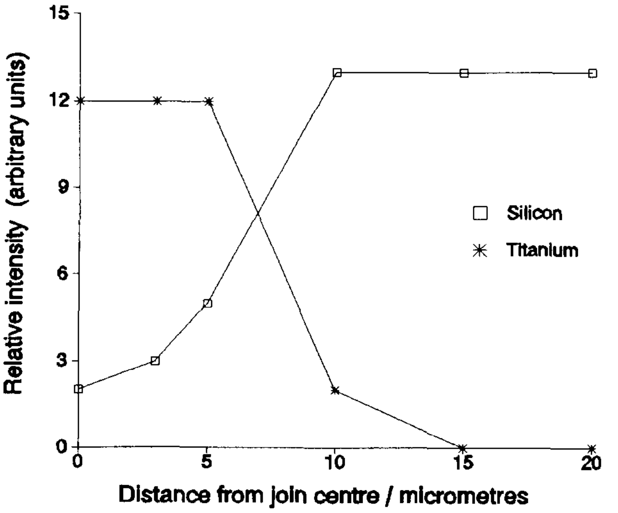

Fig. 6. Composition variation across the join for silicon carbide specimens joined using a titanium interlayer.

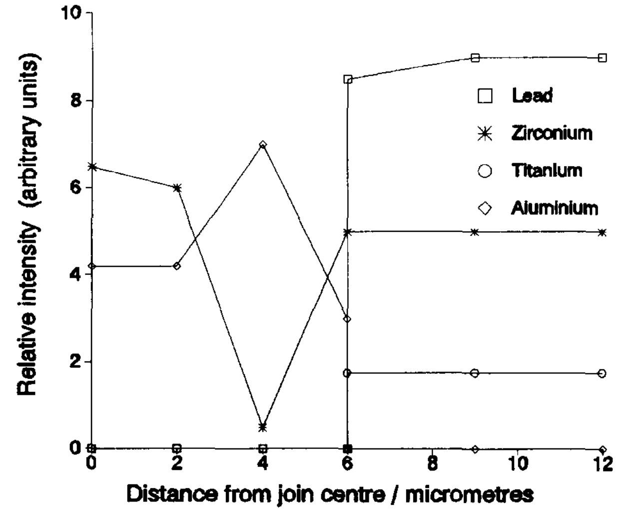

Fig. 8. Composition variation across the join for lead zirconium titanate ceramics joined using an aluminium interlayer.

the Al and Zr across the ceramic–metal interface that suggests some diffusion has occurred across the interface and reaction products exist.

Conclusions

For a ceramic–ceramic join with the aid of an active metal interlayer, then the processing parameters chosen must ensure that the metal melts, wets and chemically bonds to the surface of the ceramic interfaces. Control of the reaction zone variables such as thickness, thermal expansion coefficients and thermal stability, allows optimisation of the bond properties. Although this degree of control has not been achieved in any of the specimens provided, evidence has been found which illustrates the potential for this new processing technique.

The present study has indicated the greater wetting capabilities, and hence superior performance, of Ti-alloys over Al in electric discharge welding of sialon and silicon carbide ceramics. This agrees with the theory given in Ref. 2, in which aluminium and titanium interlayers were used in an attempt to join silicon nitride ceramics.

Limits to the range in charge energy used are: too low and bonding will not occur, too high and the ceramic fractures or metallization of the ceramic surfaces occurs. The optimum for both the foil and sputtered interlayers with the engineering ceramics studied appears to lie within the range 1·1–3·2 kV.

Since charge energy was varied simultaneously with interlayer thickness, no real indication of the effect of the latter was obtained, though the results do indicate a preference towards the use of a sputtered interlayer. A continuation in this investigation would be the introduction of a multilayer system, with the intention of reducing the thermal stresses developed due to the difference in materials used.

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