

Joining of Ceramics using Oxide and Oxynitride Glasses in the Y-Sialon System

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Abstract

Glasses in the Y-SiAlO and Y-SiAlON systems, of standard cation composition (28:Y, 56:Si, 16:Al) have been investigated as potential adhesive materials for silicon nitride-based ceramics. Glass transition and crystallisation temperatures are reported. Hardness and density have also been measured. These glass materials have been used to bond hot-pressed and sintered β -sialon ceramics containing an intergranular glass composition similar to that of the adhesive material. Joints were formed via two processes: (1) hot-pressing at temperatures from 1130°C to 1600°C for 1 h at a pressure of 5 MPa using the Y-sialon glass; and (2) pressureless sintering at 1600°C for 1 h, using both the Y-sialon and Y-sialo glasses. At a temperature of 1600°C, SEM analysis illustrates that both processes yield visibly good joints. Hardness measurements indicate that there is no degradation in value between the parent ceramic and the bonded region. Joining was dominated by the flow of glass across the ceramic interfaces, dissolution of the individual grains within the glass and reprecipitation of the Si_3N_4 across the interface reinforcing the joint. © 1997 Elsevier Science Limited.

1 Introduction

One of the needs for high performance engineering ceramics, if they are to become useful materials in the future, is the ability to fabricate them into useful and reliable shapes. Because ceramics are brittle and difficult to machine, complex ceramic structures are usually built up from simply-shaped components. For this reason, during the last few years, joining of silicon nitride has been the subject of considerable research.¹ There is no prevalent method analogous to metallurgical welding for joining two similar or dissimilar ceramics. The use of reactive metals or braze alloys that contain reactive metals has been investigated for joining

ceramics. Suganuma *et al.*² used aluminium to join silicon nitride (Si_3N_4) to itself and have reported strengths as high as 450–500 MPa. Si_3N_4 joins using commercial Cu–Ag–Ti braze alloys have produced four-point flexural strengths greater than 980 MPa.² The brazes mentioned may provide ductile strain relief when the thermal expansion mismatch is large. However, it is well known that differences in thermal expansion between metal and ceramics introduce thermal stresses in the joint upon cooling from the bonding temperatures, which may lead to catastrophic failure. Such joining methods are limited to relatively low temperature use, <800°C; thus, the high temperature capabilities of the ceramic are limited by the metals used and their inferior properties at these temperatures.

In ceramic–ceramic joining for high temperature applications, matching the elastic modulus and coefficient of thermal expansion of the ceramic and the bonding agent is more easily achieved with non-metallic joints. Silicon-nitride based ceramics are densified with various sintering aids (metal oxides). Upon cooling from the sintering temperature, the sintering aids leave an oxide or oxynitride glassy phase at the grain boundaries. One idea for joining these materials is to use glass or ceramic bonding agents with compositions that are similar to the grain boundary phase of the bulk ceramic. Chemical compatibility is guaranteed by the correct choice of glass compositions, and the joint can be made to resemble a single ceramic grain boundary. The molten glass wets the Si_3N_4 surface, penetrates the grain boundaries, and reacts with the individual grains.¹

2 Previous investigations

A number of studies have been conducted to develop materials and techniques for joining silicon-nitride based materials by effectively brazing with glasses which act as joining agents.

Early work carried out by Kaba *et al.*,⁴ investigated diffusion bonding of Si_3N_4 . Powder pellets of Si_3N_4 with 4 wt% Y_2O_3 were compressed at room temperature and sandwiched between hot-pressed silicon nitride. These were then fired at 1800°C using a pressure of 3 GPa for 1 h. No properties with the exception of hardness were measured and further scanning electron micrographs alone were used to substantiate the strong mechanical nature of the joints. Bates *et al.*⁵ successfully bonded silicon nitride (4 wt% Y_2O_3) in the green state by hot isostatic pressing (HIP) via self-bonding and slip interlayer methods. No mention of pressure, temperature or times used was made. The interlayer consisted of the same composition as that of the parent material. Room temperature strengths up to 880 MPa were achieved and strengths of 650 MPa reported for temperatures as high as 1300°C . However, drawbacks of these techniques include long processing times, high costs, and limitations of the size and of components.

Owada and Kobayashi⁶ used a MgCaSiON glass adhesive to join sintered Si_3N_4 . They used a pressure of 15 MPa and temperatures in the range of 1350 – 1600°C . Maximum strengths of 360 MPa were reported for samples joined at 1600°C but these specimens underwent rapid shrinkage. Johnson and Rowcliffe⁷ used a two-stage joining process in which one of the surfaces of the silicon nitride to be joined was first coated with a molten Mg–Si–Al–O glass. Glazing was performed at 1480°C for 15 min. Upon cooling, this glaze was ground to precisely control the thickness of the joint. A pressureless bonding heat treatment to form the joint followed, which consisted of 10 min at 1620°C . Ruptured samples that fractured in the joint area showed evidence of cracks resulting from thermal expansion mismatch between the parent material ($\sim 3.2 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$) and the adhesive glass ($\sim 5.96 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$).

The most recent break-through in the joining of silicon nitride-based ceramics has been the work carried out by Walls and Ueki in Japan.^{8,9} They have successfully self-bonded β -sialons using a glass/ceramic adhesive. The major difference between these adhesives and the pure-glass adhesives previously mentioned is that, at the bonding temperature, the joining material contains more solid than liquid phase. The main constituent is α - Si_3N_4 (45 wt%), and the remainder a mixture of oxides (Y_2O_3 , Al_2O_3 and SiO_2). The advantage of this type of adhesive is that it is closer in composition to the ceramic being joined and therefore the properties of the joint are not so different from the parent material. Ceramics were coated with a homogeneous slurry of the granulated adhesives. Joining was carried out using a small hot press

with a constant loading facility at 1600°C for 10 min (optimum conditions). A pressure of 2 MPa was applied during the heating and cooling stages of the process. A β -sialon glass ratio of 60:40 gave optimum bond strengths of 650 MPa (85% of the bond material strength). A post-joining HIP treatment improved the strength of both bonded and unbonded material by 4%, which has been attributed to a reduction in residual porosity. But, again, high processing costs of solid-state techniques may prevent their widespread application and the restriction of size and shape may prevent this method being feasible for the production of complex-shaped monolithic ceramics.

In the present study, joining techniques for Si_3N_4 -based ceramics are examined. Two different adhesives are investigated, an oxynitride and an oxide glass. Microstructures and properties are reported. Results of mechanical properties will be published in the future.

3 Experimental Procedure

3.1 Ceramic process

β -Sialon ceramics (adherent material) corresponding to a nominal z value of 1.5, densified with yttria, were prepared by ball-milling the relevant weighed powders of Si_3N_4 (H. C. Starck, LC12S), Y_2O_3 (Rare-earth products), Al_2O_3 (BDH Analar) and AlN (H. C. Starch, Grade B), for 72 h in isopropanol using sialon milling media. Weight percents used are given in Table 1. The mixtures were dried and sieved to $212 \mu\text{m}$. They were cold isostatically-pressed into $40 \times 15 \times 15 \text{ mm}$ bars and pressureless sintered in a powder bed (50 w/o silicon nitride + 50 w/o boron nitride) at 1700°C for 2 h in flowing nitrogen. Samples of the same composition were hot-pressed at 1700°C for 1 h, at a pressure of 30 MPa, under flowing nitrogen. X-ray diffraction showed that 100% β -sialon was produced in both instances and no other crystalline phase was present. It is to be mentioned that the starting stoichiometry was adjusted to give a theoretical oxynitride glass content of 11v/o of composition (in equivalent %) 28 Y; 56 Si; 16 Al; 83 O;

Table 1. Weight percent of raw powders

Composition	Y_2O_3	Si_3N_4	Al_2O_3	AlN	SiO_2	at% N
Adherent	6.51	69.49	20.61	5.39	—	—
β -sialon $z = 1.5$						
Adhesive A	49.94	9.82	12.89	—	27.35	7.8
Y–Si–Al–O–N 28:56:16:83:17						
Adhesive B	48.63	—	12.54	—	38.83	0
Y–Si–Al–O 28:56:16:100						

17 N, the same composition as that of the adherent glass material A. Glass B is of a similar cation composition but contains no nitrogen.

3.2 Glass formation

Glasses (used as adhesive materials) in the Y–Si–Al–O–N and Y–Si–Al–O systems were prepared by wet-homogenising the required weights of high purity metal oxides in a ball mill for 24 h, and then dried. Weight percents of the respective compositions are given in Table 1. Pellets of the relevant mixture were compacted in a steel die under a uniaxial pressure of 600 MPa. The samples were melted at 1700°C in molecular nitrogen in a boron nitride-lined graphite crucible so that, when the liquid melted, there was no chemical reaction with the crucibles. After melting, the crucible was withdrawn rapidly from the hot zone and the glass melt poured under air into a pre-heated graphite mould at 850–900°C and annealed within this temperature range for 1 h prior to slow furnace cooling. X-ray diffraction analysis was carried out on both glasses using a Phillips X-ray powder diffractometer using Cu–K α radiation in order to confirm their amorphous nature. Densities were measured using the Archimedes method. Hardness measurements were carried out on polished specimens using a Leco micro-hardness tester using a 200 g load for glasses and 1-kg load for ceramics. A Stanton-Redcroft 873-4 Differential thermal analyser (DTA), was used to determine the glass transition temperature (T_g) and crystallisation (T_c) temperatures under flowing nitrogen.

3.3 Joining experiments

Sintered sialon samples to be joined 15 × 15 × 5 mm, were polished to <20 μ m surface finish. Hot-pressed discs, of 20-mm diameter and 5-mm thickness, were also polished to the same finish. Samples were cleaned in acetone followed by methanol for 10 min in an ultrasonic bath prior to joining experiments. Glasses to be used as

adhesives were crushed and sieved to particle sizes less than 53 μ m. To coat adherents, the glass powder was mixed with isopropanol in a 1:3 weight ratio to form a homogeneous slurry. Mixing was carried out by ultrasonic agitation. For the joining experiments, the slurry was sprayed onto the surface of the sialons using a small spray bottle. This method allows better control of joint thickness and distribution of the adhesive layer. Hot-pressed samples were joined using adhesive A at temperatures of 1130, 1400 and 1600°C, respectively, for 1 h under flowing nitrogen. Direct contact between sialon and graphite was eliminated by use of a boron nitride lining. A pressure of 5 MPa was applied continuously to the samples during the heating, holding and cooling stages of the bonding processes.

Pressureless joined samples were bonded at 1600°C for 1 h in a nitrogen atmosphere. The same sample preparation was carried out prior to joining, as outlined above. Both adhesives, A and B, were investigated at the same time in a sandwich configuration, as shown in Fig. 1. Samples to be joined were placed in a boron nitride-lined crucible. After bonding experiments, densities and hardness were measured. SEM analysis was used to examine micro-structures of joined samples.

4 Results and Discussion

4.1 Adhesive compositions

Bulk glasses used in this investigation as adhesive materials were calculated to be the same theoretical composition as the grain-boundary material present in the sintered and hot-pressed sialon under the conditions previously mentioned. The only difference between the two glasses being the nitrogen and oxygen contents, the cation composition remaining the same. These glasses permit the observations of the effects of nitrogen content upon joining of silicon nitride-based ceramics

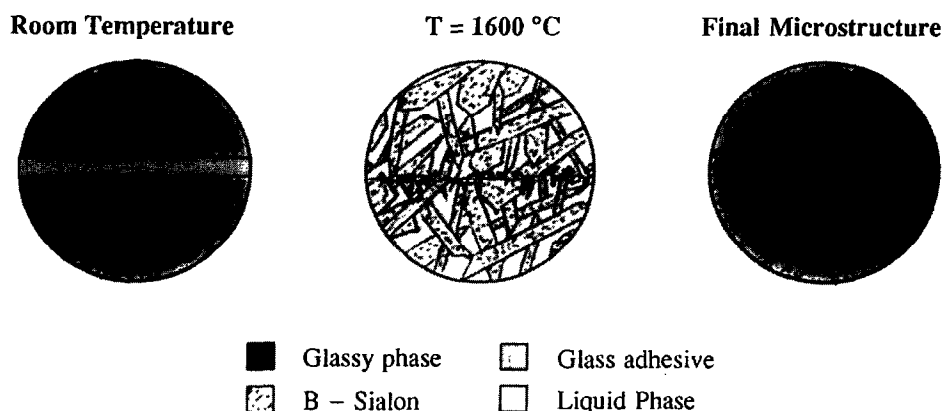


Fig. 1. Schematic representation of joining process.

under similar conditions. Each of the adhesive glasses was observed to be amorphous on the basis of X-ray diffraction data. Physical properties of the bulk glasses were investigated prior to bonding experiments. Table 2 reports densities for the adhesive glasses and shows that glass density increases with the incorporation of nitrogen into the glass structure. This is due to the closer packing of atoms in the glasses with the substitution of N, which is in three-fold co-ordination, with O, which is in two-fold co-ordination. As a result, the number of bonds between $\text{Si}(\text{O},\text{N})_x$ tetrahedra in the network would increase¹¹ and this would be expected to increase the density of the glass. The stronger covalent nature of the Si-N bond compared with the Si-O bond is also a contributing factor. Table 2 also shows the variation of the micro-hardness of the glasses and reflects the greater structural compactness of the nitrogen containing glass. Since the substitution of N for O increases the co-ordination number of the anion in the glass structure, the glass transition temperature would be expected to increase as the nitrogen content of the glass increased, and this can be observed here. The oxide glass exhibits a T_g of 937°C and the substitution of nitrogen into the network increases it to 985°C , a 48°C difference. Messier,¹² found similar trends in the same glass system. When the cation ratios were held constant, the T_g increased linearly with an increase in the atomic percent nitrogen in the glass.

Thermal expansion coefficients reported here are from work which was previously carried out on these glass systems by Hampshire *et al.*¹³ This is a very important property as mismatches between the adherents and adhesive materials may result in

thermal stresses leading to a reduction in mechanical properties of the overall ceramic material. Peterson and Tien¹⁴ reported that the thermal expansion of the glass depends on the asymmetry of the amplitude of the thermal vibrations in the glass. The amplitudes of the thermal vibrations are small when there are many strong bonds present in the network. As a result the thermal expansion decreases as the rigidity of the glass network increases, which is the case incurred by the substitution of N for O in the glass structure. Loehman¹⁵ has shown that the thermal expansion coefficient of Y-Sialon glasses decrease with an increase in nitrogen content, from $7.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ for a pure oxide glass to approximately $4 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ for a glass containing 6 at% nitrogen, composition-dependent. The average expansion coefficient may be further reduced by the dissolution of β -sialon into the glass during the bonding process (β -sialon having an expansion coefficient of $\sim 3.2 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$) making it closer still to that of the adherent material. The significance of thermal expansion mismatch to the strength of Si_3N_4 joints is discussed by Johnson and Rowcliffe.⁷

4.2 Bond investigations and microstructures

Examination of bonds produced showed that all experiments using both types of adherents and adhesive materials resulted in intact joints. Thickness of the joined regions varied between 0.5 and $10 \mu\text{m}$, depending on the glass composition and the joining temperature. Some of the joints could not be discerned visually and were difficult to detect during optical examination, primarily those joined at temperatures in excess of 1400°C . Control of the bond thicknesses down to such small detail is very promising in itself. Some authors report bond thicknesses in the region of 2 to $100 \mu\text{m}$.^{3,8,16} Table 3 summarises all test parameters and results. All testing was carried out for 1 h in a nitrogen atmosphere.

The following SEMs, (Figs 3–5) show the microstructures of joints produced via the hot-pressing

Table 2. Physical properties of as-fired glasses

Glass	Density (g cm^{-3})	Microhardness (kg mm^{-2})	T_g ($^\circ\text{C}$)	T_c (1°C)	T_c (2°C)	$\alpha (\times 10^{-6})$ ($^\circ\text{C}^{-1}$)
Y-sialo	3.62	848	937	1148	1204	6.64
Y-sialon	3.78	1025	985	1184	1250	5.67

Table 3. Summary of bonding results

Material joined	Bonding adhesive	Temperature ($^\circ\text{C}$) and pressure (MPa)	Description of joint	Hardness of ceramic (Hv) (n=10)	Hardness across joint (Hv) (n=10)
Hot-pressed sialon	Y-sialon	1130 $^\circ\text{C}$ 5MPa	Intact joint, non-uniform thickness 5– $10 \mu\text{m}$, many voids present	1549	1371
Hot-pressed sialon	Y-sialon	1400 $^\circ\text{C}$ 5MPa	Intact joint, non-uniform thickness < $1\text{--}2 \mu\text{m}$, few voids present	1572	1550
Hot-pressed sialon	Y-sialon	1600 $^\circ\text{C}$ 5MPa	Intact joint, almost indistinguishable. Uniform thickness < $0.5 \mu\text{m}$,	1535	1553
Pressureless sintered sialon	Y-sialo	1600 $^\circ\text{C}$ 0MPa	Intact joint, almost indistinguishable. Uniform thickness < $0.5 \mu\text{m}$,	1440	1431
Pressureless sintered sialon	Y-sialon	1600 $^\circ\text{C}$ 0MPa	Almost empty joint, $5 \mu\text{m}$ in thickness Small pockets of glass	1440	—

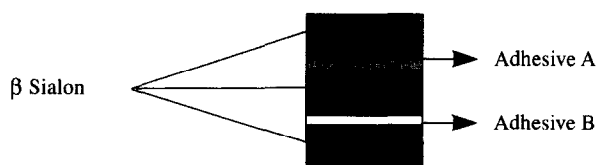


Fig. 2. Schematic representation of sample configuration for pressurelessly-bonded ceramics.

technique. The temperature at which successful joining can be conducted is limited by the kinetics of the Y-sialon system. In this system, the eutectic temperature lies at approximately 1350°C;⁸ hence, the joining temperature must exceed this. An upper limit on the joining temperature is approximately 1700°C, when decomposition becomes a problem, and any higher would not be economically viable from an industrial point of view. Another aspect which also limits the temperature range for bonding is the dissolution precipitation reaction of the β -sialon. Walls *et al.* report this to occur between 1500°C and 1600°C. Ramesh *et al.*¹⁷ report transformations occurring at temperatures as low as 1350°C for $z=0.2$ sialons. At higher temperatures, the kinetics of the solution reprecipitation process is accelerated, hence the choice of 1600°C as the maximum bonding temperature reduces the heat-treatment at the bonding temperature (Fig. 2).

At 1130°C, a non-uniform continuous glassy layer was produced across the interface. Bond thicknesses varied from 3 μm up to a maximum of 10 μm in parts. This may be a result of small irregularities in the surface finish of the ceramic parts present prior to bonding. At 1130°C, a non-uniform glassy bond up to 10 μm in thickness resulted. Many voids were present which may be due to the high viscosity of the glass at such low temperatures which can result in incomplete wetting of the ceramic. Hardness measurements decreased by a value of approximately 200 Hv across the joint region as compared to that of the parent ceramic. The glass

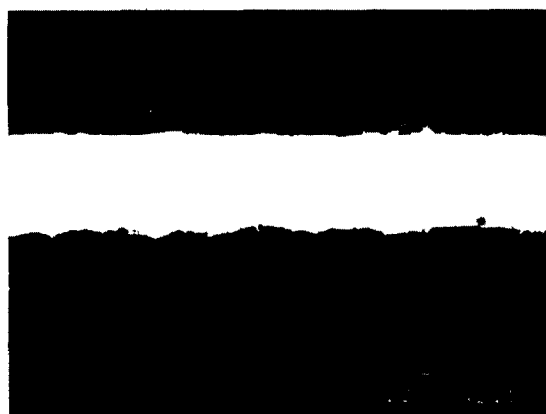


Fig. 3. Hot-pressed sialon bonded at 1130°C for 1 h, 5 MPa, using Y-sialon glass.

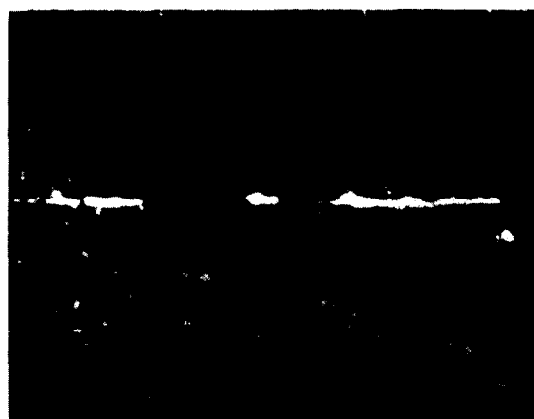


Fig. 4. Hot-pressed sialon bonded at 1400°C for 1 h, 5 MPa, using Y-sialon glass.

present in the joint was found during SEM investigation to contain very fine crystalline phases which were formed during the bonding process. The increase in hardness of the bulk glass from a value of 1025 to 1371 Hv for the glass present in the join may be characteristic of these secondary phases. However, the presence of this large glassy region is likely to depreciate considerably the properties of the overall ceramic as a result of the low resistance of glasses to crack propagation and their susceptibility to creep.

Tests carried out at 1400°C resulted in a more uniform bonded region which was difficult to ascertain in places. Bond thicknesses varied from 0.5 μm up to a maximum of 2 μm . Fewer voids were detected as compared to the samples bonded at 1130°C. This can be attributed to the decrease in the viscosity of the glass adhesive at higher temperatures and the increases in wettability of the ceramic. Diffusion of the molten glass from the joining interface into the parent material is evident, indicating that reaction has occurred at the liquid/solid interface. This result ties in with the findings of Ramesh *et al.* and shows that the solution



Fig. 5. Hot-pressed sialon bonded at 1600°C for 1 h, 5 MPa, using Y-sialon glass.

precipitation process can and has occurred at temperatures lower than 1500°C. Hardness measurements in the parent ceramic and across the bond region show no degradation in value, further substantiating our conclusions.

Figure 5 shows a sample bonded at 1600°C for 1 h; the bonded region is almost invisible. Solution and reprecipitation of the β grains has occurred and growth of Si_3N_4 across the interfaces has taken place. This reinforces the bonded region and replicates the overall micro-structure of the parent ceramic resulting in a monolithic ceramic. This again is a result of the decrease in the viscosity of the glass adhesive at this temperature which is well above its eutectic temperature and, therefore, the increases in wettability of the ceramic. Hardness values further reiterate these findings, showing no difference in value between the parent ceramic and the joint. Pressurelessly bonded samples are shown in Figs 6 and 7. These were bonded at a temperature of 1600°C for 1 h using both adhesive glasses. Figure 6 shows the ceramics bonded using the Y-sialon glass. An empty region has been shown which is characteristic of the bulk of the bonded region. Some areas were found to contain small pockets of the remaining glass. Figure 7 shows the micro-structure of ceramic bonded using the Y-sialo glass. This joint, which was very difficult to detect, is an excellent example of a good join. The interfacial contact is excellent and there is very little, if any, variation between the micro-structure of the parent ceramic and the bonded region. Hardness values also show no reduction in value. At this temperature, solution of β grains has occurred and reprecipitation of the same across the bond interface is evident. Plausible explanations for these findings would lie in the adhesive compositions used and the temperature ranges indicated. The Y-sialon adhesive may have exceeded the maximum

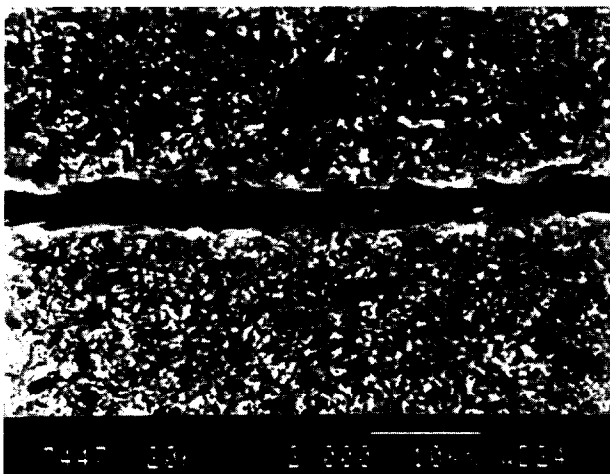


Fig. 6. Sintered sialon, pressurelessly joined at 1600°C for 1 h using Y-sialon glass.

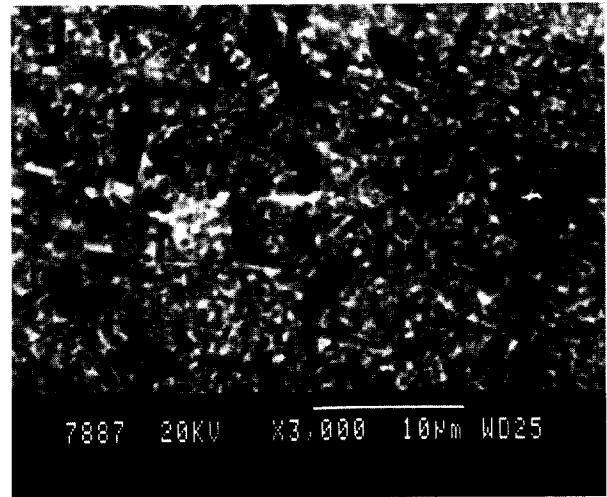


Fig. 7. Sintered sialon, pressurelessly joined at 1600°C for 1 h using Y-sialo glass.

nitrogen solubility range for reaction to occur and hence the resulting empty joint. The success of the Y-sialo glass as a bonding adhesive under pressureless conditions is due to the greater solubility of the oxide glass for nitrogen. This enhances the β - Si_3N_4 solution precipitation reaction which promotes wetting and improve adhesion at the bond interfaces.

5 Conclusions

Y-Sialon and Y-sialo glasses of the same ceramic grain boundary composition have been used to successfully bond hot-pressed and sintered sialon ceramics to themselves. Hot-pressed samples were bonded using the nitrogen containing glass, adhesive A, at temperatures of 1130, 1400 and 1600°C, respectively, under an applied pressure of 5 MPa, all producing intact bonds. At 1400°C, there is evidence of the solution reprecipitation of β -sialon occurring. Tests carried out at 1600°C resulted in favourable bond micro-structures similar to those present in the parent material. These bonding experiments indicate that these materials can be successfully joined by a diffusional reaction-bonding under very low pressures.

Pressureless-bonded samples were investigated using both adhesive glasses at a temperature of 1600°C. The nitrogen containing glass resulted in an empty joint showing no reaction between the ceramic and glass. Samples bonded using the oxide glass reveal the most promising micro-structures. The joint thickness is approximately 0.5 μm across the joint and solution and reprecipitation of β grains across the joint is evident. This joining composition is miscible with the sialon grain-boundary phase and readily penetrates the grain boundaries.

These results indicate that low-pressure bonding can be used to join silicon nitride-based ceramics, but also that bonding can be achieved via a pressureless-bonding route which allows greater dexterity for the shape, size and complexity of ceramic components.

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