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Ceramic matrix composite–metal brazed joints

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A silicon nitride fibre-reinforced cordierite glass ceramic matrix composite has been brazed to titanium and stainless steel in argon with four different interlayer materials, copper, nickel, tungsten and a metal matrix composite (mmc). Joints were tested in shear and all but one failed in the ceramic composite. The highest strength joint, using a metal matrix interlayer to join cmc to stainless steel failed in the mmc at 106 MPa. Silver–copper eutectic braze and aluminium braze can be used to join metals to titanium-coated cmc, producing joints with low levels of interfacial defects. Some joints, however, show debonding at the edges where residual shear stresses are highest.

1. Introduction

Ceramic matrix composite (cmc) materials have attracted considerable interest in recent years as a class of materials which can combine the strength and high-temperature properties of ceramics with improved damage tolerance. In many applications it is necessary to join ceramics to metals and brazing is a common means of achieving this. Much work has been done on brazing ceramics to metals [1, 2] but there is little information in the literature concerning the brazing of cmc materials to metals. Most ceramic–metal brazing is carried out in vacuum furnaces but a simpler approach involves the use of inert atmospheres. The brazing reported in this work has all been performed in a flowing argon atmosphere in a silica tube furnace which offers a less expensive method compared to using a vacuum furnace, but one potential problem with this approach is the entrapment of air within the joint which might inhibit wetting and bonding.

For a brazed joint to be successful, it is necessary that the liquid braze metal wets and bonds to the ceramic. The wettability of ceramics by liquid metals can be improved by incorporating an active metal, such as titanium or hafnium, into the braze so that it reacts with the ceramic surface [3]. Another approach, and the one used in this work, is to precoat the ceramic with a wetting promoter. This was done by sputter coating a thin layer of titanium on to the cmc surface prior to brazing. A further problem when making metal–ceramic joints is residual stress caused by the difference in thermal expansion coefficients between most metals and ceramics. These are shown in Table I. When a brazed joint is cooled the metal contracts relative to the ceramic and induces stresses in the body. In some cases these are high enough to fracture the ceramic [4] so it is necessary to introduce an interlayer between the metal and the ceramic. Complex interlayers of layered materials with incremented thermal expansion coefficients, or compliant open structures, such as corrugated sheets, may be used. More commonly, solid metal interlayers are used which deform or which have expansion coefficients near to that of the ceramic.

A range of separate interlayer materials has been used in this work. These include copper, nickel, tungsten and, more unusually, a metal matrix composite (mmc). The mmc was a continuous silicon carbide fibre-reinforced aluminium which has low thermal expansion properties, dominated by the fibres, but is also ductile on a microscale which may allow localized strains to be taken up. The use of an interlayer is likely to be important for fibre-reinforced cmc materials which tend to have relatively low interfacial shear strengths and will be susceptible to residual shear stress when bonded in certain orientations.

2. Experimental procedure

The ceramic matrix composite used in this work was a cordierite ($2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$) glass ceramic reinforced with continuous silicon carbide fibres in a [0, 90°] layup. Plaques of 4 mm thickness were cut into 5 mm × 5 mm blocks using a diamond saw. The metals used were a titanium alloy, Ti–6Al–4V, and a stainless steel, Fe–18Cr–8Ni, both cut into blocks measuring 5 mm × 4 mm × 12 mm. A silver–copper (Ag–28 Cu) eutectic alloy in 50 μm thick foil was used as the braze alloy for most of the joints. High-purity aluminium (99.999%) was used to braze the mmc interlayer to the cmc. The composite was prepared by sputter cleaning then sputter coating with 1 μm titanium. The bonding surfaces of the metals were abraded with 1200 grit silicon carbide paper then ultrasonically cleaned in acetone. Braze metal foils were cleaned by wiping with methanol.

Copper, nickel and tungsten sheet, with typical purity 99.9%, and a thickness of 1 mm and a silicon carbide (continuous fibre)-reinforced aluminium metal matrix composite with a [0, + 45°, 0, − 45°] layup, 1.5 mm thick, were used as interlayers. These were prepared by abrasion and cleaning in the same way as

TABLE I Materials

	Thermal expansion coefficients ($\times 10^{-6}$)	
	(293 K)	(500 K)
Titanium	8.6	9.9
Copper	16.5	4.6
Aluminium	23.1	26.4
Nickel	13.3	
Silicon carbide	3.3	4.2
Cordierite	2	

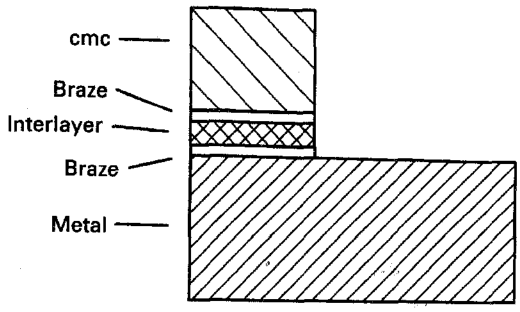

Figure 1 A brazed joint specimen showing the cmc, interlayer and metal substrate.

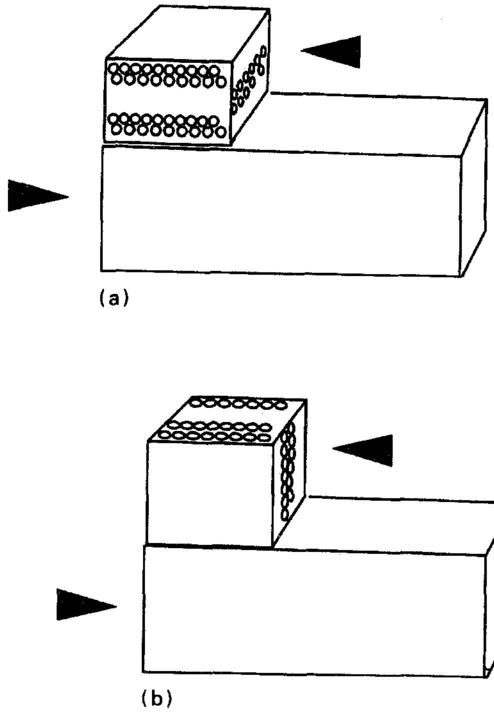

Figure 2 The two cmc orientations used for joining. Cmcf joints (a) were brazed parallel to the fibre layup planes; the cmce joints (b) were made normal to the layup plane.

the titanium. The materials used in this work are given in Table I.

Specimens comprising cmc–braze foil–interlayer–braze foil–metal were assembled as shown in Fig. 1. The cmc was joined in two different orientations to investigate the effect of joining parallel to the layup plane (denoted cmcf) and to the fibre ends (denoted cmce). The former orientation gave bond areas of 25 mm^2 and the latter gave bond areas of 20 mm^2. The different joining orientations are shown in Fig. 2. The specimens were held using a single turn of molybdenum wire which was twisted tight to clamp the pieces together before being inserted into the furnace. Electronic grade argon was flushed through the furnace for 15 min at a flow rate of 0.5 l min^{-1}. This was reduced to approximately 0.1 l min^{-1} when the furnace was turned on with a heating rate of approximately 1 °C s^{-1} until the temperature reached 800 °C for 10 min. The specimens were allowed to cool to 200 °C in the furnace under flowing argon; they were then removed to air.

One side of each brazed specimen was then polished with silicon carbide paper and 1 μm diamond paste before being gold coated for analysis in the scanning electron microscope (SEM). Joint microstructures were examined using backscattered imaging at 20 kV accelerating voltage and elemental distributions were measured with energy-dispersive X-ray analysis (EDX).

Each specimen was tested in shear using the arrangement shown in Fig. 3. The crosshead speed was 0.5 mm min^{-1} and all tests were performed at room temperature. Fracture surfaces were gold-coated and examined in the SEM.

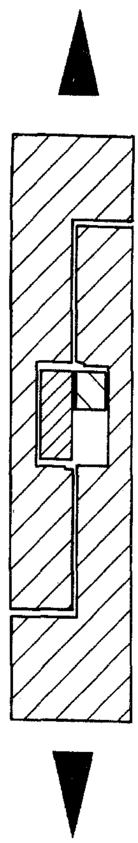

Figure 3 The shear test arrangement.

3. Results

All the brazed joints between the metal interlayers and the titanium and steel substrates were found to be defect free. Because the brazing of ceramic matrix composites is less well documented, the cmc–braze interfaces will be discussed.

3.1. Titanium–cmc joints

When the cmc was joined to titanium with copper interlayers the braze retained its eutectic microstructure but penetrated deeply into the copper, developing a convoluted interface between the interlayer and the braze. The cmcf–braze interface appeared well-bonded with very few defects. The titanium coating remained visible on the both cmcf and cmce orientations when the joint was viewed in section, but in the latter case it was less evident on the fibre ends compared with the matrix. This is seen in Fig. 4a. EDX analysis revealed lower concentrations of titanium on the fibre ends but this effect was only seen in joints brazed with copper interlayers. The braze was detached from the titanium-coated cmc at the edges of specimens where the thermal mismatch shear stresses is greatest. In testing, however, the fracture path always ran through the cmc and not through the detached regions (see below).

Joints brazed with nickel interlayers exhibited well-bonded defect-free interfaces in both cmce and cmcf orientations. Fig. 4b shows the joint between cmcf and a nickel interlayer; the titanium layer is visible and EDX measurements show that the layer contains titanium and nickel which had diffused through the braze which appears to have lost its eutectic microstructure, now consisting of a silver-rich phase with a small amount of copper-rich second phase.

Tungsten interlayers produced highly defective joints (Fig. 4c) with cracking visible at both the ceramic–braze interface and the braze–interlayer interface. Failure during shear testing, however, occurred within the cmc.

The use of a metal matrix composite as an interlayer with aluminium braze produced good joints with the ceramic appearing to be fully wetted. The titanium coating was no longer present on the ceramic although small (e.g. 5 μm) titanium-rich zones were found in the aluminium. The brazed cmc–mmc interface is shown in Fig. 4d.

3.2. Stainless steel–cmc joints

The brazed cmc interface was mostly defect-free when copper interlayers were used although the braze has detached from the titanium coating on the ceramic at some specimen edges. When nickel interlayers were used there was also extensive cracking at the braze–titanium coating interface near specimen edges where the braze and underlying metal have contacted (Fig. 5a). Segregation of the braze alloy has also occurred, leaving a silver-rich layer some 10 μm thick near the ceramic and a copper-rich layer near the nickel. Extensive interdiffusion of the braze and nickel has occurred. The nickel has also diffused through the braze to concentrate in the titanium coating.

Joints made with the tungsten interlayer are heavily cracked at both the ceramic–braze interface and the interlayer–braze interface. There is also some evidence in the cmce brazed specimens that fibres in the

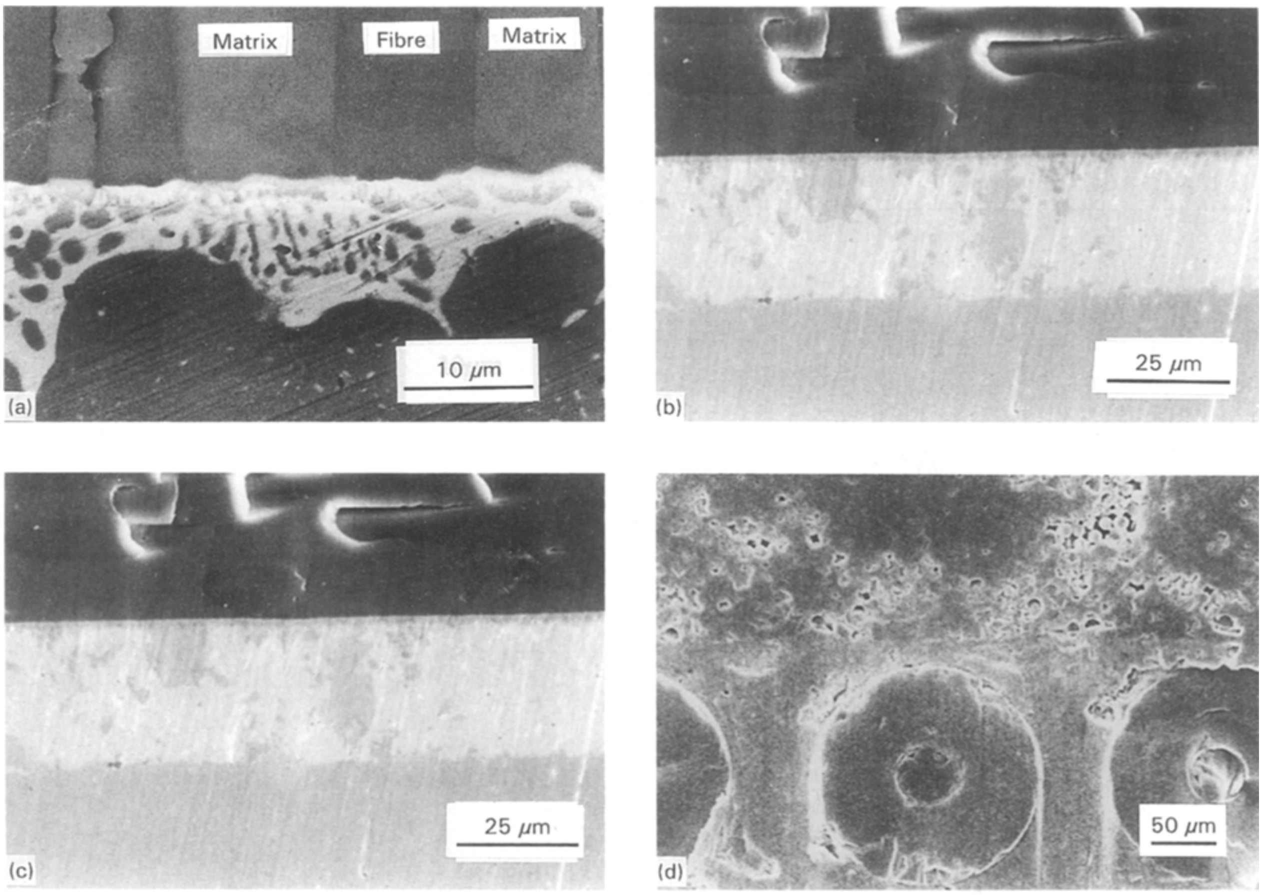

Figure 4 Titanium–cmc joints. (a) Scanning electron micrograph of a section through a brazed cmce joint with a copper interlayer. The variation in the titanium coating on the matrix and fibres is evident. (b) A cmcf joint brazed with a nickel interlayer. (c) A defective joint produced when using a tungsten interlayer. (d) The brazed interface between a cmc and an mmc using aluminium braze.

composite had cracked parallel to the brazed surface, about 5 μm into the ceramic (Fig. 5b).

Metal matrix composite interlayers produced defect-free joints using an aluminium braze.

3.3. Shear tests

All joints were tested in shear. The results are shown in Table II where a wide range of joint strengths can be seen. It is clear that those joints brazed normal to the layup plane in the composite (cmce orientation) are significantly stronger than those brazed parallel to the layup plane (cmcf orientation). All joints failed within the cmc, usually within one or two ply thicknesses of the brazed surface with the exception of one joint which was brazed with an mmc interlayer. A typical fracture surface from a cmcf specimen is shown in Fig. 6a where loose fibres are visible. A fracture surface from an edge-brazed (cmce) specimen is shown in Fig. 6b. Failure has occurred within 50 μm of the brazed interface with the crack running through the silicon carbide fibres, showing little evidence of fibre pull-out.

Joints which used tungsten interlayers are weakest in the cmce orientation but give the highest shear strength (11.3 MPa) of any of the homogeneous metal interlayer specimens in the cmcf orientation. Other face-brazed (cmcf) specimens gave poor shear strengths of a few megapascals. The mmc interlayers give an order of magnitude improvement in shear strength in the cmcf orientation for cmc–titanium joints compared with nickel and copper interlayers and produced the highest joint strength measured in this work (106.1 MPa) for cmce–stainless steel

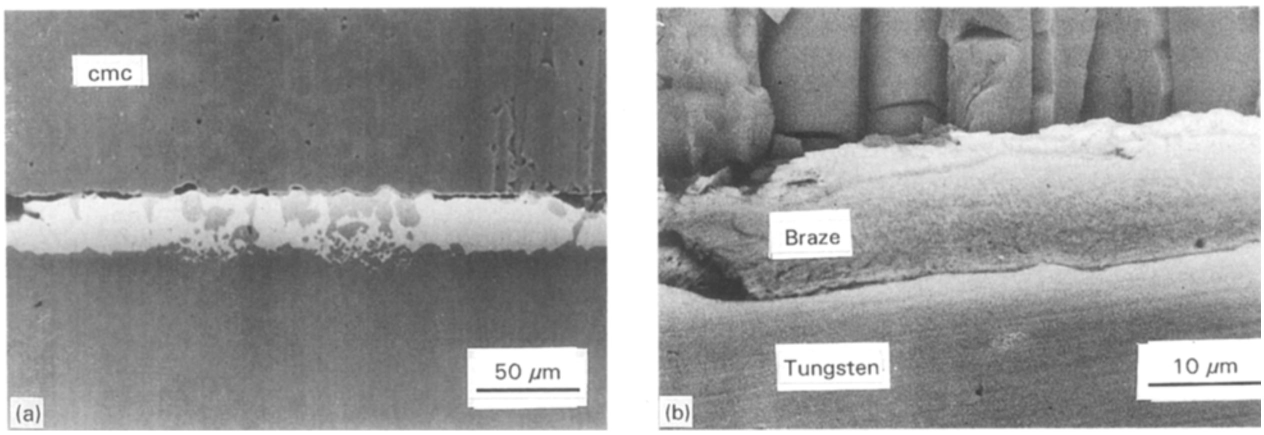

Figure 5 Stainless steel–cmc joints: (a) scanning electron micrograph of a section through a cmc-nickel interlayer joint, (b) cmce specimen brazed with a tungsten interlayer showing cracked fibres in the composite.

TABLE II Shear test results

Joint	Composite orientation	Interlayer	Shear strength (MPa)	Locus of failure
cmc-titanium	cmcf	Ni	1.64	cmc
	cmcf	Ni	2.5	cmc
	cmce	Ni	26.2	cmc
	cmce	Ni	40.6	cmc
	cmcf	Cu	1.5	cmc
	cmcf	Cu	3.4	cmc
	cmce	Cu	23.4	cmc
	cmce	Cu	91.6	cmc
	cmcf	W	11.3	cmc
	cmce	W	8.0	cmc
	cmcf	mmc	21.7	cmc
	cmcf	mmc	17.5	cmc
	cmce	mmc	25.2	cmc
cmc-stainless steel	cmcf	Ni	1.75	cmc
	cmcf	Ni	0	cmc
	cmce	Ni	53.0	cmc
	cmce	Ni	57.5	cmc
	cmcf	Cu	4.0	cmc
	cmce	Cu	41.6	cmc
	cmce	Cu	56.2	cmc
	cmcf	W	5.0	cmc
	cmce	W	8.4	cmc
	cmcf	mmc	10.5	cmc
	cmce	mmc	106.1	mmc

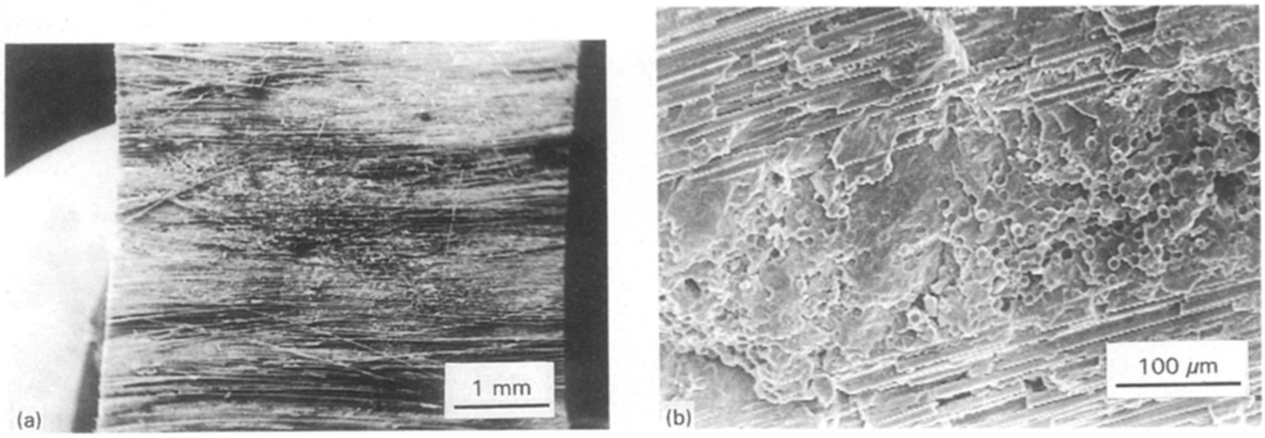

Figure 6 Scanning electron micrographs of cmc fracture surfaces: (a) cmcf orientation showing loose silicon carbide fibres, (b) cmce orientation showing broken fibres.

joints. In this combination, the mmc interlayer failed during the shear testing rather than the cmc, suggesting that there were low levels of residual stress in the ceramic.

4. Discussion

A wide range of joint strengths have been measured for cmc joints brazed to stainless steel and titanium with interlayers of copper, nickel, tungsten and mmc. Most of the joints made with the cmc brazed parallel to the fibre layup plane were very weak (less than 4 MPa) and do not represent structurally useful values. Interlayers are used to reduce high residual stresses in the ceramic caused by differences in the thermal expansion coefficients of the metals and ceramic. Tungsten is sometimes suggested for an interlayer in metal–ceramic joints due to its low expansion coefficient, but in this work its use has resulted in highly defective joints, possibly caused by the braze itself contracting and cracking during cooling. Other types of interlayer can be used for their compliant properties, taking up thermal mismatch strains by deforming. The reduction of residual stress in the ceramic is particularly important when the cmc is brazed parallel to the layup plane, because failure is induced by shearing along the fibre planes. The novel use of metal matrix composite interlayers has improved the strength of cmc joints probably by allowing small-scale deformation within the aluminium whilst the macroscopic thermal expansion properties remain low, dominated by the silicon carbide fibres in the mmc. Aluminium also has a lower melting point and yield strength than the Ag–Cu braze used in the other joints and will therefore generate lower residual stresses. The brazing temperature used for all joints was 800 °C, well above the melting point of aluminium (660 °C) but it was evident that the aluminium did not flow out of the joint during brazing. This may be due to surface tension effects keeping the liquid metal in place because the joint was not heavily loaded during heating.

All the brazed joints, with one exception, failed within the ceramic close to the brazed layer. The cmcf orientation joints failed along the fibre layup planes leaving a fracture surface covered in loose silicon carbide fibres. The edge-brazed (cmce) specimen fracture surfaces were relatively smooth with fractured fibres showing little sign of fibre pull-out. Because fibre–matrix debonding and pull-out are intended to be toughening mechanisms and might be expected, it may be that the brazing process has increased the fibre–matrix bonding and thus reduced the toughness of the cmc. Another explanation may be that the fibres had already broken near the brazed interface due to residual stresses but evidence for this was only seen in specimens with tungsten interlayers.

Despite extensive cracking in some of the braze layers, no failures occurred in the brazed regions. However, only small bond areas, 20 and 25 mm^2, were used, and when larger specimens are made, the problem of residual stress will be exacerbated. This was demonstrated by brazing a 50 mm long cmc sample to titanium with a 1 mm thick nickel interlayer. On cooling, the specimen audibly and visibly cracked over a period of days until it had minimal residual stress. Therefore, the methods used in this work to join small cmc pieces would need further refinement before large composites could be joined, although a variety of options are available.

5. Conclusion

A ceramic matrix composite (cmc) has been brazed to titanium and stainless steel using silver–copper braze in argon and four different interlayers: copper, nickel, tungsten and metal matrix composite. The cmc needs to be coated with titanium to induce wetting and bonding. Joints brazed normal to the cmc layup plane are generally much stronger than those brazed parallel to the layup plane.

The highest joint strength measured for a cmc–titanium joint was 91.6 MPa in shear using a 1.0 mm copper interlayer. The highest cmc–stainless steel joint strength was 106.1 MPa using a 1.5 mm mmc interlayer. Defect-free joints are produced with all combinations except when tungsten interlayers are used, but some debonding is evident at the edges of other specimens where residual shear stresses are highest. In shear testing, all joints except the strongest failed within the ceramic composite.

Acknowledgement
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