

Actively brazed alumina to alumina joints using CuTi, CuZr and eutectic AgCuTi filler alloys

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Abstract

Al₂O₃ to Al₂O₃ joints were produced using a one stage active brazing technique based on CuTi, CuZr and AgCuTi active brazing alloys. Single- and double-butt joints were used for microstructural (light and scanning electron microscopy and X-ray diffraction) and mechanical property (double shear test) studies, respectively. The joints produced with CuZr filler alloys (containing 2%, 4%, 6% and 8% Zr, wt%) showed low shear strengths (0.2–0.4 MPa) due to the presence of ZrO₂ at the braze–substrate interface and failed in mode I crack opening. Higher shear strengths of 15–24 and 42 MPa were obtained by using CuTi filler alloys (containing 2%, 4%, 6%, 8% and 10% Ti, wt%) (mode II/mode III dependent on the Ti content) and the eutectic (Ag–27 wt% Cu–5 wt% Ti) alloy, respectively. The high shear strengths were attributed to the small amount of Cu₂(AlTi)₄O at the braze–substrate interface and led, in the case of the ternary alloy, to the failure of the substrate rather than the braze (mode III axial splitting).

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1. Introduction

The development of new techniques to join structural ceramics has the potential of significantly broadening the applications for advanced ceramics. A commonly used method to join structural ceramics to metals is known as the moly-manganese process [1–3]. This process is well-established and is capable of producing reliable joints between ceramics and metallic alloys. However, the moly-manganese process requires two processing steps, namely, metallization of the ceramic, which is conducted at a high temperature (~1500 °C), and subsequent brazing, and is therefore time consuming. Furthermore, joint properties are sensitive to process variables and hence precise process control at high temperatures is required to obtain reliable joints, consequently this process is expensive.

There are several methods of active metal brazing. One method involves a sheet of titanium (the most commonly used active element) clad between two sheets of a conventional brazing metal [4]. Another technique uses titanium hydride powder mixed with powders of conventional brazing metals such as Cu and Ni [5]. However, the most economical brazing methods utilize an alloy of a base filler metal and an active element [6]. With such an alloy the brazing becomes a simple and economical one-step process.

Although active metal brazing has been investigated since 1940, it has not yet been widely accepted for joining structural ceramics because of inconsistent joint properties [6]. However, research on joining of metals to ceramics using active metal brazing has produced some encouraging results [7–17]. It has been demonstrated that active brazing joints require the careful selection of the brazing alloy, brazing temperature, heating rate, cooling rate, soaking time and brazing atmosphere [7–25] and of these the most important factor is the brazing alloy [9]. The most widely employed brazing alloys for metal to ceramic and ceramic to ceramic joints are systems based on Cu, Sn and Ag

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containing reactive elements such as Zr, Ti or Cr, or a combination of these elements [23–36].

Although knowledge is well advanced on the active brazing of metal to metal, and to a lesser but growing extent on metal to ceramic joints, much work is still required on both experimental and theoretical aspects of the active brazing of ceramic to ceramic. To date there are only a limited number of publications on the active brazing of carbon to carbon and ceramic to ceramic, and these are mainly concerned with carbide and nitride ceramics [31–45]; there is a dearth of information regarding the active brazing of oxide ceramics. High shear strength direct brazing between ceramic and ceramic or metal has been the focus of studies in an effort to select successful active filler metals [15–17,24–26,30,31,33–37]. For example, it has been reported that for joining partially stabilized zirconia to zirconia using a AgCuTi filler metal, both the brazing temperature and holding time have a strong influence on the strength of the joint. In this case the optimum temperature was 850 °C and the holding time was 30 min [30]. Other workers have found the selection of active filler alloys was important as it affected the failure mode of zirconia to zirconia joints [22]. Chang et al. [23] investigated the low temperature active brazing of alumina to alumina joints using a Sn based active filler alloy. Low shear strengths (less than 12.5 MPa) were obtained and this was attributed to the high thermal expansion mismatch between the ceramic and the brazing alloy. Results reported by Hongqi et al. [25] showed that the shear strength of brazed alumina to alumina joints using Ag57Cu38Ti5 filler alloy was affected by joining conditions but that high shear strengths up to 180 MPa could be obtained.

The work reported here concentrates on studying the role of the reactive elements, Zr and Ti, in reactive brazing alloys used to produce alumina to alumina single-butt and double-butt joints. Three alloy systems were investigated, CuZr, CuTi and AgCuTi; the percentage of the reactive element was varied in the binary systems. The microstructures of the resulting brazes were examined and the shear strength of the joints determined.

2. Experimental procedures

Polycrystalline alpha alumina powder of 99.8% purity and mean particle size 0.64 µm (supplied by ALM-41 Sumito, Chemical Company, UK) was used to produce substrates. The powder was cold pressed at a pressure of 120 MPa and sintered at 1600 °C to obtain alumina with porosity less than 4%. Substrates with nominal dimensions 14 mm × 14 mm × 10 mm were cut using a diamond cutting wheel. High purity Cu, Zr (Merck), Ti

Table 1

Nominal composition of alloys manufactured and used in this investigation.

Alloy designation	wt% Cu	wt% Zr	wt% Ti	wt% Ag
CuZr2	98	2	0	0
CuZr4	96	4	0	0
CuZr6	94	6	0	0
CuZr8	92	8	0	0
CuTi2	98	0	2	0
CuTi4	96	0	4	0
CuTi6	94	0	6	0
CuTi8	92	0	8	0
CuTi10	90	0	10	0
AgCuTi	27	0	5	68

and Ag (Fluka) powders were used to make the fillers. Ten Cu filler base active alloys containing different amounts of Zr and Ti, and an eutectic AgCuTi alloy were manufactured then used to produce filler paste powder mixtures. Table 1 summarizes the alloys and mixtures investigated.

The contact surfaces of the substrates were ground and polished with 1200 silicon carbide paper and 0.5 µm diamond pastes, respectively, to give surfaces with a CLA roughness of 0.05 µm. After ultrasonically cleaning in acetone, a paste of 0.15 g of the brazing filler and 0.05 g of glycerin was painted onto the prepared surface to give a thickness of paste of approximately 0.07 mm. Single brazed and double brazed alumina joints were assembled as shown in Fig. 1 and placed in a vacuum furnace (Vacuum Industry, USA), which was evacuated to a pressure of 5×10^{-5} Torr before heating up to the brazing temperature at the determined optimum heating rate of 40 °C/min. The optimum brazing temperatures were found to be 1100 °C for the copper-base binary filler metals and 980 °C for the silver-base eutectic filler metal. The samples were held at the brazing temperature for 5, 10, 15, 30, 60 and 90 min in order to investigate the effect of brazing time. After holding at the brazing temperatures for the required time, the assembly was cooled to 200 °C at a controlled cooling rate of 10 °C/min, and then left in the furnace to cool to room temperature. There was one exception to this procedure; when employing the AgCuTi braze the assembly was cooled to 800 °C, held for 10 min then cooled to room temperature as for the other alloys.

Transverse sections of the single-butt brazed samples were examined by optical and scanning electron microscopy (SEM) (Cambridge Instruments), with and without prior grinding and polishing. The polished samples were etched using a solution of 30 ml HCl, 10 ml FeCl₃ and 120 ml methanol. Also single

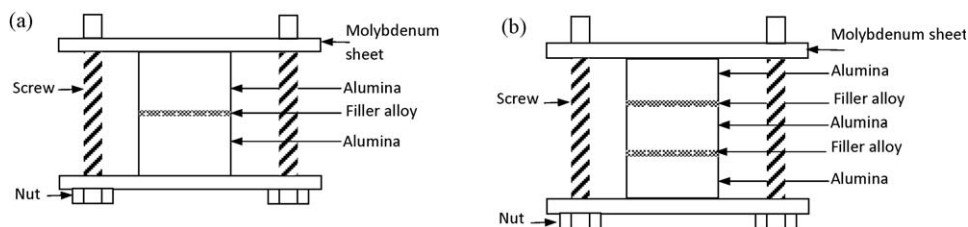


Fig. 1. Fixtures for producing (a) single-butt joint and (b) double-butt joint.

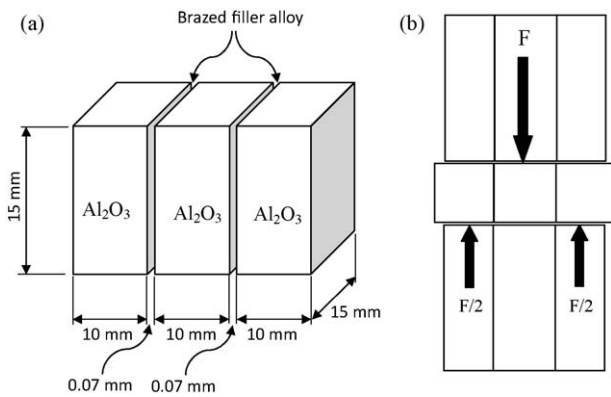


Fig. 2. Schematic illustration of the double brazed shear test (a) and the testing rig (b); the large arrows indicate the direction of the acting forces.

brazed shear tests were carried out in order to produce fracture surfaces for X-ray diffraction (XRD) analysis (Philips PW 1050 diffractometer) using a $\text{Cu K}\alpha 1$ radiation to identify any reaction product. The double brazed shear test (DBS) [6] was used to determine the strength of the joints. A diagram showing the basic principle of the DBS test is presented in Fig. 2. At least three tests were carried out on each combination of braze and processing conditions investigated in order to obtain a meaningful value for the shear strength.

It should be noted that DBS utilized in the present investigation is a new type of the shear test. The shear stresses are applied to the two brazed joints simultaneously and the

properties of the two joints can be determined. It is found, however that the shear strengths of both joints cannot be determined by a single test. This is because once one joint (the weaker one) fractures, the loading condition on the other joint becomes undesirable. Consequently, DBS test supplies the following information: (i) it provides the apparent shear strength of the weaker joint, and (ii) it can determine which joint is weaker.

Therefore when two different materials are used at the end blocks, one can measure the apparent shear strength of the weaker joint and also determine which material forms the stronger joint with the material used as the centre block. Certainly comparison of results obtained on mechanical tests of single-butt joints and DBS on samples of the same thickness would be useful but this is beyond the scope of the present study. Moreover it should be recognised that these tests do not provide the pure shear strength of a joined specimen, but just an apparent shear strength: when the crack propagates in the substrate instead of in the braze, the obtained result can not be defined as the shear strength of the joint.

3. Results and discussion

3.1. Structure of the joints

Examination of transverse sections and fracture surfaces demonstrated that long brazing times led to volatilization of the base brazing metals, copper and silver, as these have higher

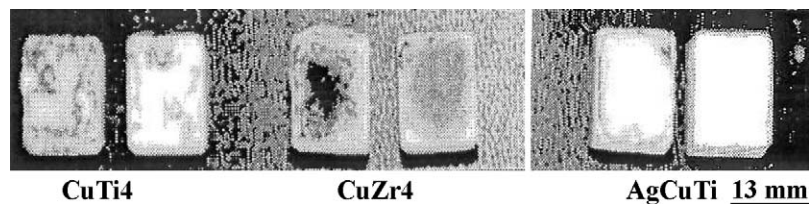


Fig. 3. Photographs showing fracture surfaces using different active filler alloys.

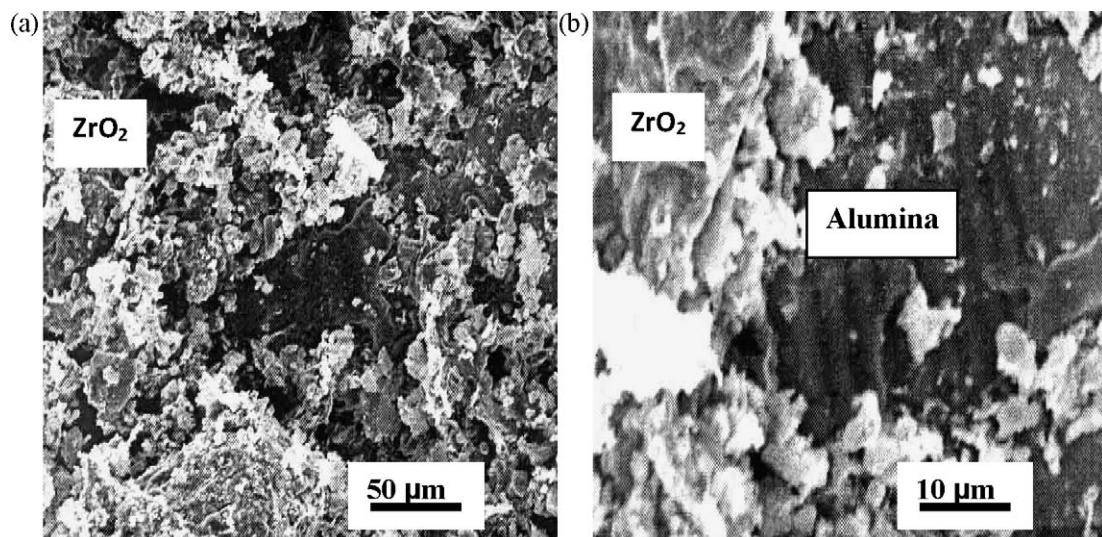


Fig. 4. SEM images showing the ZrO_2 phase at the alumina substrate: (a) low magnification and (b) higher magnification.

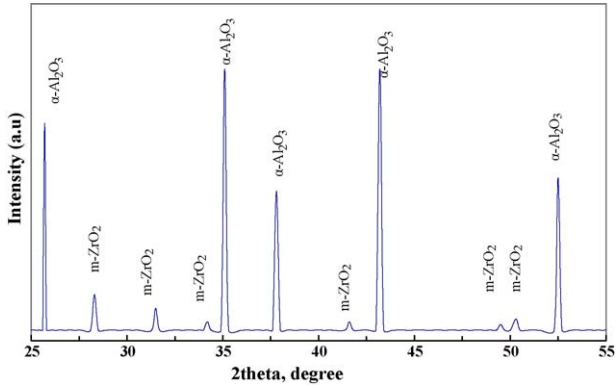


Fig. 5. XRD pattern from the fracture surface of alumina to alumina joint using Cu–4 wt% Zr filler alloy.

vapour pressures at a given temperature than the reactive elements Ti and Zr. The effective brazing processing parameters were found to be a fast heating rate of 40 °C/min and the shortest soaking time investigated of 10 min.

Poor quality alumina to alumina joints were obtained when using active brazes with Zr as an active element. Indeed some of these single-butt joints were so weak that they separated during cutting to prepare transverse sections. Fig. 3 illustrates the differences in the fracture surfaces, obtained from single-butt shear tests or from separated samples from the cutting of transverse sections, for a selection of brazes. The most noticeable feature is the large dark area in the fracture surfaces of the Zr containing brazes; this feature is shown in more detail in Fig. 4. XRD analysis

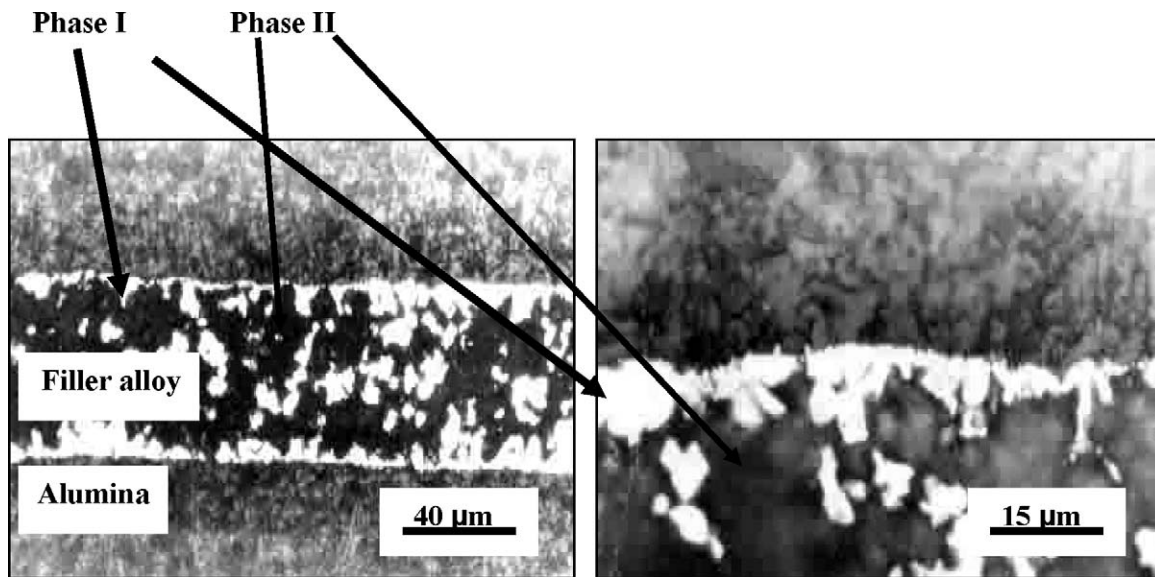


Fig. 6. SEM images of the alumina to alumina bonded with the active braze Cu–4 wt% Ti alloy showing three phases; light colored phase (phase I) is a reaction phase, the dark colored phase (phase II) is the metallic matrix and the grey colored phase is the alumina substrate: (a) low magnification and (b) higher magnification.

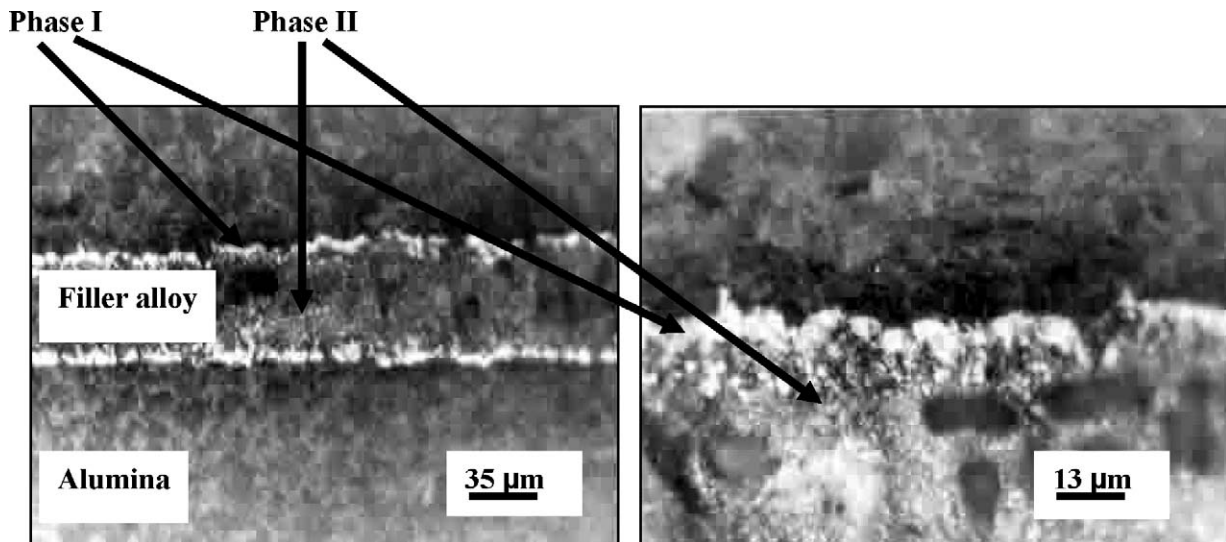


Fig. 7. SEM images of the alumina to alumina bonded with the active braze Ag–Cu–Ti eutectic alloy showing a three phases; light colored phase (phase I) is a reaction phase and the dark colored phase (phase II) is the metallic matrix and grey colored phase is the alumina substrate (a) low magnification and (b) higher magnification.

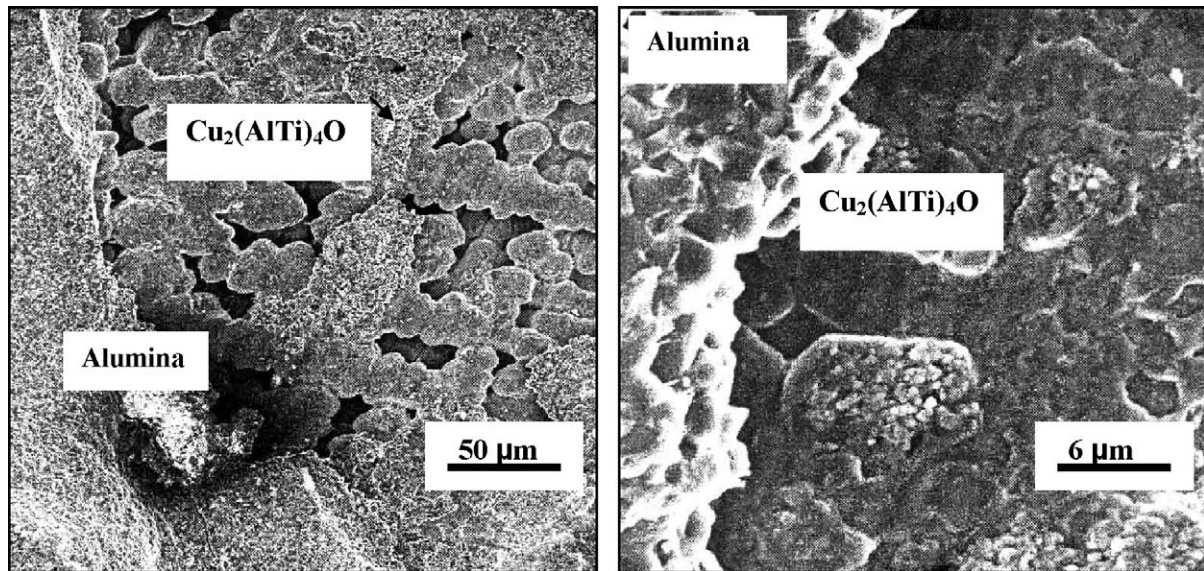


Fig. 8. SEM images of an alumina to alumina joint bonded with the active braze Cu–4 wt% Ti filler alloy. The fracture surface shows the dendritic $\text{Cu}_2(\text{AlTi})_4\text{O}$ phase.

of the fracture surfaces of the alumina to alumina joints, which had been actively brazed using CuZr alloys, identified the dark phase as ZrO_2 (Fig. 5).

There is also some evidence in the photographs of Fig. 3 that the CuTi4 and AgCuTi brazes are two-phase. This was confirmed by SEM images taken on transverse sections (Figs. 6 and 7). High magnification SEM images of the upper surface reveals the interaction between the alumina and brazed alloy containing Ti, resulting in dendritic solidification of a second phase at the brazed/alumina interface (Fig. 8). In the case of the binary braze the second phase was present at the braze–substrate interface and throughout the brazed. This second phase was shown by XRD to be $\text{Cu}_2(\text{AlTi})_4\text{O}$ (Fig. 9). There was less second phase present in the ternary eutectic brazed and it was mainly concentrated at the braze–substrate interface which, as a consequence, was not planar. This second phase was the same as found in the CuTi braze, namely $\text{Cu}_2(\text{AlTi})_4\text{O}$. It appears that this amount of reaction product with dendritic morphology and absence of porosity has a beneficial effect on bonding; XRD analysis of

the fracture surface of the ternary alloy showed the phase present to be alumina without any second phase (Fig. 10) due to the fracture path being in the substrate bulk (Fig. 11). Unlike when using CuZr fillers, the braze–substrate interface in the joints made with Ti-containing alloys showed good bonding with negligible porosity, which indicated good wetting.

3.2. Mechanical performance

The DBS tests for the joint using active brazes from the CuZr system produced a fracture that was either mode I (tensile opening) or mixed modes I and II, the latter being in-plane shear type of fracture opening. Typical fractured specimens are shown in Fig. 11, fracture having taken place through the ZrO_2 layer at the braze–substrate interface. The ZrO_2 would probably have been microcracked and the interface subjected to a residual stress due to the tetragonal to monoclinic shear transformation of ZrO_2 on cooling from the brazing temperature. Schematic diagrams of the fracture, together with the

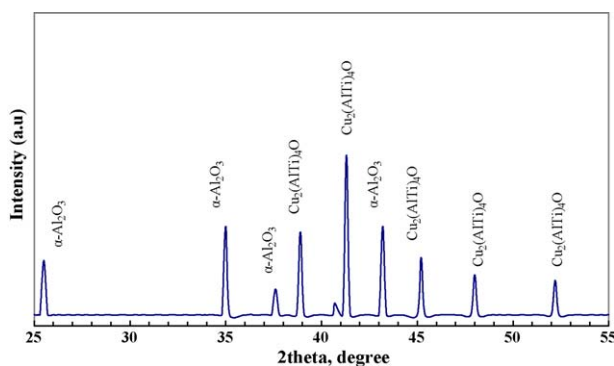


Fig. 9. XRD pattern from the fracture surface of an alumina to alumina joint using Cu–4 wt% Ti filler alloy.

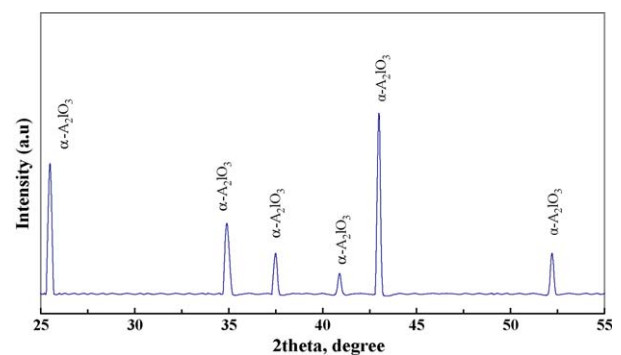


Fig. 10. XRD pattern from fracture surface of alumina to alumina joint using AgCuAl filler alloy.

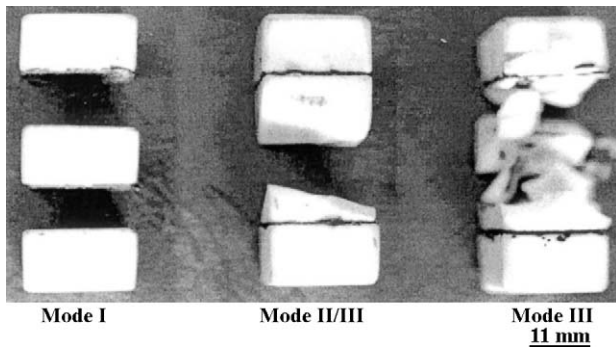


Fig. 11. Photograph illustrating the different failure modes of the actively brazed alumina to alumina using the three filler alloys.

load–crosshead displacement curve, are given in Fig. 12. The shear strength, as calculated from the maximum load divided by the total area of the braze–substrate interfaces, is very low at about 0.2–0.4 MPa (Table 2). A consequence of this low shear strength is that there was no tendency for the crack to deviate into the substrate.

For CuTi paste filler alloys, the fracture began in the plane of loading at the ceramic–filler interface then deviated into the alumina (the central alumina substrate) as show in Figs. 11 and 13. It appears that the crack initially opened in mode II in the braze then changed to mode III in the substrate (Fig. 13). The proposed schematic representation of the complete cycle of mixed modes fracture for CuTi brazing filler alloys is shown in Fig. 14.

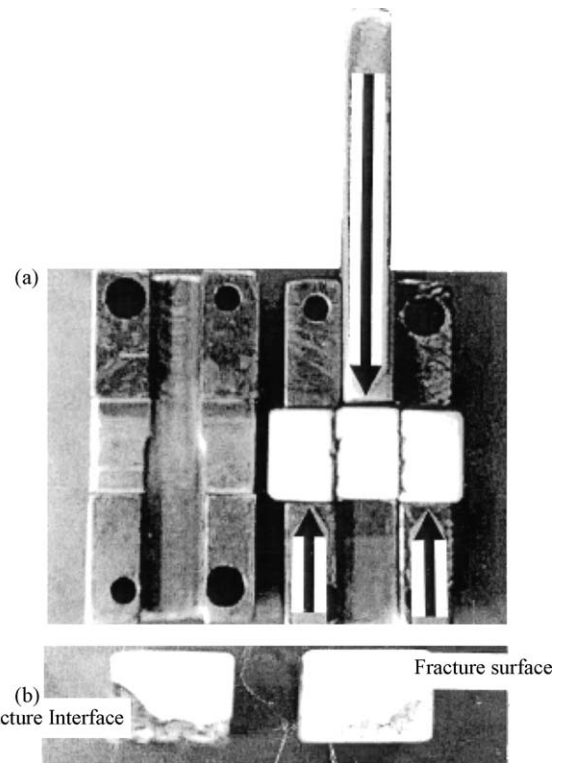


Fig. 13. Failure mode of the CuTi joints (a) showing crack initiation and open in mode II and (b) the fractures surface in mode III.

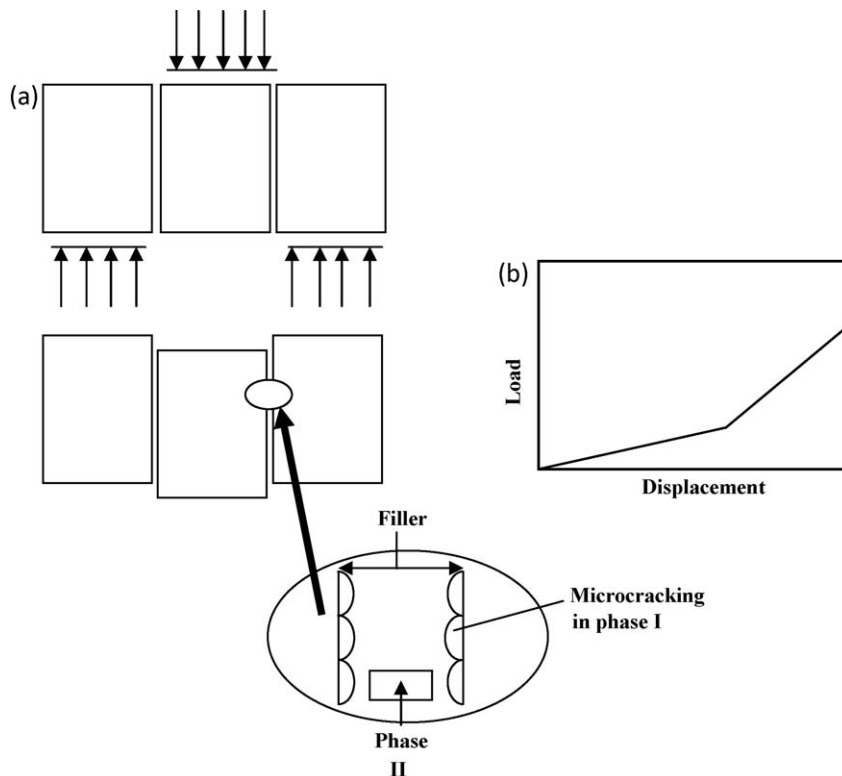


Fig. 12. Failure of the Cu–4 wt% Zr joint (a) schematic diagram of failure mode I/II and (b) load–displacement curve to failure in this mode (note the two regions of the curve).

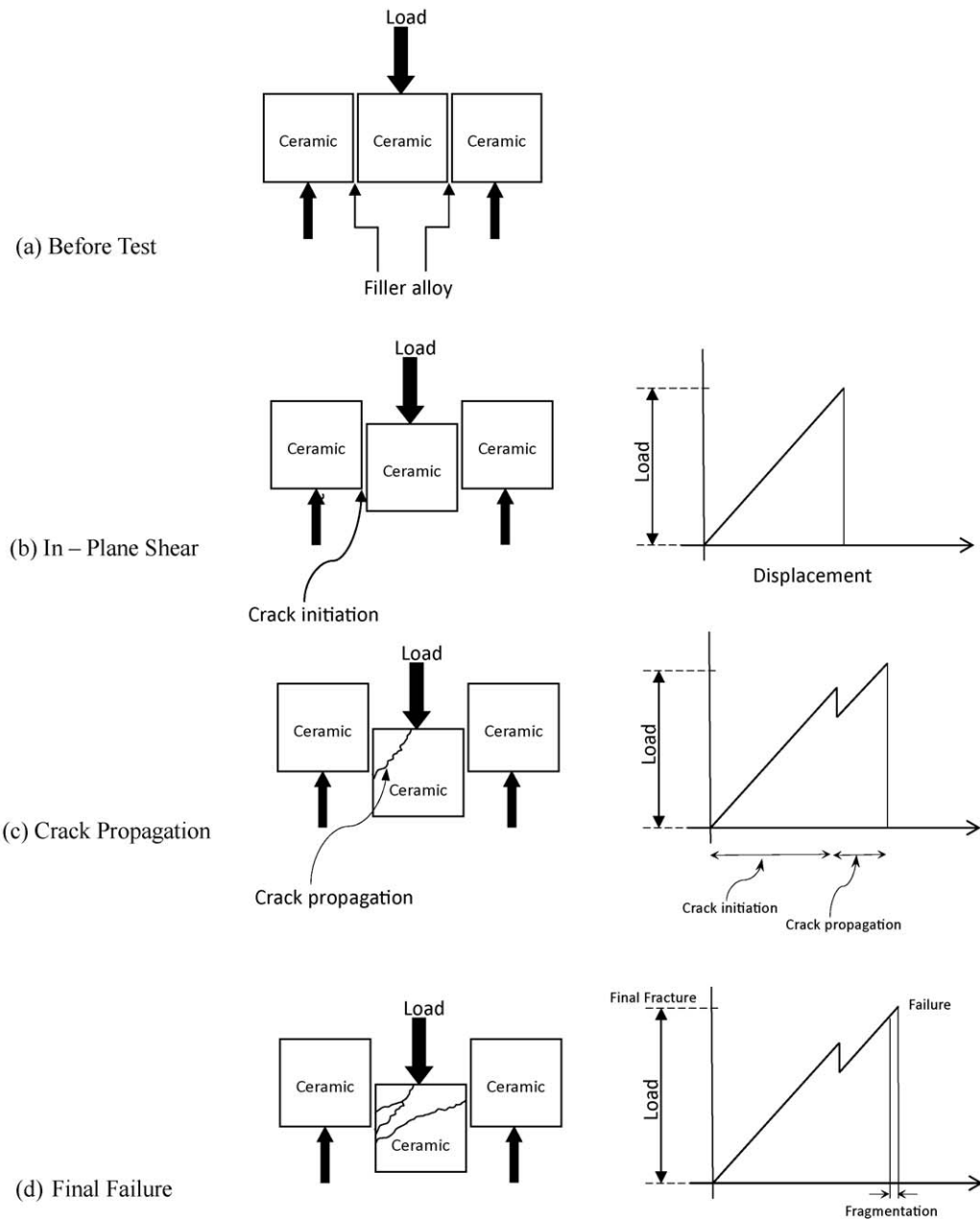


Fig. 14. Schematic representation of complete cycles of mixed modes fracture for CuTi brazing filler alloys.

Table 2
Shear strength, obtained by double braze shear test, for different paste filler alloys.

Paste filler alloy	Shear strength (MPa)	Standard deviation (MPa)
CuZr2	0.4	0.1
CuZr4	0.3	0.1
CuZr6	0.2	0.1
CuZr8	0.2	0.1
CuTi2	15	3
CuTi4	22	7
CuTi6	21	3
CuTi8	19	4
CuTi10	24	7
AgCuTi	42	3

Some typical load–displacement curves are presented in Fig. 15. In some cases the load–displacement curve exhibited a drop in load at the point the crack propagated from the brazed into the substrate; it is interesting to note that this behaviour took place in lower Ti content alloys and at varying force levels. No clear explanation was found but it may be related to details of the location, morphology and volume fraction of the second phase. The shear strength of these brazes, as calculated from the maximum load, are in the range 15–24 MPa (Table 2). Thus, the shear strength is nearly two orders of magnitude greater than that of the joint made with the CuZr filler, hence the propensity for the crack to move into the substrate. It is concluded that the presence of a small amount of $\text{Cu}_2(\text{AlTi})_4\text{O}$ at the braze–substrate interface is beneficial in contrast to the detrimental

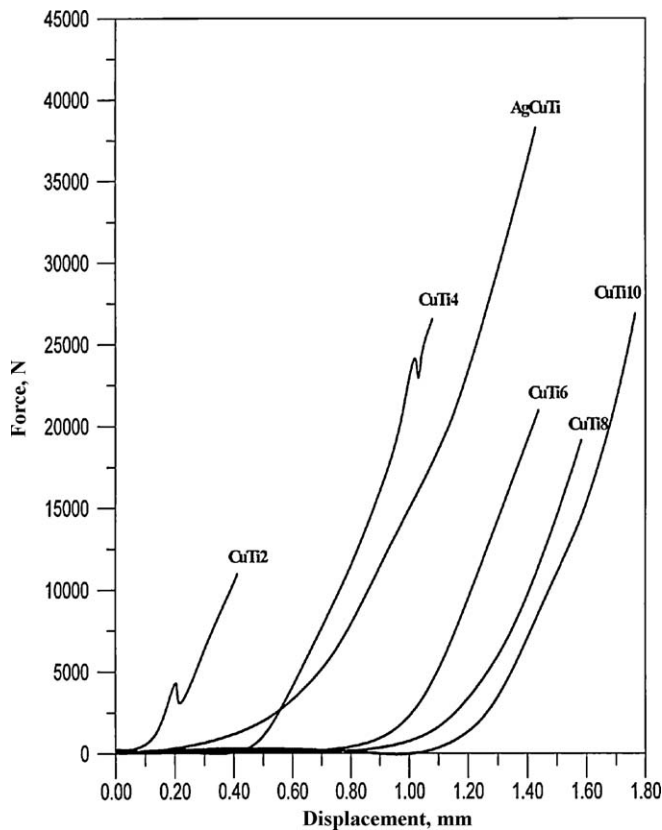


Fig. 15. Load-displacement curves for double bonding test of alumina to alumina using different Cu-Ti actively brazed alloys and AgCuTi eutectic alloy.

effect of a large amount of ZrO_2 in the CuZr brazed joints. However, taking into account the standard deviations for the strength values, it is concluded that there is no clear trend between strength and Ti content over the compositional range investigated of 2–10 wt%.

The DBS tests for the joints produced with the ternary eutectic filler metal demonstrated that the failure was predominately in the central alumina substrate with the cracks aligned primarily with the compression axis (Fig. 11). The shear strength of the interface must be at least 42 ± 3 MPa (Table 2), hence it follows that the small amount of $Cu_2(AlTi)_4O$ at the interface and the concomitant non-planar interface enhance interfacial shear strength. This value is by far the greatest shear strength recorded for the three active brazes employed in this study and that achieved with a SnAgTi(Ce, Ga) [23] but less than that previously reported for Ag57Cu38Ti5 brazing of alumina [25] (Table 3). The high shear strength of the braze accounts for the failure taking place within the substrate. The failure of the alumina substrate appears to be similar to the well-documented “axial splitting” (mode III) prominent in compression testing of unconfined ceramics, as exemplified by the failure of specimens of grout (cement and sand) discussed in the literature [46]. The fracture involves a sequence of events leading from the activation of existing flaws to the growth of cracks, their coalescence to form columns of material, the spallation of fragments and the collapse of columns is shown in this figure.

Table 3

Fracture strength obtained by different test methods.

Filler alloy	Strength (MPa)	Bulk surface	Reference
CuZr	0.4	Alumina/alumina	Present study
CuTi	24	Alumina/alumina	Present study
AgCuTi	42	Alumina/alumina	Present study
AgCuZr	132	Alumina/Ni–Cr–steel	[10]
AgCuZrSn	141	Alumina/Ni–Cr–steel	[10]
AgCuZrAl	112	Alumina/NiCr–steel	[10]
Amorphous CuTi	140	Al_2O_3/Cu	[15]
Amorphous CuTi	185	Si_3N_4/Si_3N_4	[15]
Amorphous CuTi	165	$Al_2O_3/Kovar$	[15]
Amorphous CuTi	115	Alumina/alumina	[15]
AgCu	55	AlN/Cu	[16]
AgCu	37	Al_2O_3/Cu	[16]
Cu	100	$Al_2O_3/steel$	[17]
AgCuTi	25	C/C	[19]
AgCuTi	180	PSZ/PSZ	[22]
SnAgTi(Ce, Ga)	13.5	Alumina/alumina	[23]
SnAgTi(Ce, Ga)	14.3	Cu/Cu	[23]
SnAgTi(Ce, Ga)	10.2	Alumina/Cu	[23]
CuPdTi	156	Si_3N_4/Si_3N_4	[24]
AgCuTi	180	Alumina/alumina	[25]
AgCuZn	100	TiC/steel	[26]
AgCuTi	325	TiAl/steel	[27]
$Y_2O_3Al_2O_3SiO_2Si_3N_4$	550	Si_3N_4/Si_3N_4	[31]
CuNiTiB	402	Si_3N_4/Si_3N_4	[32]
Al_2O_3/Y_2O_3	230	Si_3N_4/Si_3N_4	[33]
CuPdTi	172	Si_3N_4/Si_3N_4	[34]
Ti foils	142	Si_3N_4/Si_3N_4	[35]
CuNiTiB	402	Si_3N_4/Si_3N_4	[36]
AgCu and TiH_2	75	PSZ/stainless steel	[39]
$\alpha-Ti/Ti_2Ni$	68	Alumina/kovar	[40]
Al	60	AlN/steel	[41]
Cu	33	AlN/AlN	[41]
Al	38	AlN/AlN	[41]
BaAl borosilicate glass	30	C/C	[42]
Silicone resin	30	SiC/SiC	[43]
$Ti_2Ni + TiCu + TiNi_2Cu$	250	Ti3Al/TiZrNiCu	[44]
Foam 316 stainless steel	33	AlN/inconel 600	[45]

4. Conclusions

Selection of the correct heating cycle for joining alumina to alumina by a brazing process is critical and a short holding time of 10 min at the brazing temperature was found to be appropriate for the alloys studied in this investigation. The interface in actively brazed alumina to alumina joints using CuZr paste filler alloys consisted primarily of monoclinic ZrO_2 , which resulted in failure at the interface and low shear strength (0.2–0.4 MPa).

The formation of $Cu_2(AlTi)_4O$ at the interface of joints developed during actively brazing using both CuTi and AgCuTi filler alloys was beneficial for mechanical performance.

The intermediate shear strengths (15–24 MPa) exhibited by CuTi brazes led to the crack deviating into the substrate in the later stages of the test. A consequence of the higher shear strength of at least 42 MPa of the eutectic AgCuTi joint was that failure was largely confined to the central alumina substrate. The binary and ternary Ti-containing systems show promise for producing alumina to alumina joints by active brazing.

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