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Abstract

This work has focused on *surface engineering* coupled with brazing to join SiC_f/SiC composites and to improve the joint strength. The surface engineering was carried out through the Selective Thermal Removal (STR) of SiC fibres from a SiC_f/SiC composite to obtain "brush-like" surfaces; the modified composites were then joined by means of an AgCuTi braze alloy. In order to investigate whether a "brush-like" interface could

enhance the mechanical strength of the joint by increasing mechanical interlocking with the brazing alloy at the micron scale, the joints were tested with and without surface engineering by means of single lap offset under compression.

The treatment to form a roughened surface suitable for mechanical keying of the braze metal led to an improved ductility of the joint; the fracture surfaces demonstrated that the proposed method is promising, even though the treatment damages locally the composite.

Keywords: ceramic matrix composites; joining; surface engineering; brazing.

1. Introduction

Ceramic Matrix Composites (CMCs) are generally composed of straight or woven ceramic fibres embedded in a ceramic matrix with a weak bond between them, a process that leads to an improved fracture toughness of the composites compared to the matrix or to fibre materials. Because of their superior thermomechanical properties, CMCs based on a SiC matrix reinforced with silicon carbide fibres (SiC_f/SiC) are materials of great interest for many applications, ranging from the aerospace [1,2] to the nuclear field [3-5], which require extremely high temperature stability and damage tolerance. SiC_f/SiC are currently being used as thermo-structural materials in different components, such as aircraft brakes, body flaps, leading edges, heat exchanger components, gas turbines for power plants, thermal protection systems for space vehicles and the inner walls of plasma chambers of nuclear fusion reactors [6]. However, in many cases, their use depends to a great extent on their ability to be joined and integrated, since the manufacturing of these materials as large components or complex geometries is difficult and expensive. The study and development of reliable

joining methods to assemble CMC as large components in complex structures is a critical issue for a wider use of these materials.

The surface engineering of CMC has attracted a great deal of attention from the scientific community as it represents a smart and still unexplored technology that can be used to improve the mechanical performances of CMC joints. The design of interfaces coupled with suitable joining materials and joining technologies are key parameters in manufacturing, especially for ceramic matrix composite-based components. In a previous work [7], we proposed a technique based on the Selective Thermal Removal (STR) of SiC fibres from SiC_f/SiC composites. This process leads to a micro-sized "brush-like" structure that is able to enhance the specific surface of SiC_f/SiC. It was demonstrated that the specific surface of SiC_f/SiC significantly increased (~ 90%) when fibres were removed parallel and perpendicular to the composite surface.

In [7], the effect of the STR process on the composite surface was discussed from the morphological point of view and wetting tests, using a AgCuTi brazing alloy, on as-received and modified SiC_f/SiC were performed. In the present work, starting from those promising results, experimental parameters have been transferred from wetting test to joining process and adhesion issues are presented. Surface modified SiC_f/SiC joined samples have been manufactured and their microstructure studied. In order to assess the effectiveness of the STR process, mechanical tests (single-lap offset under compression) have been conducted on STR modified SiC_f/SiC joined by brazing and the results have been compared with those of reference SiC_f/SiC joints (obtained with as-received SiC_f/SiC). Furthermore, the mechanical strength of SiC_f/SiC after STR has been measured to verify the detrimental effects of the surface engineering process on the SiC-based composite itself.

2. Materials and methods

Keraman® SiC_f/SiC samples supplied by MT Aerospace (Germany) were used for the experimental work. The composites were manufactured at MT Aerospace, Germany, using the standard gradient Chemical Vapour Infiltration (CVI) process and supplied as rectangular shaped samples; they contained Tyranno ® S grade 1.6 K (Ube Industries, Tokyo, Japan) fibres coated with a thin C layer. SiC_f/SiC has a thermal expansion coefficient of 4×10^{-6} K^{-1}.

All the SiC_f/SiC showed pores between the fabric layers and some cracks on the surface.

Before brazing, the SiC_f/SiC were cut into 5 mm× 5 mm× 2 mm slabs and were then processed at 1450°C for 2 hours under flowing Ar in a tubular oven (according to the STR process described in [7]). The brazing alloy used to join the CMC was a commercial AgCuTi alloy (CB4, Degussa, Germany) in the form of 100 µm thick foils. This alloy consists of eutectic Ag-Cu activated with 3 wt. % of Ti (nominal composition: Ag: 57.7, Cu: 36.8, Ti: 5.5 at.%) and belongs to a family of braze media that are commonly used for mid-temperature joining processes; its melting range is 780-805 °C and it has an optimal brazing temperature in the 850-950 °C range. The braze foil was sandwiched between composite blocks and this assembly was introduced into an alumina tubular furnace set at 850 °C, held at this temperature for 30 min and then cooled down at a rate of 5 °C·min^{-1}. During the process, a vacuum lower than $5\cdot10^{-4}$ Pa was maintained by means of a turbomolecular pump; no external load was necessary as adhesion was assured by the capillary forces of the liquid which pulled the surfaces together.

The SiC_f/SiC and joined samples were characterised before and after the STR process using Field Emission Scanning Electron Microscopy (FESEM- ZEISS Supra 40) with

an Energy Dispersive Spectroscopy (EDS- SW9100 EDAX) detector and scanning electron microscopy (SEM, model: LEO 1450 VP) with an electron microprobe (EDS) (Oxford Instruments, 7353 model with Oxford-INCA software v. 4.07, type of detector: Si(Li)).

Investigation and structural analysis of the interfacial phases were carried out using Transmission Electron Microscope (TEM) JEOL 2100F UHR operated at 200kV. Image characterization was done in scanning/transmission mode employing bright field detector, in order to utilize mass-thickness contrast for enhancing the distinction of areas with different atomic number and to avoid undesirable effects of dynamical diffraction. In this mode, EDS analyses were performed by means of detector with crystal size 80mm^2 (Oxford Instruments, Xmax80, INCA software). Phase identification was confirmed by Selected Area Electron Diffraction (SAED) technique. For processing of obtained diffraction patterns licensed sofwares were used (Gatan Digital Micrograph and CMPR database). Calibration of digital camera in reciprocal space was set on MoO3 monocrystal standard.

Thin foils for purposes of TEM observations were prepared using standard preparation methods including cutting, grinding, polishing, dimpling and final step of the thinning procedure was done by ion milling (PIPS Model 691 Gatan) operated at 4.5 kV with ion-beam angles of 4° and 3°.

Complementary phases analysis was carried out by X-ray diffraction (XRD, X'Pert Pro MRD,Panalytical, Cu K_α radiation, with the aid of the X-Pert High Score software) and identified with JCPDS files.

The shear strength of the joined samples (at least 4 as-received SiC_f/SiC joints and STR SiC_f/SiC joints) was evaluated using the single lap offset (SLO) test under compression at room temperature, according to a method adapted from ASTM D905-08 (universal

testing machine SINTEC D/10) [8]; the crosshead speed was 0.5 mm/min. The maximum force was recorded and the shear strength was calculated by dividing the maximum force by the joining area.

The size of the single lap off-set shear tests for brazed samples was 10 mm × 3 mm × 4 mm with a joined area of 30 mm^2.

The SiC_f/SiC samples were subjected to 3-point bending tests (v= 0.5 mm/min; span= 40 mm), according to ASTM standard C1341-13 [9], at room temperature before and after the STR treatment. The composite size was 3 mm × 4 mm × 45 mm. The fracture surfaces of the specimens were investigated after mechanical characterization by means of SEM/FESEM.

3. Results and discussion

3.1 Surface modification and joining

The STR process was carried out on the SiC_f/SiC samples at 1450°C for 2 hours to obtain "brush-like" surfaces [7]. The appearance of the modified surface is shown in Figure 1; the SiC fibres were partially and homogeneously removed from the composite surface, and pores and detached areas formed at the fibre/matrix interface. The presence of silica microwires is evident on the fibre surface (Figure 1 d) and inset e)), both on the fibre cross-sectional area (as shown in the micrographs) and on the external part of the fibres parallel to the treated surface.

When exposed to temperatures above 1000°C, the fibres used as reinforcement in the SiCf/SiC are subjected to a thermal degradation, as discussed in [7]. The thermal degradation occurs both at the end of the fibres and on the body of the fibres, at least on

the part of the body exposed to STR atmosphere. In that sense, STR also acts on fibres parallel to the joining surface and on the overall mechanical properties of the composite. The modified SiC_f/SiC surfaces have been joined using a AgCuTi brazing alloy; the joining process ,carried out in the present work, is based on the results of the wetting tests [7]. The proof of concept of the STR modification surface technique to implement a joining process has been carried out through the morphological investigation of the cross-section of the joints; Figures 2 a and b show an example of an SiC_f/SiC - AgCuTi - SiC_f/SiC joint.

Both types of composites (as-received (Figure 3) and STR treated (Figure 4) SiC_f/SiC) showed the presence of sound joints with the brazing alloy, which was well distributed along the whole joint and had infiltrated into the pores and cracks, as can clearly be observed in the microstructures in Figures 3 and 4. The microstructures of the joint in Figure 3 a-c show that a sub-micrometric Ti-rich layer has formed at the interface and it is visible in the higher magnification figure (inset c). The wetting tests performed by the authors [7] showed that the braze perfectly spread on the SiC_f/SiC surfaces, filling all the cavities and gaps introduced by STR. The present paper reports on the experimental activity carried out to verify the beneficial effect of STR on joints manufacturing. The thermal treatment etched the fibers not only at their top but also on their side, which in turn leads to a micro-sized "brush-like" structure on both the facing surfaces of the joints.

An SEM cross section of an STR SiC_f/SiC joint is shown in Figure 4 for comparison purposes: the brazing alloy has infiltrated the composite surface along the macro-porosities, in the same way as for the as-received SiC_f/SiC, but in this case the brazing alloy has penetrated for ~ 10 μm and infiltrated the gaps obtained by the partial removal of fibres, thus a "brush-like" joint has formed with interlocked joining materials (Figure

4 b, c). ~~The interfacial composition (see Table 1) was similar for the two types of joints.~~

The interfacial composition (see Table 1) was similar for the two types of joints.

In order to improve the reliability of data from SEM-EDS, the thin Ti-rich layer was analysed by TEM to further identify the interfacial products. Figure 5 shows high-magnification images of the interface and SAED patterns identification of the resulting phases: going from SiC_f/SiC towards the brazing alloy, a ~400nm thick layer formed mostly of TiC nanograins, and a 250 nm thick Ti_5Si_3 layer were detected.

In the case of an untreated SiC_f/SiC surface (Figure 3), the Ti-rich layer is evident along the whole joint interface and it follows the surface profile (see Figure 3 c). In the case of an STR SiC_f/SiC joint (Figure 4), the Ti-rich layer follows the profile of the fibres that had partially been removed by means of STR (Figure 4 b,c) and the gap between the fibre and the matrix formed by the thermal selective process. The reaction formed Ti rich layer was continuous and homogenous, which led to braze wettability on the surface. This layer followed the profile of the selective etched fibres without obstructing the cavities, but allowed the molten braze to penetrate. In this way, continuous infiltration coupled with good wettability were achieved. The formation of this transition layer at the interface is considered a key factor for ceramic/braze joint strength. In the case of the SiC_f/SiC samples, this transition layer was developed on a larger surface, thus a larger contact area was made available for the adhesion between the brazing alloy and the SiC_f/SiC and a gradual CTE (coefficient of thermal expansion) change took place from the brazing alloy to the composite along the CMC porosities. The CTEs of the interlayer phases were 18.5 and 8.82 x 10^{-6} /°C for the AgCuTi and Ti_3SiC_2, respectively, while the CTE of ~~$Ti_5Si_3C_x$ was assumed equal to that of Ti_5Si_3, as the $Ti_5Si_3C_x$ phase is a solid solution that contains only a small percentage of C.~~

Ti_5Si_3 exhibits large CTE anisotropy: 6.11 (basal plane) or 16.62 (c-plane) [10]. In addition, thermal mismatch may be accommodated for by the relatively good plasticity of the Ag-Cu braze material [11]. This morphology of the interface in the STR samples promotes bonding, provided the interfacial roughness of the treated surface does not hamper the spread of brazing and physical contact.

3.2 Mechanical characterization

The SiC_f/SiC samples were subjected to 3-point bending tests before and after the STR treatment in order to assess the possible detrimental effect of the process (1450°C, 2 hours, Ar atmosphere) on the mechanical properties of the composite. The as-received SiC_f/SiC average flexural strength was 576±32 MPa, while the SiC_f/SiC after STR showed an average flexural strength of 387±67 MPa.

Figure 6 shows the representative flexural strength load –displacement curves of the SiC_f/SiC at room temperature, before and after the STR treatment. The STR caused a decrease in the composites flexural strength of about 30%, due to the fibre and fibre/matrix interface degradation [7]. The load-displacement curves exhibit the typical "plastic-like" behaviour that occurs in CMCs because of the toughening mechanisms of crack bridging, deflection and slippage at the interface between the fibres and matrix; this trend was observed in both of the tested SiC_f/SiC types, that is, as-received and STR treated. The lower mechanical strength of the SiC_f/SiC after STR may be ascribed to the partial degradation of the thin carbon layer at the fibre/matrix interface and of the SiC fibres; it has been reported that the residual tensile strength of Tyranno S fibres, after a heat treatment at 1450°C for 1 hour in Ar, is only 20% of the original value [12]. Despite this significant decrease in mechanical strength of the fibres [13, 14], the composite retained about 70% of its original strength after STR. The present

experimental activity is still underway to confine the STR process to the composite surface, in order to avoid or significantly reduce the decrease in mechanical strength of the whole composite.

The single lap offset (SLO) test under compression was chosen as the mechanical test for joined SiC_f/SiC in order to understand whether the STR process was useful to SiC_f/SiC to obtain statistically relevant results, and it can be useful to compare different joining materials and joining processes [15]. The measured shear strength of the joints produced with the as-received SiC_f/SiC showed a value of 21.3 ± 6.5 MPa, while the STR treated and then joined SiC_f/SiC resulted in a value of 16.5± 4.5 MPa, which roughly corresponds to a 30% decrease, as already observed for the SiC_f/SiC flexural strength before and after the same heat treatment used for the STR process.

The decrease in mechanical strength of the whole SiC_f/SiC after STR does not allow any improvement, if any, to be measured in the lap-shear strength of these joints.

XRD analysis (Figure7) was carried out on the joint fracture surfaces to detect the interfacial phases and to confirm the EDS and TEM analysis. The pattern shows several phases in good agreement with data obtained by TEM analysis: it put in evidence the formation of Ti_5Si_3 and TiC (presumably at the interface between the brazing alloy and the composite surface) and Ag and Cu mostly due the brazing alloy infiltration. The SiC/SiC composite substrate is detectable by SiC peaks.

There is no difference in detected phases on the fracture surface after the mechanical tests on the SiC_f/SiC joined samples without STR (Figure 7) and with STR.

~~However, the~~ The fracture surface analysis (Figure 8) revealed a promising morphology; as can be observed in Figures 8 a) and b), the as-received SiC_f/SiC joint underwent delamination of the composite close to the joined region, with a few spots showing macro-infiltration of the brazing alloy (already shown in the cross-section in

Figure 3 a) and a flat fracture surface. On the other hand, the infiltration of the brazing alloy all around the fibres in the STR joined SiC_f/SiC (Figure 8 d) caused a non-flat fracture surface and extensive pull-out of the fibres (Figure 8 c), in agreement with the cross section shown in Figures 4b and c. All this seems to indicate an effective role of the STR in obtaining a brush-like structure, even though this has not yet been supported by a measured increase in the mechanical strength of the joint. A schematic of the fracture path in SiC_f/SiC joints is shown in Figure 9.

The different failure mechanisms in the SiC_f/SiC joints, with and without the STR treatment, is also evident in Figure 10. The typical load/displacement curves of the SiC_f/SiC joints without STR, as tested by means of SLO, show brittle behaviour (Figure 10, full line). On the other hand, the SiC_f/SiC processed with STR and then joined exhibit a load/displacement curve (Figure 10, dotted line) with non-brittle, segmented behaviour that is typical of a composite material; this can be explained by considering the increased toughness of the joint, due to the formation of a composite joining material at the interface between the infiltrated brazing alloys and the STR SiC_f/SiC.

Other activities are currently underway in order to modify the STR process so as to avoid the degradation of all the SiC_f/SiC mechanical properties, that is, to confine the thermal removal process at the composite surface.

Moreover, the stress condition in the braze as in the ceramic composite is clearly dominated by the residual stresses. Stress level in the braze can be mitigated by plastic relaxation strain. Contraction of the brazing alloy layer is hindered by the metal cramped with the ceramic in the structured surface layer.

The ceramic opposes the reduction of the distance of the filled holes, thus leading to compression stresses in the composites. On the other hand, the thermal shrinkage of the solder in the holes leads to high tension stresses. As a consequence, the brush-like

surface can increase the residual stresses at the interface between the metal and the composite.

The tuning of the structuring depth is a key factor in order to have a balance between an improved mechanical interlocking and a reduction of detrimental stress peaks at the interface.

4. Conclusions

The proposed surface engineering treatment (Selective Thermal Removal, STR) has been successfully used to obtain "brush-like" SiC_f/SiC surfaces.

The main advantages of this process are (i) modification of the surface, due to a one-step pre-treatment, which increases the surface area and allows the joining material to be mechanically anchored, and (ii) a completely dry and environmental friendly process, in which no hazardous chemicals are used and which, coupled with a high reproducibility of the process, makes this process attractive for implementation in automatic and production machinery.

The brush-like SiC_f/SiC interface was well infiltrated by the Ag-based brazing alloy, and this led to a composite-like fracture surface and a "plastic-like" load/displacement curve after a single lap offset test under compression of the joints.

At this stage of the research, the STR process seems to be effective in increasing the joint toughness, but the treatment still needs to be modified in order to avoid degradation of the whole SiC_f/SiC. The future experimental activities will focus on a localised and less harsh thermal process, in order to find a good compromise between surface engineering and composite integrity.

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Type of SiC_f/SiC composite	Region	Elemental composition (at.%)					Possible phase
		C	Si	Ti	Cu	Ag	
As-received surface (Fig. 3)	A	-	-	0.9	11.3	87.8	Ag(Cu)
	B	-	-	2.8	92.1	5.1	Cu(Ag)
	C (7 kV)	31.5	26.5	35.0	2.7	4.3	$TiC_x + Ti_5Si_3$
STR surface (Fig. 4)	D	-	-	1.1	10.4	88.5	Ag(Cu)
	E	-	-	2.1	93.4	4.5	Cu(Ag)
	F (7 kV)	29.4	24.1	31.5	2.5	3.8	$TiC_x + Ti_5Si_3$

Table 1: EDS chemical analysis (at.%) of different regions identified in the cross-sectioned samples; at least 5 spots were analysed for each phase.

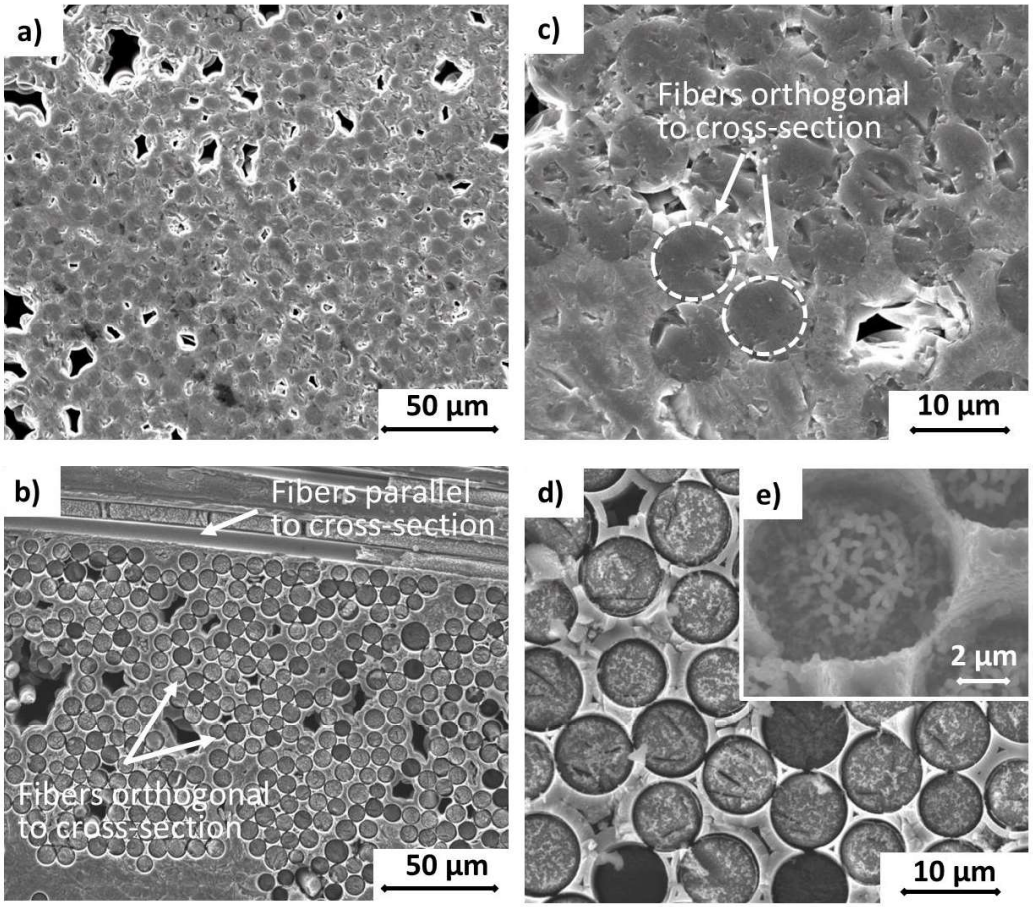

Fig. 1. SEM-SE images of the surface of the as-received (a,c) and STR treated (b,d) SiC_f/SiC; inset (e) shows the silica microwires formed on the SiC fibre surface.

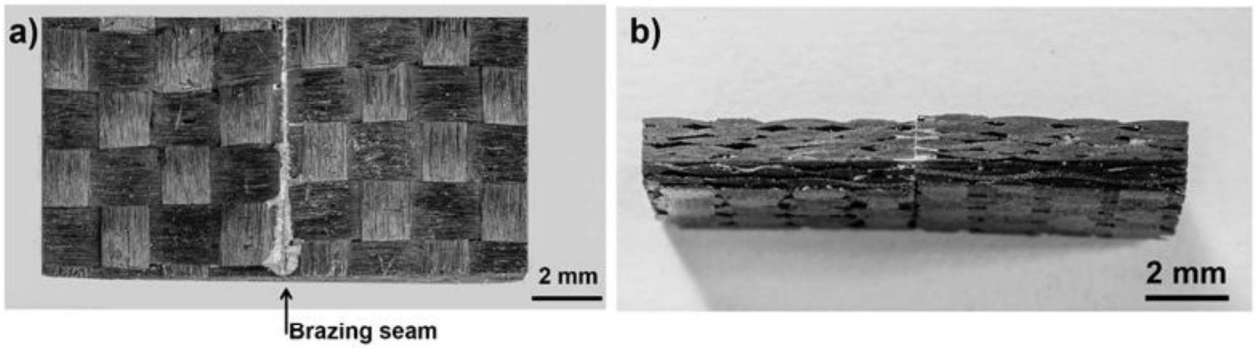

Fig. 2. Macrographs of an SiC_f/SiC to SiC_f/SiC joint obtained by means of AgCuTi : the brazing seam is visible (a), as is the infiltration of the braze into the facing composite blocks (b); good adhesion and wetting of the filler alloy along the interface with infiltration of the alloy into the holes and gaps of the composites are visible.

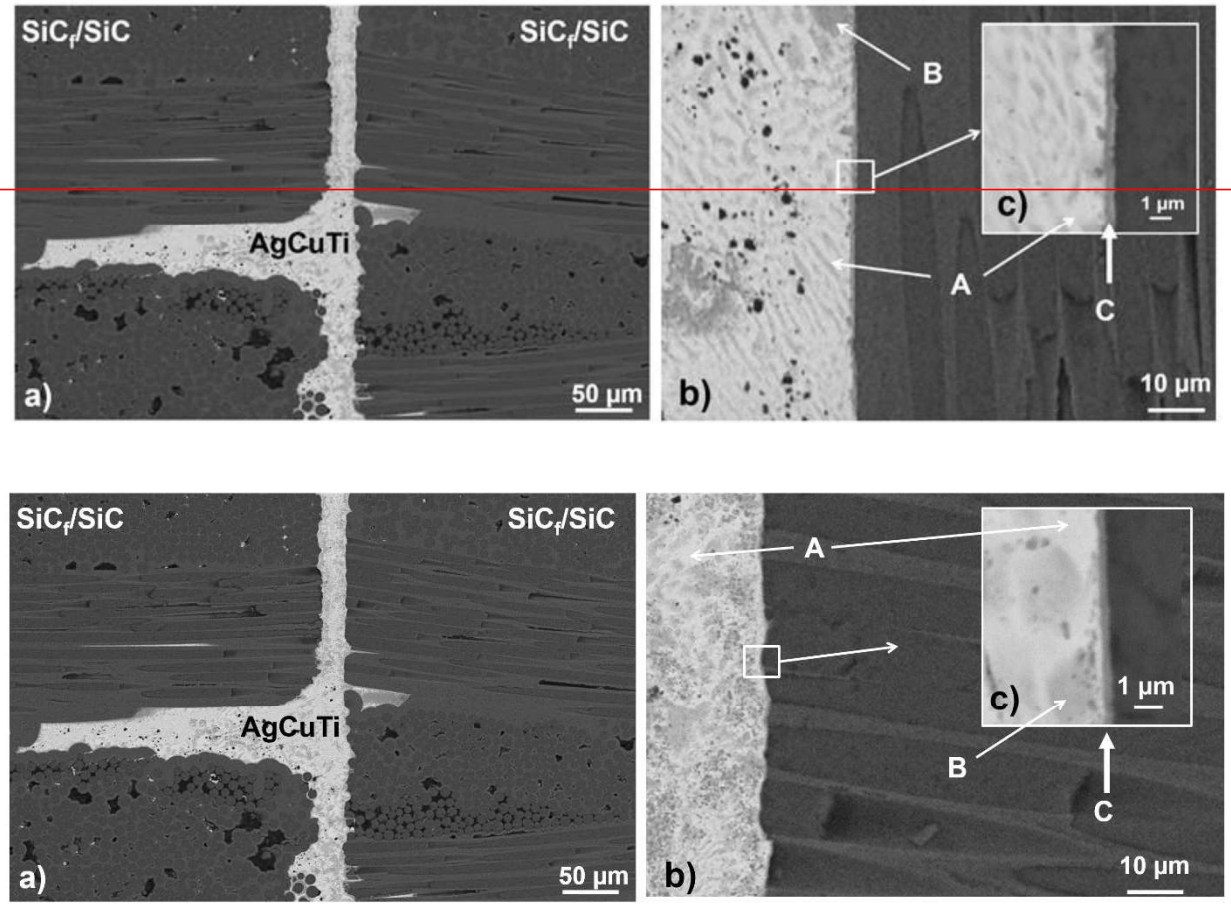

Fig. 3. SEM magnification of the cross section of an SiC_f/SiC joint (as-received composite) showing: a) a panoramic view of the brazing seam showing infiltration into the composite porosities; b) adhesion of the brazing alloy at a microscale and c) higher magnification of the interface.