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# Direct joining of CFC to copper

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## Abstract

The ITER divertor design foresees the joint between CFC (carbon fibre reinforced carbon composites) as armor material and a Cu alloy in the heat sink. The purpose of this work is to realize a new joining method between CFC and a pure copper interlayer, required to accommodate the thermal expansion mismatch between CFC and the copper alloy. The joining technique is based on the direct copper casting on CFC surface, which was previously modified by direct reaction with a transition metal of the VI B group. The formation of a carbide layer on CFC surface improves the wettability of molten copper on CFC. The process was also successfully applied to join silicon doped CFC to copper. The paper includes the results of the morphological analysis, the mechanical tests on the joined samples and preliminary thermal fatigue tests.

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## 1. Introduction

Because of their excellent properties, such as high thermal conductivity, high thermal shock and thermal fatigue resistance, the carbon fibre reinforced carbon matrix composites (CFCs) are considered as one of the armour materials for the ITER (International Thermonuclear Experimental Reactor) divertor [1]. The CFCs are located in the lower part of the divertor vertical target that has to remove the heat load coming from the plasma. The design of this part of the reactor needs an armour material, which directly interacts with plasma, and a heat sink, which transfers the heat loads from the armour to the water coolant.

One of the most critical step in the divertor manufacturing is the joint between the armour, made of CFC, and the heat sink, made of a copper alloy (CuCrZr), because of their large thermal expansion mismatch ($\alpha_{CFC} = 2.5 \times 10^{-6}\ K^{-1}$; $\alpha_{CuCrZr} = 16–17 \times 10^{-6}\ K^{-1}$) [2,3]. In order to mitigate the high thermal stress at the joint interface, a thin (1–2 mm) soft interlayer made of pure copper should be put between the CFC armour and the metallic heat sink. Unfortunately, the very high contact angle of molten copper on CFC composites ($\theta = 140°$) precludes the direct casting.

Up to now, two possible solutions are available in EU for CFC/pure Cu joints. The first one has been developed by Plansee AG and consists in the casting of pure copper on the CFC laser machined surface that is previously activated by titanium. The second method, developed by Ansaldo Ricerche, foresees the use of a brazing alloy with good wetting characteristics on CFC surface [4–7].

This paper describes a new technique to join CFC composites to pure copper developed at Politecnico di Torino; it includes two steps: the CFC surface modification by solid state reaction with a transition metal of VI B group and the direct casting of copper to CFC. [1]

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## 2. Experimental

The carbon fibre reinforced carbon matrix composites used in this work are high thermal conductivity CFC composites (Sepcarb® NB31 and NS31) specifically developed for fusion applications by Snecma Propulsion Solide. These composites have a complex 3D framework of ex-PAN and ex-pitch carbon fibres filled with a carbon matrix. The difference between NS31 and NB31 is that NS31 composites were additionally infiltrated by liquid silicon (Si: 8–10 at.%) that is finally present as pure silicon and silicon carbide [2].

The surface of the composite was modified by direct solid-state reaction at high temperature between a transition metal of VI B group and the CFC. Different metals (chromium and molybdenum) inside the VI B group were deposited on the CFC surface by a simple slurry technique (metal powder suspension in ethanol). The heat treatment performed at high temperature (>1000 °C) in a tubular oven under an argon flux or in a vacuum furnace, led to the formation of a thin carbide layer (5–10 μm) on the composite surface. The reaction products between the composites and the transition metal were detected by X-ray diffraction analysis (Philips PW1710).

The direct joining of copper to CFC was performed in a special graphite sample holder [8], where the modified CFC and the copper (in form of slurry or foil) were placed and heated at 1100 °C for 20 min.

In order to investigate the effect of the mechanical machining, the surface of some CFC samples was mechanical machined before the surface modification treatment with VI B metals. The composites surface was tool machined and the machining depth was about 100–200 μm.

The contact angles between copper and CFC (as received and surface modified CFC) were measured by the sessile drop technique in a hot-stage microscope (Leitz GmbH AII).

The cross-sections of the joined samples were observed by optical, electron microscopy and energy dispersion spectroscopy (SEM Philips 525M, EDS SW9100 EDAX).

The shear strength of the CFC–Cu joints was measured at room temperature with a compression machine (SINTEC D/10) [9].

The thermal fatigue tests on the joined samples were performed by heating the samples up to 450 °C, followed by a fast cooling from 450 to 25 °C (in air with water quench, cooling rate = 60 °C/s); this heating cycle was repeated 30 times for each sample.

## 3. Results and discussion

The CFC surface modification, described in Section 2, was performed by the slurry technique, a simple and low cost method, and is mandatory to enhance the CFC wettability by molten copper. The formation of a thin carbide layer on the composite surfaces was confirmed by the X-ray diffraction analysis; in the case of the silicon doped CFC, a little amount of metal silicides can be present.

The wetting angle of copper on the modified CFC was lower than 20° at 1100 °C [8]. The wetting angles are approximately the same when the modification was performed by different transition metals inside the VI B group. The surface modified composite could be then joined by direct casting of pure copper at 1100 °C.

The cross-section of a CFC NB31/Cu joined sample is reported in Fig. 1: the thickness of the dense carbide layer is about 5–10 μm, the two interfaces (CFC/carbide and carbide/Cu) are continuous and no cracks or pores are detected in the samples. In Fig. 1 is also detectable a less compact carbide layer (thickness = 10–15 μm), which is completely impregnated by copper: the high wettability of molten copper on the formed carbide allows the capillary infiltration of the carbide porosity.

Fig. 2 is an optical micrograph of a CFC NS31/Cu joined sample; the surface of the composite was mechanically machined before the modification by solid-state reaction. This figure shows that the thickness of the carbide layer is uniform and it is present also in the cavities of the surface, formed during the mechanical

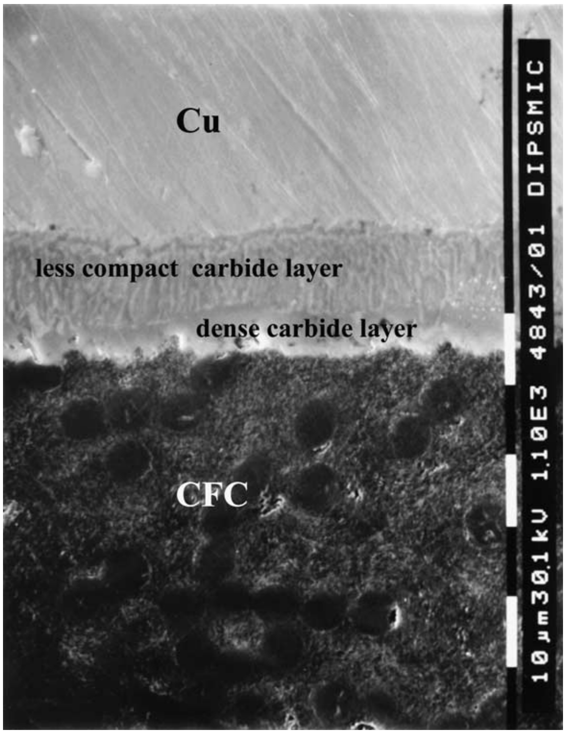

Fig. 1. SEM micrograph of the cross-section of a CFC NB31/Cu joined sample.

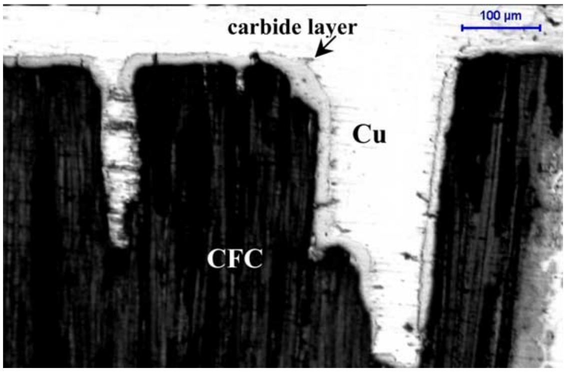

Fig. 2. Optical micrograph of the cross-section of a CFC NS31 mechanical machined/Cu joined sample.

machining. The high wettability of molten copper on the modified CFCs allows the infiltration of these cavities, avoiding the formation of voids at the copper/carbide interface. Furthermore, although the large thermal expansion mismatch between copper and CFC, no cracks are present in the composite or at the interfaces after cooling from 1100 °C (cooling rate = 6 °C/min).

This joining technique did not limit the thickness of the copper layer: up to 5 mm have been cast on modified CFC.

Shear tests at room temperature were performed on CFC NB31/Cu, mechanically machined CFC NB31/Cu, CFC NS31/Cu and mechanically machined CFC NS31/Cu samples. The results are shown in Table 1. In each case the average shear strength of the joint is above 30 MPa and it is higher than the interlaminar shear strength of the CFC (NS31 = 20–25 MPa and NB31 = 15 MPa) [2]. Moreover, the mechanical tests show no difference between mechanically machined and as received composites or between CFC NB31 and silicon doped CFC NS31. Thus, the mechanical machining did not give any improvement of the shear strength at room temperature and can probably be avoided in order to simplify the joining process.

The fracture analysis shows that cracks propagate mainly inside the composite and, some times, through the CFC/carbide interface. The fracture surfaces of a CFC NB31/Cu sample are shown in Fig. 3: on both sides it is evident the presence of the composite that failed by interlaminar shear.

Table 1
Mechanical shear test results of CFC/Cu joined sample

| CFC/Cu | Mechanical machined surface | Average shear strength [MPa] |
|---|---|---|
| NS31/Cu | No | 33 ± 6 |
| NS31/Cu | Yes | 32 ± 3 |
| NB31/Cu | No | 34 ± 6 |
| NB31/Cu | Yes | 31 ± 2 |

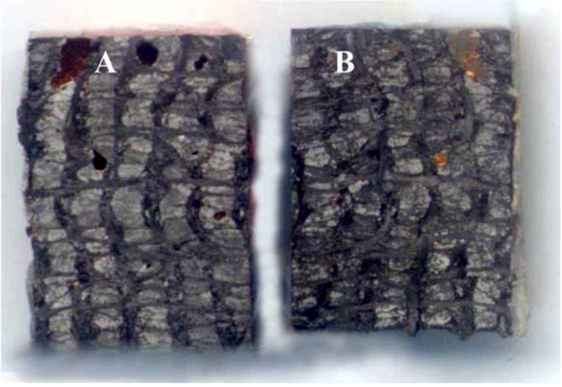

Fig. 3. Fracture surfaces of a CFC NB31/Cu joined sample after mechanical shear test. A = copper side; B = CFC side: on both sides it is clear the presence of the composite.

The joints were also characterized by preliminary thermal fatigue tests in air with water quench. After 30 cycles (from 450 °C to room temperature), the micrograph analysis of the samples did not revealed any damage: there was no separation at interface CFC/Cu and no cracks in the composite or in the copper side. Then, some CFC/Cu joined samples (19 mm × 22 mm) were brazed to the CuCrZr alloy. The brazing process, performed at Ansaldo Ricerche, includes a rapid cooling from 975 to 450 °C (>1 °C/s) and an isothermal treatment at 450 °C for 3 h in vacuum [10]. A CFC NB31/pure Cu/CuCrZr alloy joined sample is shown in Fig. 4. This

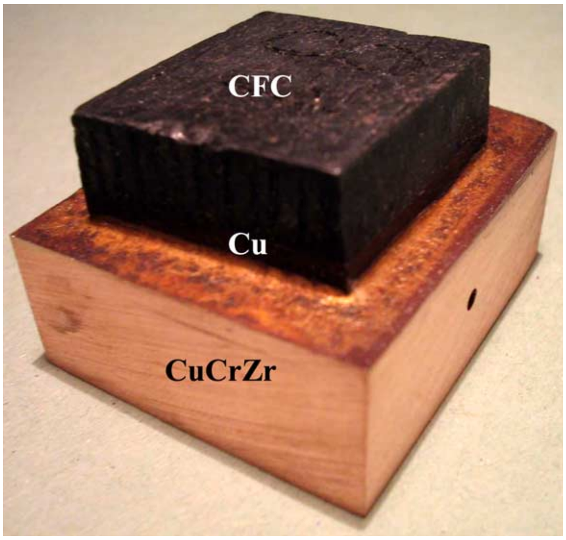

Fig. 4. Macrograph of a CFC NB31/Cu/CuCrZr joined sample after thermal fatigue tests.

sample was submitted to the thermal fatigue test described above and no damages are visible after 30 cycles.

Due to these promising preliminary results, the effectiveness of this joining technique is going to be tested by high heat flux facilities.

## 4. Conclusions

This study proposed a simple and low cost method to join pure copper to undoped or silicon doped CFC.

The shear tests showed that the mechanical machining of the composite surface is not necessary to improve the joint strength at room temperature. In addition, very promising results were obtained by thermal fatigue tests.

Future developments will be addressed towards the mock-up realization for high heat flux tests.

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## References

[1] M. Merola, M. Akiba, V. Barabash, I. Mazul, J. Nucl. Mater. 307–311 (2002) 1524.

[2] Carbon fibre composites, Materials Assessment Report, ITER EDA Doc. Series, 2001 (Chapter 2.3).

[3] Selection of copper alloys, Materials Assessment Report, ITER EDA Doc. Series, 2001 (Chapter 1.4).

[4] F. Rainer, N. Reheis, Process for the manufacturing of a cooling unit, Patent EP 0 663 670, 9 January 1995.

[5] L. Moncel, J. Schlosser, R. Mitteau, L. Plochl, in: Proceedings of the 20th Symposium on Fusion Technology, Marseille, 1 September 1998, p. 133.

[6] V. Branca, A. Federici, M. Grattarola, P. Guarnone, M. Merola, in: 10th International Workshop on Carbon Materials for Fusion Application, 17–19 September 2003, Jülich, Germany, p. 16.

[7] M. Merola, W. Dänner, J. Palmer, G. Vielder, C.H. Wu, Fusion Eng. Des. 66–68 (2003) 211.

[8] P. Appendino, V. Casalegno, M. Ferraris, M. Grattarola, M. Merola, M. Salvo, Fusion Eng. Des. 66–68 (2003) 225.

[9] M. Ferraris, M. Salvo, C. Isola, M. Appendino Montorsi, A. Kohyama, J. Nucl. Mater. 258–263 (1998) 1546.

[10] M. Merola, A. Orsini, E. Visca, S. Libera, L.F. Moreschi, S. Storai, B. Panella, E. Campagnoli, G. Ruscica, C. Bosco, J. Nucl. Mater. 307–311 (2002) 677.