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One-step brazing process for CFC monoblock joints and mechanical testing

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ABSTRACT

A new method of joining CFC to copper (CFC/Cu) and CFC/Cu to CuCrZr alloy (CFC/Cu/CuCrZr) was previously developed for the flat-type configuration. The joining technique foresees a single-step brazing process: the brazing of the three materials (CFC, Cu and CuCrZr) can be performed in a single heat treatment using the same non-active brazing alloy. The composite surface was previously modified by solid state reaction with chromium with the purpose of increasing the wettability of CFC by the brazing alloy.

The feasibility of this process also for monoblock geometry is described in this work. The thermal fatigue resistance of the joined samples (quenching from 450 °C to RT; 50 cycles) was tested and the joints were characterized by apparent shear tests before and after thermal fatigue. The apparent shear strength of the CFC/Cu/CuCrZr joined samples was unaffected after these thermal fatigue tests.

1. Introduction

Plasma-facing components (PFCs) act as actively cooled thermal shields to sustain thermal and particle loads during normal and transient operations in the next step fusion machine ITER. The plasma-facing layer is referred to as ‘‘armour”, which is made of either carbon fibre reinforced carbon composite (CFC) or tungsten (W). CFC is the reference design solution for the lower part of the vertical target of the ITER divertor, which intercepts the magnetic field lines, and therefore removes the heat load coming from plasma via conduction, convection and radiation. The armour is joined onto an actively cooled substrate, the heat sink, made of precipitation hardened copper alloy (CuCrZr) through a thin pure copper interlayer: it is mandatory to decrease, by plastic deformation, the joint interface stresses; in fact, the CFC to Cu joint is affected by the CTE mismatch between the ceramic and metallic material ($CTE_{CFC} = 0.7–1.3 \times 10^{-6}\ K^{-1}$ and $CTE_{CuCrZr} = 16–17 \times 10^{-6}\ K^{-1}$) [1].

Two configurations can be adopted for PFCs: the flat tile and the monoblock. In particular, the monoblock gives a more robust solution in comparison with flat tile for the vertical target and it is now considered the ITER reference geometry [2,3].

The monoblock design requires drilled blocks of CFC into which a CuCrZr tube is inserted and joined [4], also here, a thin layer of pure copper is necessary between CFC and the copper alloy in order to relax high joint interface stress [5]. The monoblock is preferred over the easier to manufacture flat tile design, because of the better heat flux performances and because of the observed tendency for flat tiles to suddenly and totally detach in high heat flux conditions [6].

Actually, the thermal stresses which occurred during the manufacturing process for monoblock geometry are appreciably higher than those of the flat tile, as discussed in Ref. [6] and attention should be given to solutions that mitigate or avoid the failures of armour/heat sink joints, for instance using lower manufacturing temperature.

In the past, high temperature brazing was the reference method for the ITER PFC joining. Brazes used in the past contain a certain amount of silver; today such alloys are not considered in order to avoid cadmium as a product of transmutation after neutron irradiation [7].

Currently, one of the possible solution for CFC to CuCrZr monoblock joint is the manufacturing route proposed by the Austrian company Plansee, the so called AMC® (Active Metal Casting) [8,9]. It involves the casting of Cu onto the CFC surface, which is previously structured by a laser beam to improve the joint strength and then activated by titanium in order to generate a carbide layer, which can be wetted by copper and adheres on the CFC surface. The Cu/CuCrZr joint is obtained using the hot isostatic pressing technique at relatively low temperature (about 550 °C) [4].

Other possible solutions were developed in Europe and Japan for the monoblock CFC/Cu/CuCrZr joining. A joining process for monoblock geometry was developed by ENEA [10]; the CFC monoblock tiles are prepared by pre-brazed casting (PBC) and then joined to the CuCrZr pipe by hot-radial pressing (HRP). PBC consists of a brazing process with a commercial Ti-based alloy and a successive copper casting under vacuum; the brazing alloy contains Ti which acts as an active element and allows the copper casting [11]. HRP [12] is based on pressurizing the internal CuCrZr tube and keeping the joining zone in vacuum at the required bonding temperature; with this process, a radial diffusion bonding between the cooling tube and the armour tile occurs.

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Another process for joining CFC armour to the copper-alloy heat sink was carried out by Ansaldo Ricerche [13]: the techniques involves the use of an active brazing alloy based on Cu, Ti, Al and Si where the Ti reacts with carbon to form a thin TiC layer that helps wetting. Moreover carbon fibres are added in the brazing alloy to smooth the transition between the composite and the Cu and to reduce the residual stress mismatch. The successive Cu/CuCrZr joint is obtained by brazing, by using a Cu–Ge based commercial alloy.

Finally, the Japan domestic agency (JADA) [14] developed a brazing process where CFC armour tiles were brazed to the soft copper interlayer collar by using the Ni–Cu–Mn braze filler with prior Ti–Cu metallization on CFC tile surfaces. While, the soft copper interlayer collar was brazed to the CuCrZr cooling tube with Ni–Cu–Mn (NiCuMn37) braze filler. These braze processes were simultaneously made in a vacuum environment at 980 °C [14].

In this work, the possibility to simultaneously join CFC to copper and to CuCrZr alloy in the monoblock geometry by using the same brazing alloy and by a single heat treatment is demonstrated. Laser structuring or hot pressing is not required. The morphology and mechanical strength of the joined samples were investigated. The shear strength of the joints was measured before and after thermal fatigue tests.

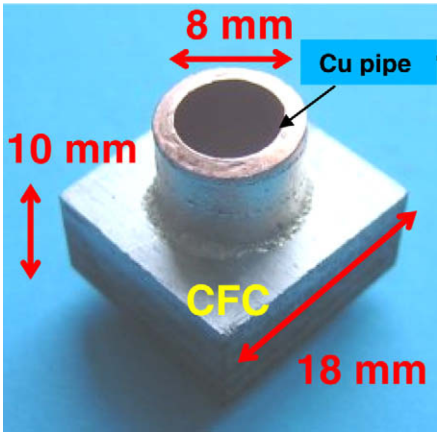

Fig. 1. CFC/Cu monoblock sample manufactured by one-step brazing process.

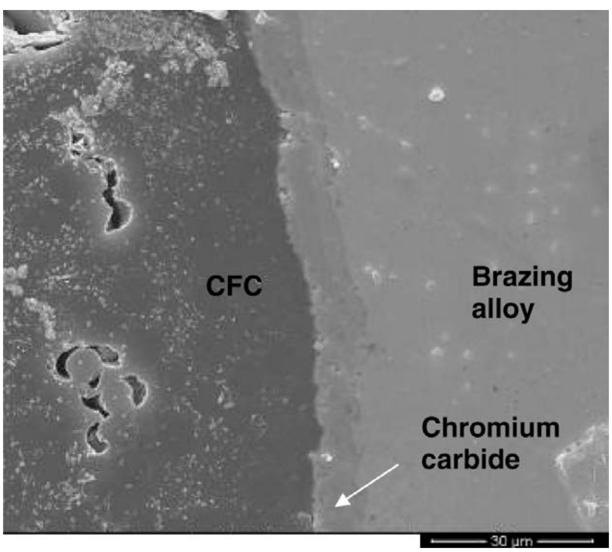

Fig. 2. Scanning electron microscopy of the chromium carbide modified CFC/brazing alloy interface.

2. Experimental

Monoblock samples were produced from high thermal conductivity 3D-CFC materials, CFC NB31 manufactured by SNECMA. Oxygen free high conductivity copper pipe (OFHC), from Goodfellow, and a pipe of CuCrZr ITER grade were used. The manufacturing of CFC/Cu alloy monoblock starts from drilling the CFC tiles along the *Z* direction (needling direction).

The process involves the modification of the CFC surface and then brazing with a commercial non-active brazing alloy, Gemco® (87.75 wt% Cu, 12 wt% Ge and 0.25 wt% Ni; Wesgo Metals). The surface modification process consists of chromium powder deposited by slurry technique on CFC and then a thermal treatment (1300 °C; 1 h; in vacuum) to obtain a continuous layer of chromium carbide through solid state reaction; this layer was proved to be wettable by non-active brazing alloys such as Gemco alloy [15]. The Gemco alloy is used for both the CFC/Cu and Cu/CuCrZr joints (brazing alloy foil thickness = 60 µm); three foils was used for each joint (CFC/Cu and Cu/CuCrZr).

The monoblock CFC/Cu/CuCrZr joints were manufactured by drilling the CFC; the Cr powder was deposited on the inner surface of the hole in the CFC. After the CFC surface modification process, three foils of Gemco were placed in contact with the modified CFC and the pure Cu pipe (10 mm external diameter and 1 mm

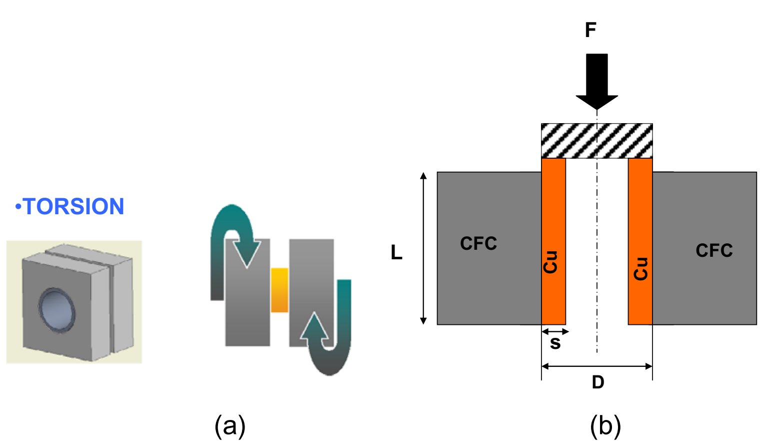

Fig. 3. Drawing of tested sample and proposed shear tests for CFC/Cu samples joined in the monoblock configuration: torsion (a) and shear by compression (b) tests. In (b) F = compression load, D = Cu tube external diameter, s = Cu tube thickness and L = CFC thickness.

thickness) was inserted; the same procedure was carried out with the CuCrZr pipe (7.5 mm external diameter and 1.75 mm thickness) and then the brazing treatment was performed. The brazing conditions (temperature, time, cooling rate, etc.) are described elsewhere [15].

Table 1
Apparent shear strengths of CFC/Cu monoblock samples. Samples "A" were manufactured using Cu pipes inserted in CFC blocks of 18 mm length × 18 mm width × 9 mm thickness (referred to as samples "A"). Samples "B" were produced using Cu pipes inserted in CFC blocks of 18 mm length × 18 mm width × 5 mm thickness.

Sample number	Apparent shear strength (MPa)	
	Samples "A"	Samples "B"
1	12.4	14.6
2	10.8	27.2
3	17.9	24
4	11.2	20.6
5	14.0	8.0
6	12.8	21.2
7	14.8	28.6

Polished cross-sections of the joined samples were characterized by optical microscopy and scanning electron microscopy (SEM Philips 525 M) equipped with EDS analyzer (EDS SW9100 EDAX). Joint apparent shear strength was measured at room temperature by a mechanical test machine (SINTEC D/10).

Preliminary thermal fatigue tests on joined samples were performed; the samples were heated in about 60 s to a temperature of 450 °C with a dwell time of 5 s and then quenched to room temperature in water; the heating/quenching sequence was repeated 50 times. The joint integrity was verified by visual examination and additional apparent shear tests were performed on joined samples after fatigue tests.

3. Results and discussion

3.1. CFC/pure Cu joining

Preliminary attempts were made by the authors to perform joining between CFC and pure copper for monoblock geometry by casting copper in the hole of the Cr-modified CFC, according to method described in [16]. The basic idea was to achieve at first

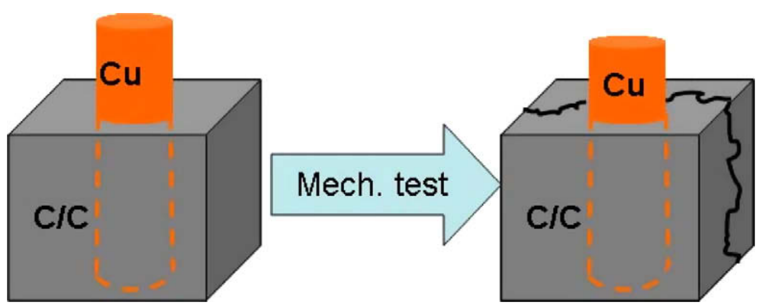

Fig. 4. CFC/Cu monoblock failure after shear test in compression: crack propagates in composite material.

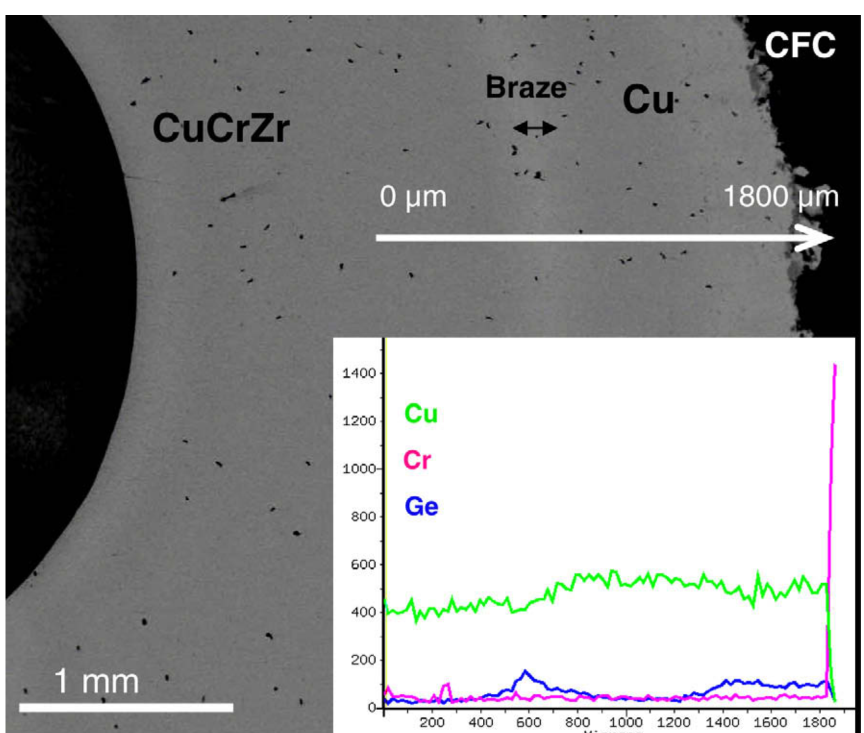

Fig. 5. BSE image of a CFC/Cu/CuCrZr sample cross-section; the arrow indicates the line of EDS analysis (Ni and Zr contents could not be determined because of their low concentrations).

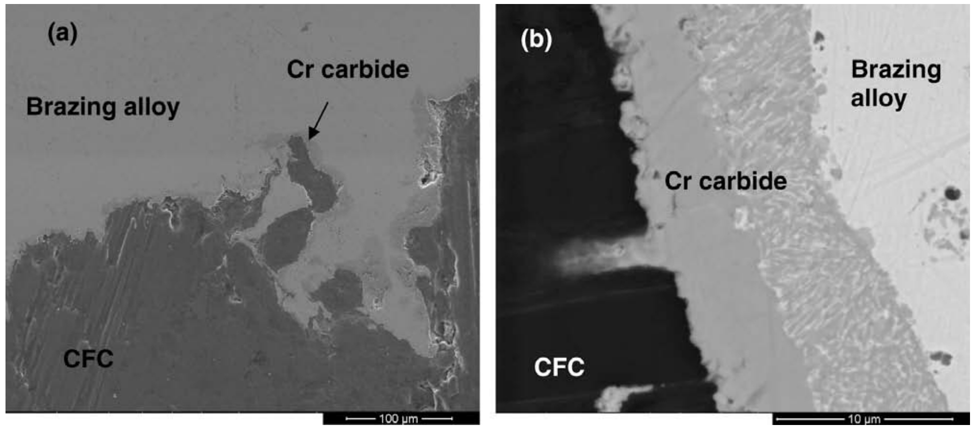

Fig. 6. SEM magnification of sample cross-section before and after 50 quenching cycles.

the joint between CFC and copper and, successively, to drill the solidified copper and reduce copper thickness to 1 mm. Using this method (copper casting) the high thermo-mechanical stresses, due to the high process temperature, lead to unsatisfactory results, i.e., the CFC/Cu interface was partially detached.

With the aim of reducing thermal stresses, further investigations were performed on the monoblock manufacturing using the one-step process. The one-step process was successfully used to produce flat tile mock-ups [15] and it does not involve melting and resolidification of Cu but only the melting of a small amount of brazing alloy, thus reducing shrinkage during cooling. A highly accurate gap size was successfully maintained and precise gap control between the CFC armour and the soft copper collar was obtained.

Preliminary experiments were carried out using only pure Cu pipe to manufacture a CFC/Cu joint; Fig. 1 shows one of the obtained samples.

Fig. 2 shows a magnification of the CFC/brazing alloy interface. An intermediate layer of Cr carbide is present between the composite and the Cu layer; this Cr carbide layer is continuous and the thickness (about 15 μm) is constant along the inner surface of the CFC. The reproducibility of the CFC surface modification

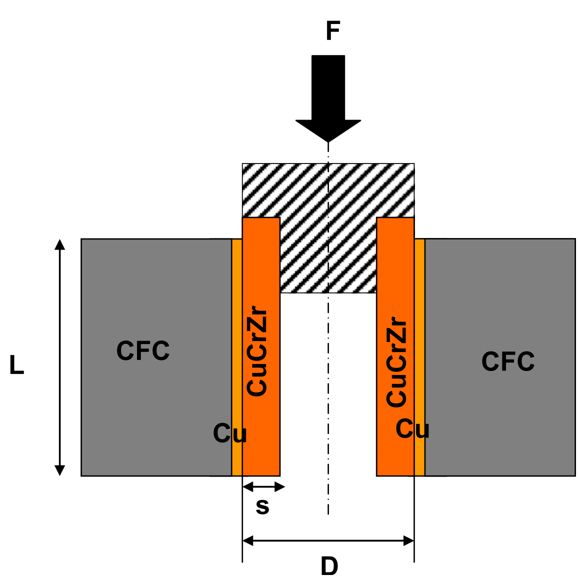

Fig. 7. Compression test on CFC/Cu/CuCrZr joint. *F* = compression load, *D* = CuCrZr tube external diameter, *s* = CuCrZr tube thickness and *L* = CFC thickness.

has been tested as for flat tile configuration [16], thus demonstrating that the Cr carbide surface modification is feasible on a curved CFC surface.

Apparent shear strength of CFC/Cu flat joints produced by one-step brazing was already measured and discussed [15]. For the flat tile configuration an average shear strength of 34 ± 4 MPa was obtained.

Further mechanical characterization was carried out on CFC/Cu samples in the monoblock geometry. Mechanical tests on curved joints require specific designs: some of them are adapted by ASTM D4562-01 [17–19], where apparent shear strengths is measured by a compression test; during these mechanical tests the joint is not stressed in uniform pure shear.

Two different mechanical tests are proposed in this work to measure shear strength for monoblock geometry: torsion (Fig. 3a) and compression (Fig. 3b). Torsion tests on monoblock geometry are recommended, because samples are stressed in pure shear, but some problems can occur while clamping CFC in the fixtures. Actually, several cracks in the CFC during torsion tests occurred and this test was then abandoned.

With reference to the compression test (Fig. 3b), the apparent shear strength of the joint was measured at room temperature with a compression machine.

A first set of joined CFC/Cu monoblock samples were manufactured using Cu pipes inserted in CFC blocks of 18 mm length × 18 mm width × 9 mm thickness (referred to as samples "A").

Mechanical test results for samples "A" are shown in Table 1; the average shear strength is 13.4 ± 1.8 MPa. Fracture surface analysis shows that cracks occurred inside the composite and not within the carbide/braze interface or the braze itself. Fig. 4 shows the typical crack propagation during compression shear strength, probably due to local compressive stresses generated by the Cu pipe lateral strain during the mechanical test.

A second set of samples (samples "B") were manufactured with lower joined area in order to induce failure in the joined area rather than in the CFC. Samples B were produced using Cu pipes inserted in CFC blocks of 18 mm length × 18 mm width × 5 mm thickness. Mechanical test results for samples "B" are shown in Table 1 and for this case the average shear strength was 20.6 ± 5.3 MPa and failure occurred in the composite, with the exception of sample 2 "B", where failure occurred within the carbide/braze interface.

The obtained values of apparent shear strength of the joints can be compared with the CFC intrinsic shear strength (about 15 MPa) [20,21]; thus indicating a joint strength higher than the interlaminar shear strength of the composite itself.

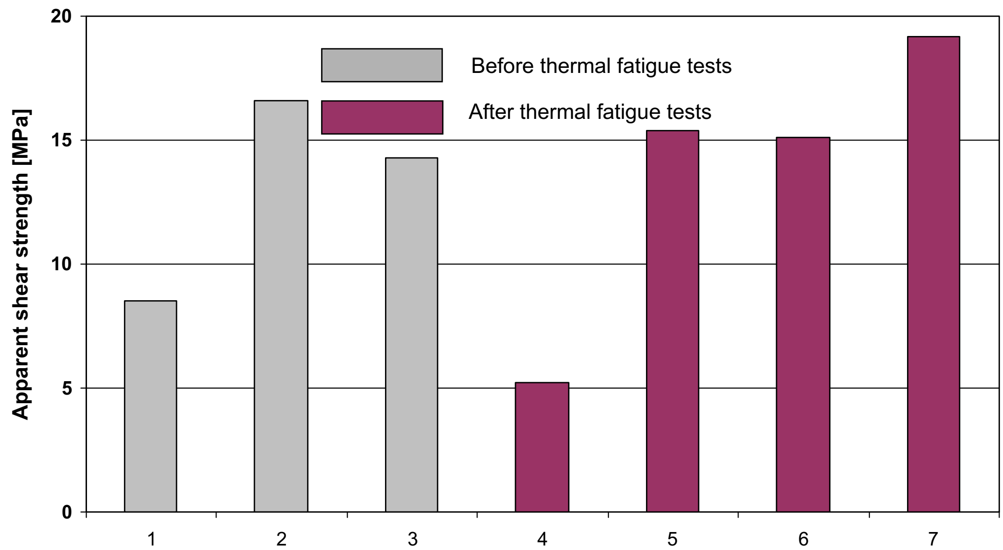

Fig. 8. Comparison of the apparent shear strength before and after 50 quenching cycles for CFC/Cu/CuCrZr joined samples.

3.2. CFC/Cu/CuCrZr joining

Successively, the complete brazing process was performed to obtain the monoblock CFC/Cu/CuCrZr samples. Fig. 5 shows a backscattered electrons (BSE) image of the joined CFC/Cu/CuCrZr sample: the braze is the lighter region between the Cu and Cu alloy pipes. The interface and the braze area are free of structural imperfections such as interfacial microvoids, shrinkage cavities, and micro-cracks. The EDS analysis across the CFC/braze/Cu interface shows evidence of stability of chromium carbide since no chromium diffusion can be detected in the Gemco alloy.

High Ge concentrations were detected at the interface in CFC/Cu across the Cr carbide layer and at the interface Cu/CuCrZr as discussed in [15].

Preliminary thermal fatigue tests on monoblock samples were performed; the samples were heated to a temperature of 450 °C (at 60 °C/s) and, after a dwell time of 5 s, were water quenched; the heating/quenching sequence was repeated for 50 times.

The comparison of the joint cross-section before and after the quenching cycles did not reveal any significant change in the interfacial microstructure (Fig. 6); in some cases, detached interfaces are detected between Cu and CuCrZr but they are probably due to lack of brazing alloy in that area.

Apparent shear tests (Fig. 7) were performed on three CFC/Cu/CuCrZr joined samples before fatigue tests and four CFC/Cu/CuCrZr samples after fatigue tests (Fig. 8). Tested sample dimensions are the same as for samples "B". In Ref. [9] it is reported that in most cases a crack is initiated at the CFC/Cu interface when a CFC monoblock or flat tile fails during the thermal fatigue experiments; consequently shear tests on samples subjected to thermal shock are performed to simulate those conditions.

The average apparent shear strength is 13.1 ± 3.1 MPa for as-brazed CFC/Cu/CuCrZr samples and 13.7 ± 4.2 MPa after thermal fatigue. Fracture always occurred inside the CFC composite, with the fracture starting at the CFC/Cu interface and propagating through the composite.

The shear strength values of these samples (CFC/Cu/CuCrZr) are lower than the ones obtained for CFC/Cu (samples "B"), this is probably due to the use of thicker high thermal expansion materials (Cu and CuCrZr pipes) that caused higher residual stresses near the CFC/Cu interface.

The comparison in Fig. 8 indicates that the applied quenching procedure does not reduce the shear strength of the interface for either brazed samples or quenched samples. Since the difference of the shear strength values before and after thermal fatigue tests is an indicator of the sensitivity to thermal shock, it can be concluded that there is a substantial capability of such interfaces (CFC/braze, braze/Cu, braze/CuCrZr) to tolerate mechanical loads after repeated thermal shocks.

4. Conclusions

A "one-step" brazing process compatible with monoblock geometry and CuCrZr heat sink properties has been developed and tested using thermal fatigue tests. The one-step brazing technique has some advantages in terms of low applied temperature, no pressure required and possibility of performing the CFC/Cu/CuCrZr joints at the same time with the same brazing filler. Moreover, the brazing alloy neither requires "active elements", since the wettability of the CFC tiles is enhanced by surface modification treatment, nor CFC laser machining.

The large thermal expansion mismatch between CFC and copper alloy is more significant than for flat tile configuration, due to curved interfaces.

Preliminary thermal fatigue tests give positive results since joined interfaces do not fail after 50 heating/quenching cycles and mechanical test results are comparable to those of as-brazed samples.

Concerning mechanical tests on joined CFC/Cu/CuCrZr monoblock samples, torsion tests are recommended since the samples are stressed in uniform shear state. Still, cracks can occur at the fixtures/CFC interface during torsion tests and the apparent shear test by compression is an alternative that can be also performed at high temperature.

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