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Tensile fracture characterization of braze joined copper-to-CFC coupon assemblies

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Abstract

A vacuum brazing process was used to join a broad spectrum of carbon-fiber reinforced carbon matrix composite (CFC) materials, machined into cylindrical coupons, between coupons of oxygen-free copper (OF Cu); the braze alloy was a copper-base alloy which contained only low activation elements (Al, Si, and Ti) relative to a titanium baseline specification. This demonstration was of particular importance for plasma facing components (PFCs) under design for use in the Tokamak Physics Experiment (TPX); the braze investigation was conducted by General Atomics (GA) for the Princeton Plasma Physics Laboratory. A tensile test of each brazed assembly was conducted. The results from the braze processing, testing, and fracture characterization studies of this reporting support the use of CFC's of varied fiber architecture and matrix processing in PFC designs for TPX. Further, the copper braze alloy investigated is now considered to be a viable candidate for a low-activation bond design. The prediction of plasma disruption-induced loads on the PFCs in TPX requires that joint strength between CFC tiles and their copper substrate be considered in design analysis and CFC selection.

I. Introduction

Approximately one-half of the carbon-fiber reinforced carbon (CFC) material specified for the plasma facing components (PFCs) of the Tokamak Physics Experiment (TPX) will require direct bonding to copper substrates. All the PFCs, of bolted or bonded designs, will require active cooling. The TPX tokamak will have long pulse capability (≥1000 s) and must accommodate high heat loads (up to 7.5 MWm^{-2} in strike zones of the divertor). In most PFC zones where high heat flux (HHF) of ≥ 0.5 MWm^{-2} is predicted, CFC tile bonding to copper is specified.

The primary goal of the TPX is to develop the scientific basis for a compact and continuously-operating tokamak fusion reactor [1]. The selection basis of CFC materials for the PFCs is addressed in a companion article [2]. As part of the early research and development tasks supporting the design of the PFCs, GA identified the need to bond a variety of CFC designs to copper with the use of a vacuum brazing approach. Particular consideration is necessary for maintaining joint adherence despite the vastly different thermal expansion coefficients of CFC (typically, $<1 \times 10^{-6}$ m/m/˚C) compared to copper and copper braze alloys ($\sim 17 \times 10^{-6}$ m/m/˚C). The technology of brazed CFC tiles and evaluation in HHF tests by electron or ion beam heating is documented [3–9] and the joints were fabricated primarily with silver-copper braze alloys containing a few percent of titanium. During brazing, the mechanism of titanium carbide formation at the braze interface with the carbon is effective for strong adherence. For achieving a design criterion of low neutron activation in the PFC materials for TPX, a braze composition of mainly copper with alloying elements comparable or lower in contribution to the generation of long-lived gamma radiation compared to copper was of interest. Incorporating titanium as an alloying element was also important. A commercial braze alloy of composition 93Cu-2Al-3Si-2.3Ti (wt %) was therefore selected as a candidate braze material for bonding demonstration; the alloy is from the class of "active" braze alloy design, which incorporates titanium as a carbide-forming element and thereby enables metallurgical bonding to the CFC surface. Braze alloy wetting of the carbon and maintaining adherence following the braze cooldown were considered as the most important criteria. Adherence can be investigated quantitatively with tensile loading of brazed joints. Correlation with elastic-plastic stress analysis offers a further opportunity to assess the joined materials. This assessment approach was followed in the effort to provide a demonstration of concept and processing feasibility.

Table 1. Summary of Tensile Test Fracture Results (at 20°C) for Carbon Fiber Reinforced-Carbon Matrix Composites Brazed to Copper

	CFC Producer →	Fiber Materials Inc.	Fiber Materials Inc.	Hercules Aerospace Co.	Science Applications Int'l Corp.	Allied Signal, Inc. ALS Div.	Allied Signal, Inc. ALS Div.	BF Goodrich Aerospace	Mitsubishi Kasei	Kaiser Aerotech	Applied Sciences Inc.
CFC Description											
	Code name	Coarseweave	Fineweave	Coarseweave	ISOCARB 140/PAN (206-B5)	13400-149B-246	865-19-4	AL-C SFB B1-2B	MKC-1PH	KK-188	VGCF-C
	Composite design	4-D C-C	4-D C-C	3-D C-C	4-D C-C	3-D C-C	3-D C-C	3-D C-C	1-D C-C	1-D C-C	1-D C-C
	Fiber architecture	Hexagonal coarseweave	Hexagonal fineweave	Orthogonal	Hexagonal coarseweave	Needled 2-D fabric	Needled felt	Needled felt	Parallel fiber tows	Parallel fiber tows	Parallel oriented fibers
	Carbon fiber type	Amoco P-25	Amoco P-25	AS-4 PAN	T-300 PAN	Amoco P-25	Amoco P-25	(Courtauld/ Zolteck) PAN	(Mitsubishi) Pitch K321	(Amoco) Pitch ThermalGraph	ASI vapor grown carbon fibers
	Matrix precursor	Coal-tar based pitch	Coal-tar based pitch	Pitch	Pitch and resin	CVI carbon	CVI carbon	CVI carbon	Pitch	Pitch	CVI carbon (by BF Goodrich) carbon fibers
Copper substrate	Braze Alloy	(a)Tensile fracture stress — MPa									
OF Cu	Copper ABA	20.2	18.2; 10.0(b) and 16.2(c)	10.1	4.0; 6.0(d)	15.2	6.7; 9.0(e)	2.2	48.8(f)	42.0	7.3
	Fracture location	(g)	(h; h; and g)	(h)	(h; h)	(g)	(h; i)	(i)	(j)	(g)	(k)

(a)Z-axis (fiber) orientation of CFC is parallel with tensile axis unless noted differently.
(b)U direction fibers parallel to tensile axis. Z-axis fibers are 90° from the tensile axis.
(c)U direction fibers at 30° angle off tensile axis. Z axis fibers are 90° from the tensile axis.
(d)Z-axis fibers at 45° to braze plane and tensile axis.
(e)X-Y fiber plane parallel to tensile axis.
(f)Test stopped; no failure.
(g)In CFC at or ≤1.0 mm from interface with braze of 0.1 mm thickness.
(h)In CFC at or ≤1.0 mm from interface with braze of 0.2 mm thickness.
(i)In CFC at ≤3.0 mm from interface with braze of 0.2 mm thickness.
(j)A cross-section metallographic examination, parallel to tensile axis, after (f), revealed no cracks in fibers nor in braze.
(k)In CFC, at approximately one-half depth of ~11 mm thickness.

2. Experimental Approach and Procedures

2.1. Materials

Ten CFC materials were obtained for braze evaluation; their composite designs represented a broad spectrum of constituent type and processing and included: different fiber types (pitch, PAN); fiber architectures 1-D, 3-D, 4-D; and different matrix types (pitch, CVI). The CFC test coupons were machined with a specified fiber axis oriented perpendicular to the end plane (to be brazed).

Cylindrical coupons of 19 mm diameter and nominal length of 20 mm were machined from the 3-D and 4-D CFCs. The z-axis fibers direction was oriented parallel to the cylinder axis (tensile test axis). In addition, for selected CFCs, other orthogonal directions of composite architecture were oriented parallel to the tensile test axis. For the 1-D CFCs, which exhibit extreme fragility in the X-Y plane, rectangular segments of 10 mm nominal thickness were used as the coupons; the 1-D fiber direction was oriented parallel to the tensile axis.

Cylindrical coupons, of 19 mm diameter and nominal length of 37 mm, were machined from oxygen-free copper, type UNS-C10100 (99.99% Cu) wrought plate. A 6.4 mm diameter hole was machined transverse to the tensile axis direction to enable the pin-to-clevis approach for load application.

The braze alloy used was of the tradename Copper ABA, in foil product form, produced by GTE Wesgo Co., California, USA, of composition 93Cu-2Al-3Si-2.3Ti (wt %). The alloy has a solidus temperature of 958˚C and a liquidius of 1024˚C. For each brazed assembly, one braze zone was achieved with a 0.1 mm thick foil. To provide a contrast, two foils, i.e., 0.2 mm thickness, were used at the opposite end of the CFC coupon. Foil diameters of 22 mm were used. The foils were cleaned ultrasonically in organic solvents before and after abrading the surfaces (≤3 µm removal) on a SiC particle (15 µm diameter) abrasive grinding paper. Prepared foil disks were stored under vacuum until ≤2 h prior to use. The end surfaces of both the copper and CFC coupons were ground on a SiC particle (23 µm diameter, nominal) abrasive paper to remove marks from the machining operation. Surface contamination was removed by ultrasonic cleaning in organic solvents. Prior to brazing, the

CFC coupons were exposed to a 1000˚C thermal cycle in vacuum to outgas impurities (≤ 5 x 10^{-3} Pa).

2.2. Braze processing

The assembly of a tensile specimen was accomplished by vertically stacking each CFC coupon between two copper coupons and positioning the braze foil(s) at the interface regions. An additional weight was rested (on axis) on top to achieve an applied normal stress of ~6 $x10^{-3}$ MPa at the braze zones. The braze thermal cycle selected incorporated a heating rate of 450˚C/h and 0.1 h holds at both 700˚C and 980˚C to enable temperature equilibration, and a braze hold at a 1030˚C ± 2˚ maximum (Fig. 1) in a vacuum of ≤2 x 10^{-3} Pa, followed by a rapid cool to 900˚C and a controlled, relatively slow cool of 180˚C h^{-1} below 900˚C.

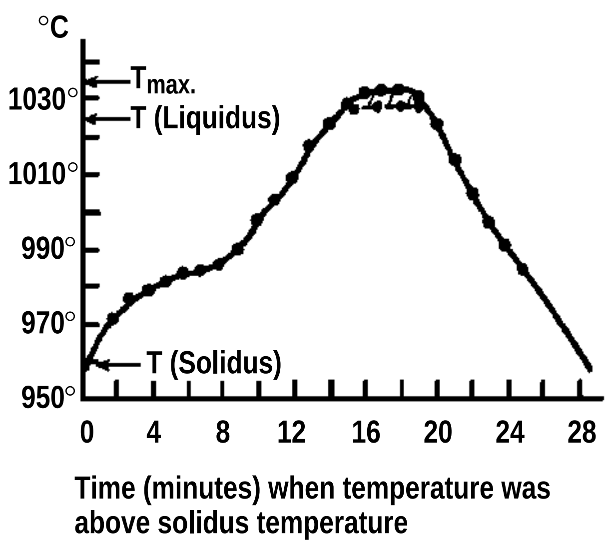

Fig. 1. Thermal profile of braze zone in upper range of thermal cycle for the joining of a CFC surface to copper; braze alloy Copper ABA (Cu-2Al-3Si-2.3Ti, wt%).

2.3. Characterization methods

Brazed specimens were examined on peripheral surfaces at ≤16x magnification using an optical stereomicroscope to verify wetting of the braze at the Cu to CFC interface. Also, the CFC coupon periphery was examined for evidence of cracking across fiber bundles and for macrocracks larger than the inherent microcracks from CFC processing which typically are present along the fiber bundle-to-matrix interface and within matrix regions.

To assess braze adherence and to achieve a measure of joint strength, a tensile test to fracture was conducted with each specimen assembly. Load application was uniaxial, perpendicular to the plane of the brazed joints. All tests were conducted in an (MTS Company) electro-servo hydraulic controlled testing machine. The linkage of the load train included the use of a universal joint and multiple pin-to-clevis pivot joints to facilitate specimen installation and to minimize bending of the specimen. All tests were conducted at an axial displacement rate of 2.1 x 10^{-3} mm/s. The test temperature was 21˚C. Using an X-Y recorder, a graph of load applied versus displacement during testing was monitored. The nominal cross sectional area of each CFC coupon was used with the failure load to determine the fracture strength. The location of fracture was recorded with reference to the nearest braze zone. Microscopy was employed to examine fracture surfaces and cross sections of a few specimens.

2.4. Stress analysis

A 2-D stress analysis was conducted for three tensile specimens to determine if the analytical results showed qualitative agreement with the strength measurements. The analysis used the ANSYS 5.1 finite element analysis and allowed for elastic-plastic behavior of the copper coupons during the braze cooldown event. The carbon composites were treated as an elastic material since behavior after localized cracking during a braze cycle cooldown is an unknown. The braze cycle was modeled as a stress-free condition at 750˚C with complete bonding between the C-C and copper. Slow cooling was assumed to 20˚C. After this, tensile load was applied to the copper. A yield strength of 34 MPa was used in the analysis for annealed copper.

3. Results and Discussion

3.1. Brazed assemblies

All of the CFCs brazed well to the copper coupons. Good wetting was observed and fillets developed on the peripheral surface of both the CFC and Cu. No evidence of gaps or voids were observed at the braze interfaces. Macrocracks were not observed on the CFC periphery. Exposed fiber bundles oriented perpendicular to the braze plane did not exhibit microcracks. In Fig. 2, two of the brazed tensile specimens are shown. The light shaded

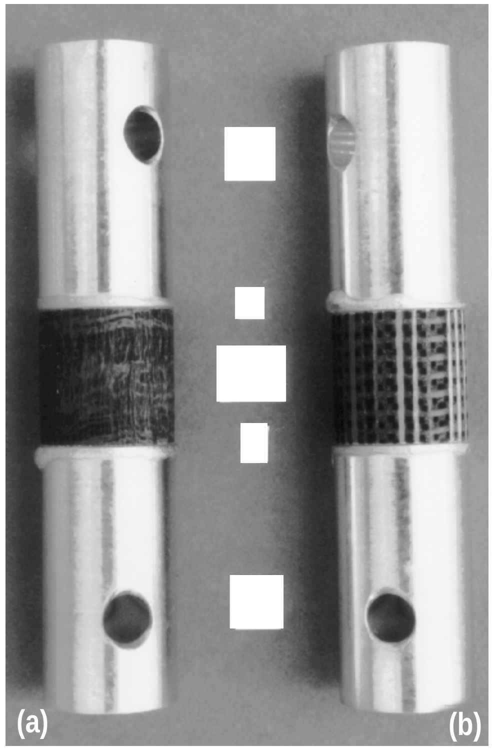

Fig. 2. Tensile specimens assembled by vacuum brazing copper coupons (19 mm diameter) to CFC coupons. *Braze alloy: Copper ABA (Cu-2Al-3Si-2.3Ti, wt %). (a) CFC type: 3-D, 865-19-4, (Allied Signal Inc.) (b) CFC type: 4-D Fineweave (Fiber Materials Inc.).

regions on the CFC reveal the locations of these Z-axis fiber bundles. The 3-D pseudo random fiber orientation, characteristic of the Fig. 2(a) CFC, is in contrast to the linear bundle orientation characteristic of the fully woven type CFC in Fig. 2(b). The braze fillets are similar for both specimens; the Copper ABA alloy did not flow beyond the fillet onto the CFC surfaces. However, for the copper coupons, localized flow beyond the fillet was typically observed.

Each 1-D CFC exhibited a few macrocracks through the coupon thickness, i.e., the crack planes were parallel to the Z-axis fiber bundles; more cracks of this form and orientation were observed after the braze cycle. This observation was not unexpected as 1-D CFCs are typically very weak in tension when stressed parallel to the X-Y plane; indeed, fracturing of this type was occasionally experienced during the machining and/or handling of the 1D CFC stock.

3.2. Braze microstructure

In Fig. 3, a photomicrograph is presented to show the microstructure features of the Copper ABA braze alloy after the joining of copper to the CFC type FMI 4-D C-C Coarseweave. The braze zone appeared uniform both in thickness and microstructural features across the examined diametral section. The joint in Fig. 3 incorporated two foils; therefore the modest normal stress of 6 x 10^{-3} MPa during brazing compressed the initial 0.2 mm of foil thickness to a braze zone thickness of 0.08 mm. Evidence of porosity or defects was negligible in the braze. After examining the joints in all of the ten CFC brazed specimens, it would appear that the use of one 0.1 mm thickness foil is sufficient to obtain a satisfactory joint. Upon examination of the CFC periphery adjacent to the braze zone, the presence of isolated microcracks appeared limited to within matrix "islands" and along fiber bundles oriented parallel to the braze plane. The dark gray phase within the braze zone of Fig. 3 was analyzed (during SEM analysis) by the energy dispersion x-ray method and was determined to be a Cu phase rich in Si and Ti, i.e., alloying elements of the specified braze alloy. At the interface of the braze and CFC coupon a phase of high Ti content was detected which is typical of titanium carbide phase which is formed there at the time of brazing.

Fig. 3. Microstructure of cross-sectioned braze joint; braze alloy (Copper ABA) between coupons of OF Copper UNS-C10100 and CFC, type FMI 4-D C-C Coarseweave composite. *Interface phase of TiC (~5 µm thickness) formed during brazing. NOTE: Z-axis fibers are perpendicular to braze plane.

3.3. Braze joint strength

The braze specimens demonstrated a linear relation of tensile load versus extension up to the load for fracture. Except for two strong 1-D CFCs, fracture occurred within the CFC coupons very near the braze zone, at a tensile stress lower than the 0.2% offset yield strength (28 MPa) of the annealed OF copper. The fracture plane was typically at an angle nearly perpendicular to the load axis, parallel to the braze plane, and between 0.1 and 0.3 mm from the braze, as shown in the photographs of Fig. 4. No lack-of-bonding type defects were observed on the fracture surface of the ten tested specimens. The strengths ranged between 2 and 20 MPa for the specimens incorporating the 3-D or 4-D CFCs; these values are low compared to the reported or estimated tensile strengths of the CFCs. In Table 1, a summary is presented of the ten 4-D, 3-D and 1-D CFCs brazed and tested. Pertinent pedigree of processing is provided in the upper half of the table. The lower part of Table 1 lists the fracture strength values from the tensile test of each specimen assembly of the corresponding CFC and copper coupons brazed with the Copper ABA alloy. The footnotes clarify the orientation of the CFC fiber architecture in relation to the braze plane and the uniaxial direction of the tensile test. The bottom row of Table 1 identifies the location of the fracture in relation to the nearest braze plane and more specifically to which braze zone, i.e., the zone of one 0.1 mm thickness braze foil or two foils (0.2 mm).

3.4. Fracture surface microstructure

Scanning electron microscopy (SEM) analysis of the fracture surface of the FMI 4-D Coarseweave CFC coupon revealed a characteristic which helps explain the mode of fracture and relatively low strength level. In Fig. 5, the SEM micrograph presented delineates the fiber bundle ends which were perpendicular to the braze plane. A Ti and Cu rich zone (detected by energy-dispersion x-ray analysis) covers the bundle ends. This feature was typical of a majority of the bundles. This observation strongly supports an explanation of CFC strength as being a function of the fiber volume fraction of the CFC that is oriented at a steep angle to the braze plane. The examination of a cross-section oriented perpendicular to the braze plane (of the opposing fracture half) revealed no voids or cracks in the braze layer. Further, the braze layer appeared adherent to the copper coupon. For two CFCs listed in Table 1 information known about the composite strength and fiber volume enabled a comparison of composite strength, braze joint strength and the product of fiber volume fraction and CFC tensile strength (for a specific orientation). Table 2 presents this

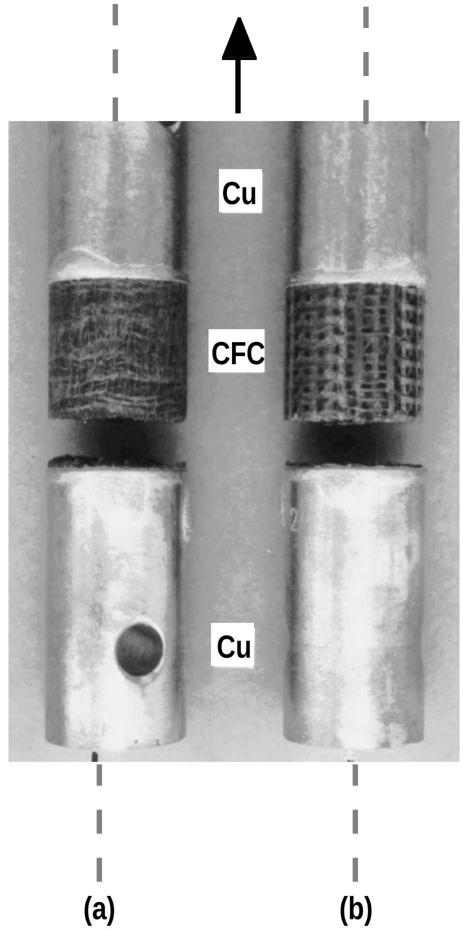

Fig. 4. Typical fracture locations in brazed specimens following tensile test to failure, at 21˚C. (a) CFC type: 3-D, 865-19-4 (needled fibrous felt and CVI matrix); (b) CFC type: 4-D Fineweave (hexagonal fully woven and pitch matrix).

information. The rationale for assuming fiber ends (brazed to copper) are the primary carrier of tensile load would appear to be reasonable on a first order basis.

3.5. 1-D CFC fracture characteristics

Two of the 1-D CFC braze specimens were exceptionally strong as compared to the 3-D and 4-D type CFCs. Loading of the specimen with the MKC-1PC CFC was stopped at 48.8 MPa and unloaded. The copper coupons had experienced appreciable plastic strain. A

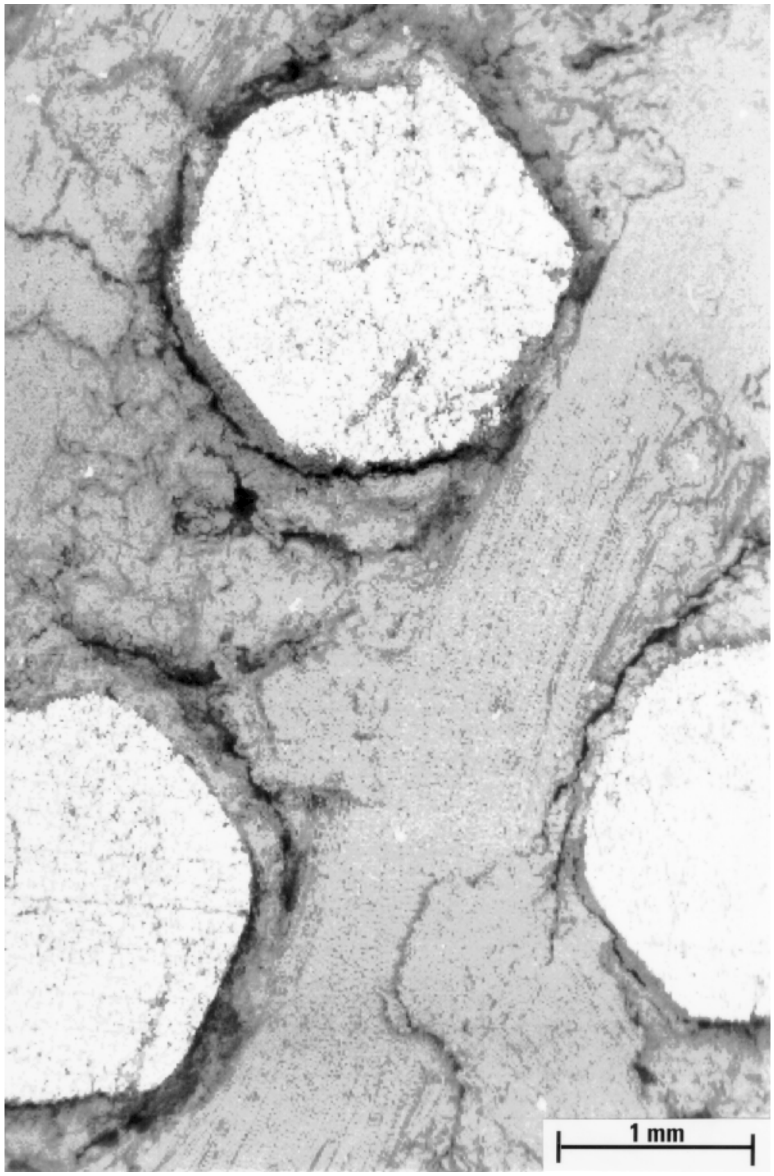

Fig. 5. Fracture surface microstructure of the CFC coupon. Ends of the Z-axis fiber bundles (where they were joined to the braze) are revealed by light shading by the Ti-rich copper layer of the braze-zone interface. SEM micrograph image achieved with back-scattered electrons. Fracture surface of the adjacent CFC matrix and fractured fibers (parallel to braze plane) are not covered with braze alloy and are ≤1 mm from the braze zone.

cross section made perpendicular to the braze planes revealed no braze defects and no cracks transverse to the unidirectional carbon fibers .

Table 2. Correlation of Brazed Joint Strength and CFC Composite Parameters

		Fiber Volume Fraction for CFC Composite Orientation[a]			(Fiber Volume Fraction) x
CFC Composite Type[b]	CFC Producer's Estimated Tensile Strength for CFC (MPa)	CFC Axis Perpendicular to Braze Plane	V_{fiber}/V	Brazed Joint (Cu-to-CFC) Strength[b]	(Estimated Tensile Strength of CFC) (MPa)
4-D Fineweave CFC (FMI)	200	Z	0.209	18.2	41.8
	55	U	0.092	10.0	5.1
3-D needled fibrous felt 865-19-4 (Allied Signal Inc.)	100	Z	0.065	6.7	6.5
	100	X	0.098	9.0	9.8

[a]The CFC fiber volume fraction Vfiber/V is the produce of overall fiber volume of CFC multiplied by the fiber volume distribution of a specified composite orientation.
[b]CFC descriptions and fracture strength values from Table 1.

The VGCF-C CFC is different from the other two 1-D CFCs in that the fiber lengths are typically much shorter and not continuous across the 11 mm coupon thickness. Inherent strength of the CFC would therefore be influenced substantially by the carbon matrix.

3.6. Stress analysis

The 2-D elastic-plastic analysis predicted very high tensile residual stresses near the specimen periphery in the CFC following cooldown from the braze temperature because of the extremely different thermal coefficients-of-expansion (CTE) between the Cu and CFC. However, with the 1-D CFC, the relatively high CTE and lower modulus of elasticity in the X-Y plane directions enables residual tensile stress to remain below the reported ultimate strength of the MKC-1PH in the 1-D, i.e., Z-direction. For the 3-D and 4-D CFCs, fracture was predicted during cooldown from the braze temperature due to tensile stresses exceeding the reported CFC strength values. In fact, no brazed specimens exhibited fracture after the braze cycle. Plastic straining of the annealed copper helps to relieve stresses in the CFCs. However, the additive stored residual stress in the CFC segment of the brazed specimens may be a factor in causing fracture during the tensile tests at stress levels substantially lower than values reported for unbrazed CFC. In addition, microcracking within the carbon matrix zones may aid in the relief of CTE derived residual stresses.

4. Conclusions

The results from these processing, testing and characterization studies support the use of CFC composite designs, i.e., of varied fiber architecture and matrix processing, in bonded CFC-to-copper PFC designs for TPX. To proceed further, HHF testing of CFC tiles bonded to copper substrate is warranted to evaluate actively cooled CFC flat block and macroblock designs. Further, the braze alloy Copper ABA (Cu-2Al-3Si-2.3Ti wt%) has been shown to be a viable candidate for achievement of the low-activation braze designs of specific interest to TPX. In addition to the stresses arising at a joint interface from plasma operational and vessel bakeout cycles, plasma disruption-induced loads predicted for TPX will develop impulsive stresses on CFC tiles and therefore require joint tensile strength levels to be considered in PFC design and CFC selection; the reported results should be useful in conducting these engineering tasks.

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