ADVANCED X-RAY IMAGING OF METAL COATED/IMPREGNATED PLASMA-FACING COMPOSITE MATERIALS

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Abstract

A combination of X-ray imaging techniques is employed for the characterization of coated/impregnated carbon based composite materials to be used as Plasma Facing Components (PFC) in fusion devices. X-ray micro-tomography (μXCT) is applied for the visualization of Carbon Fiber Composites (CFC) – Cu joined samples and also for the CFC material porosity network characterization. The quantitative determination of the tungsten coating thickness on carbon materials is performed using a combined absorption/fluorescence X-ray technique (μXRFT). The method was applied at the analysis of W coated fine grain graphite (FGG) and CFC tiles. The X-ray imaging techniques provide fast analysis and high spatial resolution.

1. Introduction

Due to their excellent resistance to excessive high heat and thermal shock loads, CFC is one of the favoured candidate armour materials for ITER's divertor strike point plasma facing components [1]. However, the intense heat flux received by these components implies active water cooling. This is achieved through a welding - active metal casting (AMC) [2] - between the metallic water loop made of Cu alloys and the CFC material. To improve the relatively

weak mechanical bond between CFC and Cu the composite surface is often coated by Ti/TiC. This procedure leads to an increased wettability of CFC by molten copper [3].

The CFC porosity plays a major role in both fabrication and operating of PFCs. The reason is twofold: First, the quality of the AMC of the monoblock geometry developed for ITER depends on the metal impregnation inside the CFC and second, a part of the fuel retention is due to the co-deposition mechanism inside the CFC porosity. The present paper presents results concerning the analysis of relevant PFC materials coated with refractory metals (W, Mo) and/or welded on Cu heat sink structures. μXCT is applied for the visualization of CFC – Cu joined samples and also for the CFC material porosity network characterization.

Tungsten erosion, subsequent tungsten transport, and redeposition are of great interest, because a full tungsten divertor is foreseen to be used during the deuterium-tritium operational phase of ITER [2]. Upgrade and work is currently in progress to completely replace the existing JET CFC tiles with tungsten-coated tiles within the JET ITER-like wall project. The need for a fast and nondestructive method which allows the quantitative determination of the thickness of a tungsten coating on a carbon material on large areas motivated us to develop a combined absorption/fluorescence X-ray technique [4]. We show in this paper that this method provides fast analysis with high spatial resolution of the deposited layers.

2. Methods and materials

The X-ray tomographic experiments were performed on our newly upgraded cone-beam X-ray tomography facility [5-6] (Fig. 1). The system is equipped with a high performance Nanofocus X-Ray source for non-destructive inspection. The source is operational in micro- or nano-focus regime in which case it is capable of sub-micron feature recognition, at a tube voltage up to 225 kV and a maximum power of 10÷20 W. X-Ray images can be acquired by using three different high resolution detector types: Image Intensifier (768x576 pixels of 132x132 μm^2) for rapid

non-destructive examinations and CMOS flat panel (pitch size 48 μm) as very high resolution 2D imaging detectors and a line detector (pitch size 400 μm) for the slice by slice scanning of high density samples. Positioning and turning around of the sample are ensured by a set of seven high precision motorized micrometric manipulators. Automation, control and data acquisition were obtained by means of an in-house software package. The tomographic reconstruction for the cone-beam scanning is based on an optimized implementation of the modified cone beam filtered back-projection algorithm. Using a parallelization technique on multiprocessors workstations, experimental data consisting of several hundreds of large radiographic images (1220x1216 pixels) are processed for building the 3D reconstructions of typically 1024x1024x1024 voxels in less than 12 min.

The method for tungsten coating analysis was implemented using the Tomo-Analytic system, which was developed especially for fusion materials analysis [4]. Tomo-Analytic is a combined X-ray fluorescence (μXRF) and cone-beam μXCT system for the noninvasive 3D morphology and composition mapping. The key element of the μXRF component is a policapillary lens which provides a focal spot size of few tens of micrometers. A significant increase of the X-ray intensity (up to three orders of magnitudes) is obtained [7] which allows improved detection sensitivity.

Tomo-Analytic is a configurable and versatile tool in which different measuring methods can be accommodated for the characterization of the thickness uniformity of FGG/CFC with metallic coatings:

μXRF) the coating X-ray fluorescence peak intensities are converted to elemental concentrations and/or film thicknesses.

μXRFS) the X-ray fluorescence radiation emitted by the substrate is attenuated by the coating material; a correlation can be derived between the secondary emissions and the coating thickness.

μXRFB) the coating thickness is determined from the correlation with the attenuation of the X-ray back-scattered radiation by a substrate with low effective atomic number. This procedure has the advantage to be more suitable to carry out reference free thickness measurements.

μXRT) the geometry for the μXRT method is presented in Fig. 2. The X-rays are detected by an energy selective detector after passing through the investigated sample where they are attenuated accordingly with the composition and thickness of the materials.

The optimal measurement configurations and the irradiation parameters were obtained by MCNP-5 Monte Carlo simulations [8].

In order to analyse the brazing of NB31 CFC to Cu one scans volumes of 5x5x5 mm^3 which can be considered statistically relevant for the CFC material while allowing a maximum possible space resolution.

N11 CFC samples of a relatively small volume (2x2x6 mm^3) were provided to us in the frame of the Deuterium Inventory in Tore Supra (DITS) post-mortem analysis [9]. By μXCT information about the pores connectivity as well as the metal impregnation inside the CFC macroscopic pores, in case of heat sink region of the TS CFC, can be retrieved.

The tungsten coatings have been deposited on FGG and CFC samples by the Combined Magnetron Sputtering and Ion Implantation (CMSII) technology. This was recently used for W coating of about 2,000 CFC tiles for the ITER like Wall project at JET and approx. 1,000 FGG tiles for the ASDEX Upgrade tokamak at IPP Garching. The CMSII technique involves simultaneous magnetron sputtering deposition and high energy ion bombardment. A high voltage pulse discharge (U = 30 – 50 kV, τ = 20 μs, f = 25 Hz) is applied on the substrate alternatively with the DC bias. The periodical ion bombardment increases the surface mobility of the deposited atoms which leads to a high densification of the coating. Glow Discharge

Optical Spectrometry (GDOS) is currently used for measurement of the coating thickness and impurities as a quality control technique for industrial production. More details about the fusion relevant W coatings including equipment and technology can be found in [10-12].

3. Results

μXCT is used to compare the overall quality of the brazing of CFC to Cu with or without coating of CFC by Ti or TiC. A relatively large volume of 5x5x5 mm^3 at the interface between Cu and CFC was scanned at a voxel resolution of 6.6 μm. In Fig. 3 the bright regions correspond to the impregnated Cu. To enhance visualization of this phenomenon, additional image post-processing was performed. The tomography images were orthogonally aligned and an image fusion was obtained by summing the transversal (Fig. 3 a1, b1, c1) and longitudinal (Fig. 3 a2, b2, c2) cross-sections. The integral of the volume of the Cu impregnated in CFC can be considered a good measure of the brazing efficiency. Based on this measure Ti coating apparently leads to better brazing although with a maximum penetration length less than 0.3 mm. Due to the more than one order of magnitude difference between the X-ray attenuation coefficients of carbon and Cu the image reconstruction display strong artefacts. However, the μXCT analysis clearly reveals the tendency of the infiltration of Cu along the macroscopic pores formed between the ex-pitch and ex-pan fibers. An optimized μXCT scan of the CFC region helps to visualize the morphology of the NB31 CFC: ex-pitch fibers are aligned in vertical perpendicular planes and the ex-pan fibers bundles are perpendicular on the image surface (Fig. 3 d).

Fig. 4 illustrates the 3D structure of the metal inside the macroscopic pores of a N11 CFC sample cut from the interface with the Cu heat sink. One can see that the CFC macroscopic pores are nicely coated by metal (Ti) and the slimmest ones are totally filled by Cu. The identification of the materials was certified by X-ray fluorescence analysis. In this case Ti plays the beneficial role of a CT contrast medium, which emphasizes the pores contours. The

presence of the raster of conical drillings, with a base diameter of 0.125 mm and a maximum height of 0.4 mm, totally coated by Ti and partially filled by Cu, is due to the way the fingers of the TS Toroidal Pump Limiter (TPL) are built. The Cu heat sink and CFC tiles are joined using AMC [13]. One can think of imaging the 3D reconstructed model of the infiltrated metal as input data for the evaluation of the thermal properties of the CFC – heat sink assembly.

3D CT images are used to describe the distribution of macroporosity with respect to the position of the carbon fibre bundles and its correlation with the impregnation pattern of the heat sink material. As the first CFC developed by SNECMA for fusion technology, N11 displays a rich porosity network with elongated pores, which have sizes up to a couple of mm (Fig.5). The main difficulty for the estimation of the porosity factor is to obtain a closed bordering surface also in the regions where the macroscopic pores touch the exterior surface of the sample. With the procedure introduced in Ref. 6 one obtains an open porosity factor of ~11%, in good agreement with the nominal value of 12% specified by the manufacturer.

The sensitivity and the calibration of the X-ray methods (μXRFB and μXRT) for the W coating thickness analysis were carried out on a non-exposed, tungsten coated FGG sample with three thickness steps determined by metallographic cuts by SEM (Fig. 6). A detailed discussion of the SEM and the μXRFB thickness values is to be found in Ref. 4. Accordingly, the reliability of the μXRT method is limited if a polychromatic incident X-ray spectrum is used. Therefore a new experimental geometry was implemented (Fig. 2). It comprises a quasi-monoenergetic X-ray source (MoKα line of 17.48 keV) and an energy selective detection system (150 eV energy resolution). Table 1 summarises the W thickness values and their associated standard deviations. The relatively large scattering of the SEM values is attributable to the roughness of the W layer. For the μXRT method one uses the SEM samples with a FGG substrate of only 0.5 mm. This FGG substrate has an X-ray transmission coefficient of 95% as calculated with the program of Ref. 14. The errors associated to the thicknesses values are standard deviations

calculated over series of several tens of measurements points and do not include the uncertainty of the X-ray attenuation cross-sections, which has a direct influence on the absolute accuracy of the method.

The calibration procedure proves that the μXRT method ensures fast and high resolution analysis and could resolve well below 5% in W thickness difference. Another advantage of the μXRT method is that it can be used for thicker W layers as it does not reach a saturation thickness required by the methods based on X-ray fluorescence. Thus, with the Mo anticathode X-ray tube used in our configuration, one can measure up to 20 μm thick W. This value represents the W thickness range of the W coated JET ITER-like wall tiles and of the FGG coated tiles of AUG.

To demonstrate the applicability of this technique we have measured a 5 mm thick FFG sample coated with ~10 μm W on top of a Mo interlayer of ~2.5 μm. The average result expressed in W equivalent thickness is 10.6 ± 0.2 μm. The GDOS analysis on a witness sample coated simultaneously with the FGG sample gives 9.2 μm for W and ~ 2.5 μm of Mo. As 2.5 μm of Mo is equivalent with ~0.3 μm of W in terms of X-ray attenuation at 17.5 keV one can notice a difference of ~10% of the W layer thickness. If the GDOS values are to be considered as reference, one possible reason of this overestimation is the inaccurate compensation of the non-linear pulse pileup effect for the measurements with and without W coating layer.

Also we have mapped with high space resolution (150 μm step) the W thickness of similar coating layers on a 5 mm thick NB31 CFC sample (Fig. 7). One can see the quasi-monoenergetic energy spectra of the X-ray beam transmitted through the coated and uncoated NB31 CFC sample (right panel). A transmission coefficient (T=0.185) is obtained by normalizing the intensity of the X-ray beam transmitted through the coating to that transmitted through the coating free area resulted during a pulling test. With the program in [14] one can

estimate an average W equivalent thickness of 9.9 ± 0.2 μm. No GDOS data is available for comparison.

4. Conclusion

High resolution cone beam μXCT was optimized for the analysis of metal coated/impregnated CFC samples. It is proved that the tomographic analysis provides substantial new information about the pores connectivity and, in case of heat sink region of the TS CFC TLP, about the 3D structure of the metal impregnation inside the macroscopic pores. The results obtained by 3D μXCT analysis of statistically relevant volumes of CFC can be considered as a good basis for the monitorization of the development of actively cooled PFC.

An instrument as well as associated measuring methods based on X-ray imaging have been developed and qualified as a non-invasive solution for investigation of the thickness of W coatings on carbon materials substrates. The Tomo-Analytic instrument is a combined X-ray fluorescence (μXRF) and cone-beam μXCT system for the non-invasive 3-D morphology and composition mapping. The μXRT component ensures fast and high lateral resolution (≅ 20 μm) analysis of large areas of carbon materials coated with tungsten. The combined use of X-ray transmission and fluorescence methods represent a unique instrument for the post-mortem analysis of large area coatings. These techniques are currently being applied on W coated FGG tiles from the all-tungsten divertor of AUG and in the postmorten analysis of ITER-like wall W coated tiles.

Acknowledgements

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Table 1 – Calibration of the X-ray methods for W coating thickness analysis.

Layer no	Layer thickness / standard deviation		
	SEM	μXRFB	μXRT
1	2.78 ± 0.06	2.74 ± 0.08	2.69 ±0.05
2	2.94 ± 0.07	2.84 ± 0.10	2.87 ±0.05
3	3.15 ± 0.09	2.97 ± 0.11	3.10 ±0.06

FIGURES CAPTION

Figure 1 - View of the NILPRP NanoCT facility. Inset shows the diamond target – sample configuration.

Figure 2 - Tomo-Analytic geometry for combined fluorescence/transmission experiments.

Figure 3 - Image fusion of axial and longitudinal tomographic cross-sections: uncoated CFC (a1, a2), CFC coated by Ti (c1, c2), CFC coated by TiC (b1, b2). Bright regions correspond to impregnated Cu. A longitudinal cross section of the NB31 CFC samples which shows the ex-pitch and ex-pan fibers (d).

Figure 4 - Tomographic analysis of the Cu heat sink region of a DITS sample: 3D reconstructed volume (top-left), axial (top-right), sagital (bottom-left) and transversal (bottom-right) cross-section. Bright regions correspond to Cu and gray regions correspond to Ti-coated macropores (cross-section width 2 mm, voxel resolution: 2.75 μm).

Figure 5 - Transversal (a), longitudinal (b) and sagittal (c) cross-section through the tomographic reconstruction of a N11 CFC sample after image thresholding for porosity factor calculation (same reconstruction parameters as in Fig. 4).

Figure 6 - FGG calibration sample with three W thickness steps determined by metallographic cut on SEM.

Figure 7 - W coating uniformity mapping by μXRT. An area of 10x5 mm^2 was scanned with 0.15 mm step (left). The quasi-monoenergetic energy spectra of the X-ray beam transmitted through the coated and uncoated NB31 CFC sample (right).

Figure 8 -

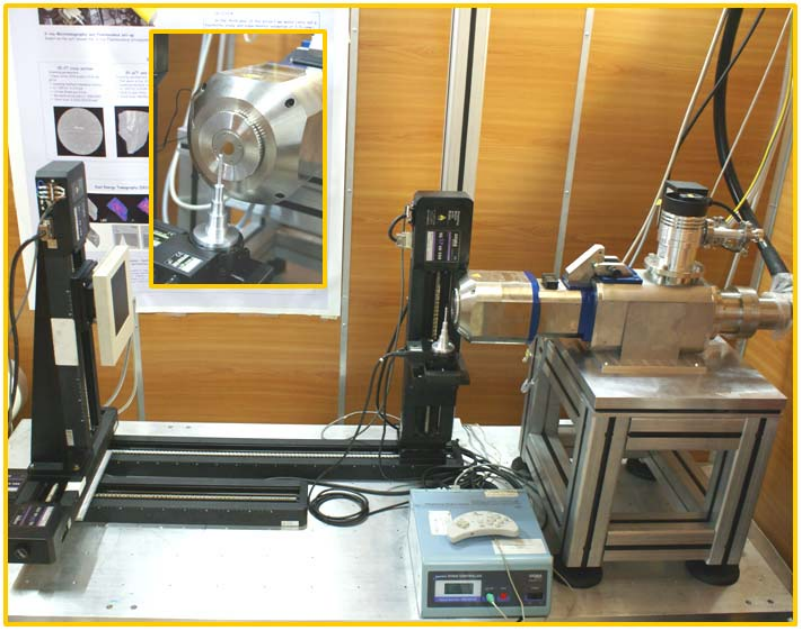

Figure 1

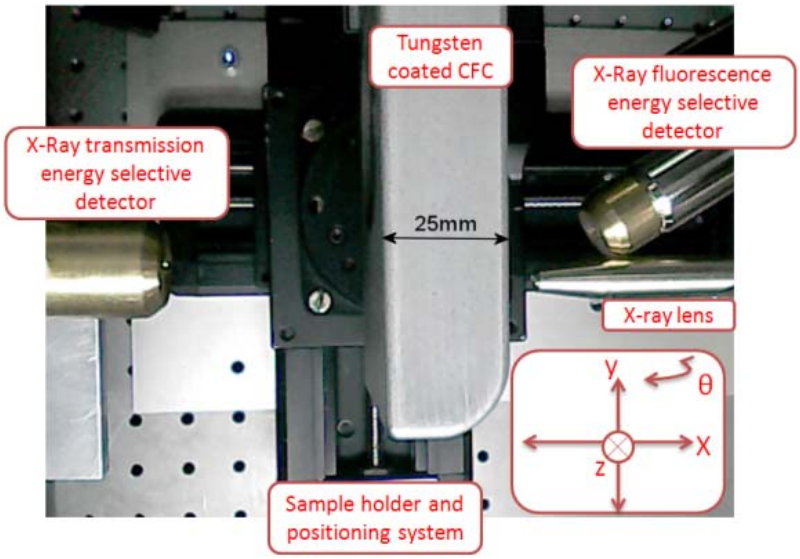
Tungsten coated CFC
X-Ray fluorescence energy selective detector
X-Ray transmission energy selective detector
25mm
X-ray lens
y
θ
X
z
Sample holder and positioning system

Figure 2

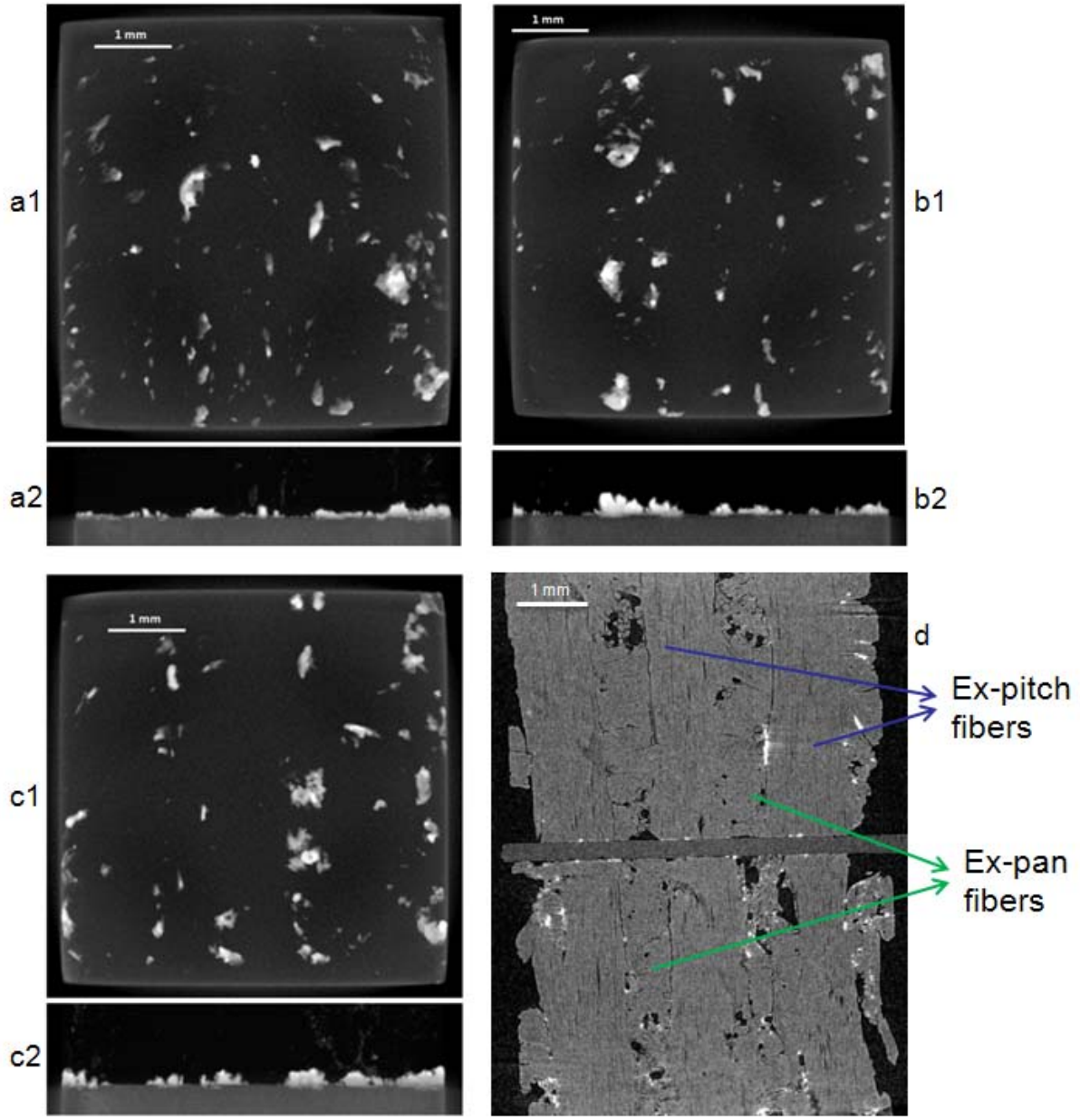

Figure 3

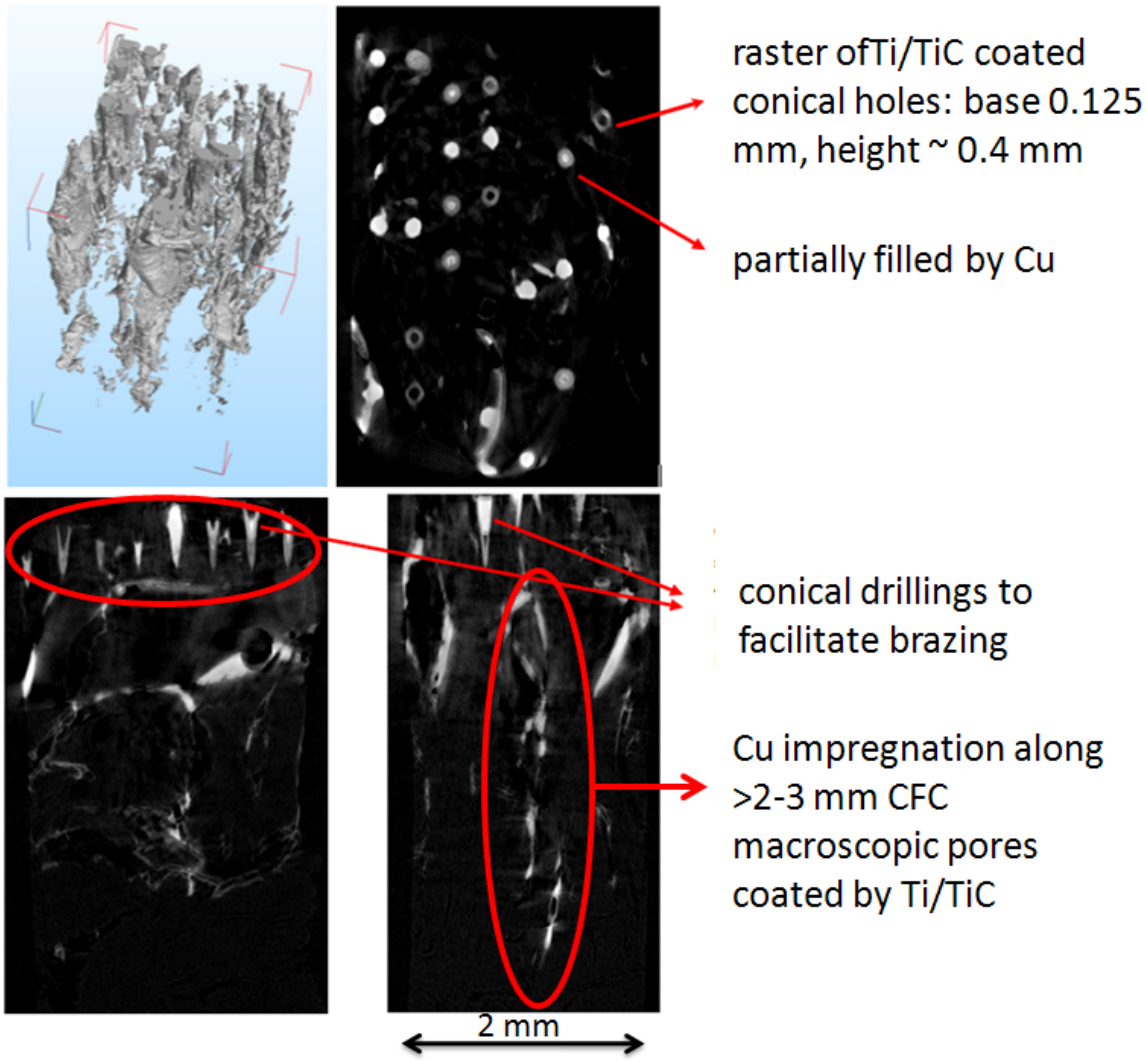
raster ofTi/TiC coated conical holes: base 0.125 mm, height ~ 0.4 mm
partially filled by Cu
conical drillings to facilitate brazing
Cu impregnation along >2-3 mm CFC macroscopic pores coated by Ti/TiC
2 mm

Figure 4

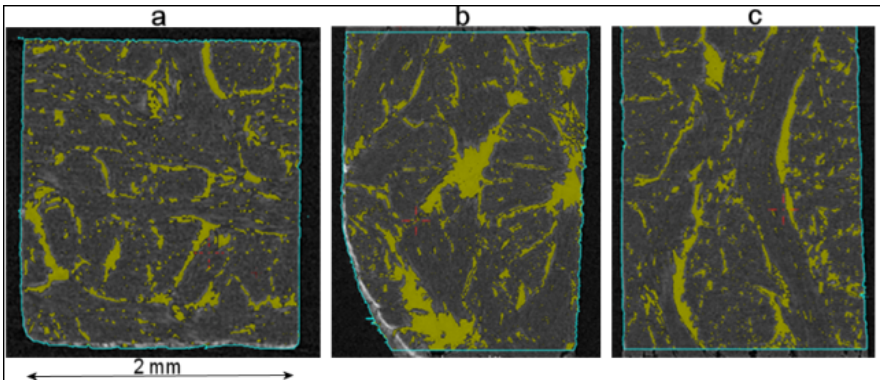

Figure 5

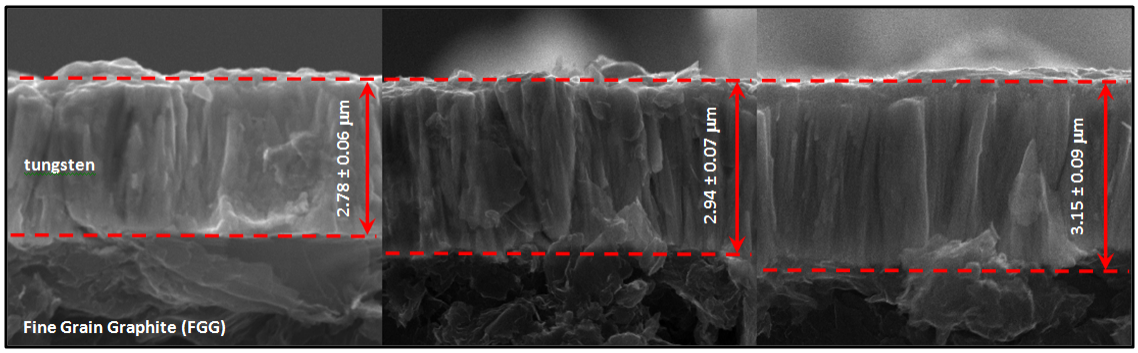
tungsten
2.78 ± 0.06 µm
2.94 ± 0.07 µm
3.15 ± 0.09 µm
Fine Grain Graphite (FGG)

Figure 6

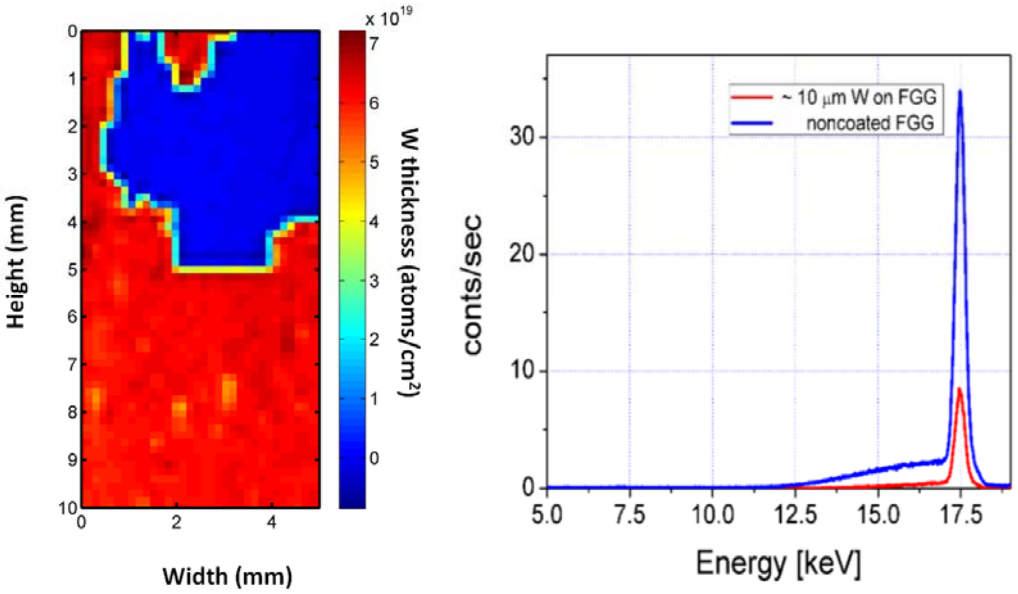
x 10^19
W thickness (atoms/cm^2)
Height (mm)
Width (mm)
~ 10 μm W on FGG
noncoated FGG
cnts/sec
Energy [keV]

Figure 7