X-RAY MICRO-TOMOGRAPHY STUDIES ON GRAPHITE AND CFC SAMPLES FOR POROSITY NETWORK CHARACTERIZATION
- WP09-PWI-04 (AS_6) -

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1. Introduction and objectives

The CFC monoblocks of the ITER divertor vertical target must sustain high heat fluxes of 10 MW/m^2during 400 s (normal operation) and up to 20MW/m^2 during 10 s (off-normal event). Carbon–carbon fibre composites (CFCs) have a unique combination of high conductivity, low Z and resistance to damage due to high heat loads. Given the demanding environmental requirements in the ITER divertor, specially developed CFCs are required as plasma facing components (PFC) materials.

The problem of fuel retention in carbon material is a major concern because tritium is radioactive, and the amount allowed within the ITER installation is an essential safety issue.

There are two main fuel retention mechanisms: i) carbon eroded from PFCs can be re-deposited in form of amorphous layers, containing significant fraction of fuel of up to H/C~1, ii) H isotopes from plasma can be implanted into carbon PFCs and diffuse into the material bulk [1,2].

From earlier investigations the retention in the material bulk was considered to be less critical than the retention in co-deposited layers. Recent gas balance experiments in Tore Supra [3,4] indicated, however, that significant fractions of fuel can be stored in CFC materials via this mechanism. It was attributed to a higher effective diffusion of H in the bulk of CFC due to a relatively high porosity of these materials of ≈10-15%.

Hence, the accurate 3D porosity description of the CFC materials would provide an essential input for the quantization of the fuel retention in material bulk. In this project we address the problem of quantitative characterization of the porosity structure of the carbon reinforced fiber (CFC) materials by high resolution X-ray tomography. This topic is included in the EFDA 2009 Work Programme, Task Agreement TA-1 “Fuel retention as a function of wall materials foreseen for ITER" organized under the Special Expert Working Group (SEWG) "Gas balance and fuel retention".

In 2009 the main **objectives** of this project were the following:

- Establish an working environment for high resolution X-ray tomography (μCT) on miniaturized graphite and CFC samples;
- Determination of microtomography scanning parameters for optimum space and density resolution;
- Quantitative evaluation of the sample porosity factor.

During the reporting period (January-December 2009) we have carried out the following research activities: i) determination of microtomography scanning parameters for optimum space and density resolution on miniaturized graphite and CFC samples ii) optimized tomography measurement of the 3D model of relevant volumes of CFC materials and iii) development and validation of an image processing technique for volume analysis in terms of absolute porosity factor.

2. Results and discussion

Most experiments were carried out at our newly upgraded X-ray tomography facility NanoCT (Fig. 1). In http://tomography.inflpr.ro one gives a more detailed NanoCT system description and its overall parameters. A limited series of experiments were performed on a high end tomography facility which uses an advanced transmission X-ray tube with a high power diamond target and a 16 bits flat panel detection system.

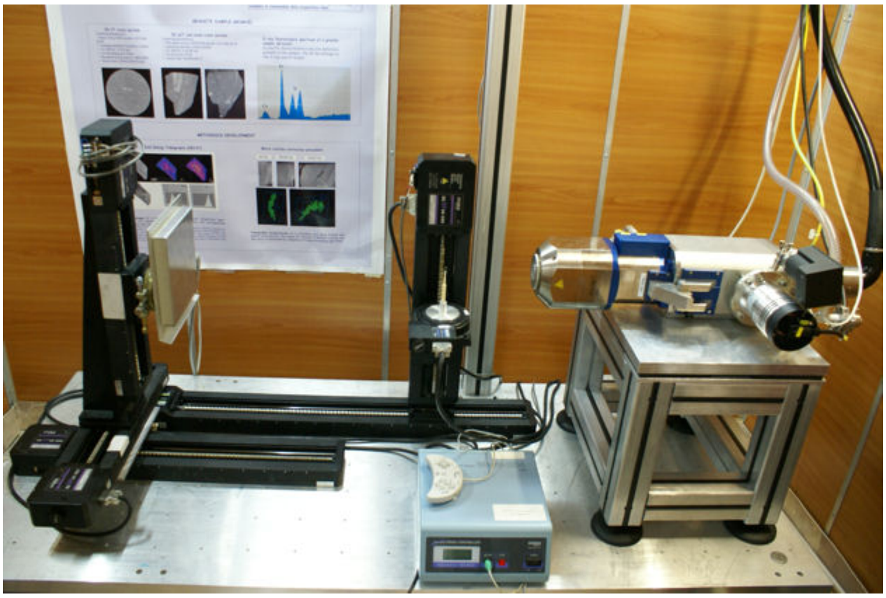


Figure 1 – View of NanoCT facility

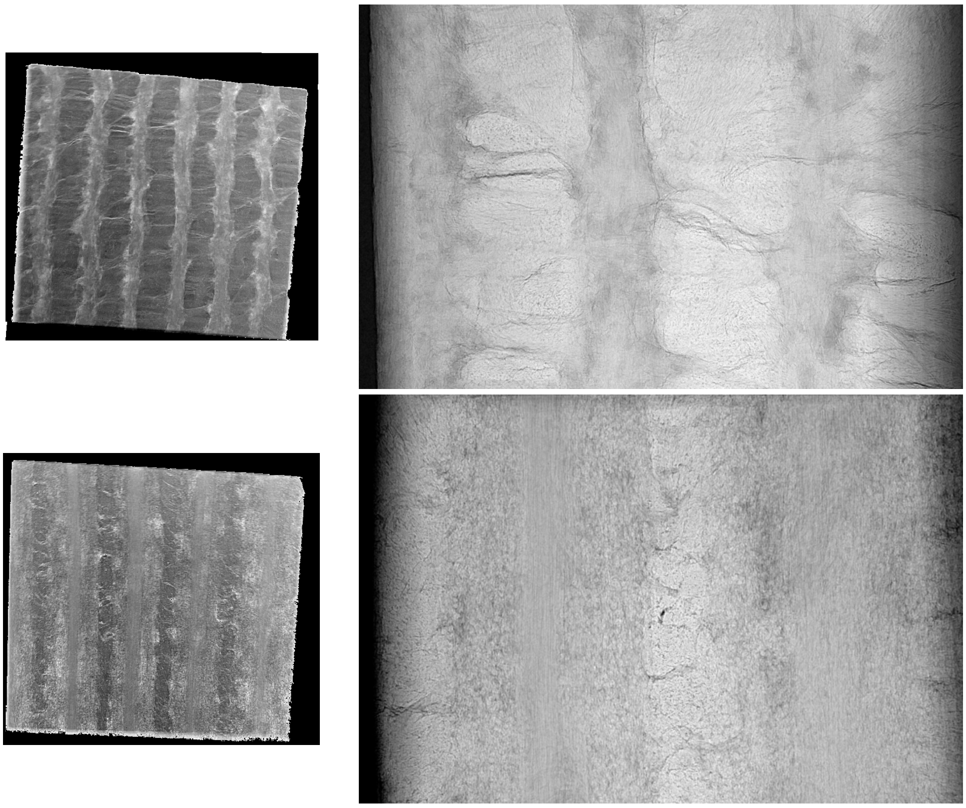
Tomography measurements were performed on relatively large volumes of two type of CFC samples (former ITER reference CFC NB31, JET CFC DMS780 and for reference on a porosity free fine-grain graphite EK98.

Moderate resolution measurements of CFC rods of 5x5x5 mm^3 at voxel resolution of approx. 14 µm with an estimated minimum detectable feature of cca. 15-20 µm have been reported in the semiannual report of January-June 2009. These measurements were devoted to the optimization of the X-ray source working parameters (high voltage and current) as well as the detection parameters (pitch size and integration time).

Comparative digital radiographies with space resolution of 14 µm and 2.5 µm are presented in Figure 2. Already from the radiographies one can note a clear difference in the signature of the two CFC materials.

CFC
NB31

CFC
DMS780



Sample size 10x10 mm^2 space resolution 14 µm

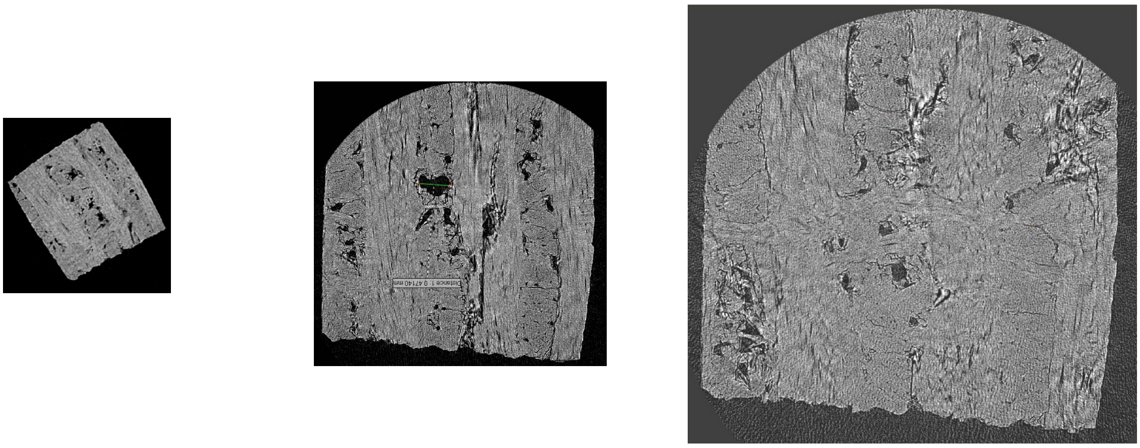
Sample size 5x5 mm^2 space resolution 2.5 µm

Figure 2: Comparative digital radiographies with space resolution of 14 µm and 2.5 µm

Once the optimum parameters have been determined one has proceeded to high resolution measurements (2.5 µm/voxel) on same CFC samples by the “offset tomography” technique [7].

A set of up to 1440 of radiographies at equidistant angles have been used for high resolution fully 3D tomography. Figure 3 shows representative tomographic images with 14, 5 and 2.5 µm per voxel of CFC of type Nb31. The images with 5 µm and 2.5 µm were obtained with the newly implemented "offset tomography" scanning method. A set of up to 1440 of radiographies at equidistant angles have been used for high resolution fully 3D tomography. Offset tomography is a powerful technique to almost double the magnification factor (accordingly doubling the space resolution) for a given detector size. Same high resolution (2.5 µm) tomography cross-sections for the CFC DMS780 sample are also measured and reported elsewhere.

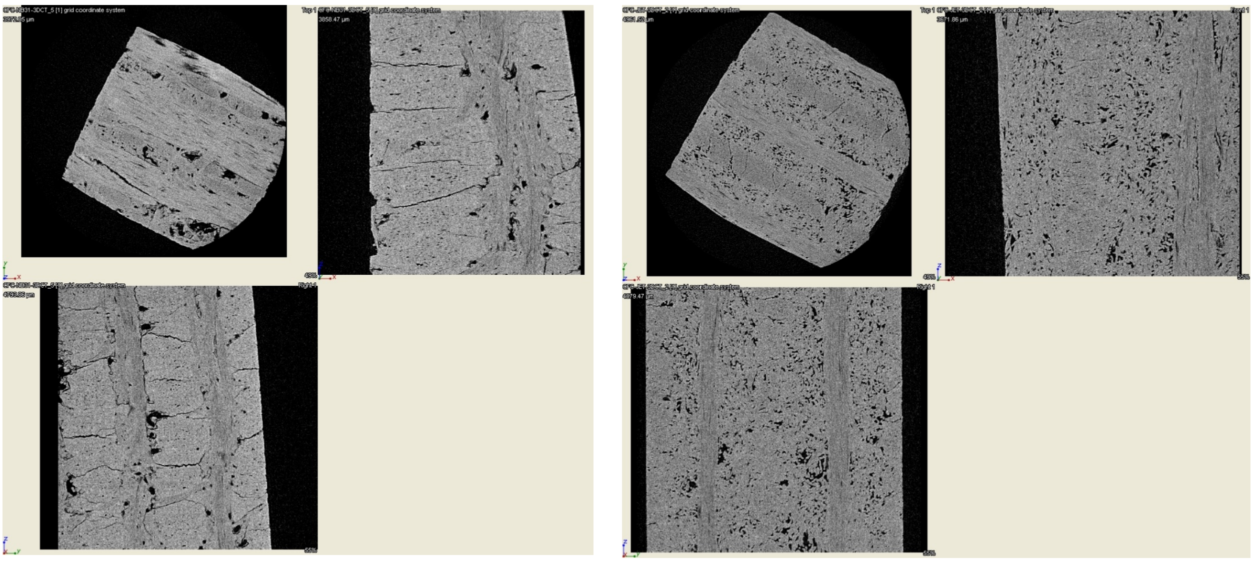
While the maximum resolution of 2.5 µm per voxel on a relatively large sample is a remarkable performance the images noise might be not sufficient for accurate porosity factor evaluation. In order to reduce the image noise we have carried out following steps: i) reduction of reconstruction artefacts like “ring artefacts” and “beam hardening artefacts” and ii) repeat the measurements on a high-end tomography facility which uses an advanced transmission X-ray tube with a high power diamond target and a 16 bits flat panel detection system. Both the high power target and the upgraded digital output of the detector are instrumental in the noise reduction on the tomographic images. The diamond target provides a significantly higher X-ray output and the 16 bits flat panel detector is able to display the X-ray transmission factors on a broader gray values scale.



14 µm 5 µm 2.5 µm

Figure 3 - CFC Nb31 - representative images of tomographic reconstructions with 14, 5 and 2.5 µm per voxel. The images with 5 µm and 2.5 µm were obtained with the newly implemented "offset tomography" scanning method.

Images shown in Figure 4 are representative for the results of high resolution tomography measurements on a CT facility with high power diamond target and 16 bits flat panel detector.



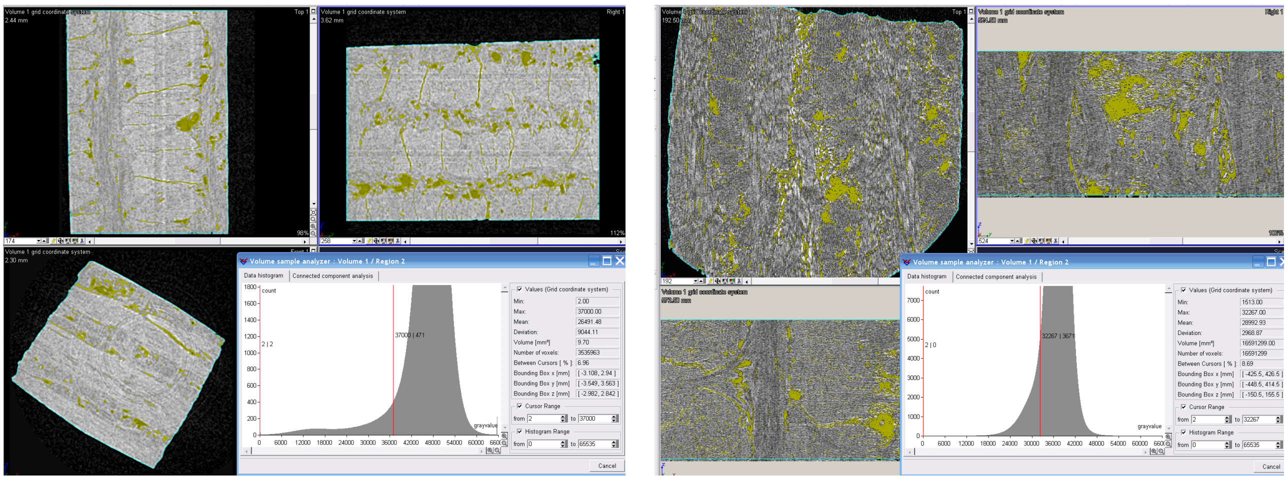
CFC Nb31 *CFC DMS780*

Figure 4 - CFC Nb31and CFC DMS780 high resolution tomography on a CT facility with high power diamond target and 16 bits flat panel detector. Sample size: ≥ 4x4x4 mm^3; voxel resolution: 6 µm.

Already from the visual inspection on can note the high quality of these images which a totally free of *ring* and *beam hardening* artefacts. The ring artefacts reduction was performed by a special scanning method in which the sample and/or the detector were randomly shifted. The beam hardening artifacts were mitigated by a linearization of the CFC transmission curve with the help of lookup tables obtained for calibration CFC materials.

Quantitative evaluation of the sample porosity factor

The 3D reconstructed volumes can be processed in order to determine the main CFC network porosity characteristics. For the quantitative analysis of the porosity structure in terms of total void fraction, network connectivity, wall thicknesses we used a powerful 3-D visualization and measurement software VG Studio MAX of Volume Graphics GmbH, Germany. In Figure 5 we introduce some of the data post-processing steps. The first one is to find an optimal choice for the threshold level, in order to create a correct border between CFC and porous regions. A detailed inspection of this demarcation is performed while navigating through the reconstructed volume along transversal, longitudinal and sagital cross-sections. After validation, the reconstructed volume is segmented and the porous structure is extracted as an independent object which can be represented also as a 3D structure. With the volume analysis module we determined the absolute value of porosity factor. The defect analysis tool can be used to determine the voids volume/size/projected area distribution.

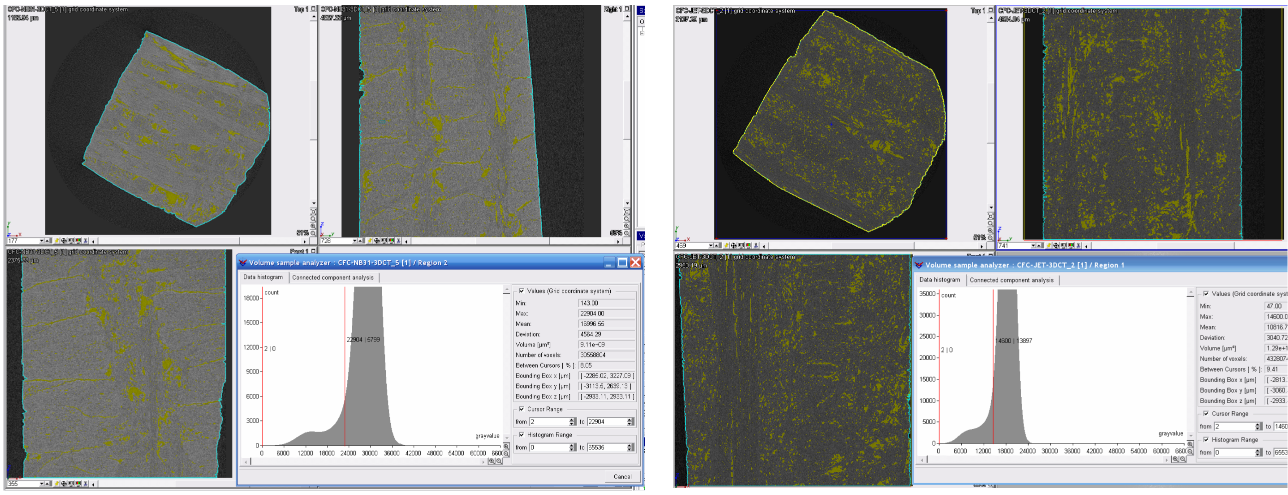


14 µm/voxel; porosity factor 7.0% 5 µm/voxel; porosity factor 8.7%

Figure 5: Post-processing steps of the reconstructed volume data in order to obtain the CFC NB31 porosity factor: the differentiation between CFC materials and porous regions is determined by the threshold levels.

From Figure 5 -left panel- one can note that the moderate resolution of 14 µm/voxel coupled with the relatively intense "ring artefacts" could be the possible cause of the underestimation of the porosity factor. The much higher resolution achieved in the reconstruction images depicted in the right panel could better provide the absolute porosity factor.

Finally, the porosity factors calculation procedures for the two types of fusion technology relevant CFC samples are illustrated in Figure 6. As input data we used the reconstructed volumes delivered by the high end tomography facility. The porosity factors values are in good agreement with the manufacturer nominal specifications.



CFC NB31: 6 µm/voxel; porosity factor 8.05% CFC DMS780: 6 µm/voxel; porosity factor 9.41%

Figure 6: Post-processing steps of the reconstructed volume data in order to obtain the CFC NB31and DMS780 porosity factors. The input data were obtained at a high end CT facility with diamond target and 16 bits flat panel detection system.

The radical improvement of the images quality has convinced us to purchase and install a diamond high power target on the NanoCT facility from INFLPR.

3. Conclusion and future work

High resolution cone beam tomography has been optimized for CFC samples. One important result was establishing the dependence of the porosity evaluation accuracy on the space resolution and the amount of tomography images noise.

A procedure for the quantitative evaluation of the sample porosity factor has been introduced and tested. For example for CFC NB31 we obtained porosity factors of around 8% and for CFC DMS780 of around 9.4%, in very good agreement with the manufacturer nominal values.

We believe that the main goal of the project was reached and that we are fully equipped to participate at the Deuterium Inventory in Tore Supra (DITS) post mortem analysis by providing high resolution tomography measurements on CFC samples. Our current 3D micro-tomography reconstructions for relatively large samples of CFC (NB31 and DMS780) could be considered a good basis for the characterization of the initial porosity of the new CFC ITER reference material NB41.
Preliminary results on the "X-ray micro-tomography studies on graphite and CFC for porosity network characterization" were presented at the Annual meeting of Special Expert Working Groups (SEWG) on «Fuel retention » organized at CEA-Cadarache. Recently, our results were also presented at The Annual General Meeting for the EU PWI Task Force which took place in Warsaw 4-6 November 2009.

4. Acknowledgement

The research activities to be conducted in this project are relevant for the EFDA 2009 Work Programme, Task Agreement TA-1 “Fuel retention as a function of wall materials foreseen for ITER" organized under the Special Expert Working Group (SEWG) "Gas balance and fuel retention".

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