	Experiment title: Micro-Tomography of Carbon/Carbon Composites	**Experiment number**: ME-148
Beamline: ID22	**Date of experiment**: from: 20-Sept-2000 to: 24-Sept-2000	**Date of report**: 14-Feb-2001
Shifts: 12	**Local contact(s)**: Christoph Rau	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Oskar Paris**

Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, and Metal Physics Institute, University of Leoben, Jahnstrasse 12, A-8700 Leoben, Austria

Herwig Peterlik and Dieter Loidl**

Institute of Materials Physics, University of Vienna, Boltzmanng, 5, A-1090 Vienna, Austria

*Christoph Rau**

European Synchrotron Radiation Facility, B.P. 220, F-38043 Grenoble Cedex 9, France

Report:

Carbon/Carbon composites (carbon fibers in carbon matrix, C/C) are the most promising material for aerospace applications due to their high specific strength (strength to density ratio), which is maintained up to temperatures more than 2000°C. For the extraordinary mechanical properties of the composite, fiber and matrix as well as their interface are responsible [1-3]. The interface properties can be adjusted by a different final heat treatment temperature (HTT), the so-called graphitization, which additionally leads to cracks in the matrix due to the different thermal expansion coefficients of matrix and fibers. The aim of the present experiment was to use X-ray micro-tomography with high spatial resolution [4,5] to determine for the first time the three-dimensional network of cracks in C/C for different HTT and for different pre-deformations.

Samples of bi-directionally woven C/C with three different HTT (1800°C, 2100°C and 2400°C) were deformed by three-point bending tests. This allowed to introduce tensile as well as compression stresses up to 80 MPa into the composite material parallel to the weaving planes. From the deformed samples, rod shaped specimens where machined perpendicular to the weaving planes for micro-tomography experiments at ID22 (specimen length: 10 mm, cross-sectional edge-length: ≈ 0.4 mm). Thus, these specimens contained a gradient of deformation along the long axis from tensile to compressive stresses with a neutral zone in the center. Due to restricted time, only three “volumes” per specimen (≈ 1 mm x 0.4 mm x 0.4 mm), i.e. the regions of maximum tension, neutral zone and maximum compression, were investigated with micro-tomography. Additionally, specimens where also prepared from undeformed material and also studied for comparison. In total 12 tomograms (3 temperatures times 4 mechanical states) were recorded.

The specimens, mounted on a goniometer-head, were illuminated with monochromatic radiation (E=18keV) with a sample to detector distance of d=10mm. These parameters were determined by imaging a thin section

(<50 μm) of C/C under different conditions, giving optimum contrast not only for the imaging of cracks, but also for the single carbon fibers within the carbon matrix due to phase contrast (see Fig.1). For each tomography, 1250 projections of the specimens were taken with rotation angles from 0° to 180°, with measurement times ranging from 1 to 5 seconds per single projection. A LAG:Eu scintilator with a point spread function of 1μm coupled to a Frelon-CCD (a pixel resolution of 1.4 μm was chosen for the tomography experiments) was used for the imaging of the projections. Three-dimensional (3D) reconstruction was performed using a filtered backprojection algorithm [4,5].

Fig. 2 shows a 3D view of a specimen (HTT=2100°C, tension part) with the weaving plane lying in the x-y plane, i.e., the fiber bundles are alternately oriented along the x and the y direction. Three types of intra-bundle cracks are clearly visible: 1) large cracks in the y-z plane, running through the whole bundle, 2) small cracks in the y-z and the x-z plane, not extending over the whole bundle, and 3) small cracks in the x-y plane. It is, however, not yet clear if type 3) are cracks originating from the HTT / mechanical load or elongated voids arising from the production process of the composite.

A first qualitative comparison between the different specimens yielded differences in crack density and –distribution, in particular concerning the different heat treatments (HTT). We are currently working on a quantitative (semi-automated) determination of crack-types, -density and length-distributions. The results are to be compared with model predictions on the effects of cracks on the elastic and mechanical behavior of the Carbon/Carbon composites [6,7].

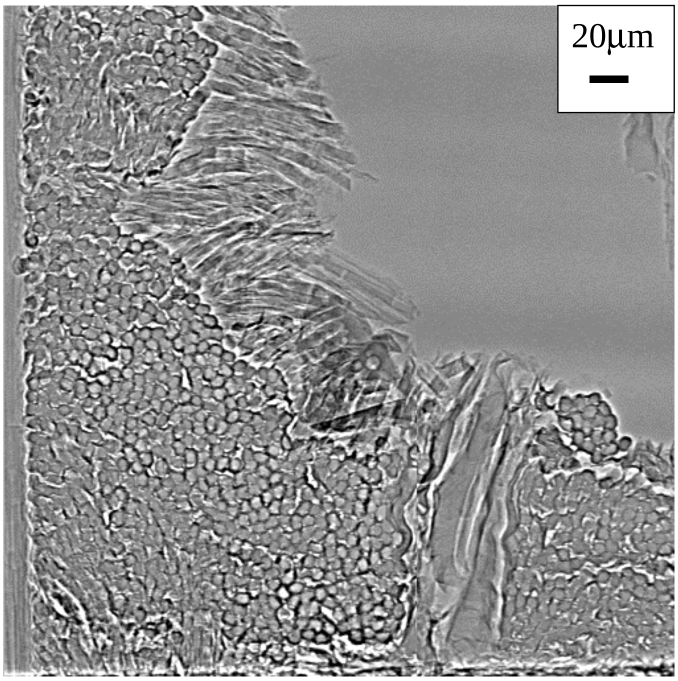

Fig.1: X-ray image of a thin section of C/C at E=18keV and d=10 mm. The pixel size was 0.7mm. Due to the contrast enhancement using phase-contrast, the cross-sections of the single carbon fibers (≈7 μm diameter) within the carbon matrix are clearly visible

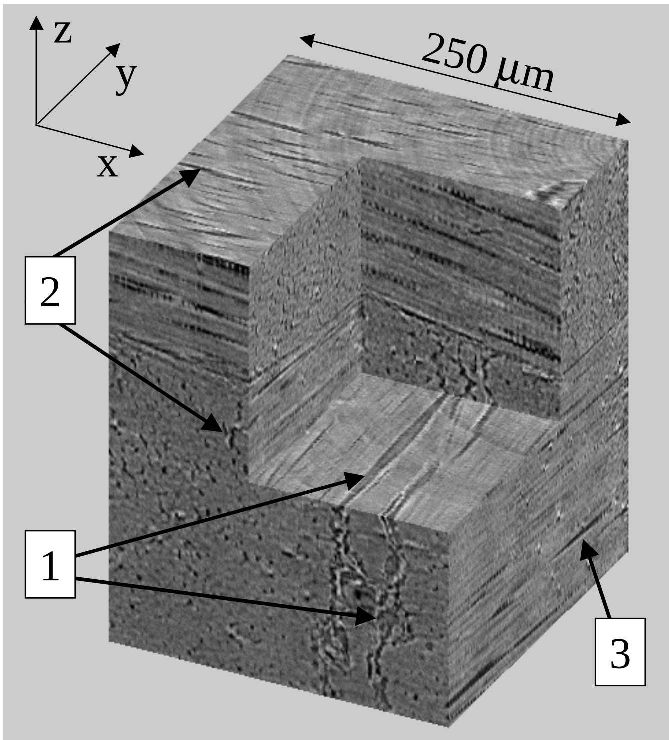

Fig. 2: Reconstructed 3D- view of a specimen of C/C. The fiber bundles run alternately in x-direction (upper part) and in y-direction (lower part). The numbers 1) – 3) indicate different types of cracks in the composite (see text).

References

[1] E. Fitzer and L.M.Manocha, *Carbon Reinforcements and carbon/carbon composites* Springer, Berlin (1998).

[2] A.Domnanovich, H.Peterlik,A.Wanner, and K.Kromp, *Comp.Sci.Techn*. **53** (1995) 7.

[3] A.Domnanovich, H.Peterlik, and K.Kromp, *Comp.Sci.Techn*. **56** (1996) 1017.

[4] J.Y.Buffière, E.Maire, P.Cloetens, G.Lormand and R.Fougères, *Acta mater*. **47** (1999) 1613.

[5] P.Spanne, C.Raven, I.Snigireva and A.Snigirev, *Phys.Med.Biol*. **44** (1999) 741.

[6] J.W.Lee and I.M.Daniel, *J. Compos. Mater*. **24** (1990) 1225.

[7] A.C.Kimber, *J. Mater. Sci. Lett.* 11 (1992) 1700.