ESRF'	Experiment title: High resolution X-ray tomography in the study of damages inside microheterogeneous materials	Experiment number HS 255:
Beamline: ID19	**Date of experiment:** 16-20 april 1997 (8 shifts) ; 22-24 april 1997 (4 shifts)	**Date of report:** **February 98**
Shifts: 12	Local contact(s): Jose BARUCHEL	*Received at ESRF:*

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Report:

Possibilities afforded, at ESRF, by **phase contrast** methods for imaging microstructural features and damage initiation inside composite materials has already been demonstrated (cf. Reports on experiments HS 538 and HS 69). In this report, we discribe new results on the quantitative study of damage in metal matrix composites, obtained by the same method.

Experimental methods

As was the case during the earlier experiment, the white beam of beamline ID19 is monochromated at 23keV. A detector, based on the FRELON camera developed by the ESRF Detector Group allows to obtain a 6.65 µm pixel size. The fluorescent screen of this detector is set at a distance of 82 cm of the sample, in such a way to clearly detect the diffraction features due to Fresnel diffraction and corresponding to sharp phase variations of the transmitted x-ray beam. The tomographic reconstruction was performed using a 3D extension of the conventionnal 2D filtered backprojection algorithm. We demonstrated that the result of the reconstruction is the sum of a plain **absorption** image and of a **phase contrast** image, which is sensitive to the second derivative of the real part of the refractive index of the material [Cloetens 19971.

A model metal matrix composite made of a 6061 aluminium alloy reinforced by Silicon Carbide (SiC) particles has been studied. This material has been elaborated by rheocasting under air and extruded at 530 °C.The average size of the reinforcing particles was 120µm, their volume fraction was 12%. Small flat tensile samples were spark cut from the extruded bars and submitted to a standard T4 or T6 heat treatment after mechanical polishing of their main faces.

Monotonic tensile tests were performed using a special in-situ tensile testing device which frame was made out of a transparent polymer (PMMA). Thanks to this tube, a 180 degree rotation of the sample under load was possible, without cutting the x-ray beam. The PMMA gave negligible absorption on the 2 D recorded images. All the mechanical tests were performed at room temperature using a constant crosshead displacement rate of 150 $\mu m.mn^{-1}$. During the tests, the load and the crosshead displacements are recorded on a computer. Five tomographic scans were performed on the same sample at different strain levels including the initial undeformed state. During the tomographic scan, about 2 hours for 600 2D projections, the crosshead position was maintained constant resulting in a slight drop of the applied stress.

Results.

Examination of the interior of the T4 specimen revealed that the extrusion processs has induced some decohesions between the matrix and the SIC particles as can be seen on Figure 1. Those decohesions were hardly visible on the surface of the samples because the polishing process had filled most of the voids with the ductile matrix.

The chronology of damage mechanisms occurring during the plastic deformation of the sample can be described as follows:

1 Mode I rupture of the SiC particles
2 Particle matrix new decohesions
3 Propagation of extrusion-induced decohesions.

From a qualitative point of view, our results show that the damage mechanisms in the bulk do not differ from those observed at the surface. Quantitatively, however, there is a difference between surface and bulk observations. Indeed, **the number of broken SiC particles, appears to be larger in bulk than at the surface.** It is worth emphasising that it the first time that such results are obtained using a non destructive method. They shows that quantitative surface observations of damage during in situ experiments must be taken with great care.

Those experiments are very promising because they provide a unique information on the microstructure and deformation processes in the bulk of micro-heterogeneous materials in a non destructive way.

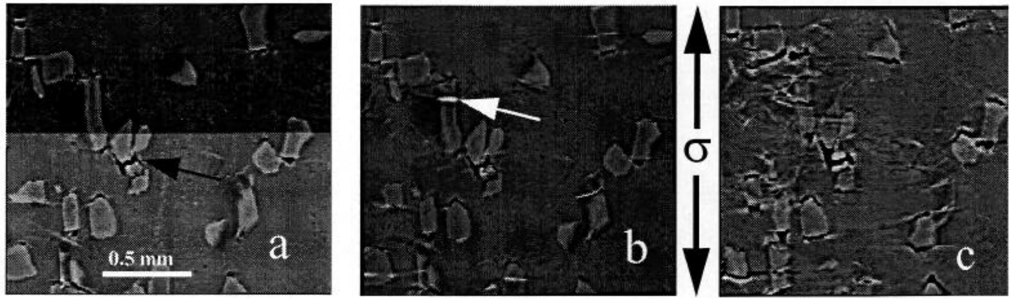

Figure 1 Reconstructed images of the same zone in the interior of a T4 Al/SiC sample at different strain levels **(a:** undeformed state, **b:** 0.5 % plastic strain, and c: 6 % plastic strain). Some particle/matrix decohesions induced by the extrusion process can be seen on the initial state (black arrow). The first cracks in the SIC particles appears in white (white arrows). All the observations have been done under load.

Publications on the subject by the applicants

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[2] CLOETENS P.et al, Journées de la matihe condensée, ORLEANS, (France), August 1996.
[3] CLOETENS P.et al., J. Appl. Phys, 81 (9) 1997, 5878.
[4] BUFFIERE J.Y.et al., ICSMA 11, PRAGUE& (Tch), Aug. 1997.
[5] BARUCHEL J. et al., SPIE's Int. Symposium, SAN DIEGO (California), 27 July-l August 1997.
[6] PEIX G. et al., SPIE's International Symposium, (cf. supra).
[7] BUFFIERE J.Y. et al., 17th Europ. Crystallographic Meeting; LISBONNE (Portugal), Augt 1997
[S] PEIX G. Et al.,Congrès COFREND sur les essais non-destructifs, NANTES (France), Sept. 1997.