ESRF	Experiment title: High resolution X-ray tomography in the study of damages inside microheterogeneous materials	Experiment number HS 69:
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Shifts: 6	Local contact(s): José BARUCHEL	*Received at ESRF:* 24.2.97

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Report:

From the materials science point of view, one of the most fundamental properties of high strength structural materials is their durability. As lifetime predictions require a sound understanding of the microscopic damage mechanisms leading to failure, there is an obvious need for an experimental method such as high resolution computed tomography (HRCT), which provides three-dimensional (3D) images and quantitative information on damage occuring in materials during strain/stress application. In the present work, the potential of phase contrast methods for imaging microstructural features in materials has been demonstrated. Strain-induced cracks with openings below the micrometer range are visible through the phase modulation they introduce. 3D images of damage resulting from monotonic tensile tests have been obtained on two metal matrix composites consisting of a 6061 aluminium alloy reinforced by :

1) a unique silicon carbide (SiC) fiber, 140 μm in diameter, with a 30 μm diameter graphite core.

2) a 10 % volume fraction of SiC particles with a mean size of 120 μm. For this Particle Reinforced Composite (PRC), the initiation and evolution of damage were monitored on the same sample at three different strain levels and compared to an initial undeformed state. For each tomograph, fhe sample was removed from the tensile testing device and set up on the beam line. Therefore, all damage observations were performed in the unloaded state.

Experimental methods

The white beam emitted on beamline ID19 was monocbromated by a practically perfect silicon single crystal, at an energy of 23 keV. A detector based on the FRELON (Fast REadout LOw Noise) CCD camera developed by the ESRF Detector Group and on a 10 μm fluorescent screen was used. Due to optical magnification, an effective pixel size of 6.65 μm was obtained. The phase sensitive HRCT was performed wifh a sample-detector distance of 0.82 m. For each sample, 600 images of 512 x 512 pixels were acquired over 180°. 3D images of 256x256x512 isotropic voxels were reconstructed. The extreme simplicity of this instrumental setup is to be stressed. The possibility of merely using free-space propagation, i.e. Fresnel diffraction, is associated with the very high lateral coherence of the photon beam.

The tomographic reconstruction was performed using a 3D extension of the conventional 2D filtered backprojection algorithm. While this is a temporary and obviously unsatisfactory approach, pending the development of efficient detectors and codes for phase mapping (“holographic reconstruction”), it works very effectively for detecting the edge contrast associated with phase singularities.

Results

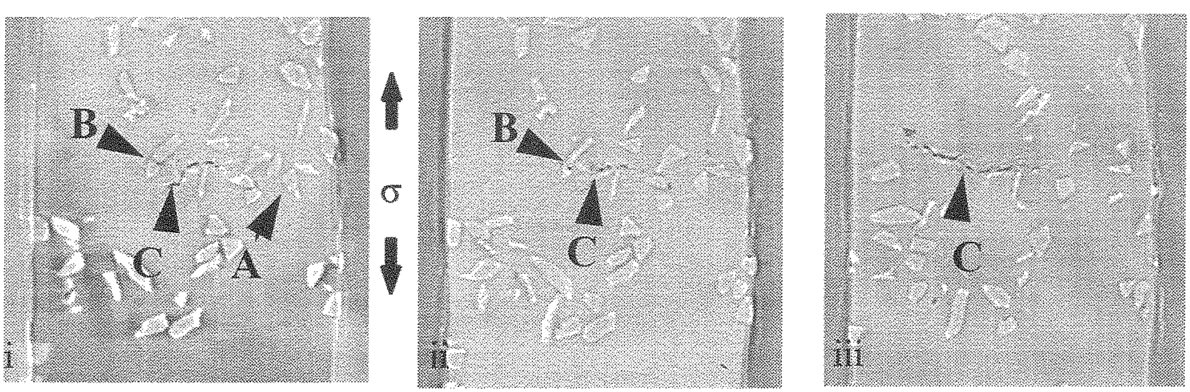
In the case of the fiber reinforced composite (FRC) three cracks were detected in the SiC fiber, as reported in the previous report. A comparison with post-mortem observation using a scanning electron microscope (SEM) reveals a perfect correlation between both images, but it is important to notice that some SIC fragments, observed in the tomographic slices, had disappeared during the specimen grinding for SEM examination, which shows that specimen preparation can cover up some characteristic features of the rupture process and underlines the unique advantage of HRCT.

In the case of the PRC, Figure 1 shows several slices of the reconstructed 3D volume of the studied sample, after a plastic deformation of 1 %. The SiC particles are clealy identified and cracks within particles (detail A and B) or within matrix (detail C) are detected by the microtomographic method. The tomographic reconstruction of the sample surface (Figure li), is very similar to the corresponding SEM micrograph (Figure liv). Thanks to the fringe structure of phase contrast images, cracks with an opening as small as 0.5 µm, much smaller than the experimental pixel size of 6.6 µm, can be detected (see for example detail A on Figure li and the corresponding detail on Figure liv).

Discussion

Concerning the FRC, no obvious correlation was found between the presence of voids inside the matrix and the cracks inside the fiber. Tomography revealed that monotonic tensile testing leads to multiple fracture of the fiber, as already reported for the same kind of material, strained under similar conditions. The numerous cracks observed in the fiber, giving SiC fragments, are thought to be due to constructive interferences of shock waves corresponding to the sudden release of elastic energy in the fiber when it breaks.

Concerning the PRC, two main damage mechanisms are observed when the plastic regime is reached: i) the cracking of the matrix on brittle oxides resulting from the processing of the material; ii) the breaking of SiC particles. Quantitative analysis of the reconstructed 3D images of the sample revealed that the aspect ratio of the broken particles along the tensile direction is high and that the damage accumulation rate is different at the surface and in the bulk of the material. As a matter of fact, HRCT reveals that a characterisation of damage restricted to the surface underestimates the number of broken particles by a factor 4.



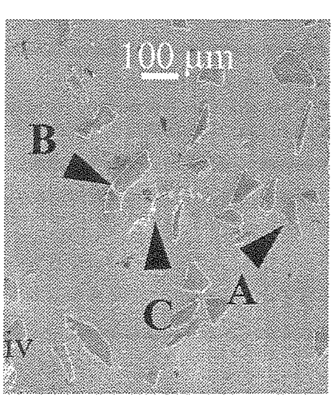


Figure 1 Observation of damage in an Al SIC composite after monotonic tensile testing at room temperature (1% plastic deformation). i) Reconstructed image of the sample surface. ii) and iii) Reconstructed images at 25µm and 75 µm below the surface, respectively. iv) SEM micrograph of the sample surface. **A** and **B** indicate some cracks in the SIC particles. C indicates a crack in the matrix.