ESRF	**Experiment title:** Damage assessment in micro-heterogeneous materials using high resolution X-ray tomography	**Experiment number:** HS-554
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Report

This report describes a first attempt to correlate damage nucleation observations during deformation (imaged by X-ray tomography) and internal stress built up (measured by X-ray diffraction) within a metal matrix composite (MMC). Besides, new high resolution 3D observations of internal features within industrial materials are also reported.

Experimental methods

The material used for the coupled tomographic and stress measurements experiments is a model Al/SiC composite with large ceramic reinforcements. Both measurements were performed simultaneously at different stages along the tensile curve of the sample. The complete procedure for the mechanical tests and the tomographic measurements have been described in a previous experimental report (HS 455)

1) In-situ measurements of internal stresses within the matrix have been performed through the recording of diffraction rings on high resolution X-ray films
2) The fracture of the reinforcing particles, is studied simultaneously using phase-contrast tomography. The tomographic scans are performed in-situ on a sample under load thanks to a specially designed tensile testing device set directly on the tomograph turntable. The tensile machine as well as the details of the procedure for the mechanical tests are described elsewhere (exp. report # 455).
3) A new fluorescent screen with a very high resolution, allowing 3D tomography with a voxel size of 2 μm^3, has been used.

Results

As a matter of fact, our attempt to perform in-situ stress measurements proved to be unsuccessful. It must be pointed out that for detecting an internal stress variation of 10 MPa in the material, one should be able to measure a variation of the diffraction ring diameter of around 20 μm on the films which are likely to suffer non homogeneous thermal distortions during their processing. Those thermal distortions have unfortunately occured in our case and have impeded a precise measurement of small changes in the shape (or diameter) of the diffraction rings which we wanted to measure. The present experimental arrangement could possibly be improved by the use of an internal reference sample, in powder form, to calibrate the diffraction rings on each film.

On the other hand, phase contrast tomography with a voxel size of 2 μm^3 delivered a considerable amount of excellent data. The materials investigated here were, for the first time, "real" industrial materials with an heterogeneity size in the 20- 40 μm range. A set of different kinds of materials was investigated :

- Engineering ceramics
- Glass reinforced polymers
- 7010 aluminium alloy
- Sintered steel

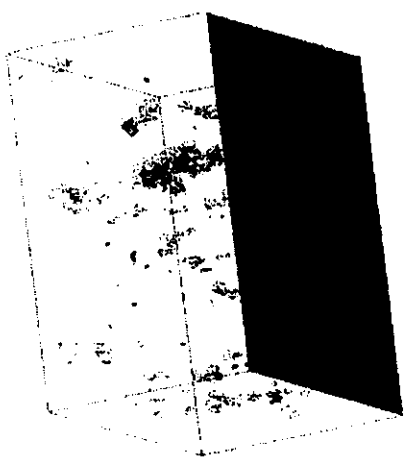

Figure 1 3D view of intermetallic phases in a 7010 aluminium alloy as observed by X ray Tomography with the 2 μm camera. The average size of the particles shown is around 30μm.

Moreover, a preliminary experiment on an Al/Cu model composite has shown that it was also possible to use the 2 μm camera for the observation of damage under load using an in situ tensile testing device. This device had been previously used at a lower resolution (6 μm) on a model composite reinforced with quite large particles (see exp. Report # HS455 and Newsletter n°30). The possibility to investigate damage nucleation and development with an improved resolution is very promising and offers vast possibilities in the field of industrial structural materials for which the average size of the reinforcing phase is quite small. Possible candidates for such studies could be the 7000 Al alloys for which intermetallic phases which are shown on figure 1 play a key role in damage development under stress.

Publications on the subject by the applicants see attached sheet

Publications

[1] BUFFIERE J.Y., VERDU C., PEIX G., CLOETENS P., PATEYRON M., BARUCHEL J., « Damage assessment in an Al/SiC metal/matrix composite during monotonic tensile tests using synchrotron x-ray high resolution microtomography, Proc.of ICSMA 11, Pragues Aug. 1997 Mat. Sc. Eng. 1997, A234-236.
[2] CLOETENS P., PATEYRON-SALOME M., BUFFIERE J.Y., PEIX G., BARUCHEL J., PEYRIN F., SCHLENKER M., « Observation of microstructure and damage in materials by phase sensitive radiography and tomography », J. Appl. Phys, 81 (9) 1997, 5878.
[3] BARUCHEL J. *et al.* "X-ray optics and imaging with hard coherent synchrotron radiation" SPIE's Int. Symposium, SAN DIEGO (California), 27 July-1 August 1997.
[4] PEIX G. *et al.* "Hard x-ray phase tomographic investigation of materials using Fresnel diffraction of synchrotron radiation", SPIE's International Symposium, (cf. supra).
[5] BUFFIERE J.Y. *et al.* "Principle and applications of phase contrast microtomogarphy using coherent synchrotron radiation" 17th Europ. Crystallographic Meeting; LISBONNE (Portugal), Augt 1997
[6] PEIX G. *et al.* Caractérisation de l'endommagement dans les matériaux de structure par tomographie haute résolution par rayons X"Congrès COFREND sur les essais non-destructifs, NANTES (France), Sept. 1997.
[7] BUFFIERE J-Y *et al.* "A new method for the 3D characterisation of microstructure and damage during in situ tensile tests using synchrotron X ray computed tomography. Application to Al based materials", Proc. of 6th Int. Conf. on Aluminium alloys Toyohashi July 1998 T.Sato et al. eds. p.529.
[8] SAVELLI S. *et al.* "Characterization by synchrotron X-ray microtomography of internal microstructural features and their detrimental effects with respect to the fatigue properties in an aluminium cast alloy", Proc. of 6th Int. Conf. on Aluminium alloys Toyohashi July 1998 T.Sato et al. eds. p.571.
[9] BUFFIERE J-Y *et al* "Damage assessment in a metal matrix composite during monotonic tensile tests using phase contrast microtomography" ESRF Newsletter April 1998 p.20.
[10] BUFFIERE J-Y *et al* Characterisation of internal damage in a particle metal matrix composite during monotonic tensile testing using X-ray synchrotron phase contrast microtomography Accepted for publication in *Acta Materialia.*
[11] CLOETENS P. *et al* Hard X-ray phase imaging using simple propagation of a coherent synchrotron radiation beam Journal of Physics D : Applied Physics 1999 *under press.*