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.

Jean-Yves Buffière*, Eric Maire*, Peter Cloetens**, JoséBaruchel** and Roger Fougères*.
*GEMPPM UMR CNRS 5510 INSA LYON 20 Av. A Einstein 69621 Villeurbanne Cedex France.
**ESRF BP 220 38043 Grenoble France

ABSTRACT

A Synchrotron X-ray source has been used to perform computed tomography on micro-heterogeneous materials. Thanks to the large specimen source distance (100 m), the X-ray beam exhibits a high lateral coherence which is used to obtain images of micro-heterogeneous materials containing phases with similar X-ray attenuation with an improved contrast This technique has been used for the characterisation of phase distribution in over-aged Al/Cu alloys and for the assessment damage initiation and development during in situ mechanical tests on an Al based composite.

Keywords: *X-Ray tomography, Damage 3D characterisation.*

1. INTRODUCTION

Micro heterogeneous materials form the vast majority of structural materials. Hence, much attention has been paid, in the scientific literature, to the mathematical modelling of their mechanical behaviour. For such modelling, the required experimental characterisation is twofold: first, one should be able to describe accurately the distribution of the phases in the material (volume fraction, size, shape...) and second, a good characterisation of damage occurring in the material during strain or stress application is needed. So far, the non destructive imaging of microstructural features or damages rely on surface observations. For example, the initiation of damage can be observed during in situ mechanical tests in a Scanning Electron Microscope (SEM)[1,2]. Such observations show that, in most cases, damage initiates either inside reinforcement material (which can be found in the form of fibres, particles or precipitates) or in the vicinity of the matrix (interface or interphase). This damage then propagates and leads, through a percolation process, to a major crack which induces the material failure.

From a theoretical point of view, damage initiation and evolution is only well described in the bulk. At the surface of a sample, prevails a complex stress state which is difficult to take into account for modelling. Thus, there is an obvious need for a non invasive technique providing three dimensional (3D) images of microstructural features or damage within materials. For the moment, X ray tomography is the only technique which can provide such images with a resolution in the micrometer range. This technique has been used, indeed, to characterise defects induced in materials by processing or by stress application (for a review see for example Reference [3]). In the present paper we show how synchrotron X-ray radiation can be used to improve the resolution of tomography for imaging microstructural features in Al-based materials. Preliminary results on damage induced by material processing and by in situ straining are presented.

2. EXPERIMENTAL METHODS.

2.1 Materials

Two kinds of Al based micro-heterogeneous materials have been investigated in this study: an aluminium/copper alloy and a metal matrix composite reinforced by Silicon Carbide (SiC) particles. For the Al/Cu alloy, the objective was to produce two kinds of spatial distribution of the intermetallic particles and to be able to discriminate the two microstructures by means of X ray tomography.

Hence, the Al/Cu materials were fabricated by the powder metallurgy route from granules of Al containing 5, 17 and 24 wt. % Cu. Given its initial copper concentration, each kind of granule exhibits a different fraction of Al_2Cu particles (approximately 0, 11 and 38 vol. % respectively) embedded in a same homogeneous matrix (Al + 5 wt. % of Cu in solid solution). Two kinds of samples were produced : a homogeneous sample with the 17% powder only, and a clustered sample with a mixture of 5 and 24% powder. The primary granules of powder used in this study were produced by the impulse atomisation process (IAP) [4]. This is a single fluid atomisation technique which generates atomised granules in a N_2 atmosphere. The granules had a large d 50 (around 500 µm) but a low geometric standard deviation (1.33). Bulk samples were obtained by pressing the granules at 500 °C under vacuum at 70 MPa during 2 hours in a graphite cylindrical die (diameter 26mm, length 26mm). The cylinders were extruded at 500°C with a 40:1 ratio. The diameter of the resulting bars being too small to cut a 10 mm wide sample, the bars were compressed at 530°C. They were finally annealed for 50 days at 530°C and quenched in water. The resulting size of the Al_2Cu particles was of the order of 20 µm in the homogeneous sample and 30µm in the particle rich grains of the heterogeneous sample.

The metal matrix composite has been obtained by mixing SiC particles with an average size of 120 µm with a 6061 alloy through a rheocasting route under air. The unusual large size of the particles was chosen with respect to the experimental resolution of the 3D images. After solidification, the material has been extruded at 530°C with a 16:1 ratio. Before testing, a T4 heat treatment (1h at 530°C + water quench +2 h at 175 °C) was performed on the samples.

2.2 Imaging technique

The tomography experiments were conducted at the European Synchrotron Radiation Facility (ESRF) in Grenoble on line ID19. The sample, set on a rotating stage, was placed in a monochromatic X-ray beam. Several radiographs corresponding to 2D projections were recorded on a 1024*1024 CCD detector for 600 different angular positions of the sample along a 180° rotation. A whole scan of the sample lasted approximately two hours. The energy of the beam was set at 23 keV giving a ratio of incident/transmitted intensity of about 15%.

In a classical X-ray tomography experiment, the contrast observed on the projections is based on the attenuation of X-rays by regions of the sample exhibiting different local absorption coefficients. In the present case, the distance between the X-ray source and the sample being large (around 120 m) the X-ray beam exhibited a high transversal coherence and gave an extra contribution to the contrast. This contribution, called phase contrast imaging [4], is due to constructive interferences between X-rays diffracted at the interface between phases exhibiting different refraction index (e.g. SiC/Al, Al/air, SiC/Air...). Those interferences were found to be detected more effectively when the detector/sample distance was large (about 80 cm for an X-ray energy of 23 keV). The voxel size in the reconstructed volume, determined by the experimental settings of the detector, was about

6.65*6.65*6.65 μm^3. A back-filtered reconstruction algorithm initially developed for standard attenuation tomography gave very good results for the reconstruction of the volumes of the samples.

2.3 Mechanical tests

A special mechanical testing device has been realised in order to record the 2D projections of the samples under load. This machine is set on the rotating stage and experienced the same rotation as the sample. Therefore, the central frame of the machine is made out of a PMMA tube which gives negligible absorption on the 2D projections along the 180° rotation. The device can be used in tension or in compression with loads up to 1500 N. The crosshead displacement and the load are recorded with a computer and monitored during the tests.

Tensile tests were performed at room temperature using a constant crosshead displacement rate corresponding to a strain rate of 5.10-4s^{-1}. Double shouldered specimens similar to those used during in situ mechanical tests in a SEM were used [1]. Their cross section was 1.5*1.5 mm^2 their gage length was 5 mm long and was imaged entirely on the CCD. Before testing the samples were mechanically grinded using SiC paper and diamond paste down to 1µm. A first scan of the sample was performed in the undeformed state with the sample in the tensile machine. The load was then increased and the 2D projections were recorded while maintaining the crosshead position constant. During the scan, a slight drop of the load was observed corresponding to a reduction of the stress on the sample of less than 10%. Four scans corresponding to different plastic strains levels were performed on each sample.

3.Results and discussion.

3.1. Characterisation of the spatial distribution of Al_2Cu nodules.

The different spatial distributions of the Al_2Cu nodules were clearly observed in the Al/Cu samples. This is illustrated on Figure 1. The alloy with granules containing 17% of copper showed an homogeneous distribution of nodules, while the alloy made with a mixture of granules containing 5 and 24 % of copper showed a clustered distribution of nodules.

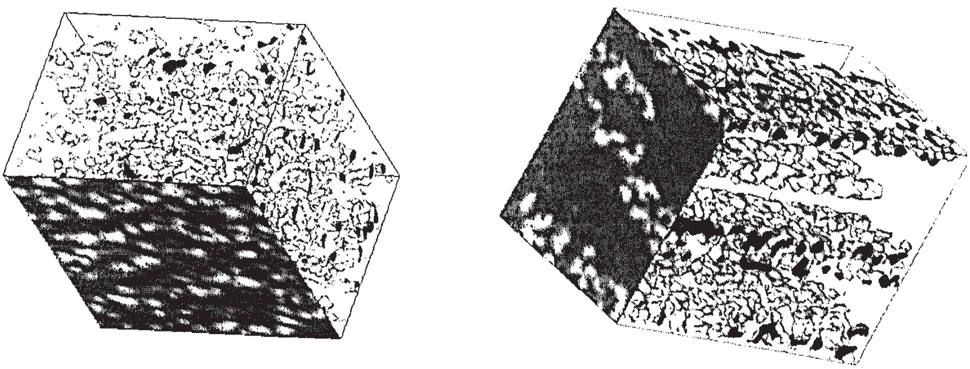


Figure 1. *3D reconstructed images of the interior of the Al/Cu alloys containing granules with 17 % Cu (left) and a mixture of granules containing 5 and 24 % Cu (right). One of the faces of the reconstructed volume is visualised in grey level while the volumes of Al_2Cu nodules are visualised by a simple threshold value on the grey levels.*

The difference in X-ray attenuation of Al and Al_2Cu being is rather large: 2.31 and 13.5 $cm^2.g^{-1}$ respectively. Hence the Al_2Cu nodules can be extracted for visualisation by simply thresholding the images. Automatic 3D image analysis is currently being carried out to extract the stereological parameters allowing a precise description of the two kinds of distribution which will be used for the modelling of their mechanical behaviour.

3.2. Characterisation of the Al-based composite

Three reconstructed images of the interior of the Al/SiC composite can be seen on Figure 2. For this material the difference in X-ray attenuation between Al and SiC is small: 2.31 and 2.25 $cm^2.g^{-1}$ respectively. Thanks to the phase contrast technique, however, the interface between the matrix and the reinforcing particles are underlined by a set of diffraction fringes (Figure 3) which greatly enhances the contrast and allows a very good detection of the particles [4]. The contrast given by a crack in a SiC particle is also greatly enhanced by the phase contrast technique. Comparisons between reconstructed images and SEM images of the surface of the sample have shown that cracks with an opening down to 0.5 μm could be detected [3].

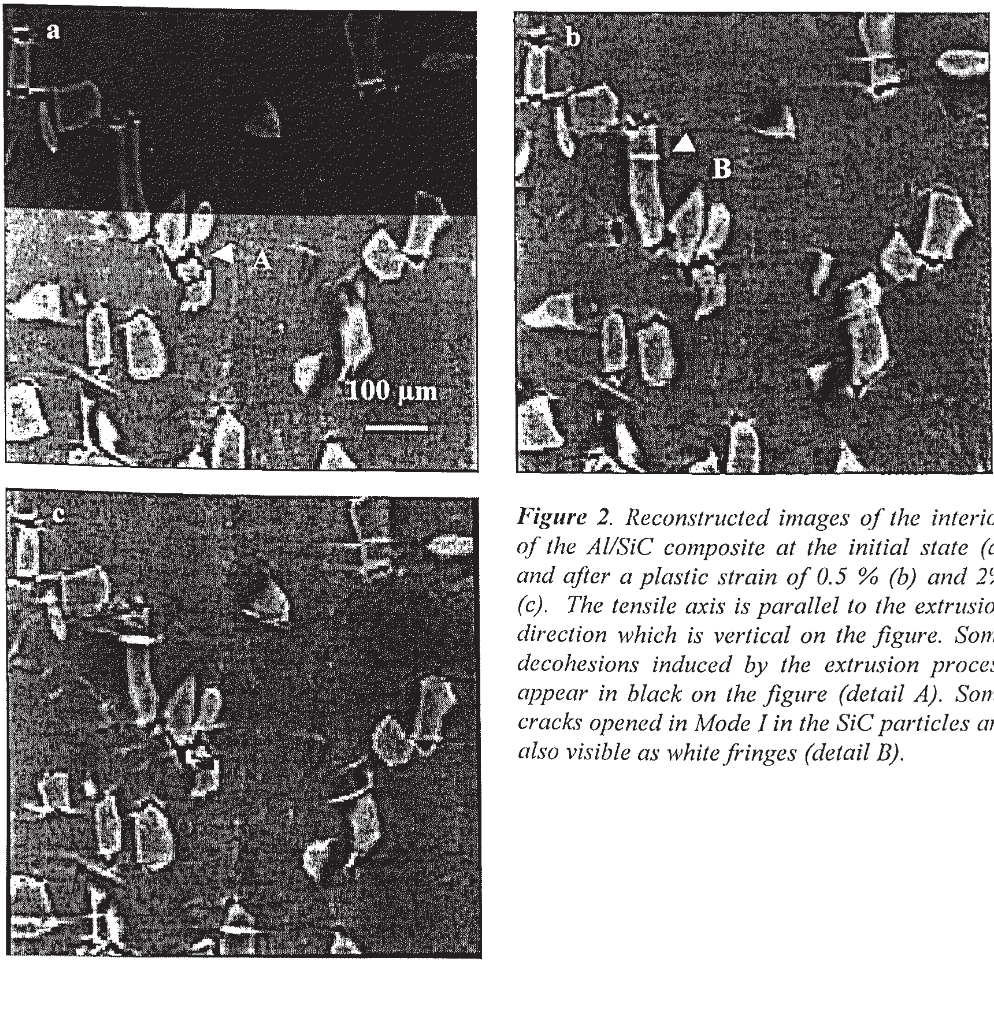
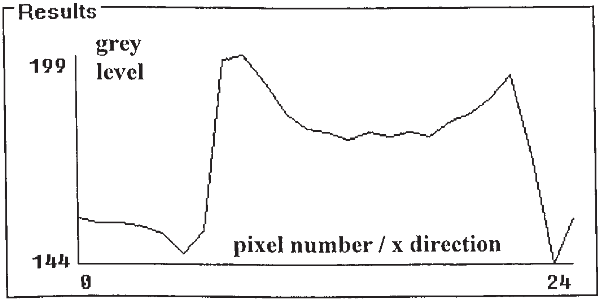


Figure 2. Reconstructed images of the interior of the Al/SiC composite at the initial state (a) and after a plastic strain of 0.5 % (b) and 2% (c). The tensile axis is parallel to the extrusion direction which is vertical on the figure. Some decohesions induced by the extrusion process appear in black on the figure (detail A). Some cracks opened in Mode I in the SiC particles are also visible as white fringes (detail B).

It can be seen from Figure 2 that the extrusion process has induced some internal decohesions between matrix and reinforcement. It is important to note hat those defects were hardly visible on the surface of the sample after inspection in a SEM. The polishing process had indeed filled the holes with ductile aluminium. The volume fraction of those decohesions has been measured by automatic image analysis and found equal to 0.1 %, An histogram of their size distribution, is shown on Figure 4.



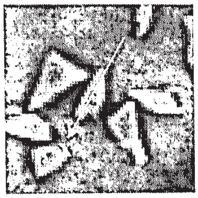


Figure 4 *Grey level profile obtained across a SiC particle on a reconstructed image of an Al/SiC composite. Thanks to the phase contrast technique, SiC/matrix interfaces are underlined by a set of bright and dark fringes which greatly favours the detection of the particles.*

A detailed analysis of the reconstructed volumes of the strained sample has been undertaken in order to study the damage mechanisms induced by the plastic deformation both at the surface and in the interior of the sample. As the plastic deformation increases, the following events are observed:

1 Rupture of the SiC particles in mode I
2 Creation of new interfacial decohesions between the SiC particles and the matrix
3 Propagation of the decohesions induced by the extrusion process.

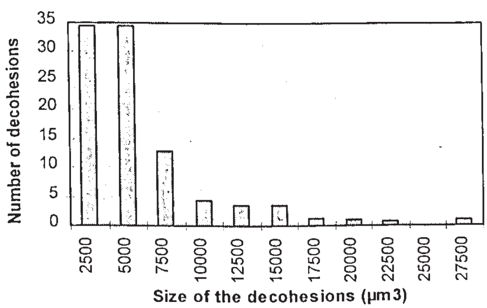


Figure 4 *Histogram of the size distibution of process induced decohesions measured in the bulk of the Al/SiC composite by image analysis of the reconstructed volumes.*

Qualitatively, those damage features do not differ from those observed at the surface of the same kind of micro heterogeneous material during mechanical tests in a SEM [1]. Quantitatively, however, the number of broken particles in the bulk of the sample appears to be larger than that measured at the surface of the sample. This is in agreement with previous results obtained on the same material prior to the extrusion process [6] The complex stress state which exists at the surface of the sample could be responsible for the observed differences in damage growth rate. Therefore, great care should be taken, when analysing in situ surface observations during mechanical tests on micro-heterogeneous materials as already suggested by other authors [7].

Finally, our observations tend to show that the interfacial decohesions induced by the extrusion process do not play a significant role in damage initiation as they tend to grow only at the later stages of the deformation process. It is likely that the load transfer is less efficient on a particle with a decohesion than on a particle with a perfect interface. Therefore, less elastic energy can be stored in a SiC exhibiting a decohesion which could lead to a delayed rupture of the particle. FEM calculation of the stress state within a hard intermetallic particle with an adjacent pore in an aluminium matrix are in agreement with this assumption [8].

4. Conclusion

Modelling the mechanical behaviour of micro-heterogeneous materials requires a sound characterisation of the phase distribution in the materials as well as of the initiation and development of damage during stress/strain application. This characterisation can be achieved through X-ray tomography which is, for the moment, the only non destructive technique able to provide 3D images of the interior of materials with a resolution in the micrometer range. The results presented in this paper show that the use of synchrotron radiation as an X-ray source for tomography greatly enhances the contrast of 3D images. Thanks to the high lateral coherence of the beam, an interface in the material (matrix/reinforcement, matrix/crack...) is underlined by a set of diffraction fringes. Thus a very good contrast is obtained on the reconstructed images of micro-heterogeneous materials showing phases with similar X-ray attenuation. This technique, called phase contrast imaging, also allows a very good detection of damage features like cracks.

Preliminary results on model materials have shown that X-ray tomography can be used first to quantify micro-structural features in Al/Cu alloys showing different distribution of the Al_2Cu nodules and, second, to assess damage development in Al-based composites during in situ tensile tests. For this material, the damage mechanisms observed in the bulk do not differ from those observed at the surface but their respective growth rate appears to be different.

REFERENCES

[1] E. Maire,C. Verdu, G. Lormand and R. Fougères Mat. Sc. Eng. A 196 (1995) 135-144.
[2] L. Manoharan and J.J.Lewandowski Scripta Met. 23 (1989) 1801.
[3] P. Cloetens, M. Pateyron, J-Y. Buffière, G. Peix, J. Baruchel, F. Peyrin, M. Schlenker, J.Appl. Phys. 81 (9) (1997) 5878.
[4] M. Morin, M. Rieder, J. Meja and H. Henein: Proceedings of the International Symposium on Light Metals 1996, M. Avedesian, R. Guilbault and D. Ksinsik, Eds., Met. Soc. of CIM, Montreal, 1996, 293-304.
[5] P. Cloetens, R. Barrett, J. Baruchel, J.P. Guigay M. Schlenker, , J.Appl. Phys. D 29 (9) (1996) 133.
[6] J-Y. Buffière, E. Maire, C. Verdu, P. Cloetens, M. Pateyron G. Peix, J. Baruchel, Mat. Sc. Eng. A 234-236 (1997) 633-635.
[7] P.M. Mummery B. Derby Journal of Materials Science 29 (1994) 5615-5624.
[8] M. Gharghouri, Research report on the study of fatigue of 7010Al alloys GEMPPM INSA Lyon, Dec.1996.

ACKNOWLEDGEMENTS

The authors are grateful to Luc Salvo, from GMP2 Grenoble, for processing the Al/SiC material.