ESRF	**Experiment title:** High resolution tomography on micro-heterogeneous materials coupled with internal stress measurement	**Experiment number:** HS 690
Beamline: ID 19	**Date of experiment:** from: 08/02/99 to: 11/02/99	**Date of report:** 28/07/99
Shifts:12	**Local contact(s):** P Cloetens	*Received at ESRF:* 05/08/99

Report

During this experiment several cast aluminum alloys and two different metal matrix composites have been investigated by high resolution X ray radiography tomography (resolution 6μm) and X-ray diffraction.

Results on the cast aluminium alloys.

A model cast aluminium alloy (AS7G03) with different pore contents has been studied as well as an industrial alloy. In a previous study, the AS7G03 alloy had been studied by X-ray tomography in order to characterise the 3D characteristics of the pores resulting both from "natural" solidification process and from gassing of the melt. This very valuable 3D data is currently used to describe the distribution of defects in the alloy and is used as an input for a physical model predicting the fatigue life of the material. However, detailed observations of fatigued samples after failure have revealed some crack initiations on very large pores which had not been detected in the previous experiment. The reason is that those pores being very large (typically around 500 μm) the probability of finding one in a small tomography sample (a cylinder of a few mm^3) is very small. Therefore the same alloy has been characterised again but this time using X-ray radiography. Although the radios only provide a 2D projection of the pores, the volume of material that can be investigated in a reasonable time is considerably larger than with tomography.
A series of 60 radios has been recorded on samples showing the same size and shape as the ones used for tomography. On top of the pores which had already been observed some very large pores were also detected on the radios. Three tomography of the samples containing the larger pores in the different model alloys have been performed . From the radio data an equivalent size of the pores has been measured. This new data fits well with the data obtained by tomography and has allowed the tail of the pore distribution function to be completed.
Besides, for the first time, an industrial cast alloy has been studied by radio and tomography. In this case, only rather large pores were observed (typical size around 200 μm). Again, the data obtained by tomography and radio fits well with the previous data of the model alloy. The analysis of the 3D data tend to show that the pore shape in the industrial alloy is similar to the gassing pores shape of the model alloy. This is probably due to the gas introduced in the melt to avoid micro-shrinkage during solidification in the industrial process route.

It is worth mentioning that, in the industrial alloy, only the very large pores (around 1mm in size) could be detected with high resolution x-ray films and a classical x-ray laboratory source. This illustrates, once more, the enormous improvement brought by the phase contrast for defect detection in such materials.

Results on MMCs

Three different materials have been investigated. The first one is composed of a pure aluminum matrix reinforced by 20% (volume fraction) of zirconia/silica particles. This model material has been elaborated in order to investigate the damage mechanisms under stress. Although the resolution of the tomography was a bit too low for the average size of the reinforcing particles (around 40 μm) the reconstructed images have shown that the reinforcement distribution can be considered as homogeneous and that the processing route has not induced large defects such as matrix reinforcement decohesions. Further investigation of this material loaded in situ will soon be carried out at a better resolution.

The second composite is a 6061 aluminum alloy reinforced by Silicon Carbide particles (SiC). which had been studied previously by tomography (exp rep HS 255). Here the aim of the experiment was to take advantage of the monochromatic x-ray beam on ID19 for recording diffraction rings of the material under load. Those rings, once analysed, give access to the average stress in the SiC particles and in the matrix. The rings are recorded on high resolution films. To avoid thermal distortion of the film during processing a retractable micro tube filled with a copper powder was installed in the tensile rig. This powder was used to calibrate the diffraction rings on the film. The only rings which were analysable were those of the aluminum matrix. The SiC particles were too big, and in a too small number in the beam to give diffraction rings. The evolution of the relative deformation of the Al cristallographic planes were then measured. This evolution is linear in the elastic regime. The stress increases then more rapidly due to a higher stress partitioning between fiber and matrix in the plastic regime.

The third composite is a titanium alloy (TA6V) reinforced by long SiC fibres. One sample of this material has been loaded at constant strain rate and room temperature on an in situ tensile testing device. A series of 18 radios were taken all along the tensile curve. The microstructure of the material is simple enough (only one layer of fibres) for a qualitative evaluation of internal damage on 2D projections. The radios show, first, that the studied sample has been damaged during its preparation (voids at the fibre ends) and that one fibre is broken. When the load is increased the fibres tend to crack perpendicular to the stress direction. No increased damage could be observed near the broken fibre for the investigated experimental conditions.

A similar sample has also been imaged at the initial state. For that sample, the evolution of the average stress in the matrix and in the reinforcements during a similar in situ mechanical deformation has been studied by diffraction. This has been done on beamline ID11 during a different experiment (collaboration with Prof. Withers Manchester Univ.). In that case the diffraction rings were recorded with a CCD camera and the size of the beam is small enough to investigate the stress very locally. Those results are very promising: by combining X-ray tomography/radiography and X-ray diffraction on the same sample (which will be moved from one line to the other) one should be able to obtain unique information on damage initiation/development and stress relaxation in this kind of material. The corresponding proposal has already been accepted at ESRF (HS 1047). The experiment will be carried out in January 2000.