ESRF	**Experiment title:** Connectivity in semi-solid alloys during thermal treatment or mechanical deformation using phase contrast tomography	**Experiment number:** HS555
Beamline: ID 19	**Date of experiment:** from: 1 0/04/98 to 14/04/98	**Date of report:** **28/07/98**
Shifts: 12	**Local contact(s):** LUDWIG Wolfgang, CLOETENS Peter, BARUCHEL Jose	*Received at ESRF:* 0 6 AOUT 1998

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Report:

High resolution tomography on ID 19 allows to obtain 3D images either using absorption or phase contrast. This technique was used to study the microstructure of aluminium alloys that are semi-solid during forming and especially the connectivity of the solid phase during thermal treatment and forming : 5 Al-Cu samples and 7 Al-Si samples were investigated during the 12 shifts.

Experimental methods :

The Al-Cu alloy is a binary alloy made of pure aluminium and copper (Al 10% in wt Cu). We investigate the microstructure of such an alloy after partial remelting at a temperature where the liquid volume fraction is about 0.2. Five different microstructures were produced by appropriate thermomechanical treatment before partial remelting in order to get different solid connectivities. Owing to the good contrast in the absorption mode, all experiments were made with the detector placed at lmm with respect to the sample.
The Al-Si alloy is a A356 (Al 7%wt Si 0.3%wt Mg, 0.l%wt Fe . ..). This material is especially designed for semi-solid forming. In order to study the microstructural evolution during thermal treatment, the duration of the partial remelting at 583°C was varied before quenching (5,15,30 minutes). In order to study the microstructural evolution during mechanical testing, 3 samples have been deformed under compression at 583°C at low speed, medium speed and high speed. Finally one A356 sample obtained under different casting conditions was also investigated to compare with the A356 optimized for semi-solid forming. For the Al-Si system it was not possible to get enough contrast between the two metallurgic phases by absorption contrast, therefore we used phase contrast: the detector was placed 600 mm downstream of the samples for all the Al-Si samples except the last one for which we changed the distance to 800 mm.

Results :

For the Al-Cu samples as well as for the Al-Si samples, we obtained good quality 3D images with a pixel size of 2 microns which allow to detect the connection between solid particles. It should be mentionned that until now 3D images of semi-solid structures were only obtained by serial cutting of the sample and taking 2D micrographs in planes parallel one to another.

To our knowledge only three attempts were made: the first one by [Ito et al] on Al-Si samples but with very low solid fraction (0.2) and with serial cutting every 40 microns. The second by [Voohrees et al] on Sn-Pb alloy, with serial cutting every 20 microns and the third on Al-Si material but only on a few solid particles with serial cutting every 10 microns. The conducted experiment allows us to get cutting every 2 microns on a volume of 0.5mml x 0.5mm x 0.5mm. Figure 1 shows two slices, separated by 20 microns, out of the reconstructed volume of an Al-Si alloy. It appears that such a distance between sections may lead to a loss of information on solid particles: this confirms the great interest of getting directly the 3D volume. Concerning the connectivity and the microstructural evolution we can only present partial results presently: the experiments were conducted in April and 3D analysis of images obtained using X-ray tomography is not always obvious since the segmentation of the volume is not an easy task especially when phase contrast is employed. The scientific approach of the problem is the following : we first work on the segmentation of Al-Si and Al-Cu volumes since segmentation is absolutely necessary to get quantitative information about the solid phase. This work was done using different image analysis systems (Matlab, Image pro plus, Aphelion) to select the most appropriate. In the case of Al-Cu we have just managed to binarize the volumes. For Al-Si volumes (those obtained with phase contrast) no analysis system can provide good segmentation to study the connectivity of the solid : some solid particles are not closed, or some necks are missed. We are currently writing our own program and the first results are interesting. In parallel, we developped some tools to get some idea of the connectivity of the solid phase without any need of segmentation. As an example of the first results obtained concerning the connectivity, we are able to get the 3D skeleton from the 3D images even on volumes obtained with the phase contrast mode. Figure 2 shows the skeleton of the solid phase for one of the AI-Si samples and it can be seen that this material presents a high level of connectivity. This of great interest since usually this material is described as solid particles embedded in liquid.

Conclusion

These first experiments on semi-solid materials using X-ray tomography provide good quality 3D images in absorption or in phase contrast mode. We show that this technique is the only way to get information on the connectivity of the solid phase with high resolution (2 micron pixel size). Furthermore the first results indicate that the connectivity of the solid phase in the Al-Si alloy is high. Some of these results will be presented by J. Baruchel at the SF2M meeting in Paris during autumn 1998.

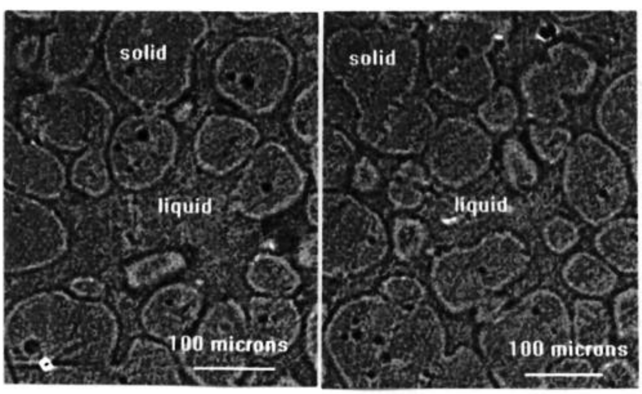

Figure 1 : Two slices, separated by 20 pm,
out of the Al-Si volume, obtained in the phase contrast mode.

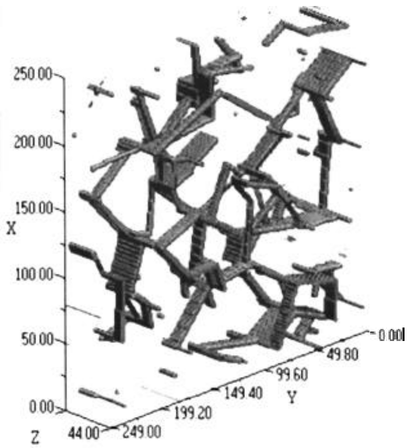

Figure 2 : Solid skeleton of an Al-Si volume (1 unit = 2 microns).