ESRF	Connectivity in semi-solid alloys during thermal treatment or mechanical deformation using phase contrast microtomography	**Experiment number:** **HS894**
Beamline: **ID19**	**Date of experiment:** from: 15/04/99 to: 20/04/99	**Date of report:** **28/07/99**
Shifts: 3	**Local contact(s):** P. Cloetens	02 SEP 1999 *Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

BRACCINI Muriel	Laboratoire GPM2-ENSPG-INPG
VERRIER Sandra	Laboratoire GPM2-ENSPG-INPG
SUERY Michel	Laboratoire GPM2-ENSPG-INPG
SALVO luc	Laboratoire GPM2-ENSPG-INPG
JOSSEROND Charles	Laboratoire GPM2-ENSPG-INPG
BOLLER Elodie	ESRF
CLOETENS Peter	ESRF

The aim of this experiment was to study the microstructure of aluminium alloys in the semi-solid state : the microstructure of semi-solid materials is characterised by the solid phase and the liquid phase. Two materials have been investigated an Al 10%Cu alloy and an Al 7%Si alloy. In both case the energy used was 17.5 keV and the 2 micron CCD camera was used, which is a good compromise according to the microstructure. The Al-Cu alloy was studied in a temperature range where the solid fraction is about 80% while the Al-Si alloy was studied in a temperature range where the solid fraction was around 50%. According to the composition of each alloy, the absorption mode (distance camera – sample =3 mm) was used for the Al-Cu alloy, while the phase contrast mode was used for the Al-Si alloy. In this latter case several experiments at different distance were performed in order to choose the optimum distance which was set to 500mm. The evolution of the microstructure was studied in two ways :

1) for the two alloys Al-Cu and Al-Si we studied the evolution of the microstructure during a thermal treatment in the semi-solid state
2) for the Al-Si we studied the evolution of the microstructure with deformation. In this case the material was deformed in the semi-solid state at various strain rates and for various strain and x ray tomography has been performed on each sample.

In this report we will focus on the first point since not all the volume have been completely analysed to give definitive conclusions about the second point.

Influence of thermal treatment on semi-solid microstructure :

The originality of such experiments was to attempt to make in situ measurements. Owing to the rapid evolution of the microstructure(within few minutes) it is not possible to make real in situ measurements even if the scan lasts 9 minutes. So we decided to do interrupted "in situ" measurements. The process was the following : the sample was held in a furnace at the desired temperature (555°C for Al-Cu and 587°C for Al-Si) for a time t quenched in water and put on the x ray tomography device. Then after tomography, the sample was put in the furnace again and so on. Of course care was taken to follow the same volume owing to marks made on the sample. The time in minutes for each material are given in the table below :

Al-Cu	0	5	15	30	60
Al-Si	0	5	10	20	30

Figure 1 presents for the Al-Cu two micrographs of 2D section at the same z for the time 30 minutes and 60 minutes. One can see that there is evolution in the microstructure within the 30 minutes treatment. This evolution concerns the solid phase but also the liquid phase.

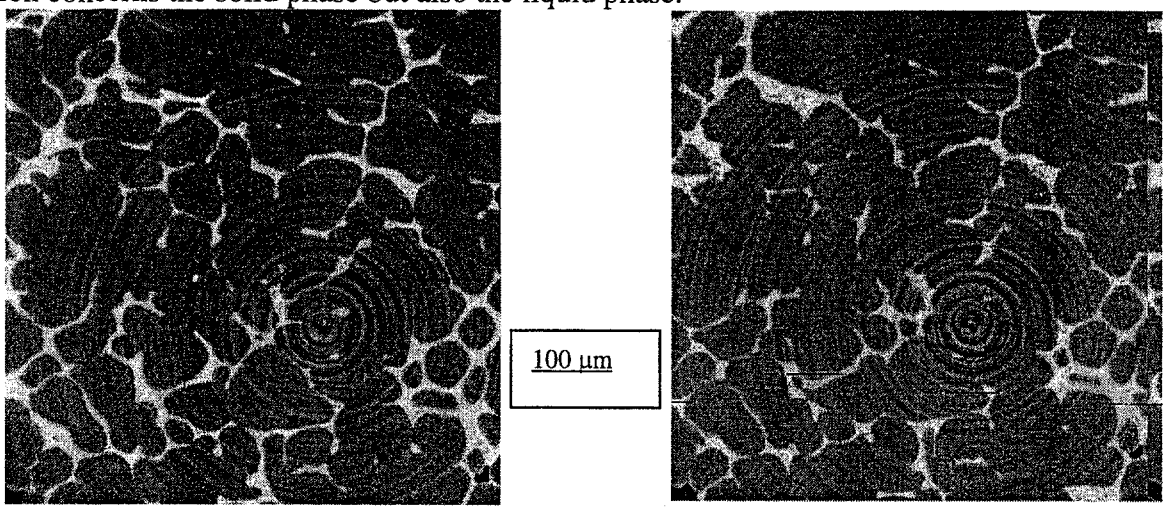

Figure (1) : microstructure of Al-Cu material for two time of remelting (30 min left 60 min right)

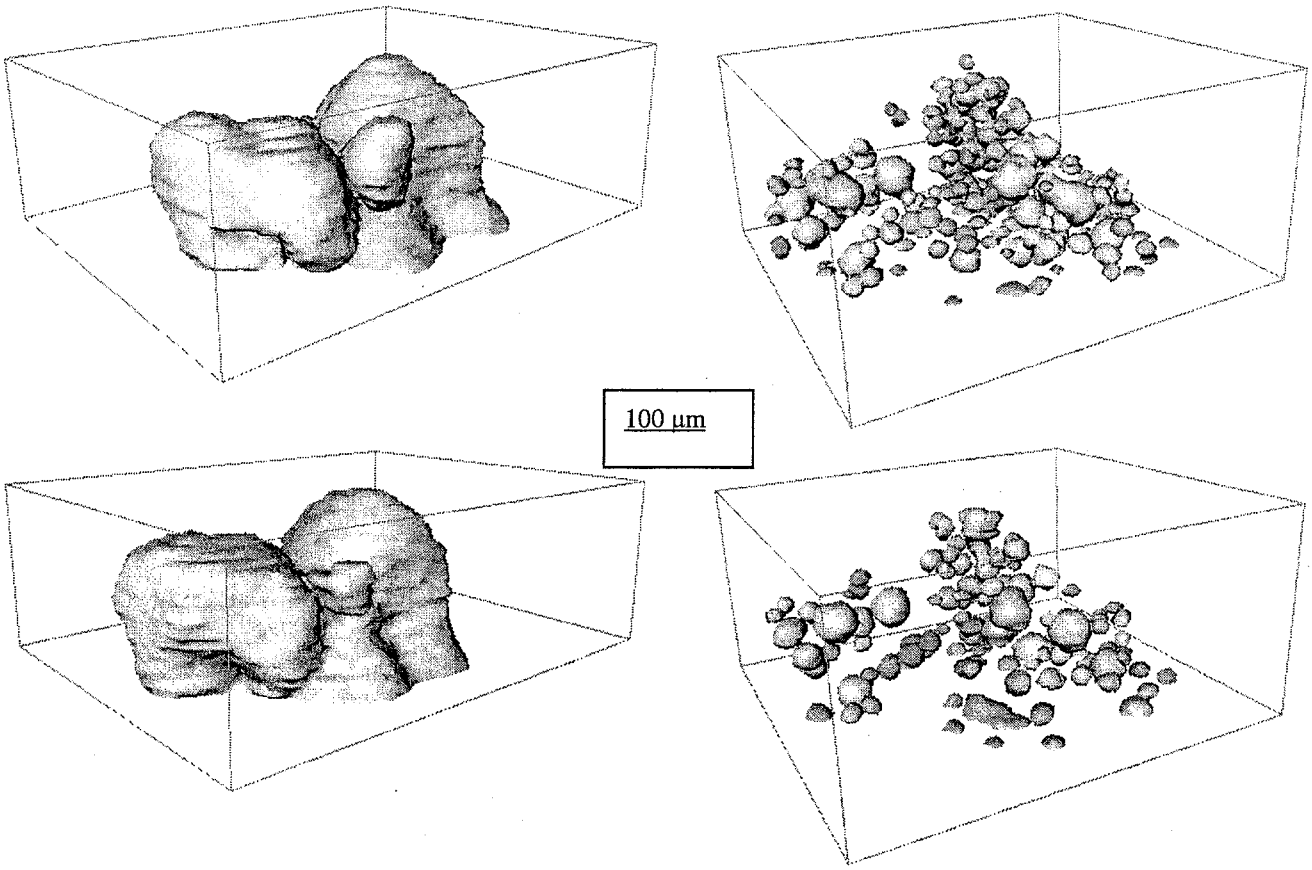

Figure 2 : solid phase in Al-Si material after 5 minutes in the semi-solid phase (up) and 10 minutes (down)

Figure 3 : entrapped liquid in solid of figure 2 after 5 minutes in the semi-solid phase (up) and 10 minutes (down)

Figure 2 and 3 present some results obtained for the Al-Si alloy. In figure 2 a group of solid particles have been isolated and one can clearly see that during the 5 minutes treatment the solid phase smoothen and that the small particle becomes smaller. This is typical of the Ostwald ripening mechanism. Figure 3 is focused on the entrapped liquid in the solid phase presented in figure 2. As it can be seen this entrapped liquid evolves also with time and it can be noticed that the number of liquid dropplets. However the entrapped liquid fraction decreases which indicates liquid dropplets dissolve to the benefit of interconnected liquid.

Conclusion : This results are to our knowledge the first 3D visualisation of in situ microstructural evolution of the solid phase and entrapped liquid phase. They will be presented at the international conference which will be held in Lyon about X ray tomography (invited conference).