ESRF	**Experiment title:** **Microtomographic investigation of grain boundary wetting (GBW) and liquid metal embrittlement in the Al-Ga system**	**Experiment number:** HS 1046
Beamline: **ID 19**	**Date of experiment:** from: 19.11.99 to: 22.11.99	**Date of report:** 29.02.00
Shifts: 12	**Local contact(s):** P. Cloetens, E. Boller	*Received at ESRF:*

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Report:

Synchrotron radiation X-ray microtomography and microradiography are used to investigate the penetration of liquid gallium into the grain boundaries of aluminium. Due to the large difference in absorption, micrometer and sub-micrometer thick liquid Ga layers can be directly observed in the bulk of polycrystalline Al samples [1]. The aims of the experiment were: 1) to characterise the kinetics of the penetration process by in-situ imaging 2) Application of the grain boundary visualization method to study fracture propagation in the bulk of aluminum alloys and 3) to apply the method to other relevant liquid -solid metal couples.

1. In-situ imaging

The initially foreseen method of repeated *tomographic* imaging during the penetration process (stepwise penetration by successive solidification and melting of the liquid Ga) turned out difficult due to the significant supercooling tendency of Ga and the high penetration rate. In-situ measurements were therefore performed via fast *radiographic* imaging of thin polycrystalline Al samples. Figure 1 shows the vicinity of a triple line as observed during one of the penetration experiments. In the configuration used (1µm pixel size, FRELON camera, 4 channel parallel readout, 800 MB image buffer) series of 400 images with a frame rate of up to 15 images per second could be recorded showing the penetration of several polycrystalline and also two bicrystalline samples.

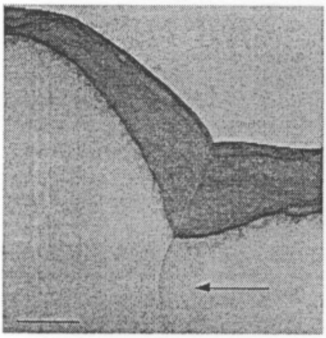

Figure1: Micro-radiography of a 0.7 mm thick Aluminum (99.8%) multi-crystal penetrated by liquid Ga. The image shows the vicinity of a triple line. Considerably less Ga is present in the lower grain boundary (arrow) - an indication for low specific energy of this grain boundary. The width of the Ga film increases during the first minutes of penetration before it reaches an equilibrium thickness. Note that there is a surplus of Ga at the intersection of the grain boundary with the free sample surface (liquid grooving). The cloudy contrast near the surface intersection is attributed to surface diffusion of liquid Ga.

2. Analysis of fracture propagation path

Al-Si alloy samples (pre-fatigued in dynamic tension experiment) were imaged before and after infiltration of liquid Ga. Pre-existing, micrometer-sized cracks are easily detected in the volume of the sample (1 x 1 x 2 mm) using coherent imaging techniques ("edge detection" regime, [2]). After infiltration of Ga, the grain boundaries can be visualized due to the enhanced absorption. The observed grain boundary structure was verified with a EBSP mapping of the sample surface (Fig 2). This 3D visualisation method allows (post mortem) distinction between intra- and intergranular crack propagation throughout the volume of the sample and is proposed as a technique for fracture characterisation [3].

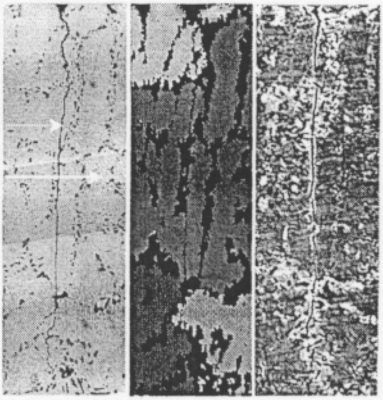

Figure 2: Same zone on the surface of a pre-fatigued Al-Si sample observed with three different techniques: a) optical micrograph, b) grain orientation mapping (EBSD): different colors correspond to different crystalline orientations, c) tomographic reconstruction after infiltration of Ga: cracks, Si particles and grain boundaries (enhanced absorption due to Ga) are easily distinguished. Note that a) & b) are restricted to the sample surface whereas the tomographic dataset contains information throughout the whole volume.

3. Application to other metal systems

Comparative studies in a few selected metal systems were performed. Interesting observations were obtained for the Ni-Pb system, where penetration seems to be restricted to triple lines (Figure 3) and for the Al-Sn system showing a pronounced dewetting after solidification of the liquid phase (Figure 4). The tomographic characterization in these systems was performed post-mortem (after solidification of the liquid phase) - and clearly shows the interest for future *in-situ* observations at high temperatures.

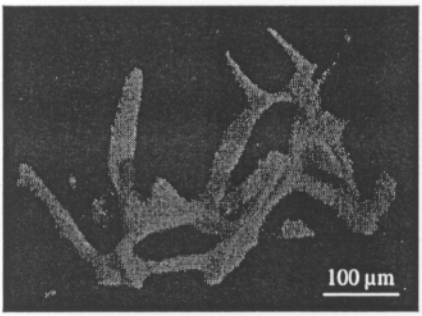

Figure 3: Volume rendering of Pb network in polycrystalline Ni showing finger like structures along triple lines.

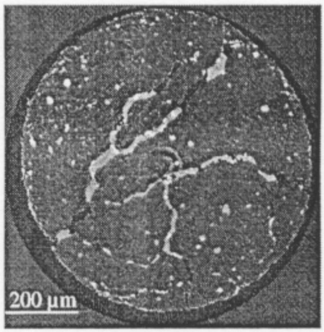

Fig 4: Al sample penetrated by liquid Sn (tomographic reconstruction after solidification).

References

[1] W.Ludwig, D.Bellet, Materials Science & Engineering, in press

[2] P. Cloetens, M. Pateyron-Salomé, J.Y. Buffière, G. Peix, J. Baruchel, F. Peyrin,M. Schlenker, J. Appl. Phys. 81 (1997) 5878

[3] W. Ludwig, J-Y. Buffière, S. Savelli, D. Bellet, to be published