ESRF	**Experiment title:** **Phase imaging investigation of liquid metal embrittlement in the Al/Ga system**	**Experiment number:** **HS-890**
Beamline: **ID19**	**Date of experiment:** from: **14.2.99** to: **18.2.99**	**Date of report:** **1.3.99**
Shifts: 12	**Local contact(s): P. Cloetens, E. Boller**	*Received at ESRF:*

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Report:

Scientific background:

Liquid Gallium (T_m=302.9K) when in direct contact to aluminum surfaces penetrates rapidly along the grain boundaries. Tensile loading of samples in direct contact with liquid Gallium gives rise to a fracture mechanism called 'Liquid Metal Embrittlement' (LME).

The feasibility of in-situ observation of grain boundary wetting and Liquid Metal Embrittlement (LME) was studied for the Al-Ga and Al-Sn systems by means of high resolution X-ray tomography and radiography.

Experimental setup

We used the ID19 micro-tomography setup consisting of a precision mechanics sample stage (rotation & translation) and a high resolution detector system. The spatial resolution of the detector system (fluorescent screen, microscope optics, ESRF Frelon camera,1024^2 CCD) was measured with the knife-edge method to 1.7 µm. The effective pixel size was 0.98 µm. After some test scans with the standard double crystal monochromator (Silicon 111) it turned out, that there will be no need for fine energy tuning round the absorption edges of Ga and Sn as initially assumed. We decided therefore to switch from the double crystal setup to a Rb-B_4C multilayer. The latter system has a energy bandwidth which is about a factor100 times larger and typical scan times (1000 projections) could be reduced from >7 hours to less than one hour. All scans were taken at 15 keV

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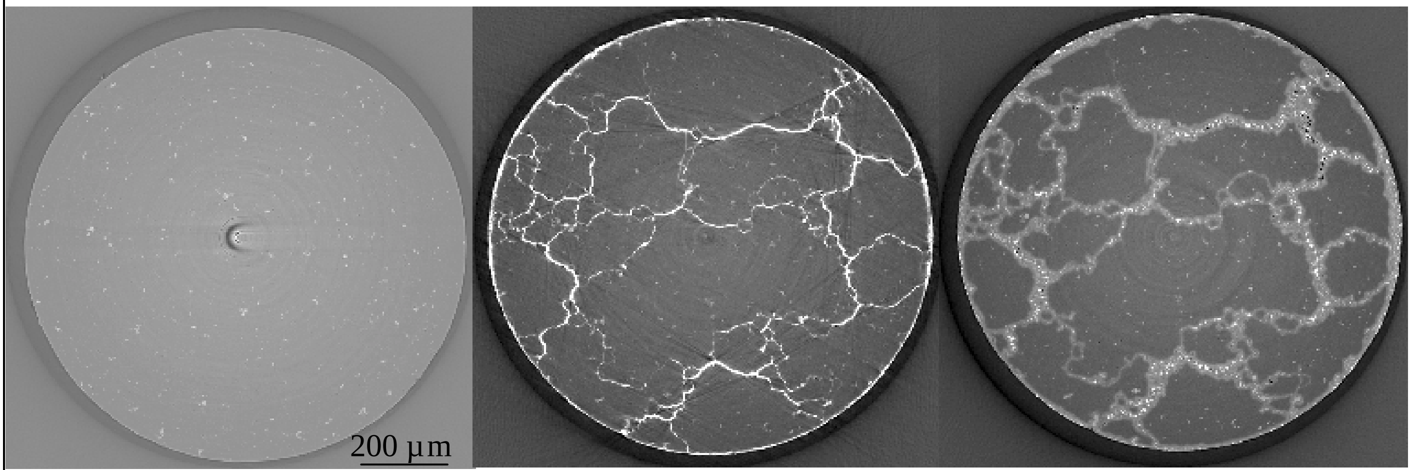
Results:

Micro-tomography

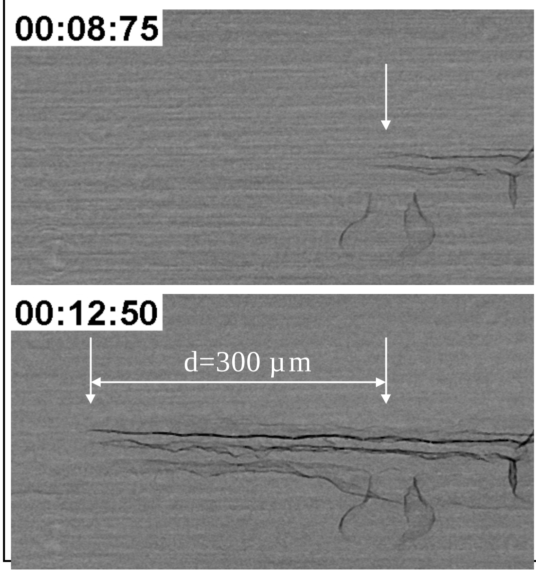
After alignment, determination of the detector transfer function (for later image deconvolution) and optimization of the experimental conditions we performed about 15 tomographic scans (800 projections each) of differently prepared samples (annealing time, annealing temperature…).
As most striking result we may state, that for some aluminum alloys the penetration by liquid Gallium leads to complete wetting of the grain surfaces in the whole sample. The combination of this grain boundary 'decoration' method' (which is widely used for surface techniques) with X-ray microtomography is a unique tool to obtain three-dimensional information about position, shape and size of the grains in the **bulk** of the samples. Figure 1 shows reconstructed slices of the same position in the sample before and after application of liquid gallium.



a b c

Figure 1: 2D tomographic reconstruction showing grain boundaries in polycrystalline aluminum: a) before application of Ga b) after application of Ga & 4hours annealing at 50 °C c) after 2 hours of additional annealing at 300 °C.

Radiography



In-situ observation of penetration of liquid gallium in aluminum samples was demonstrated. The combination of multilayer optics, the Frelon camera and fast data-acquisition software allowed us to take sequences of 100 images with up to 5 images per second (data rate: 10 MByte /s). The penetration speed can be estimated to 50-100 µm /s.

Figure 2: absorption radiographs of a thin aluminum platelet (400 µm) taken at different moments of the penetration process (⚒t = 3.75 s). The platelet is in direct contact with a reservoir of liquid Gallium 3 mm further to the right.