ESRF	**Experiment title:** In situ investigation of the Ga Liquid Metal Embrittlement of Al samples using high resolution microradiography.	**Experiment number**: Me679
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Shifts: 9	**Local contact(s)**: Eva Pereiro López	*Received at ESRF:*

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Report:

The grain boundary (GB) penetration of liquid Ga in Al bicrystals and polycrystals has been studied by synchrotron radiation X-ray microradiography. This experimental technique allows for non-destructive in-situ characterization of dynamic processes in the bulk of millimetre sized Al samples with a temporal resolution in the sub-second range and spatial resolution of about 1 µm. The minimum detection limit for nanometric liquid Ga layers in flat GB's (as for bicrystals) is of the order of 5nm in the case of edge-on observations.

Systematic studies concerning the influence of individual parameters (e.g. the nature of the GB, the presence of internal or external stress, the thermo-mechanical sample's history etc.) have been performed using identical bicrystals, cut from the same ingot. Comparative studies with polycrystalline samples were conducted as well, in order to reveal possible differences in the penetration process, caused by the changed boundary conditions, i.e. geometrical constraints on the movement of individual grains or presence of internal stresses.

The kinetics of the penetration process has been characterized for both kinds of samples by insitu measurements of the propagation and thickening rates of the liquid Ga layer [1]. A set of microradiographs showing the GBP of liquid Ga in an Al bicrystal is presented bellow in fig.1a, b & c; calculations of the liquid thickness (fig.1d) demonstrate that the propagation and thickening of the Ga are linear. Moreover, experiments applying external stress to the samples have shown the major role of stress in the process, even for very low stress levels as the ones provoked by the sample fixation. Higher penetration rates have been observed for stressed samples and a clear relation between stress and the occurrence of GBP for different grain boundary energies has been underlined. Fig.2 presents the experimental results for Al polycrystals demonstrating, in this case, that we are dealing with a discontinuous process. GBP in polycrystals can be divided in three steps:

1) a "propagation phase" where initially nanometric liquid Ga films penetrate along certain GB's simultaneously to a continuous increase of the layer thickness up to a hundred nanometers (as for bicrystals).

2) a discontinuous and quasi-instantaneous thickening of the nanometric Ga layer along certain percolation pathways of the GB network.

3) a saturation of the Ga layer (as for bicrystals).

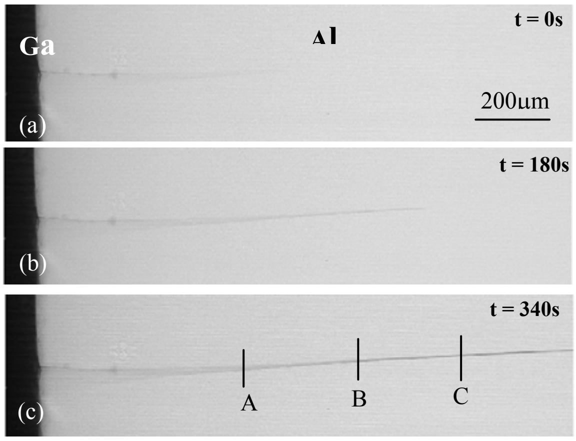
The discontinuous behaviour for polycrystals is the most significant feature of our observations when compared to the monotonous behaviour of bicrystals. This can be interpreted considering that the formation of micrometric thick wetting layers is very likely governed by stress relaxation and associated grain movements in already penetrated GB networks [2].

As stress influences strongly this phenomenon, preliminary investigations on samples under controlled external loading conditions (using the in-situ load cell developed by INSA, Lyon) have been carried out, too. These preliminary results are promising but more systematic studies have to be performed, thus more experiments in loading conditions are foreseen.

References

[1] Pereiro-López E., Ludwig W., Bellet D. & Baruchel J., Nuclear Instr. and Methods in Physics Research B 2003; 200: 333-338.

[2] Pereiro-López E., Ludwig W., Bellet D., Acta Materialia (submitted).



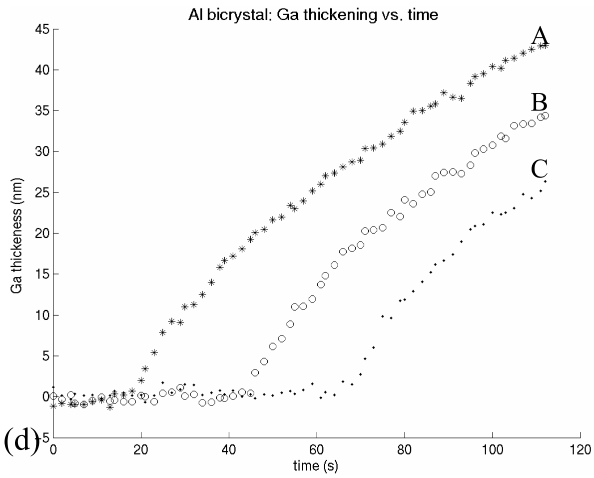


Fig1

Fig.1: (a-c) Set of radiographs showing the in situ Ga penetration along the GB of an Al bicrystal. (d) Evolution of the Ga thickness layer in regions A, B & C depicted in fig.1c.

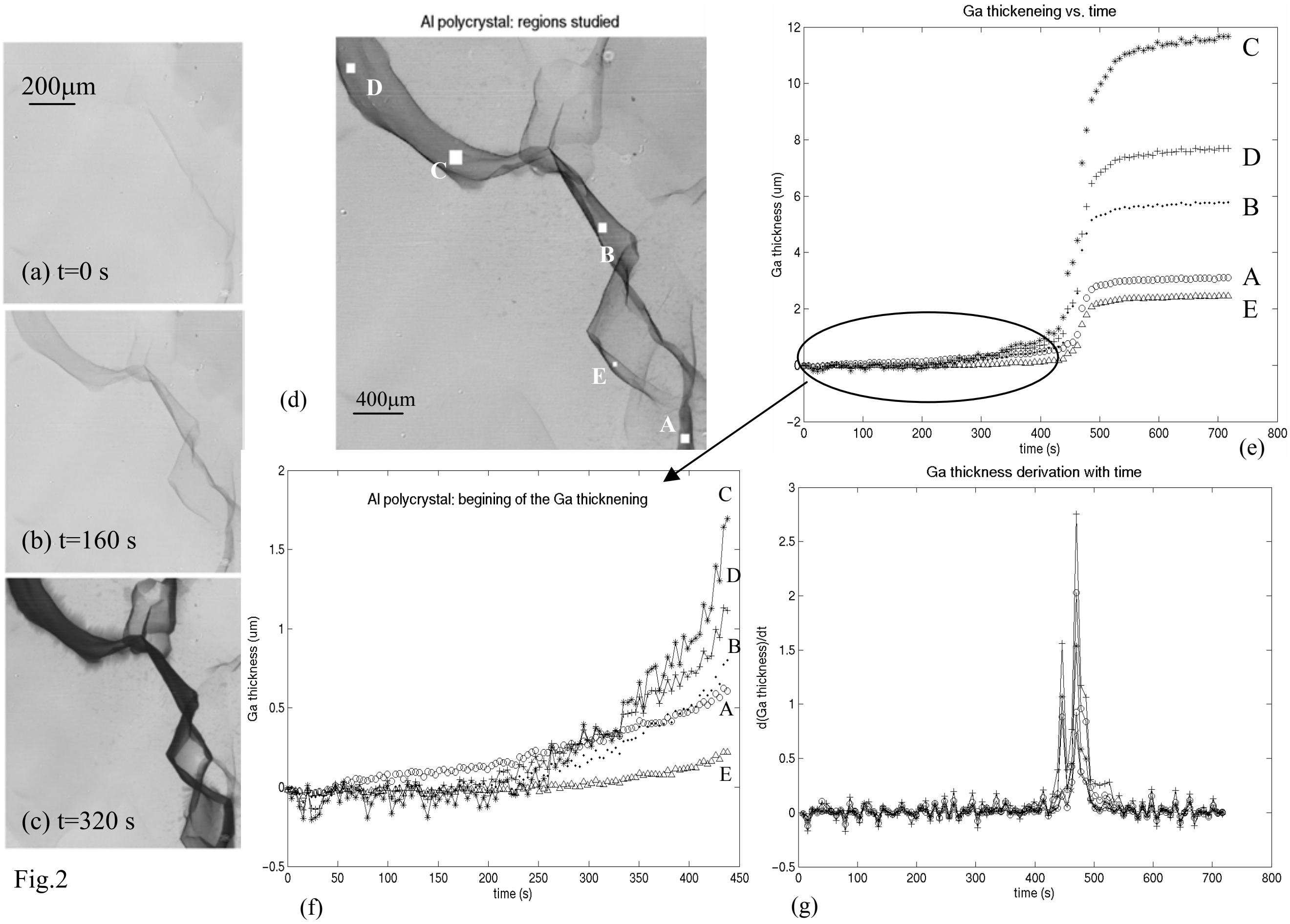


Fig.2

Fig.2: (a-c) X-ray microradiographs of an Al polycrystal at different times during GBP by liquid Ga. (d) location of the regions studied inside the GB. (e) Evolution of the Ga layer thickness in these regions. (f) zoom of the propagation and thickening before the discontinuity. (g) derivation of the Ga layer thickness vs. time showing the discontinuous behaviour.