ESRF	**Experiment title:** Application of combined radiography and topography to the characterization of Al-based solidification process	**Experiment number**: MA-308 (a)
Beamline: ID19	**Date of experiment**: from: 04/10/2007 to: 04/14/2007	**Date of report**: 02/04/2008
Shifts: 9	**Local contact(s)**: Dr. Juergen HAERTWIG	*Received at ESRF:*

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Scientific background

In the last decades the research of solidification of metals, alloys and compound crystals has developped to a large extent. The solidification microstructure largely and often definitively controls the properties and quality of grown materials. Thus, a precise understanding of growth processing, from the microscopic to the macroscopic scale, is essential to tailor reproducibly products of specified quality.

The development of brilliant third-generation synchrotron X-ray sources, together with advances in X-ray optics and detectors, has timely provided efficient tools for in-depth understanding of physical phenomena in a broad spectrum of situations. Synchrotron X-Ray radiography enables in situ and real-time observation of microstructure evolution, i.e. a direct access to dynamical phenomena which could not be anticipated from post-mortem analysis (morphological observations and compositional measurements on samples after completed or interrupted solidification). As most phenomena involved during solidification are dynamical, our objective is to perform in situ and real-time investigations of the solidification of Al-based alloys by using synchrotron imaging techniques. By this way, we intend to provide benchmark experiments both for the metallurgical and the material sciences communities.

Experiments

The MA-308 experiments was divided in two parts. During the first set of experiments, noted MA-308(a), we adressed mainly on the application of the combined synchrotron radiography and topography to the characterisation of the quasicrystal growth. Several oustanding results were obtained, presented in International Conferences and published in Journals with referees ([1], [2], [3]).

In particular, we show for the first time the in situ and real-time growth of dodecahedral quasicrystals. The QC character of grains was checked by post-mortem measurements of composition and indexation of diffraction diagrams. Measurements of the growth velocity of facets, based on synchrotron radiography, highlighted interaction between grains due to solute rejection. The combination of radiography and topography allowed to visualise evolution of strains during the growth of a monograin and showed that grains are strongly constrained, even in the early stages of their growth.

List of recent publications in International Journals with reviewers

[1] *J. Gastaldi, B. Billia, H. Nguyen Thi, T. Schenk, G. Reinhart, N. Mangelinck, H. Klein, H. Härtwig and J. Baruchel*
In situ synchrotron x-ray imaging of the solidification progress in metallic alloys
Trans. Indian Inst. Met. ***60*** *(2007) 281-286,*
Abstract: *In situ X-ray imaging of the solidification progress can be carried out, nowadays, in front of the synchrotron sources, by combining X-ray radiography with X-ray topography. Thereby it is possible to obtain information directly, on the one hand on the shape and the velocity of the growing interface by means of X-ray radiography, and on the other hand, thanks to X-ray topography, on the strains and the defects generated at the moving interface by the growth process as well as in the grain volume by internal stress centres and external thermomechanical stresses. In addition, the interaction of strains and defects, contributing to the formation of the solidification microstructure observed after cooling, can also be followed during this combined imaging. Some of these feasibilities are highlighted by presenting various examples of the solidification of opaque alloys, displaying facetted or dendritic growth, we have studied with the apparatus we have developed to perform such experiments at the ID 19 ESRF (European Synchrotron Radiation Facility – Grenoble - France) beamline.*

[2] *B. Billia, J. Gastaldi, H. Nguyen Thi, T. Schenk, G. Reinhart, N. Mangelinck, B. Grushko, H. Klein, J. Härtwig and J. Baruchel*
Growth structures, interface dynamics and stresses in metallic alloy solidification: in situ synchrotron X-ray characterisation
Trans. Indian Inst. Met. ***60*** *(2007) 287-291,*
Abstract: *In solidification processing, the physical properties are primarily controlled by the microstructure built in the solid. Thus, materials engineering requires detailed understanding of microstructure formation dynamics. Using in situ and real-time synchrotron X-ray imaging at the European Synchrotron Radiation Facility, the solidification progress in thin metallic alloys is characterized. On Al - 3.5 wt% Ni alloys, disorienting induced by stress accumulation with the development of columnar dendrites is observed. In particular, X-ray radiography shows bending of secondary dendrite arms which, in the columnar to equiaxed transition, can be furthermore precipitated by equiaxed crystal sedimentation. Ledge growth and facetted solid-melt interface are found on AlPdMn quasicrystals. Increasing the solidification rate, the kinetic undercooling becomes sufficient for nucleation and growth of new grains ahead of the columnar grains.*

[3] *J. Gastaldi, G. Reinhart, H. Nguyen Thi, N. Mangelinck, B. Billia, T. Schenk, J. Härtwig, B. Grushko, A. Buffet, J. Baruchel, H. Jung, P. Pino and B. Przepiarzynski*
In situ study of the quasicrystal growth by synchrotron X-ray imaging
Phil. Mag. ***87*** *(2007) 3079-3087,*
Abstract: *One of the main challenges of the quasicrystal science is to elucidate how the quasiperiodic order can extend so far, i.e. up to several centimeters according to the size of the single grains of various alloys routinely grown nowadays [1, 2, 3]. Noticing that most of the present knowledge on the growth of quasicrystal grains has been collected after their cooling at room temparature, we have carried out the first in situ and real time observation of this peculiar process which has clearly disclosed both, the shape of the growing interface and its defective state. Therefore we have studied the solidification of an AlPdMn alloy giving quasicrystal grains, by synchrotron X-ray imaging, combining thereby the radiography and X-ray topography techniques. Radiography allowed us to clearly evidence a facetted growth, proceeding by lateral motion of ledges at the solid-melt interface and controlled by the interface kinetics rather than by the local heat flow as widely thought. Thus a realistic estimate of the kinetic coefficient was deduced from the solid-melt interface undercooling which indicates that the quasicrystal growth is more likely comparable to both semiconductor and oxide growths than to pure metal growth. The X-ray topographs, we recorded simultaneously with radiographs, revealed that a lot of strains and defects are generated in the quasicrystal grains during their growth, which could be related to the growth process itself and very informative on the origin of the stability of the quasicrystal lattice.*