ESRF	**Experiment title:** Application of combined radiography and topography to the characterization of Al-based solidification process	**Experiment number**: MA-308 (b)
Beamline: ID19	**Date of experiment**: from: 07/11/2007 to: 07/14/2007	**Date of report**: 02/08/2008
Shifts: 9	**Local contact(s)**: Dr. Juergen HAERTWIG	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*NGUYEN-THI Henri**, L2MP, Campus Saint-Jérôme, Case 142, 13397 Marseille Cedex 20, France
*BERGEON Nathalie**, L2MP
MANGELINCK-NOËL Nathalie, L2MP
JUNG Hyejin, L2MP

*SCHENK Thomas**, LPM, Ecole des Mines de Nancy, Parc de Saurupt, 54042 Nancy cedex, France

BUFFET Adeline, PhD (ESRF), BARUCHEL José** (ESRF), *HÄRTWIG Jürgen** (ESRF)

Scientific background

[See report MA-308(a)]

Experiments

The MA-308 experiments was divided in two parts. During the second set of experiments, noted MA-308(b), we adressed mainly the study of directional solidification of Al-based alloys, using the combination of synchrotron radiography and topography [1].

During the dendritic columnar growth of non-refined Al - 3.5 wt% Ni alloy, dynamical phenomena linked to mechanical effects were characterised in particular: bending of secondary arms induced by gravity and interaction of eutectic growth with dendrites [2]. The related paper has been chosen by the Editors to appear on Springer's website as an "Open Access" article, which is "*reflective of the comprehensive nature of the paper and its overall excellence"* as precised by the Editors.

The Columnar to Equiaxed Transition (CET) during directional solidification of Al -3.5 wt% Ni refined alloy was studied. Applying a sharp change of pulling rate provokes grains nucleation on refining particles that block the dendritic columnar growth [3]. The fact that both columnar and equiaxed grains are blocked before the grains are touching each other show that the blocking is due to solutal interaction. This observation was recently quoted in a review paper on CET (*J.A. Spittle, Inter. Mat. Rev. 51 (2006) 247*) Equiaxed solidification continues by successive nucleations and growth. Analysis of the final equiaxed microstructure as a function of the velocity jump shows that refining particles efficiency reaches a limit due to the mutual interaction of equiaxed grains [4]. The changes in grains shapes are also characterised and comparison with numerical simulations is in progress.

List of recent publications in International Journals with reviewers

[1] *A. Buffet, G. Reinhart, T. Schenk, H. Nguyen Thi, J. Gastaldi, N. Mangelinck-Noël, H. Jung, J. Härtwig, J. Baruchel and B. Billia*
Real-time and in situ solidification of Al-based alloys investi-gated by synchrotron radiation: a unique experimental set-up combining radiography and topography techniques
Phys. Stat. Sol., vol.204, n°8 (2007) 2721-2727
Abstract: *A unique experimental set-up combining X-ray radiography and topography has been recently developed at the ESRF. It allows direct observation of the solidification microstructure by these two complementary imaging techniques. This combined observation offers new possibilities, in particular for the investigation of strains, stresses and defect formation during directional solidification. In this paper we present in detail the experimental device and selected results obtained for two Al-based alloys: Al-3.5wt%Ni and ϑ-Al3Mg2. For the first time, we were able to characterize and quantify (by using topography) mechanical phenomena observed by radiography during the growth process.*

[2] *G. Reinhart, A. Buffet, H. Nguyen Thi, B. Billia, H. Jung, N. Mangelinck-Noël, N. Bergeon, T. Schenk, J. Härtwig and J. Baruchel*
In situ and real time analysis of strain and microstructure defects formation during solidification of Al – 3.5 wt% Ni alloys
Metall. Mater. Trans. A (2008) in press
Abstract: *Alloy solidification was investigated by using a unique experimental set-up developed at the European Synchrotron Radiation Facility (ESRF) combining both synchrotron X-ray radiography and topography. While synchrotron X-ray radiography allows in situ and real-time observation of the solid – liquid interface of metallic alloys, white-beam synchrotron X-ray topography offers the possibility of investigating strain and defect formation within microstructures during their growth. In this paper, we present results obtained during directional solidification experiments performed with Al – 3.5 wt% Ni samples. The initial state after thermal stabilization is characterized. Then, the transition from a motionless initial interface to the columnar growth regime after pulling is thoroughly investigated. Disorientations of parts of the microstructure occur during dendrites development. Moreover, bending of secondary arms is quantified and a value of the yield stress at the melting point for aluminum is estimated. Lastly, entrapment of dendrite structures by the eutectic phase induces stress deformations of the growing solid and eutectic grains also grow strained.*

[3] *H. Nguyen Thi, G. Reinhart, N. Mangelinck-Noël, H. Jung, B. Billia, T. Schenk, J. Gastaldi, J. Härtwig and J. Baruchel*
In-Situ and Real-Time Investigation of Columnar to Equiaxed Transition in Metallic Alloy
Metall. Mater. Trans. A ***38-7*** *(2007) 1458-1464,*
Abstract: *In this paper, we present a review of observations during Al-3.5 wt% Ni alloys solidification experiments performed at the European Synchrotron Radiation Facility (ESRF) in Grenoble. These experiments provide direct access to dynamical phenomena during columnar growth (initial transient and breakdown of planar solid - liquid interface), and for the first time to the transition from columnar to equiaxed microstructure (nucleation ahead of columnar front, blocking of columnar front by equiaxed microstructure) and fully equiaxed growth (propagation of an effective front). Based on these experimental observations, critical parameters such as columnar growth velocity variation during the transition or equiaxed-grain diameter are measured and discussed.*

[4] *G. Reinhart*
Dynamique de formation de la microstructure de solidification d'alliages métalliques: caractérisation par imagerie X-synchrotron
Université P. Cézanne (2006) Marseille, France