ESRF	**Experiment title:** Live characterization of metallic materials during solidification processing by means of Synchrotron X-ray imaging	**Experiment number**: HS-2789
Beamline: ID19	**Date of experiment**: from: 12/07/2005 to: 12/13/2005	**Date of report**: 06/12/2006
Shifts: 15	**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
BILLIA Bernard, L2MP
*REINHART Guillaume**, L2MP
*MANGELINCK-NOEL Nathalie**, L2MP

*GASTALDI Joseph**, CRMCN, Campus de Luminy, Case 913, 13288 Marseille cedex 9, France

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

*GRUSHKO Benjamin and PRZEPIORZYNSKI Bartosz**,, IFF, Forschungzentrum, Jülich, GmbH, 52425 Jülich, Germany

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

Report:

The solidification microstructure, with the accompanying solute segregation profile, 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.

Experimentally, most studies on metallic alloys have characterized the solid-liquid interface by post-mortem analysis (morphological observations and compositional measurements on samples after completed or interrupted solidification). As most of 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. During this set of experiments, we adressed the following issues using X-ray radiography and topography:

- Defects, crystallographic orientation and mechanical effects during the columnar growth of Al-3.5wt%Ni alloys [1].
- Columnar to equiaxed transition during directional solidification of Al - 3.5 wt% Ni alloys [2,3]
- Quasicrystal growth mechanism [4,5]
- Twinning during the growth of Al_3Mg_2 and pores in Al_3Mg_2.

[1] *G. Reinhart, H. Nguyen-Thi, J. Gastaldi, B. Billia, N. Mangelinck-Noël, T. Schenk, J. Härtwig, J. Baruchel. In situ and real time investigation of directional solidification of Al - Ni alloys by synchrotron imaging Material Science Forum, **508** (2006) 75-80*

Abstract. *Solidification is a dynamic phenomena and, as a consequence, it is of major interest to be able to investigate this process by in situ and real time observation. With synchrotron sources, this can be achieved by applying X-ray Imaging techniques (Radiography and Topography). Hence it is possible to follow the dynamical selection of solidification pattern on metallic alloys and to observe strain effects during growth process. In this paper, we present results obtained by using separately the two imaging techniques for the study of the microstructure formation during Al – Ni alloys solidification.*

[2] *G.Reinhart, N. Mangelinck-Noël, H. Nguyen-Thi, T. Schenk, J. Gastaldi, B. Billia, P. Pino, J. Härtwig, J. Baruchel*
Investigation of Columnar-Equiaxed Transition and Equiaxed growth of Aluminium Based Alloys by X-Ray Radiography
Materials Science and Engineering A, ***413-414*** *(2005) 384-388*
Abstract. *Among solidification processes, the columnar to equiaxed transition (CET) and equiaxed growth are still raising issues both from the metallurgical point of view and for the understanding of the fundamental related physical phenomena. The phenomena involved are complex and most of the time intimately related to the dynamical events happening during the solidification. Bridgman solidification of Al – Ni alloys is performed at the ID19 beamline of the European Synchrotron Radiation Facility (ESRF) in Grenoble. The use of a 3rd generation synchrotron X-ray source allows in situ and real-time observation of the solid – liquid interface, in particular at the CET. To provoke the CET, refined alloys are used and the pulling rate is increased following several profiles. The experiments give direct access to specific behaviours and enable the measurement of several key parameters during the CET and equiaxed growth, which can be discussed.*

[3] *Reinhart G., Nguyen-Thi H., Mangelinck-Noël N., Schenk T., Billia B., Gastaldi J., Härtwig J., Baruchel J.*
In-situ observation of transition from columnar to equiaxed growth in Al-3.5 wt% Ni alloys by synchrotron radiography
Modelling of Casting, Welding and Advanced Solidification Processes - XI, Proceeding of MCWASP - XI conference (May 2006, Nice, France), 359-366
Abstract. *We present results on Columnar to Equiaxed Transition (CET) induced by applying pulling rate jumps during the solidification of refined alloys. Solidifications of Al-3.5 wt% Ni alloys are performed in a Bridgman furnace at the European Synchrotron Radiation Facility (ESRF) in Grenoble. The use of synchrotron X-ray radiography allows direct observation of the solid-liquid interface for metallic alloys, in particular during the CET where most of events are dynamical. The morphology of growing columnar or equiaxed dendrites can be well resolved, in real time and with high image contrast. These experiments provide direct access to specific behaviours during CET and equiaxed growth.*

[4] *Gastaldi J., Schenk T., Reinhart G., Klein H., Härtwig J., Mangelinck-Noël N., Grushko B., Nguyen Thi H., Pino P., Billia B., Baruchel J.*
In situ observation of pore evolution during melting and solidification of Al-Pd-Mn quasicrystals By Synchrotron X-ray radiography
Phil. Mag., ***86*** *(3-5) (2006) 335-340*
Abstract. *It is now generally admitted that pores are intriguing special features of quasicrystals. Therefore, we have performed an "in situ" and real time observation of the pore evolution during directional solidification and melting cycles of an icosahedral Al-Pd-Mn bi-grained sample by synchrotron X-ray radiography. Rather surprisingly, no pore was observed to grow during the solidification stages. Nucleation and growth of pores were firstly seen during melting. These pores were subsequently shrinking either just being absorbed or during resumption of directional solidification. It is concluded that the vacancy origin of pores is certainly valid whereas the vacancy supersaturation needed thereby to explain their growth would be more probably related to the peculiar structure of quasicrystal than to the destruction of the thermal equilibrium.*

[5] *H. Nguyen-Thi, J. Gastaldi, T. Schenk, G. Reinhart, N. Mangelinck-Noel, V. Cristiglio, B. Billia, B. Grushko, J. Härtwig, H. Klein, J. Baruchel*
In situ and real-time probing of quasicrystal solidification dynamics by synchrotron imaging
Physical Review E, submitted
Abstract. *Quasicrystals growth remains an unsolved problem in condensed matter. The dynamics of the process is studied by means of synchrotron live imaging all along the solidification of icosahedral AlPdMn quasicrystal. The lateral motion of ledges driving facetted growth at the solid-melt interface is conclusively shown. When the solidification rate is increased, nucleation and free growth of new facetted grains occur in the melt, due to significant interface recoil induced by slow attachment kinetics. The detailed analysis of the evolution of these grains reveals the crucial role of aluminium rejection, both in the poisoning of grain growth and driving fluid flow.*