ESRF	**Experiment title:** In situ and real-time investigation of solidification of Al-based alloys by X-ray imaging	**Experiment number**: MA-95
Beamline: ID19	**Date of experiment**: from: 04/04/2006 to: 04/11/2006	**Date of report**: 01/02/2007
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Report:

The solidification microstructure, with the accompanying solute segregation profile, is the key link between materials processing and materials behavior. Thus, a precise understanding of growth processing, from the microscopic to the macroscopic scale, is essential to tailor reproducibly products of specified mechanical 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). Recent improvements in synchrotron X-ray imaging offer the possibility to study dynamical phenomena which occur during metallurgical processes. 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]
- Quasicrystal growth mechanism [3, 4]

[1] *B. Billia, H. Nguyen-Thi, G. Reinhart, N. Mangelinck, J. Gastaldi, T. Schenk, J. Hartwig, J. Baruchel, V. Cristiglio, B. Grushko, H. Klein*
Advances in Science and Technology ***Vol.46*** *(2006) 1-10*
Abstract*. The solid microstructure built in the solid governs the properties of materials elaborated from the melt. In order to clarify the dynamical mechanisms controlling solidification processing, we use in situ and real-time synchrotron X-ray radiography at ESRF (European Synchrotron Radiation Facility) to analyze microstructure formation in thin aluminum alloys solidified in the Bridgman facility installed at the ID19 beamline. During directional solidification of Al - 3.5 wt% Ni alloys, global mechanical constraints induced by the shape are found to act on the solid microstructure. In particular, radiography videos of dendritic growth show disorientations of sidebranches induced by mechanical stresses. In the solidification of AlPdMn quasicrystals, live imaging reveals that facetted growth proceeds by the lateral motion of ledges at the solid-melt interface. When the solidification rate is increased, the kinetic undercooling becomes sufficient for grain nucleation and growth in the liquid. These grains develop specific features that can be attributed to grain competition and concomitant poisoning of growth caused by the rejection of aluminum in the melt.*

[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] *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 ***E74*** *(2006) 031605*
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.*