	Experiment Title:	Experiment Number:
ESRF The European Synchrotron	In-situ Ultra fast X-ray tomography to investigate pore formation and kinetics in a commercial Ni-based superalloy	MA3483
Beamline: ID19	**Date of experiment:** from: 2017/06/14 to: 2017/06/17	**Date of report:** December 2019
Shifts: 9	**Local Contact:** Elodie BOLLER	

Names and affiliations of applicants:
Pierre Lhuissier (SIMaP - CNRS - Univ. Grenoble Alpes)
Luc Salvo (SIMaP - CNRS - Univ. Grenoble Alpes)
Edouard Chauvet (SIMaP - CNRS - Univ. Grenoble Alpes)
Alexis Burr (SIMaP - CNRS - Univ. Grenoble Alpes)
Jean-Jacques Blandin (SIMaP - CNRS - Univ. Grenoble Alpes)
Guilhem Martin (SIMaP - CNRS - Univ. Grenoble Alpes)

REPORT:

Aims:
The experiments aim at understanding the formation and kinetics of pores during solidification of a commercial Ni-based superalloy. Practical aims were:

- to characterize in-situ the solidification path of the investigated materials by varying the cooling rates: volume fraction of liquid as a function of temperature and estimation of the dendrite arm spacing as a function of the cooling rate.
- to track pores during solidification in order to provide new insights into their formation and kinetics depending on the cooling rates.

Experiments:
Two different sets of experiments were carried out:

(i) in-situ solidification with controlled cooling rates;
(ii) in-situ high temperature tensile tests performed in the mushy zone.

Solidification experiments were carried out within a furnace dedicated to the ID19 beam line ("Four Ecole des Mines"). The solidification consisted of melting the material at 1400°C and to cool it down at controlled cooling rates, namely 5°C/s, 25°C/s and 50°C/s while performing 3D-scans. A thermocouple located at the close vicinity of the solidified material provided a good estimation of the temperature during the solidification.
High temperature tensile tests were performed using an induction furnace (home-made SIMaP device) with a dedicated homemade tensile micro-mechanical machine.
A high energy beam of 68 keV was used and two different pixel size were used: 1.22 and 2.44 with a scanning time from 0.3 s to 3 s

Results:
Time-resolved in situ microtomography has been employed to track the nucleation and growth of individual pores during solidification of a commercial Ni-based superalloy. The effect of the cooling rate on the dendritic structures as well as on the formation of porosity has been investigated. Phase contrast obtained with a coherent X-ray beam is used to visualize the evolution of dendritic structures in absence of a sufficient absorption contrast, see example in **Fig 1.a-b**. Two different mechanisms leading to shrinkage pores have been identified. An example of pore forming through mechanism B is given in **Fig 2.a-b**. The first mechanism (mechanism A) is associated with the coalescence of secondary dendritic arms at temperature during the early stages of solidification. The second mechanism (mechanism B) is related to

insufficient liquid feeding in the interdendritic region during the last stages of solidification, at lower temperatures. We found that a variation of cooling rate by a factor 2 does not affect the nucleation rate of pores generated through mechanism B. However, it seems to affect the nucleation rate of small pores obtained through mechanism A. A detailed study based on those results has been published in [1].

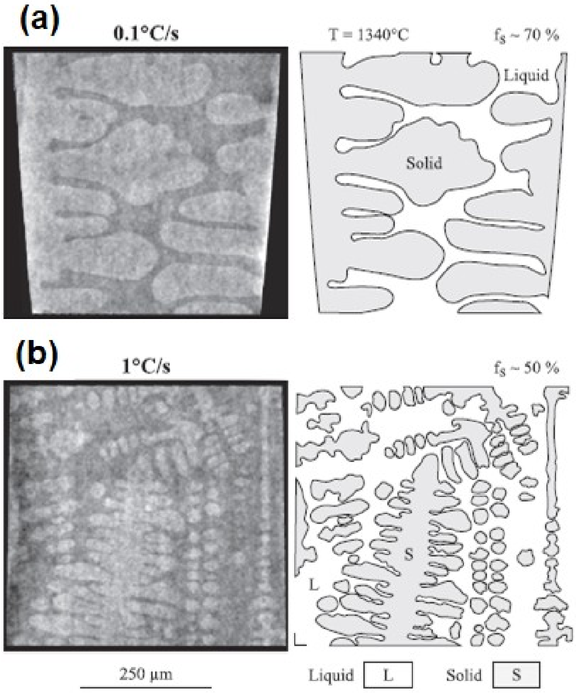

Fig 1. Effect of cooling rate on the dendritic solidification structures: (a) 0.1°C/s and (b) 1°C/s. For each sample a slice of the reconstructed volume is given. For a sake of clarity, a schematic is also shown.

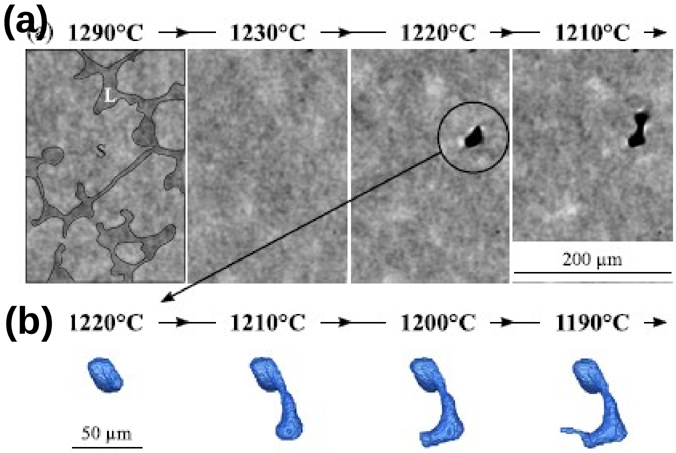

Fig 2. Formation of pores forming in the interdendritic region (cooling rate of 0.5°C/s). (a) Evolution of a single cross section showing the apparition of a pre after apparent complete solidification. (b) 3D rendering of the pore showing the growth mechanism during the last stages of solidification process.

Valorisation:

Those experiments were carried out in the framework of the PhD of Edouard Chauvet (defended on November 2017). The results were preliminary analyzed by Edouard Chauvet (PhD Student) and deeply analyzed by Emeric Plancher (post-doctoral fellow). Materials and methods, partial results are reported in Edouard CHAUVET PhD thesis [2]. Parts of the results obtained in this MA3482 proposal are now published, see Ref [1].

[1] Emeric Plancher, Paulien Gravier, Edouard Chauvet, Jean-Jacques Blandin, Elodie Boller, Guilhem Martin, Luc Salvo, Pierre Lhuissier. *Tracking pores during solidification of a Ni based superalloy using 4D synchrotron microtomography*. Acta Materialia vol 181 (2019) 1-9. https://doi.org/10.1016/j.actamat.2019.09.040

[2] Edouard CHAUVET, Université Grenoble Alpes, PhD Thesis, *Mise en oeuvre de superalliages base Nickel par Electron Beam Melting* (2017). http://www.theses.fr/2017GREA1084