| ESRF | **Experiment title:**<br>Application of synchrotron X-ray imaging to the study of Ni-base alloy solidification | **Experiment number**:<br>MA-5713 |
|---|---|---|
| **Beamline**: | **Date of experiment**:<br>from: 3 May 2023 to: 6 May 2023 | **Date of report**:<br>11 October 2023 |
| **Shifts:** | **Local contact(s)**:<br>Marta MAJKUT – Alexander RACK | *Received at ESRF:* |


**Names and affiliations of applicants** (* indicates experimentalists):

* REINHART Guillaume, IM2NP, Campus Saint-Jérôme, Case 142, 13397 Marseille Cedex 20, France

* MANGELINCK-NOEL Nathalie, IM2NP, Campus Saint-Jérôme, Case 142, 13397 Marseille Cedex 20, France

* REGULA Gabrielle, IM2NP, Campus Saint-Jérôme, Case 142, 13397 Marseille Cedex 20, France

* NGUYEN-THI Henri, IM2NP, Campus Saint-Jérôme, Case 142, 13397 Marseille Cedex 20, France


**Scientific objectives:**

Ni-base superalloys are nowadays the reference material family to produce blades for advanced turbine engines. A major issue during turbine blade production is the formation of defects such as stray grains, freckles, and segregated areas that are linked to the development of convective flows during the solidification step. The aim of the MA-5713 experiments was to study the impact of thermo-solutal convection on the dendritic microstructure formation as well as dendrite deformation and misorientation during metallic alloy solidification. To this end, in-situ and real-time observation of the directional solidification of a Ni-based superalloy was carried out. The ID19 team had successfully implemented simultaneous recording of radiograph images and of a diffraction spot images in the framework of a collaboration with our team on the study of silicon growth. This new observation configuration was successfully applied for the first time during the solidification of a Ni-based superalloy in the framework the MA-4862 campaign.

**Experimental Procedures:**

The unique IM2NP device named GaTSBI (Growth at high Temperature observed by Synchrotron Beam Imaging) was used (Figure 1, left part). This device consists of a high temperature furnace allowing melting and solidification of materials up to 1800°C to be performed. This device is also compatible with two X-ray imaging techniques: radiography to collect absorption images and diffraction imaging to visualize Bragg spot images. For the absorption image, the white beam was made monochromatic after illuminating the sample by using a post-specimen monochromator. A sCMOS pco.edge 5.5 camera was used with a a pixel size of 6.5 µm x 6.5 µm and a field of view of 8 mm x 8 mm. Enough contrast was obtained by using a monochromatic beam at an energy E ≈ 35 keV. The images of a Bragg spot were recorded by placing another sCMOS pco.edge camera with a pixel size of 4.5 µm x 4.5 µm in the path of a diffracted beam (Figure 1, right-hand part).

Two samples sample were prepared at IM2NP: a CMSX-4 commercial alloy and a Ni-11%pdsAl model binary alloy. Thes samples were 45 mm in length, 7.8 mm in width and 0.3 mm in thickness and inserted into an alumina crucible. Only the CMSX-4 sample could be investigated due to a failure of the GaTSBi device chiller early in the campaign, and the time necessary to detect the failure and find an alternative solution.
6 directional melting/solidification sequences could be been performed by cooling down the sample at cooling rate of -0.3 and -1°C/s in an applied temperature gradient of 30°C/cm.

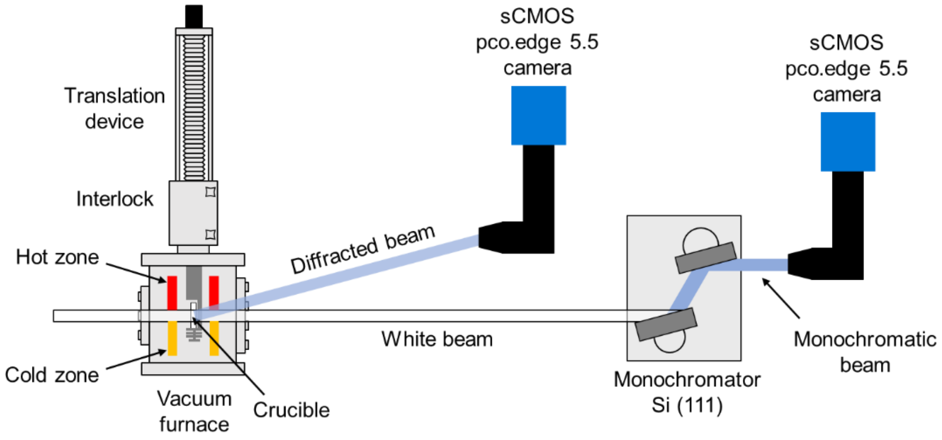


Figure 1: Sketchs of the GaTSBI high temperature vacuum furnace and of the configuration used to combine X-radiography and diffraction imaging in a synchronized manner during directional solidification.

**Results:**

The experiments showed that the innovative approach of combining X-radiography and diffraction imaging provides complementary information on the microstructure development. An accurate determination of the solid-liquid interface position is made possible by looking at the radiographs while the sample is cooled down (Figure 2-a1 to d1). Meanwhile, additional information were obtained by looking at the images of the Bragg spot. For example, the shape of the dendritic grain is visible in the first images (Figure 2-a2 to c2), but the diffraction image progressively deforms, which is accompanied by an increase of the diffraction spot intensity. Eventually, the dendritic shape isn't recognizable anymore (Figure 2-d2). This can be attributed to the developpment of strain during the cooling down of the grain. Further experiments would be necessary to investigate the impact of the solidification parameters on the dendrite deformation. It is also envisaged to extend the experimental configuration to enable the concomitant observation of two Bragg spots, which would be helpful to provide quantitative information on the misorientation. As previously mentioned, the solidification of the binary alloy could not be performed due to a technical failure. Such an investigation would be useful to look at larger microstructures where the impact of strain development would be easier to characterize.

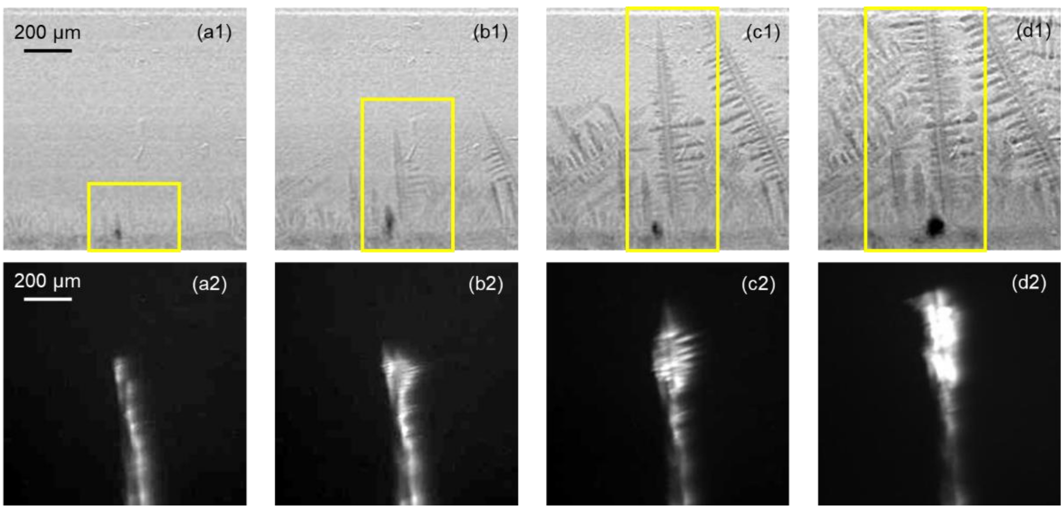


Figure 2: Sequence of radiograhs (top line) and topographs (bottom line) showing the development of a dendritic grain. The experiment duration is approximately 10 minutes.