ESRF	**Experiment title:** Application of synchrotron X-ray imaging to the study of Ni-base alloy solidification	**Experiment number**: MA-4862
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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-4862 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 applied for the first time during the solidification of a Ni-based superalloy in the frameworkof the present experiments.

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 camera in the path of a diffracted beam (Figure 1, right-hand part).

A CMSX-4 sample was prepared at IM2NP. The sample was 45 mm in length, 7.8 mm in width and 0.3 mm in thickness and inserted into an alumina crucible. 8 directional melting/solidification sequences have been performed by pulling down the sample from the hot to the cold zone at velocities, ranging from 2 µm/s to 10 µm/s.

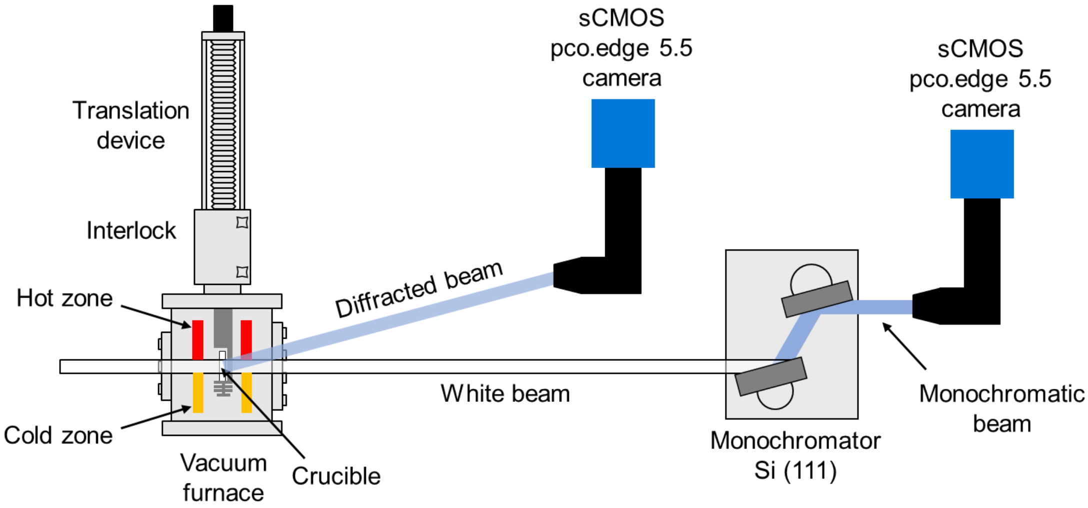
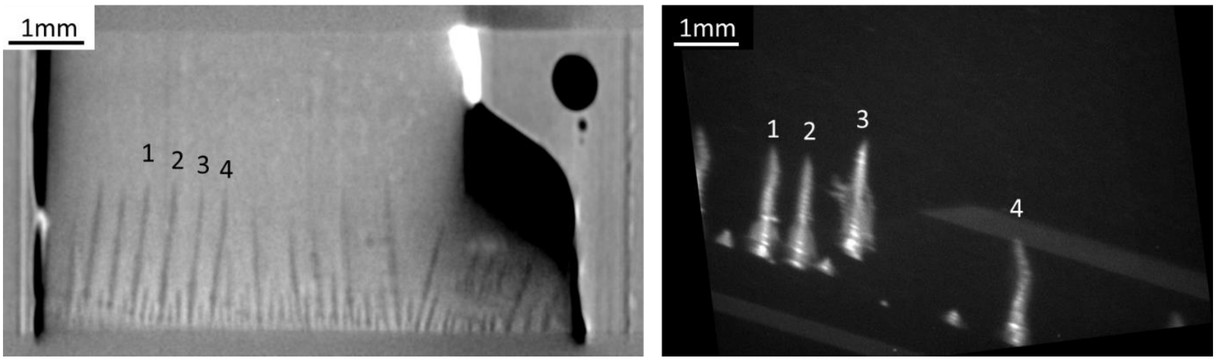


Figure 1: Sketchs of the GaTSBI high temperature vacuum furnace and of the configuration used to combine X-radiography and diffraction imaging in a synchronized manner during directional solidification.

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 pulled down. Meanwhile, additional information were obtained by looking at the images of the Bragg spot. For example, the deformation of the initial planar solid-liquid interface is made clearly visible by an increase in intensity at the locations where the morphological instabilty is occuring.

Following the morphological instabilty, cellular and dendritic microstructure developed. It was possible to identify individual dendrites in both the radiographs and diffraction images (Figure 2). Another interesting observation was the successive misorientations of the dendrite secondary arms during growth. The origin of this phenomenom is still under investigation. It could be related to the development of strain at the neck connecting the arm to the primary trunk. Solutal interactions induced by thermo-solutal flow can cause a decrease of the neck radius and a progressive shift of the arm orientation due to buoyancy. 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 qualitative information on the misorientation.



(a) (b)

Figure 2: (a) X-radiography showing the development of a columnar dendritic micrsotructure. (b) Diffraction image recorded at the same time. The dendrite images appear deformed due to the development of strains and misorientations during growth (G = 20 °C/cm, V = 5 µm/s).