ESRF	**Experiment title:** Examination of Single-Crystal Dendrite Structure and Porosity in Nickel-Based Superalloys Using X-Ray Microtomography	**Experiment number:** MA-1275
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Report:

1. Experimental

Material

The second generation single-crystal nickel-based superalloy CMSX-4 was used to investigate dendritic microstructure and porosity as a function of withdrawal rate and heat-treatment. Its full chemical composition (wt.-%) is as follows: Ni-6.5Cr-9.0Co-0.6Mo-3.0Re-6.0W-5.6Al-6.5Ta-1.0Ti-0.1Hf.
The cylindrical rods of 180 mm in height were produced in a Bridgman type vacuum induction melting (VIM) furnace. Cast alloys were heat treated under Ar atmosphere in two steps: 1) 16 h at 1340°C followed by 2) 40 h at 870°C.
There were two samples prepared from each casting in as-cast and heat-treated state; circular and rectangular one cross-sectioned perpendicular to- and along the dendrite growth direction, respectively. The thickness of samples was in the range from 0.2 to 0.4 mm.

Synchrotron Investigation and Image Processing

Projections of dendritic microstructure were collected with the use of micro-Computed Tomography (μ-CT) technique. The scans were performed at the European Synchrotron Radiation Facility (ESRF) in Grenoble at the beamline ID19.
The samples were installed on a motorized stage and rotated vertically. The single projections of transmitted samples were collected with the use of a CCD camera. The rotation axis of the samples was set up to be in the middle of a CCD camera. Due to the high density of material caused mostly by refractory elements (Re, W), the energy of parallel electron beam was set to 65 keV. The effective image resolution was controlled by

the applied optics. There were two objectives used resulting in an effective image pixel size of 0.78 μm (high resolution – HR) and 5.02 μm (low resolution – LR). In each case the rotation range was set to 180° to reduce time of operation. The setup for high resolution objective resulted in 1500 projections collected every 0.12° (1500 x 0.12 = 180°). For the low resolution one there were 2000 projections recorded every 0.09° (2000 x 0.09 = 180°).
In each case there were also a flat field and dark field images collected, what allowed processing homogenous illumination of samples.

<u>Reconstruction of projections and image processing</u>

The projections were reconstructed in the software NRecon with the use of a Filtered Back Projection algorithm. This procedure resulted in obtaining a set of grayscale cross-sections through the material.
The real three-dimensional reconstruction of the volume and 3D volume calculations were performed with the use of FEI's Visualization Sciences Group AvizoFire software.
The obtained grayscale images exhibited a lot of common tomographic artifacts, like ring-artifacts. For their elimination the projections were processed using following filters. The 3D despeckle algorithm was used to remove the single pixels which had the values differing highly from their neighbors in three directions. Then the 3D median filter was used to further reduce the noise preserving the edges. Finally the adaptive histogram equalization algorithm was applied to obtain the homogenous grayscale distribution removing the residual illumination at the edges. Such prepared images gave the final 3D dendrite morphology and played as an input to reveal the pores. For this purpose the thresholding was applied to cut off the gray levels corresponding to dendritic morphology. This operation resulted in obtaining the binary images revealing only the porosity in the volume. The residual ring-artifacts observed by 3D inspection were deleted by tracking their coordinates in the volume.

2. Results

Representative images of the dendritic structure perpendicular to the growth direction are given in Figure 1a anb 1b, for the low- and high resolution, respectively. The results obtained in low resolution mode reveal more accurate morphology of dendrites, whereas the morphology of pores is better visualized when the high resolution objective was applied. In HR mode images seem to exhibit to much noise to give the reliable information about the dendrite morphology. In turn, the effective voxel size of 5.02 μm does not support obtaining the reliable information about the morphology and volume of pores. This is the reason why for the detailed study the scans of different resolution were used to visualize dendrites and pores.

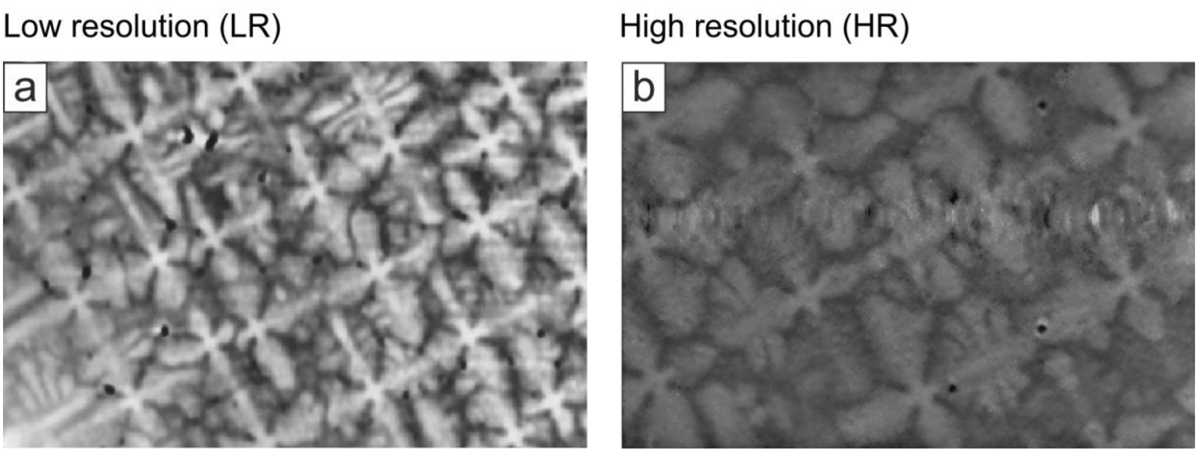

Fig. 1 Morphology of the dendrites on (100) plane obtained in a) low resolution (voxel size – 5.02 μm) and b) high resolution (voxel size – 0.7 μm) scanning

A representative image of the reconstructed microstructure is given in Fig. 2. Due to the selected level of grayscale thresholding the interdendritic regions were cut off to reveal the real 3D morphology of the dendrites. The total volume was decreased to 0.63 mm^3 in order to present the primary and secondary arms more precisely. The front plane is (100). In this view dendrites are shown along the growth direction [001]. On the left side of the scanned volume two dendrite cores are perfectly visible. This allows the quantitative analysis of the PDA and SDA spacing, as indicated by the narrows.

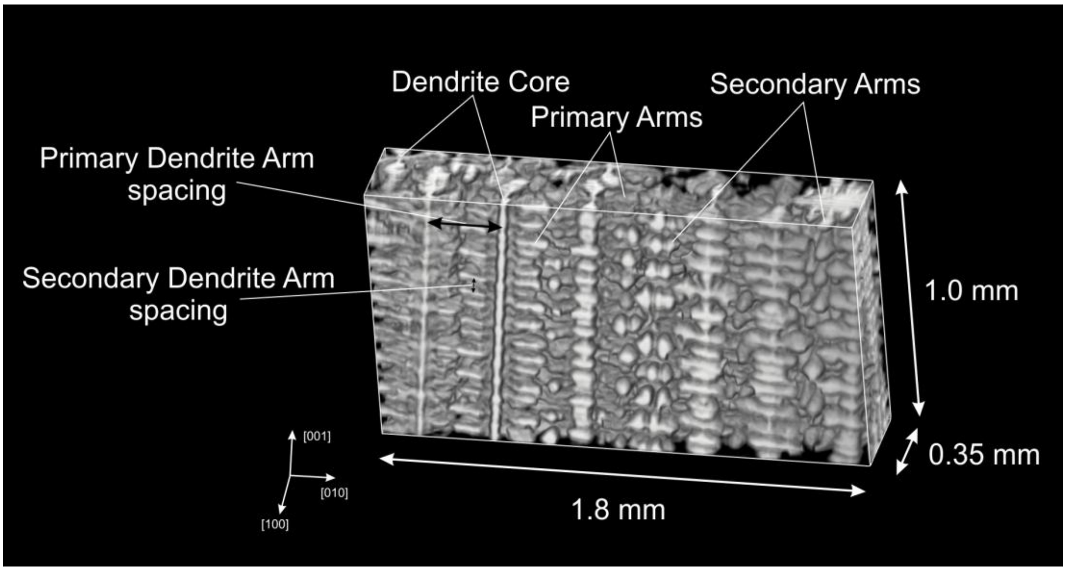

Fig. 2 The three-dimensional morphology of the dendritic structure obtained in a low resolution scanning. The front plane (100) shows the cross-sections through dendrite cores, primary and secondary dendrite arm spacing.

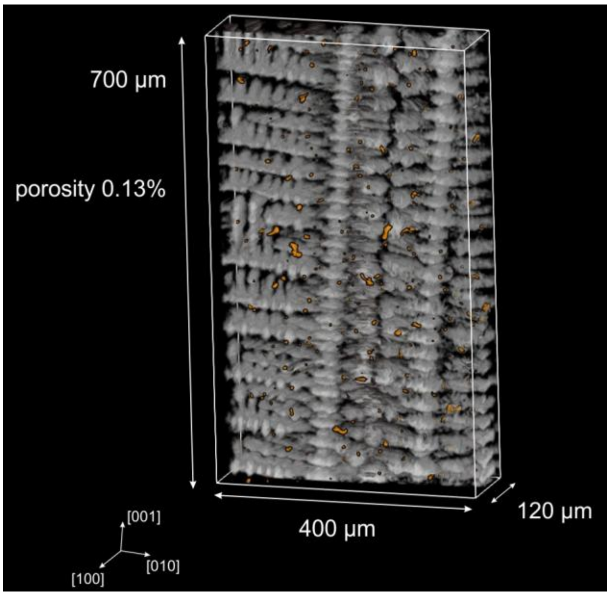

Figure 3 demonstrates the 3D distribution of pores within the dendritic structure. Dendritic morphology is imaged in gray and pores in orange color. As expected, pores are located in interdendritic areas.

The study on the pores morphology reveals that porosity in as-cast state consist of solidification pores (S-pores) formed during solidification in the mushy zone and gas porosity. The S-pores are much larger in size and exhibit irregular shapes depending on the morphology of already solidified dendrite arms. The gas porosity is mostly characterized by small pores of regular circular shape.

Fig. 3 Pores located in interdendritic areas; view in [001] direction.

3. Results

Current investigation demonstrate that synchrotron µ-CT is a promising technique for investigation of dendritic microstructure and porosity in single-crystal nickel-base superalloys in 3D. Results are especially important, as they allow not only qualitative but also quantitative characterization.