	Experiment title: Local x-ray surface grain mapping of niobium	**Experiment number**: HC-4065
Beamline: ID01	**Date of experiment**: from: 07/10/2020 to: 12/10/2020	**Date of report**: 25/02/2021
Shifts: 15	**Local contact(s)**:Ewen Bellec	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

This was a remote experiment, with only the local contact being present at the beamline.

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Report: We are engaged in a project which aims to optimizing the properties of niobium for its use in radiofrequency cavities, by controlled annealing procedures in different gas environments. The incorporation of oxygen and nitrogen in the Nb lattice and its influence on carbide and hydride formation has been identified as a key ingredient. **The aim of this experiment was to locally map the surface structure of individual grains and grain boundaries of niobium, using (sub-)micron resolution. Samples, pre-characterized concerning the exact grain shape and orientation in selected marked areas, were used.**

Polished polycrystalline crystals were characterized by SEM and Electron Backscatter Diffraction (EBSD) at Desy Nanolab. These microscopy techniques resulted in maps of grain orientation and designated areas marked by platinum. These samples were mounted on the ID01 diffractometer and the designated areas were found back by using the optical microscope and by mapping out the samples using the specularly reflected beam: due to absorption of the beam through the Pt markers, there is a relatively good intensity contrast. The results of finding the markers back at the beamline in shown in Figure 1.

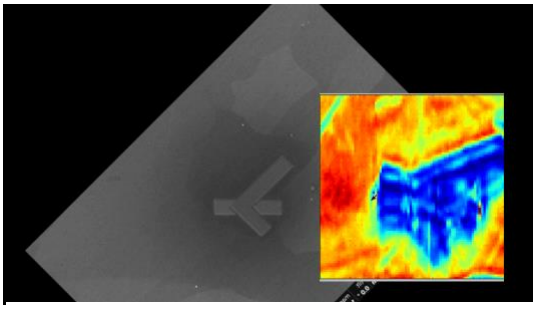

Fig. 1 Correspondance between the large Pt markers, imaged by the SEM and as resolved by x-rays at the beamline

Once the correlation with the marker structure between SEM and XRD was made, further nanoXRD measurements were taken in reflectivity geometry. Figure 2 shows a typical result of comparing the microscopy and grain orientation maps with a map obtained from the reflected x-ray beam at an angle of incidence of 0.6 degrees. Together with a

beamsize of 200x70 nm^2 (HxV), this gives a footprint of 200x7000 nm^2 (HxV) on the sample surface. Such a lateral resolution showed to be sufficient to resolve the individual grains and distinguish between the middle and border (close to grain boundaries) of the grains. It was noted that for even smaller angles of incidence the lateral position is not accurate anymore, due to the coupling between centre of rotation and sample height alignment.

The different techniques clearly allow us to distinghuish between the different grains. Details knowledge about the grain orientation is very helpful in further analyzing and interpreting the data and results. For example, it is clear that different grain orientations will on averag have different atomic surface structures. These can be closely packed or more open or be a combination of terraces and steps. The segregation behavior of dissolved species like N and O is expected to be different on these grains and at the grain boundaries.
Figure 3 shows detailed XRR curves obtained at different positions on the sample. The subtle but still significant changes from position to position allow it to obtain a better picture of the heterogeneity of the selvedge, which here consists mostly of a native Nb-oxide layer.

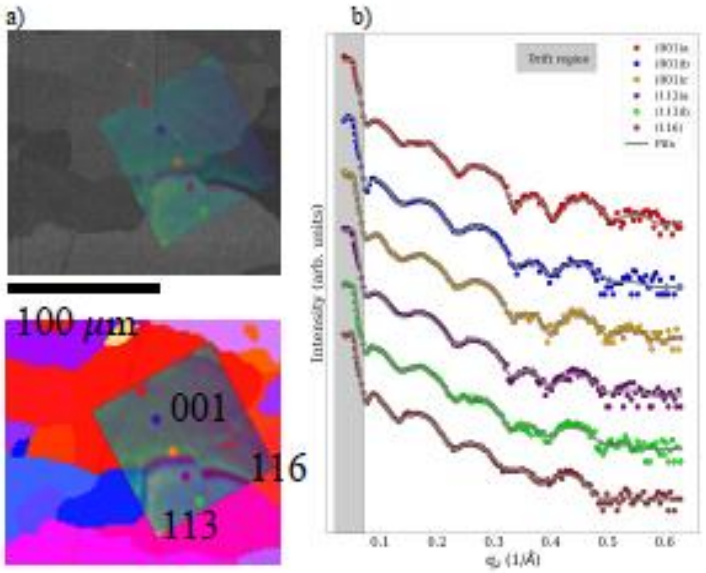

Fig. 3 a) shows the SEM of a selected region and b) the corresponding grain orientations. Overlayn in a transparent color is the region mapped out by nanoXRR. The colored dots in a) indicate the positions where full XRR curves were taken. The results in b) show these XRR curves and fits (lines) using the same color coding.

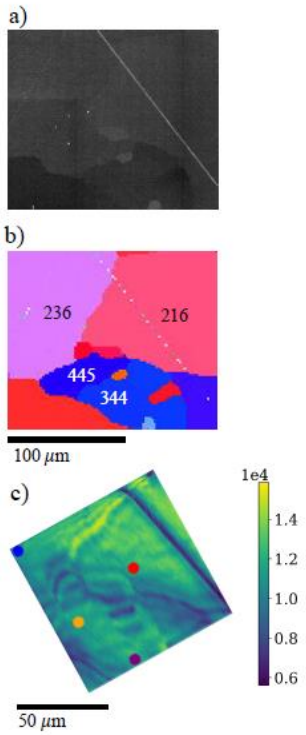

Fig. 2 Imaging of the same surface region using a) SEM, b) EBSD c) nanoXRR. Most clearly visible are a straight scratch on the surface and some of the grain boundaries. In c) the colored dots indicate the positions where full XRR curves were taken.

The most important information that we could have obtained from this experiment, is unfortunately still missing. The local changes after a controlled anneal in a pure nitrogen atmosphere were not measured. Due to a leak in the oven, the oxygen content was too high and the sample was severely oxidized after heating. This also increased the surface roughness considerably and prevented from obtaining further high quality XRR curves. This is a real pity, because all the other aspects of this challenging experiment worked out very well.

Conclusion

In principle a very successful experiment has been performed. The preparation of the markers, finding them back on the beamline and performing local XRR all worked. Also noteworthy is that everything worked as a remote experiment, thanks to the tireless efforts of the local contact. Therefore it is even more unsatisfying that the final goal, namely characterization before and after the anneal in nitrogen, was not reached. Since this issue deals with a technical detail concerning the furnace, we are very confident that in a future experiment, it is absolutely feasible to perform all the measurements.