	Experiment title: Local x-ray surface grain mapping of niobium	**Experiment number**: HC-4545
Beamline: ID01	**Date of experiment**: from: 24/11/2021 to: 29/11/2021	**Date of report**: 12/09/2022
Shifts: 15	**Local contact(s)**:Ewen Bellec	*Received at ESRF:*

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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.**
The initial proposal HC-4065 was granted beamtime and took place under pandemic measures as a remote experiment. Unfortunately, during that experiment the annealing step didn't work. The present proposal was a resubmission, based on the previous experimental difficulties.

Main results
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 correlating the EBSD maps with the Kmaps recorded at the beamline are summarized in Fig 1. There are also clear changes in the maps before and after the annealing.

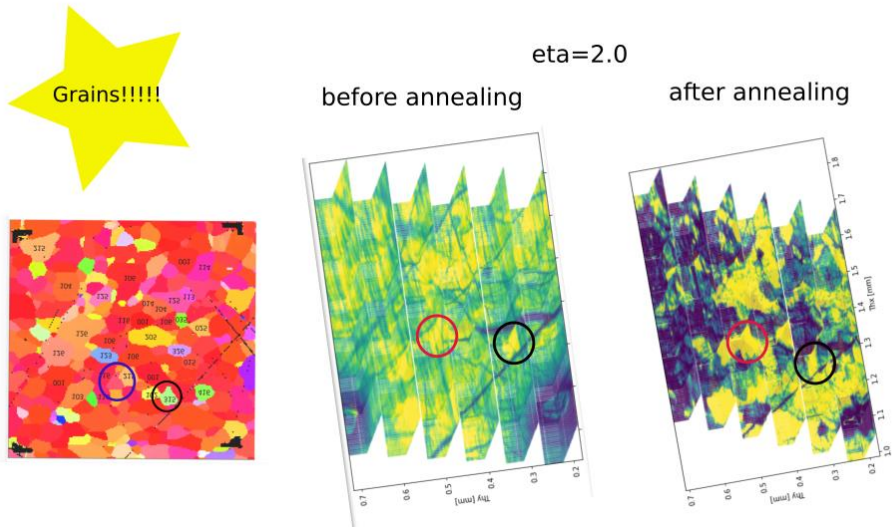

Fig 1. Correlation between EBSD and diffraction maps. Two characteristic grains, with 126 (red circle) and 213 (black circle) orientations are xclearly identified. Compared to previous experiments, a relatively large non-linear disortion of the xy-plane of the piezo arose (Kmap), which results in the particular reconstructed maps.

Complete XRR curves at the selected positions, approximately 10, yield very good data for detailed fitting of electron density profiles, as seen in Fig. 2 for the 213 oriented grain.

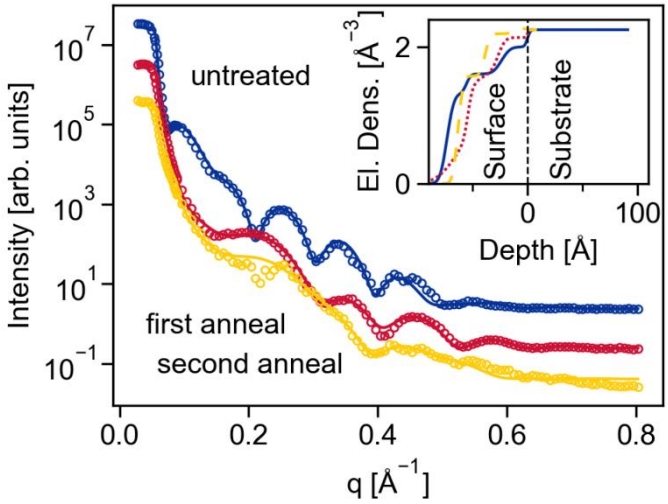

Fig 2. XRR curves taken from a 213-oriented grain before and after annealing. The data clearly show a reduction of the oxide thickness.

Conclusion

A very successful experiment has been performed. The preparation of the markers, finding them back on the beamline and performing local XRR before and after annealing all worked. Currently we are finalizing the data analysis and summarizing the results for a publication.