	Experiment title:	Experiment
Beamline: ID01	**Date of experiment**: from: 12/04/2023 to: 17/04/2023	**Date of report**:
Shifts: 15	**Local contact(s)**: Edoardo Zatterin	*Received at ESRF:*

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Report: We are engaged in a project which aims to optimize 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 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 interstitial oxygen concentration, using diffuse scattering, of individual grains and grain boundaries of niobium.**
The strategy that we follow for these kinds of experiments is as follows: in our home lab, samples are characterized by EBSD, from which the grain orientation is obtained. Then certain areas are selected, by writing Pt markers on the samples. These Pt markers can be found back at the beamline by scanning the sample and measuring their diffraction signal. Once the area of interest is found, the sample needs to be locally aligned to perform XRR and to put in the right geometry to measure a certain *hkl* point.

Main results

-Pt-marker area and correlation with EBSD
As in our previous experiments at ID01, it was quite straightforward to find the markers. Mapping out a region using a reflectivity contrast, made it possible to identify individual grains (see Fig 1.)

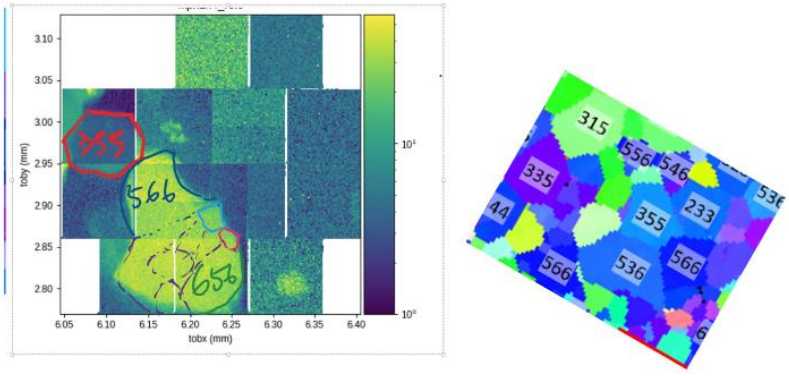

Fig 1. Correlation between EBSD and diffraction maps. A characteristic grain with a 566 orientation is crearly identified.

-Radiation damage
When starting to search for the diffuse scattering signal, the sample was kept in air without mounting the PEEK dome in order to prevent unwanted scattering influencing the measurement. However, under these circumstance, severe radiation damage effects were observed. Fig. 2 shows XRR curves taken sequentially at the same sample position. Clearly, a thicker layer, probably oxide, grows under these conditions. By using an Ar flow and attenuators, no radiation effects were observed anymore.

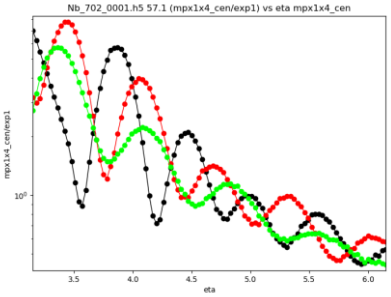

Fig 2. Reflectivity measurement on the same spot demonstrating a local change of the surface as a result of beam damage.

-Sample alignment for GI geometry

A particular spot on the sample needs to be placed in the incident angle rotation axis. A complicating factor to achieve this, is given by the sample surface and lateral sample positioning motor directions (Piezo table and hexapod) not being parallel to each other. As a result, while moving the sample around, the spot of interest very often is not in the rotation centre anymore and reliable use of the GI geometry is not given anymore. This effect can be different every time a sample is mounted and in our previous beamtime it appeared not as severe as this time.

We established a correction routine, which helps in placing the sample such that reflectivity is measured at the same spot. The lateral sample position (toby) for different angles of incidence (eta) was determined at which a particular Pt marker was in the beam. Fig 3 shows the result, together with an analytical correction function based on the geometry. XRR curves were now measured by explicitly correction the sample position while changing the angles. The results is also shown in Fig 3.

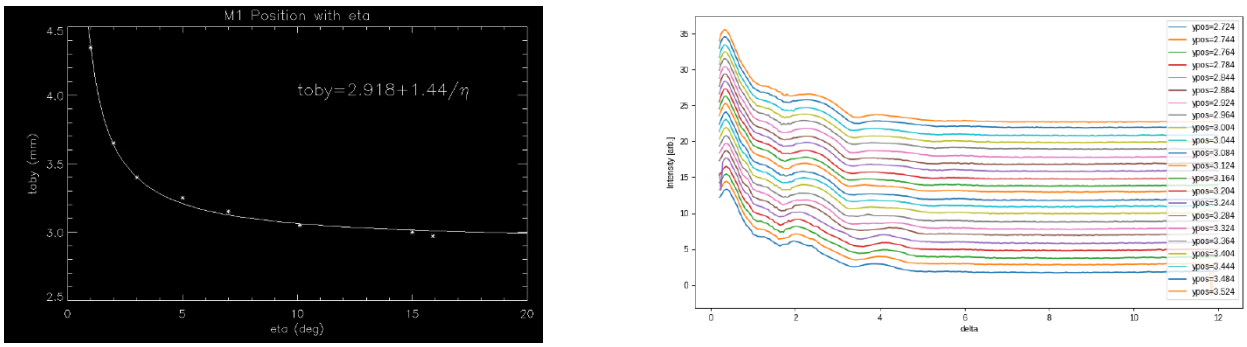

Fig 3. (left) Lateral sample position where a Pt marker was found vs the angle of incidence (eta) . (right) Reflectivity measurements with obatined with this correction, assuring that the curve is taken at the same sample spot.

-Moving through reciprocal space

A crucial issue for the experiment is to be able to find the right positions in reciprocal space. Unfortunately, with the beamline's control software changing from SPEC to BLISS, the hkl operation mode was not available anymore. The work-around was to use an off-line version of SPEC (PSIC) from our own lab and use that to calculate the motor positions and perform scans along certain trajectories. Although it was straightforward to find 2 Bragg peaks and set up an orientation matrix, a lot of time was then lost due to 2 issues. First of all, there appeared to be a large software issue deep in BLISS, which prevented the use of a3scans. This was also a problem for the XRR measurements, which needed to be done while correcting the sample position (see previous point). Approximately 2 shifts were lost due to the software problem. Then, it turned out to be difficult to verify whether the geometry (especially sense of rotation directions) of the ID01 diffractometer and our own PSIC version were identical. This issue also led to loss of beamtime and would be best addressed in a commissioning round. Nevertheless, distinct diffuse scattering peaks (see Fig 4.) have been measured and the exact hkl-labeling is ongoing.

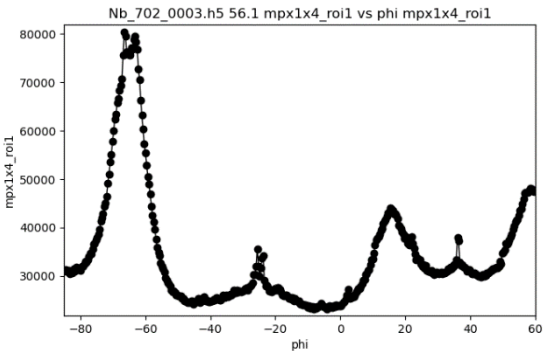

Fig 4 Phi scan through 112 Bragg peak (phi ~-65) and several diffuse scattering peaks (phi ~-25, 18)

-Conclusion

Due to a combination of several issues, related to beam damage, sample alignment and beamline control software, we have not been able to reach our goal and measure all the relevant data. We have established a way to perfrom these measurements and established the feasibility. We have also obtained local XRR data, from individual grains with known orientation. These results are important in view of our project concerning niobium and for testing the whole overall new measurement method for polycrystalline sample surfaces.