ESRF	**Experiment title:** In situ investigation of the interaction of oxygen with Nb surfaces by surface sensitve x-ray diffraction	**Experiment number**: SI-1149
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Report:

The aim of this experiment was to study the interaction of dry and wet oxygen with Nb(110) single crystal in the near surface region as a function of oxygen pressure and sample temperature. We wanted to get detailed information about the oxidation mechanism of Nb as well as the role of sub-interfacial oxygen for the oxide growth, by combining three different in-situ surface sensitive techniques: grazing incidence measurements of the diffuse scattering originating from the interstitial oxygen monitored by a 2D detector, x-ray reflectivity and crystal truncation rods (CTR) measurements which would allow us to follow the evolution on an atomic scale of the metal/oxide interface structure.

We successfully performed, during the beamtime, in-situ and simultaneous measurements of the diffuse scattering with the 2D detector and the reflectivity measurements for three different conditions of oxidation and thermal treatments from room temperature up to 2273K. We successfully ran the new high temperature setup installed on the diffraction chamber allowing temperature up to 2273K. Depth-resolved diffuse scattering linescans have as well been measured by tuning both incident and exit angles reaching a region from ~2.5nm up to ~2000nm with a nm resolution. Unfortunately, the quality of the sample appeared not to be good enough to get reliable results due to damages that occur during the first step of its preparation.

Prior to the experiment, the Nb(110) single crystal has to be annealed at 2500K under UHV conditions in order to remove all oxygen contaminations from the bulk. The sample is afterwards exposed to air at room temperature in order to grow few nm of a quasi-amorphous Nb oxide layer under atmospheric conditions and then mounted into the in-situ surface x-ray diffraction chamber. During the annealing process, the sample has been partially damaged when the temperature reached in the system seems to have been too high for a moment. Among others, small droplets visible on the border of the surface (Figure 4) appeared and gave rise to a huge background in the region where the diffuse scattering should be measured as one can see clearly by comparing Figure 1 and Figure 2. This background make a quantitative analysis of the oxygen induced diffuse scattering impossible.

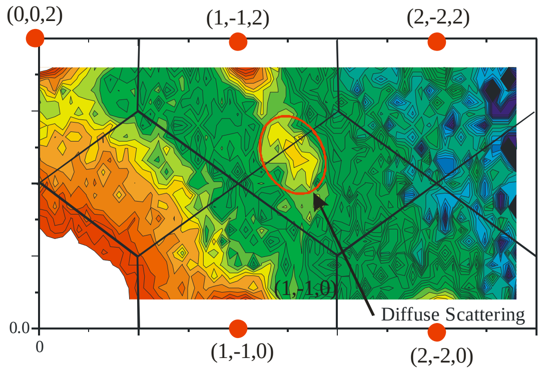

Figure 1 Reciprocal map of the diffuse scattering induced by interstitial oxygen in the Nb lattice (measured at ANKA)

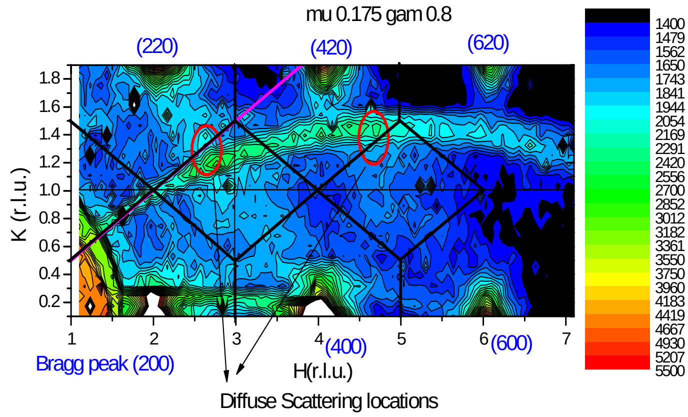

Figure 2 Background originating from droplets on the surface of the sample.

The in-situ reflectivity measurements performed allowed us to follow the evolution of the different oxides layers during the successive thermal treatments or oxidation conditions applied on the sample. In Figure 3, reflectivity measurements are plotted obtained after successively heating the sample previously oxidized in air from room temperature to 200°C and then to 300°C. The reflectivity measurements show the progressive dissolution of the oxide layer in different steps. Afterwards, the sample has been annealed at 2273K in order to clean the surface and to follow after different conditions of oxidation another sequence of thermal treatments. Three different sequences (air oxidation, 4.10^{-4}mbar and ~200mbar of dry oxygen) could be performed after annealing to 2200K and for each sequence heating from room temperature to ~350°C has been investigated.

The CTRs measurements which would have allowed us to analyze the changes in the interfacial structure between the natural Nb oxide layer and the Nb bulk during the different steps of the program have not been possible also because of the damage of the sample after the annealing process.

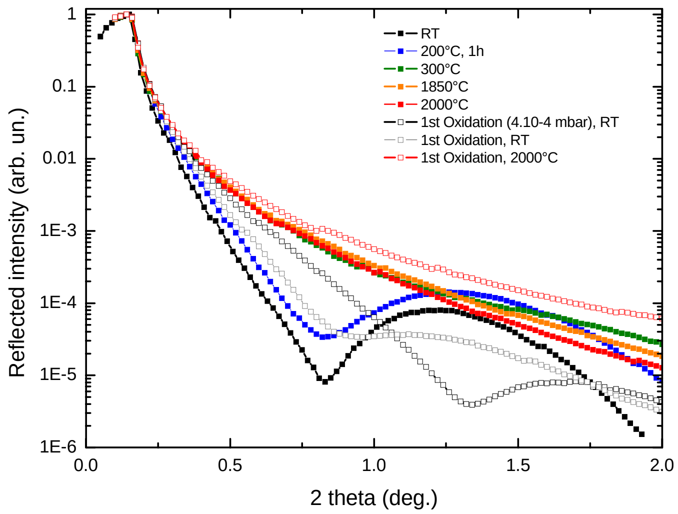

Figure 3 In-situ reflectivity measurements showing a sequence of thermal treatments applied on the sample oxidized in air, and oxidized in dry oxygen after annealing at 2273K.

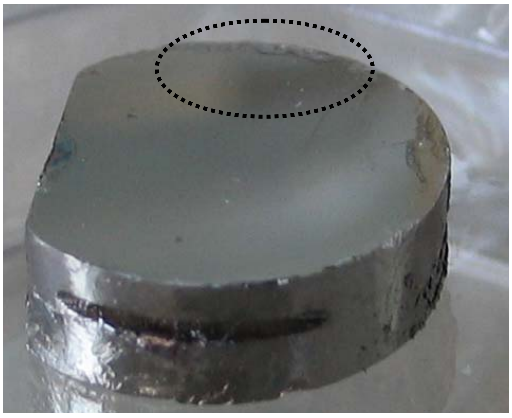

Figure 4 Some of the molten droplets on the border of the surface due to too high temperature during the annealing process