ROBL-CRG	Experiment title: <i>Anisotropic Deformation of Nb Films during H Load</i>	Experiment number: 20_02_004
Beamline: BM 20	Date of experiment: from: 15.04.1998 to: 22.04.1998	Date of report : Mai 1998
Shifts: 16	Local contact(s): Dr. Norbert Schell	<i>Received at ROBL:</i> 01.06.1998

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Report:

Some metals like niobium can dissolve large amounts of hydrogen, up to concentrations of one H atom per Nb atom. Hydrogen is thereby located in the Nb host lattice on tetrahedral interstitial sites and expands the lattice isotropically. The resulting long range displacement field causes an indirect elastic interaction between the H atoms. In thin Nb layers on sapphire, elastic displacements are influenced by the film surface as well as by the interface between film and substrate. It is well known that for low H concentrations thin epitactically grown Nb films will expand only perpendicular to the surface. With higher loading pressures a lateral anisotropic lattice expansion will also result.

The aim of our measurements at ROBL was to observe the cross over from one dimensional to three dimensional lattice expansion by measuring the lattice parameters under in-situ conditions and to determine the degree of anisotropy of the lattice expansion in dependance of the H content.

The lattice parameter in the growth direction is measured by conventional Bragg diffraction; the lateral lattice parameters are accessible under grazing incidence and exit angles (GID). Specular reflectivity under small angles shows the change in film thickness as well as interface roughness at the cross over from one to three dimensional expansion.

Previous measurements showed that loading the sample with hydogen causes an irreversible change in its morphology. Thus, it is absolutely necessary to keep the sample in a constant environment and to allow all three measuring modes (Bragg, GID, reflectivity) at the same time. In order to achieve those demanding conditions a specially adapted UHV chamber for the ROBL six-circlediffractometer was constructed. It allows in-situ H loading without restricting the necessary diffractometer's degrees of freedom. Two sample systems with different Nb film thicknesses were available. Both were covered with a protecting layer of palladium appr. 100 Å thick. The samples were exposed to H pressures from 0.1 to 50.0 mbar at temperatures between 200 °C and 300 °C. The Nb films were (110) oriented. With a scintillation counter angular and radial scans were measured around the Bragg reflexes (110), (110), (112) and (002).

The data evaluation is not yet complete. However, the following figures for the appr. 750 Å thick Nb film show as typical examples the changes of the data curves without H atmosphere and with 50 mbar H atmosphere.



Figure 1.

Figure 2.

Figure 1 to the left shows the Bragg measurement in radial direction around Nb (110). The H induced shift of the Bragg reflex corresponds to a relative lattice expansion of 4 %. While the unloaded sample shows so called Laue fringes (which serve as a measure for the crystalline quality of the film), they are no longer recognizable in the loaded sample. Figure 2 to the right shows the change in specular reflectivity after H loading and thereby reveals the change in film thickness.



Figure 3.

Figure 3 above shows the lattice expansion in two lateral crystallographic directions, vertically oriented to each other (GID data). The relative lattice expansion in $(1\underline{1}0)$ is 2.4 %, in (002) direction 2.8 %.