 ROBL-CRG	Experiment title: Characterization of structure and morphology of buried layers of transition metal oxides	Experiment number: 20_02_613
Beamline: BM 20	Date of experiment: from: 23.07.2003 to: 26.07.2003 and from: 22.11.2003 to: 24.11.2003	Date of report: 15.01.2004
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Report:

Thin buried layers of molybdenum oxide have received great attention due to their possible use as protective layers, insulators, optical coatings or sensors [1, 2]. However, up to now, the structural phase formations in such buried implanted layers were not investigated by *in-situ* XRD analysis during implantation and annealing, thus yielding no direct information on the kinetics.

The polycrystalline molybdenum samples were implanted with oxygen ions at an energy of 1.5 MeV up to a fluence of $1.6 \times 10^{18} \text{ O}^+\text{-ions/cm}^2$ without additional heating. The pressure in the implantation chamber was $\leq 10^{-6}$ mbar. A rise in the target temperature of $\sim 300^\circ\text{C}$ was observed during the implantation process due to ion beam heating.

The first part of the ROBL experiment was aimed at the *ex-situ* XRD characterization of structure and morphology of the thin oxide layers by the high intense synchrotron radiation. In the second part the structure changes in the buried molybdenum oxide layer were studied by *in-situ* XRD during annealing.

The XRD patterns of the *as-implanted* Mo samples show that ***by high dose implantation the MoO₂ precipitates have been formed in the molybdenum substrate***. The structure evolution of MoO₂ phase during annealing was studied by *in-situ* XRD analysis in Bragg-Brentano geometry. Up to 500°C the pattern remains unchanged, but with increase of the temperature the intensity of the MoO₂ peaks grows. This indicates the ***formation and growth of the crystallites***. ***After annealing at 600°C (1 h) the MoO₂ crystallite size is about 8 nm*** as calculated by the Scherrer formula.

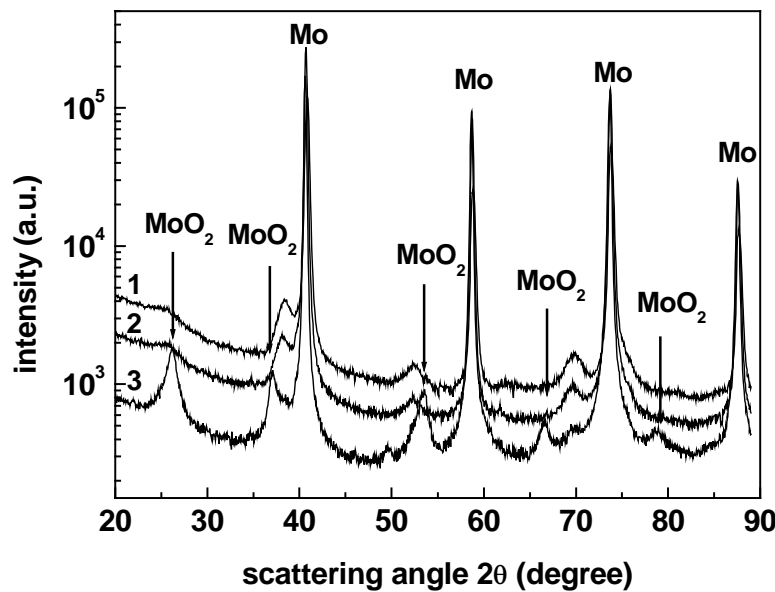


Fig. 1: Synchrotron radiation XRD pattern of the O⁺-implanted and post-annealed Mo sample recorded at the incidence angles of 0.5° (1), 1° (2), 4° (3) and wavelength of 0.154 nm.

The *ex situ* recorded XRD patterns (Fig. 1), taken under different grazing incidence, reveal the generation of a ***buried layer and MoO₂ phase formation after implantation and annealing***. In the scans taken at 0.5 and 1° incidence angle the penetrating radiation reaches only the near surface region and MoO₂ was not detected, but for incidence angle of 4° the x-rays penetrate deeper (1/e-depth ~500 nm) and the occurring strong reflections come from a buried MoO₂ layer.

The evaluation of the *in-situ* annealing measurements is not yet completed.

References

- [1] C. Hammerl, B. Rauschenbach, Nucl. Instr. and Meth. B **178** (2001) 220.
- [2] C. Hammerl, B. Renner, B. Rauschenbach, *et al.*, Nucl. Instr. and Meth. B **148** (1999) 851.