Phase Formation in Mo and Ti after High Dose Nitrogen Implantation

J.W. Gerlach [a], D. Manova [a], H. Neumann [a], S. Mändl [a], B. Rauschenbach [a], W. Assmann, F. Grüner, and M. Schubert

[a] Leibniz-Institut für Oberflächenmodifizierung Leipzig

The surface modification of modern metal alloys is a very urgent problem with great technological significance as the material composition of components is mainly determined by the bulk properties. Hence, increased wear and surface hardness have to be accomplished posteriorly. High temperature, high dose nitrogen implantation is one convenient way for obtaining such goals. However, fundamental information on phase formation and diffusion rates is missing for most cases.

In the present experiment, plasma immersion ion implantation (PIII), where positive ions are accelerated from a plasma by applying negative high voltage pulses on the substrate, was used for nitrogen implantation into Ti and Mo substrates. Subsequent ERDA measurements were performed with the position sensitive ionization chamber in the ERDA beam line using 200 or 210 MeV Au ions.

Pure samples (99.5%) were polished to a mirror-like finish and treated in a HV system at a base pressure of 2×10^{-6} mbar and a working pressure of 3×10^{-3} mbar with nitrogen PIII. High voltage pulses of 15 kV were used. Additional heating by IR lamps was used to reach elevated temperatures up to 750°C. The final implanted dose was 2-4$\times10^{18}$ atoms/cm^2.

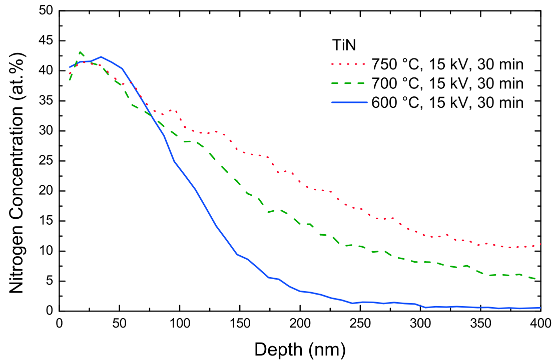

Fig. 1: Nitrogen depth profiles for high temperature, high dose implantation into Ti.

The depth profiles are composed of two parts, a surface layer with a constant nitrogen concentration, followed by a deep diffusion tail extending beyond the depth accessible by ERDA (see Fig. 1). The surface concentration was between 25 and 35 at.% for Mo and about 40 at.% for Ti. The surface layer was always thicker than the ion range, indicating an additional growth process of this layer. All profiles indicate a thermally activated inverse parabolic growth law. An activation energy for the nitrogen diffusion toward the bulk of about 0.8-1 eV was obtained for both materials. However, the prefactor D_0 is lower for Mo by one order of magnitude. Thus, the layer thickness will always be lower for Mo than for Ti by a factor of 3.

However, strong differences between the two investigated systems were found with X-ray diffraction. In Mo, the formation of Mo_2N was observed across the whole temperature range from 300 up to 750°C (see Fig. 2), corresponding to the observed nitrogen/molybdenum ratio. Albeit, a transition from the cubic Mo_2N to the tetragonally distorted Mo_2N occurred near 600°C. This can be clearly seen by the asymmetric peak splitting near 45° and 65°. This is a kinetic transition from the metastable toward the stable tetragonal phase, induced by a higher mobility of the atoms in the lattice [1].

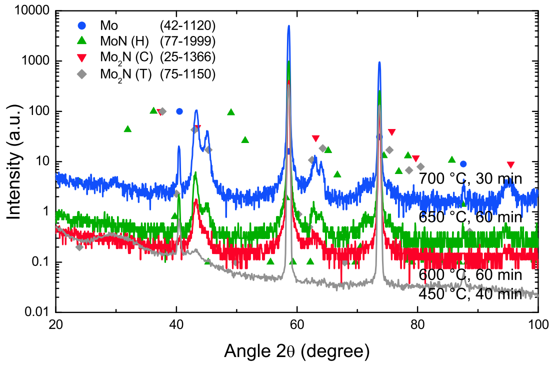

Fig. 2: X-ray spectra for Mo implanted with nitrogen at different temperatures. Symbols mark peak position of different phases.

In contrast, a completely different situation is observed for nitrogen in titanium. Beyond 500°C, no more TiN could be detected despite the same surface concentration, while this phase was observed for 500°C and below (see Fig. 3). Apparently, the higher (thermal) mobility induces no phase formation. The dominating process is diffusion, coupled with a lattice expansion, as can be deduced from the left shoulders appearing on the Ti substrate peaks.

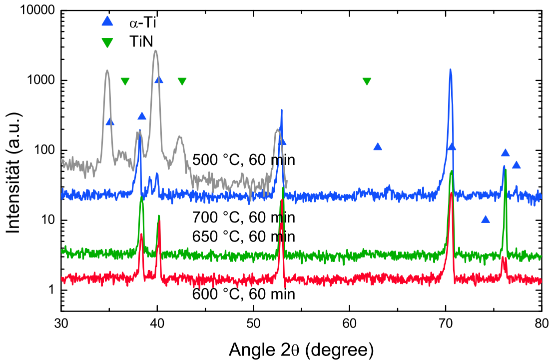

Fig. 3: X-ray spectra for Ti implanted with nitrogen at different temperatures. Symbols mark peak position of different phases.

References

[1] D. Manova *et al.*, EMRS Spring Meeting, Strasbourg, 2003