ROBL-CRG	**Experiment title:** **Depth resolved XRD analysis of nitrogen implanted Ti-6Al-4V alloys**	**Experiment number**: 20_02_031(c)
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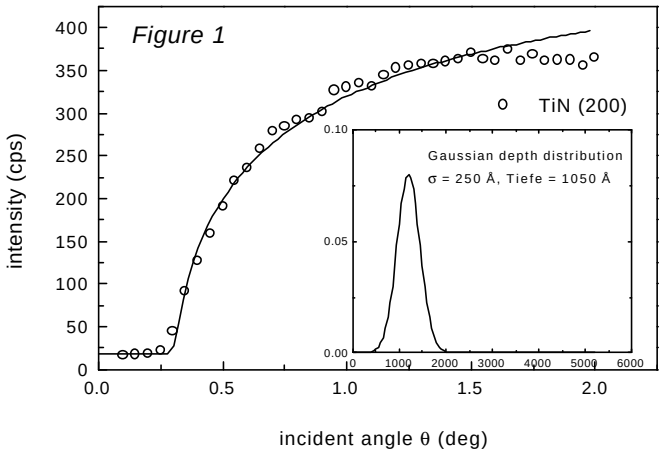
Report:

With the x-ray diffraction analysis adapted for grazing incidence diffraction (GID) geometry it is possible to investigate depth distribution of crystalline phases. It is based on the changes the average penetration depth of the x-ray beam under variation of the incident angle ($_i$). However, it is not easy to detect from such experiments the real depth distribution of a crystal structures. The scattering volume is not well defined because of the exponential attenuation of the x-ray intensity in the sample. The scattering is accumulative, so the signal from the surface will be present at all greater $_i$. A difference method is not applicable because of different absorption coefficients. To find the real phase distribution the theory of the standing wave field was used. The scatterted intensity is given by:

$$\mathrm{I}(a_i) = \mathrm{I}_0 \int_{-\infty}^{0} \left|E_t(r,k)\right|^2 g(z)dz$$

Were I_0 is the normal intensity, E_t is the electrical field calculated from the standing wave field [1] and $g(z)$ is the distribution function of the scatterers. Thus by fitting $\mathrm{I}(\ _i)$ with a model function $g(z)$ to the measured values of a certain sample the depth distribution can be derived.

The aim of the experiment was to show the different phase distribution of TiN and Ti_2N in the nitrogen implanted Ti-6Al-4V alloys. The used samples were implanted with ion beam implantation technique at different energies (from 20 keV to 180 keV) and different fluences (1 - 6)x10^{17} N^+/cm^2. So, it was possible to compare in the GID implantation depth profiles of 100 up to 200 nm. Further samples were implanted by plasma ion immersion (PII) with a



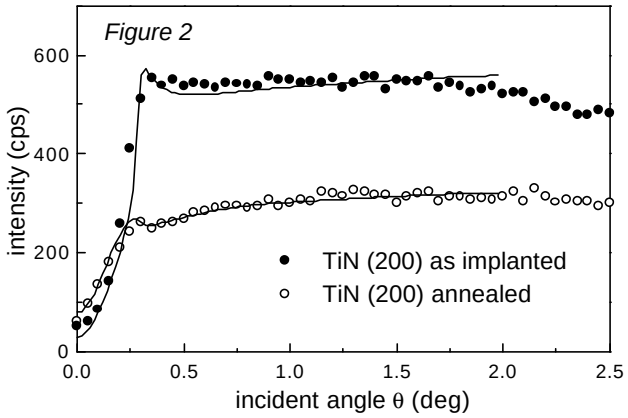
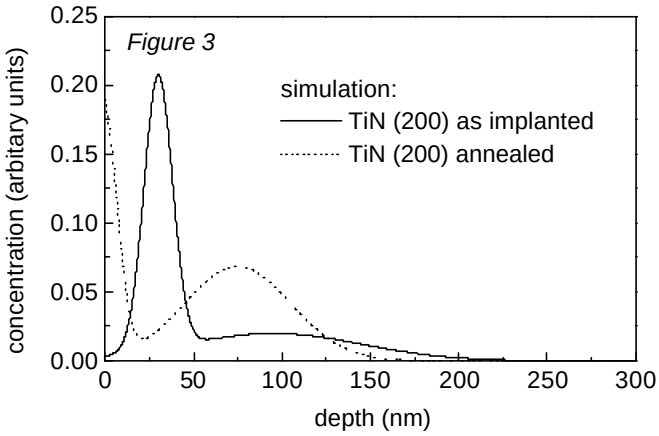


Figure 1: *measured and simulated depth distribution of TiN (200) reflex of a ion beam implanted Ti-6Al-4V sample. Implanted fluence: 6x10^{17} N$^+$/cm^2 with an implantation energy of 80 keV*

Figure 2 *measured and* ***Figure 3*** *simulated depth distribution of TiN (200) reflex of a PII implanted Ti-6Al-4V sample. Implanted fluence: 2x10^{17} N$^+$/cm^2 with an implantation energy of 40 (20) keV.*



fluence of 2.0x10^{17} N$^+$/cm^2. With this technique nitrogen is deposited only very near to the surface.

All samples were studied in the as-implanted and in the annealed state. For all samples the depth profile of characteristic Bragg reflections were recorded by varying the incident angle form 0° to 2° at a wave length of = 0.154 nm.

The result for a ion beam implanted sample in the as-implanted state is given in Figure 1. The simulation shows the maximum of the Gaussian depth profile at a depth of 135 nm. Former investigations with the elastic recoil detection analysis [2] have indicated that the maximum of the nitrogen concentration lies at a depth of 146 nm with also a Gaussian depth profile.

Figure 2 depicts the result for a PII implanted sample in the as implanted state and after annealing at 680°C for 2 h. Both curves show in principle the same behaviour: the occurance of a resonance peak near the critical angle of Ti-6Al-4V of $_{crit}$= 0.3°. For this example it is only possible to get a well adapted curve if two Gaussian distributions are used as it is shown in Figure 3. One at the surface, the second one in the deeper region of about 80 nm. The ratio of the two normalized concentrations give a hint to a starting diffusion process and beyond to the phase formation in deeper regions.

[1] D. Windt, Computers in Physics, IMD--Software for modeling the optical properties of multilayer films: 12 (4) (1998), 360

[2] F. Berberich, W. Matz, E. Richter, N. Schell, U. Kreißig and W. Möller, Surface and Coatings Technology, **128-129** (2000), 450-454