ROBL-CRG	**Experiment title:** **Structural changes during annealing processes of PII implanted Ti64 alloys**	**Experiment number**: 20_02_031(a)
Beamline: BM 20	**Date of experiment:** (long term) from: 26.01.2000 to: 07.03.2001	**Date of report**: 04.04.2001
Shifts: 12	**Local contact(s)**: N. Schell	*Received at ROBL:* 15. 4. 2001

Names and affiliations of applicants (* indicates experimentalists):

*# F. Berberich, *+W. Matz, *#N. Schell, +E. Richter

+Forschungszentrum Rossendorf, Institute of Ion Beam Physics and Materials Research, P.O. Box 510119, D-01314 Dresden, Germany

#ROBL – CRG at ESRF

Report:

The surface hardness of the titanium alloy Ti-6Al-4V, widely used as construction material, may be improved by nitrogen implantation [1]. Plasma immersion ion implantation (PIII) produces a nitrogen profile directly below the surface. In-situ X-ray diffraction experiments were performed using a high-temperature diffraction chamber. At the wavelength of 0.154 nm grazing incidence technique was applied. By varying the angle of incidence from 0.5° to 4° depth dependent structural information is obtained from 0.1 to 1 µm assuming an average density of 4.43 g/cm^3 for Ti-6Al-4V.

In order to characterise the structural changes of different PIII samples during annealing, which should cause the increased hardness, in-situ XRD experiments were performed. Ti-6Al-4V samples implanted with a fluence of 1.7x10^{17} N^+/cm^2 were used. The scanning time for each diffraction pattern was 30 min. So the duration of the in-situ experiment corresponds to the typical annealing time used for such alloys.The spectra measured at incidence angles of 1° are most sensitive to the surface layer and show the formation of the TiN phase much clearer than those ones received at higher incidence angles. Figure 1 depicts the spectra at the incidence angle of 1° for RT (before and after annealing) and at the various annealing temperatures.

At 500°C the pattern consists of the Bragg peaks of both Ti phases (α + β) and TiN formed by implantation. The TiN peaks are very broad, indicating a small crystallite size of about 10 nm calculated with the Scherrer formula. The lattice constant of TiN was determined to a = 0.4217 nm. This value is -0.7% different from the value for

the stoichiometric phase [2] which may indicate a nitrogen deficit. With increasing temperatures the intensity of the TiN peaks decreases. In Figure 1 this can most clearly be observed for the (200)TiN peak at 2θ of 42.6°. At 750°C, however, a new peak appears at slightly lower Bragg angle than the TiN peak observed so far. It remains in the pattern after the annealing process when cooling the sample to RT. At isothermal time dependent experiments it was found, that this peak does not arise from a peak shift, but it is growing only after the disappearence of the original TiN peak. The two other peaks of low intensity, marked with '▼', which belong to the pattern are also observed. After annealing the lattice parameter of the phase TiN* is slightly different from the initial one and amounts *a* = 0.428 nm.
The start of Ti_2N formation is observed at temperatures between 600 and 650°C. At the final temperature of 750°C, as well as at the RT measurement after the annealing process, the Ti_2N peaks are clearly detected. These peaks are much sharper than the former TiN peaks, indicating crystallites of Ti_2N with average sizes of 35-40 nm.
The data were correlated with hardness, ERDA, and SEM results [3].

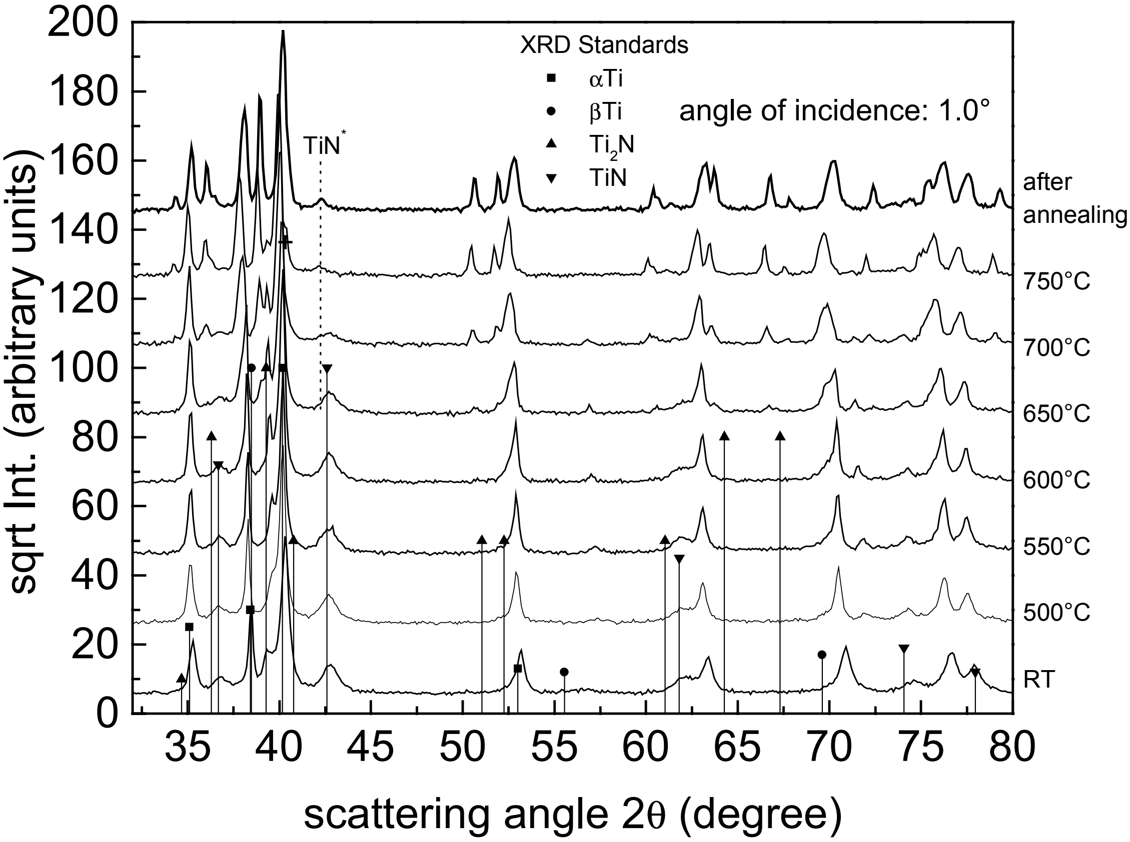

Figure 1: XRD spectra recorded at the angle of incidence of 1° during in-situ annealing of nitrogen implanted TiAlV alloy using PIII

[1] F. Berberich, W. Matz, E. Richter, N. Schell, U. Kreißig and W. Möller, Surface and Coatings Technology, **128-129** (2000), 450-454
[2] Powder diffraction file PDF-2, No. 38-1420; ICDD, Newton Square, PA 19073-3273, USA
[3] F. Berberich, W. Matz, U. Kreißig, E. Richter, N. Schell, and W. Möller, Appl. Surf. Sci. (2001) in press (Proc. AOFA 11)