ROBL-CRG	**Experiment title:** *in-situ* temperature dependent measurements of phase transformations in N-implanted Ti6Al4V alloys	**Experiment number**: 20_02_019
Beamline: BM 20	**Date of experiment**: from: 9.5.99 to: 10.5.99 19.6.99 22.6.99	**Date of report**: 26.08.99
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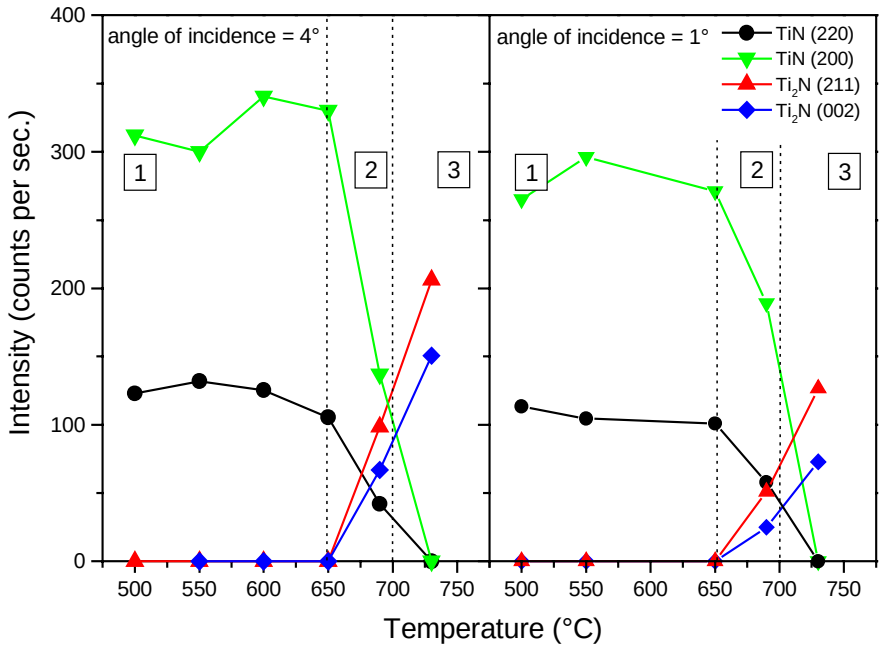
Report (first part):

The experiments are part of a general work to investigate the structural mechanisms of the enhanced hardness of the technical alloy Ti-6Al-4V due to N^+ implantation and its degeneration. Samples were implanted with doses (1 - 6) x 10^{17} N^+/cm^2 at 80 keV. The structural implications of the loss of hardness during annealing were studied by in-situ XRD-experiments with synchrotron radiation at a sample implanted with a dose of 6x10^{17} N^+/cm^2. Additional sample characterisation was performed by SEM, ERDA and Vickers hardness.

The as-received sample shows only Bragg peaks of the hexagonal α – phase, which is the main phase, and peaks of the cubic β-phase. After the ion implantation we observe the $TiN_{0.3}$ at first and then the TiN Bragg peaks. The intensity of these peaks show a direct correlation to the implanted doses. The Bragg peaks of both nitride phase are significantly broadened. From the line broadening a mean crystalline size of 5 nm was estimated. It was concluded that the observed hardening of the alloy is of precipitation type rather than the formation of a TiN layer.

The in-situ experiment with a Ti-6Al-4V sample implanted with a dose of 6 x 10^{17} N^+/cm^2 was performed at ROBL with a high temperature diffraction chamber. The investigated temperature region was from 500°C to 750°C in steps of 50K. At each temperature two X-ray patterns were recorded; for an angle of incidence of 1° (approximate penetration

depth of 200 nm) and another one for 4° (up to 1000 nm depth). 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 these alloys.
At 500°C the pattern consists of the Bragg peaks from both Ti phases and from TiN formed by implantation. With increasing temperature the intensity of the TiN peaks decreases. The formation of Ti_2N begins at a temperature between 650°C and 690°C. At the final temperature of 730°C all TiN peaks have completely vanished and only Ti_2N peaks are observed besides the Ti-phases. From a plot of the integrated intensities for TiN and Ti_2N in Fig. 1 it can be seen, that this phase transformation is a continuous process.



ing in-situ annealing

th the α-Ti and β–Ti
g.2 . Especially, the
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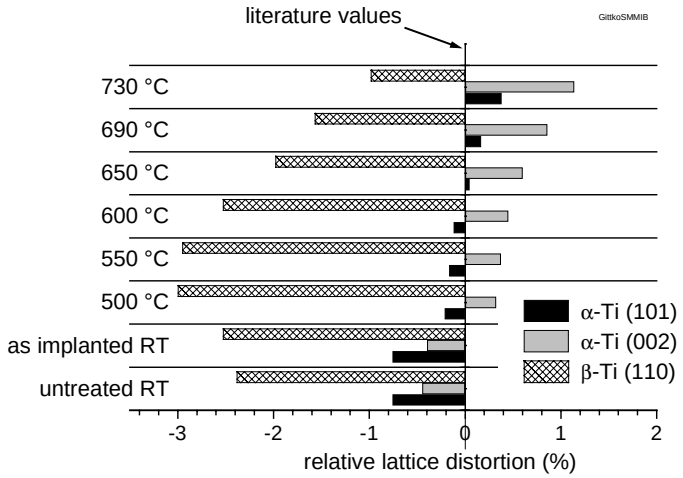
ealing is connected
with a reduction of the V-content in the β-Ti. This is suggested as an additional process for hardness reduction besides the phase transformation.



annealing