	Experiment title: Alpha- and Beta-residual stress variations during selective surface aging of shot peened Timetal 21s	**Experiment number**: ME 9
Beamline: ID11B	**Date of experiment**: from: 3-3-00 to: 8-3-00	**Date of report**: 9-10-00
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Report:

Experimental: Timetal 21s samples of 4.5 mm thickness were shot peened on the 4×10 mm^2 surface. The incoming X-ray beam had an energy of 90 keV and size of 7×300 μm^2. The samples were mounted in a furnace such that the narrow beam dimension was perpendicular to their surfaces. Complete diffraction rings up to $\beta\{4\ 0\ 0\}$ were recorded on a PSD (FReLoN CCD coupled to an image intensifier). Exposure times were 1 sec for strong and 10 sec for weak reflections. Depth profiles were recorded before and after annealing treatments (350 – 450^{o}C for a few hours). During the annealing the beam was positioned nominally 40 μm below the surface. In order to detect a triaxial stress state diffraction patterns were recorded for three angular sample settings (-30^o, 0^o, 30^o) with respect to a rotation axis perpendicular to the peened surface. Strain released reference samples were prepared and measured to provide the strain free lattice parameter.

Evaluation: A signal independent image pre-conditioning was performed using Fit2D (Dark field subtraction, Correction of spatial distortions, Transformation to polar coordinates). The 2θ center positions of the diffraction rings were then determined by profile fitting of individual azimuthal pixel rows. This

procedure avoids complex peak profiles originating from grain overlap and provides information on the grain size and degree of plastic deformation. The complete strain tensor was evaluated for several reflections from the azimuth dependent angular 2θ shifts relativ to a reference sample and by fitting to the 2D general equation.

Results: The depth dependent strain tensor profiles of the shot peened β-phase samples showed the expected compressive in-plane (11 and 22) and tensile out-of-plane components (33) and negligible shear components (Fig. 1). However, the quantitative differences between several reflections are much larger than expected from the elastic anisotropy. Very large and hkl dependent Poisson ratios were observed. Both findings indicate the existence of strong intergranular strains. A diffraction signal of the precipitated α-phase was observed (Fig. 2) indicating epitaxial growth and oposite strain as compared to the β-phase.

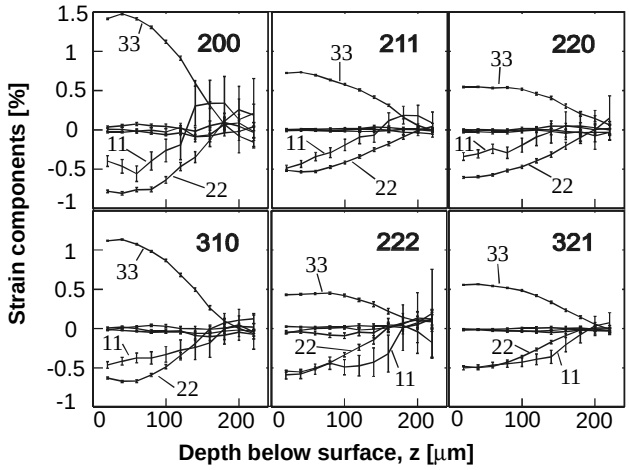

Fig. 1: Depth dependent strain tensor components of a shot peened Timetal sample. The error bars represent ± 2 standard deviations. The large errors for the 11 component result from the restricted sample rotation of only ± 30° around the surface normal.

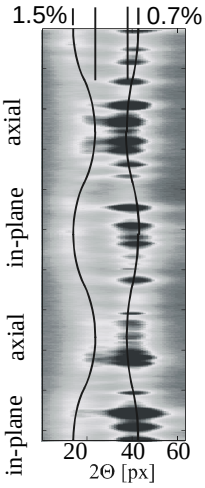

Fig. 2: α{102} (left) and β{200} (right) diffraction rings in polar coordinates indicating epitaxial growth and micro strain.

Conclusion: It was demonstrated that the combination of micro-focussed high energy synchrotron radiation and 2D detectors provides a unique tool for the *in situ* investigation of steep strain gradients. The obtained results provide strong evidence of the existence of intergranular strains and of an epitaxial nature of the α phase precipitation. Figures of merit of the 2D detector to be used for this application are stability, large area, low spatial distortion and fast read out. A state of the art detector was not available for the experiment. Improvement of the detector performance would be highly beneficial for the technique. Further experiments should be performed with an improved 2D detector and elastic constants should be measured. It will be possible to follow the real time strain relaxation and α precipitation during annealing.