ESRF	**Experiment title:** Residual Stress Distribution in Tungsten Thin Films with Ripple Morphology Deposited on TSVs Inner-Wall	**Experiment number**: MA 2785
Beamline: ID 16B-NA	**Date of experiment**: from: 11/12/ 2015 to: 15/12/2015	**Date of report**: 09/09/2016
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Names and affiliations of applicants (* indicates experimentalists):

J. Todt*, D. Gruber*, J. Keckes
Chair of Materials Physics, Montanuniversität Leoben, Jahnstraße 12, 8700 Leoben, Austria

M. Tkadletz*
Chair of Functional Materials and Materials Systems, Montanuniversität Leoben, Roseggerstraße 12, 8700 Leoben, Austria

Report:

1. Introduction

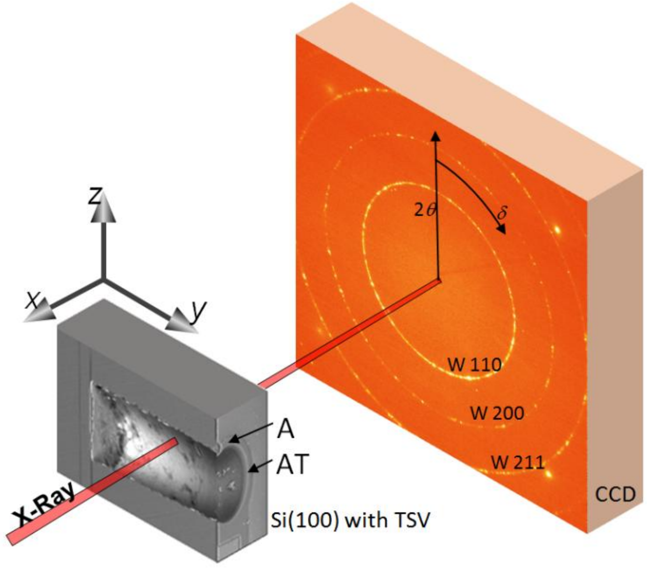

Figure 1: Schematic sketch of the experimental set-up at ID 16B-NA. Axial scans were made for TSVs with different geometries at the positions marked A and AT.

As described in the proposal, the purpose of the experiments at beamline ID 16B-NA was to measure stress profiles along the rippled inner wall of hollow W-lined TSVs with a spatial resolution better than 100 nm. The ripples are due to the specific manufacturing process which is used to fabricate the TSVs by repeated step-wise etching of Si and deposition of the inner wall liner stack containing the W functional layer [1].

The beam diameter (FWHM) of approx. 70 nm, coupled with a photon energy of 29.6 keV, which were available at the time of the experiment were well-suited for the purpose of recording powder diffractograms in transmission through the 200 nm thick W film, the absorption of the Si substrate and the other layers of the TSV liner stack did not pose any problem.

The stages available at the beamline proved sufficient for the alignment of the samples, although the additional possibility of a rotational alignment around the *x*-axis (*cf.* Fig. 1) would have been helpful. Below we give a short summary of results obtained from the beamtime (MA-2875) at ID 16B-NA.

2. Stress distributions along the TSV inner walls

The evaluation methodology for stresses relies on the biaxial nature of the stress state in the W film deposited on the TSV inner walls, which is due to the free surface effect in the radial direction. By making two axial scans on cross-sectioned TSVs (along the axis and along the edge) it is thus possible to obtain the axial and tangential stresses along the length of the TSVs (*cf.* Fig. 1). This method was already published along with results obtained from other measurements performed at beamline ID 13 [2].

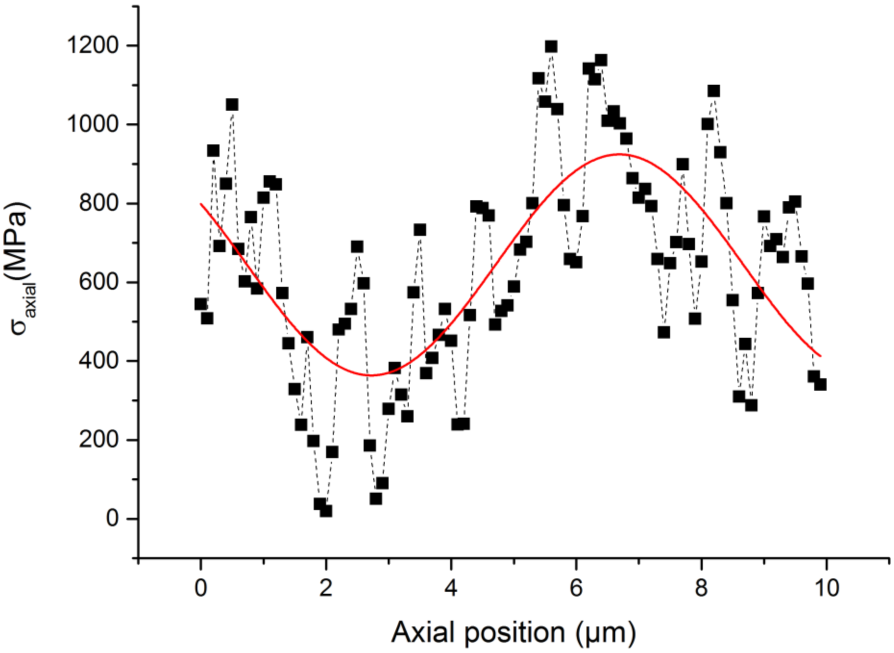

Figure 2: Distribution of axial stress along the axis of one of the measured TSVs. Black squares represent the measured data, the red line is a sinusoidal fit. The relatively large scatter of data points is due to a rather poor diffraction statistic.

TSVs from four different cross-sectioned TSV arrays, each with different diameter, seperation and ripple geometry were measured. In Fig. 2 the results from one of the measurements are presented.

Due to a rather poor diffraction statistic with not quite continuous Debye-Scherrer rings on the detector, the measured data were fitted with a sinusoidal function. The fitted values are: a mean stress of 644±21 MPa, an amplitude of 280±31 MPa and a period of the osciallation of 7.9±0.3 µm. These values correlate well with the ripple geometry and with expectations based on prior results [2].

Results from the other measured TSVs are qualitatively similar, also match well with expectations from prior results and recently developed FEM simulations and are being incorporated into the model to enhance its predictive power.

3. Summary

Distributions of axial and tangential stresses were successfully measured in rippled W films that are used as a liner for the inner wall of TSVs. Four different kinds of geometries were analysed. The results will help to enhance the accuracy of a FE model that was recently developed to identify critical stresses in TSVs with rippled inner wall liner stacks [2].

The investigation of TSVs that cracked due to fatal tensile stresses will be of future interest. Especially the morphology of the crack path will help to elucidate defects in the inner wall liner stack, where the crack might originate. Beamline ID16B-NA would be well-suited for this purpose, as it also offers the possibiliy of nano-tomography coupled with XRD phase contrast and fluorescence phase ID.

3. References

[1] C. Krauss, S. Labat, S. Escoubas, O. Thomas, S. Carniello, J. Teva, F. Schrank, *Thin Solid Films* **530** (2013) 91-95

[2] J. Todt, H. Hammer, B. Sartory, M. Burghammer, J. Kraft, R. Daniel, J. Keckes, S. Defregger, *J. Appl. Crystallogr.* **49** (2016) 182-187