Application Note

alemnis
Mechanical testing at the micro- and nano-scale

In situ micromechanical testing combined with electron backscatter diffraction (EBSD)

Introduction

The recent development of in situ mechanical testing in combination with direct observation and analysis is leading to an enhanced understanding of deformation mechanisms in materials [1-3]. High resolution electron backscatter diffraction (HR-EBSD) [4, 5] straddles the mid-level resolution scale (~ 100 nm) between studying individual dislocations in the Transmission Electron Microscope (TEM) and the bulk deformation behavior using techniques such as X-ray diffraction. One of the advantages of EBSD is that it is a surface technique and therefore is suitable for combination with a mechanical property testing technique such as nanoindentation, where the high spatial resolution is ideal for small-scale sample geometries.

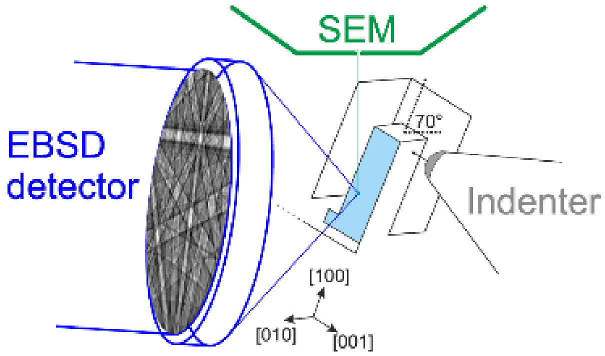

Figure 1. Typical experimental arrangement for nanoindentation on a prefabricated cantilever where the EBSD detector can acquire diffraction patterns in the {100} plane

The application of HR-EBSD to in situ micro-compression has been reported on Ga As [6, 7] and titanium [3] micropillars, showing how the combined technique can be applied to different material systems, loading geometries and specimen geometries.

Experimental

The example shown in Fig. 1 shows a typical experimental arrangement for micromechanical tests performed by nanoindentation on a micro-cantilever which has been fabricated on a mechanically and electro-chemically polished edge of a tungsten single crystal specimen using focused ion beam (FIB) milling. Cantilevers of this type have final dimensions of ~ 20 μm in length and 5-7 μm in thickness and width.

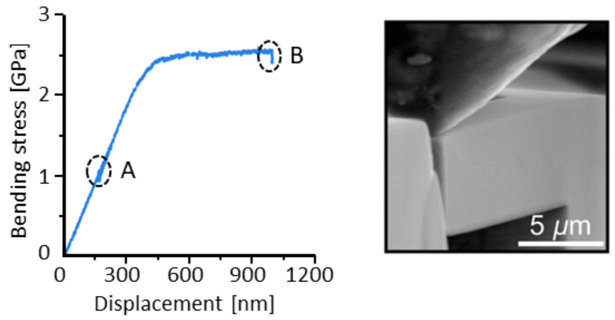

Figure 2. Example of a typical bending test performed on a cantilever beam fabricated from single crystal tungsten. Point A shows the elastic portion of the deformation and point B the point of fracture.

Mechanical tests are performed using an Alemnis nanoindentation system (Alemnis SA, Switzerland) mounted with a cono-spherical tip of relatively large diameter.

The sample and the indenter stage are aligned in such a fashion as to simultaneously perform micro-cantilever deformation tests with the indenter, visualize the experiment and acquire diffraction patterns using the EBSD detector. Typical test methodology on a cantilever beam comprises a displacement-controlled test with interrupted holds at various displacement steps, to collect diffraction patterns under loaded conditions. The cantilever surface facing the EBSD detector was the {100} plane in the example shown. Full displacement gradient tensors were calculated from each EBSD scan, using cross correlation software CrossCourt 4.0 (BLG Vantage, UK) that allows determination of elastic strains with high strain sensitivity (~ 2×10^{-4}) [8]. Typically, 20 regions of interest with sizes of 128 x 128 pixels are selected on the recorded 2 x 2 binning Kikuchi patterns for the analysis. The elastic strains can be converted into stresses by applying the anisotropic Hooke's law. The accumulated geometrically necessary dislocations (GND), created in the plastic regime, leave a measurable contribution to the local rotational gradient which can be determined with high angular resolution (10^{-4} rad) [9].

Results and Discussion

An example of a typical bend test performed on a single-crystal tungsten micro-cantilever is shown in Fig. 2 where two points have been defined below and above the yield point. The displacement of the indenter was held constant at these 2 values (points A and B respectively in Fig. 2) so that the corresponding EBSD scans could be performed. The nanoindentation data was converted to stress-displacement data by using the dimensions of the micro-beam.

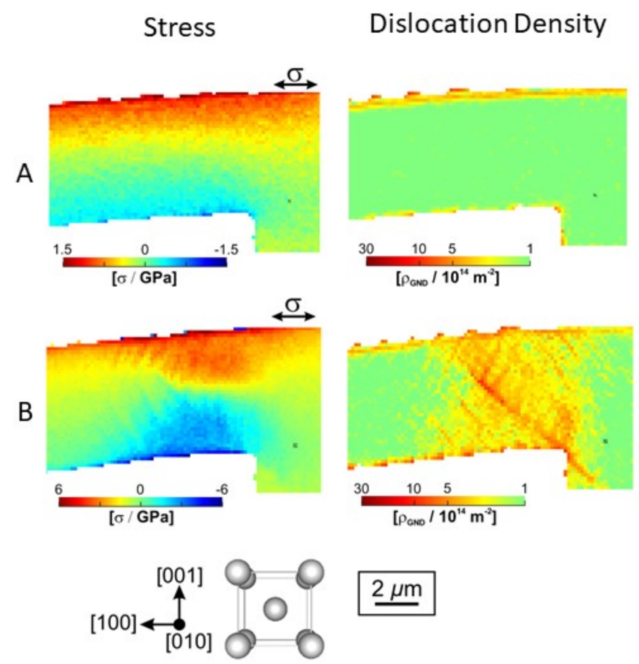

Figure 3. EBSD diffraction data for stress and dislocation density for points A and B as defined in Fig. 2.

The EBSD scans for the two aforementioned stress levels are shown in Fig. 3 where a clear transition can be observed from tensile to compressive stress along the cantilever width.

A maximum stress of ~ 1.2 GPa, measured by CrossCourt at point A, was found to be in good agreement with the bending stress-displacement data. On loading the cantilever beam into the plastic regime, the measured stresses increase significantly (note the different scale bars). The maximum stress at point B was ~ 5 GPa in tension and compression. The stress distribution becomes more complex due to localization of plastic deformation in the vicinity of the cantilever support. However, zero stresses in the central region, corresponding to the neutral axis, can clearly be seen.

The EBSD maps, on the right, show the distribution of GNDs for both stress levels. While no GNDs can be seen for point A located in the elastic regime, pronounced dislocation evolution is observed for point B in the fully plastic regime. Slips systems of type <111> {100} were identified based on the crystallographic orientation of the cantilever beam. GND pile-ups along the neutral axis, where the tensile stress is zero, were observed with local dislocation densities as high as 1 x 10^{15} m^{-2}.

Note that the cantilever edges are slightly rounded which leads to the falsified dislocation evaluation at the top and bottom edge of the beam. The stress field and the GNDs at point B overlap showing the strong influence of the nucleated dislocations on the stress measurements.

Conclusions

The results presented here summarize one of the first successful efforts to combine HR-EBSD with *in situ* micromechanical testing in a compact and user-friendly setup. Such a system can be applied to different material systems and loading geometries such as pillars, beams, tensile and shear specimen geometries. It can also be applied to complex microstructures where deformation mechanisms at interfaces, grain boundaries and precipitates are of interest. To summarize, HR-EBSD and *in situ* micromechanical testing are powerful complimentary analysis techniques that allow detailed investigation of the elastic-plastic deformation behavior of many materials at small scales.

References

[1] J. Ast, G. Mohanty, Y. Guo, J. Michler, X. Maeder, *In situ* micromechanical testing of tungsten micro-cantilevers using HR-EBSD for the assessment of deformation evolution, Materials and Design 117 (2017) 265-266

[2] J. Ast, M. N. Polyakov, G. Mohanty, J. Michler, X. Maeder, Interplay of stresses, plasticity at crack tips and small sample dimensions revealed by in-situ microcantilever tests in tungsten, Materials Science & Engineering A, 710 (2018) 400-412

[3] Y. Guo, J. Schwiedrzik, J. Michler, X. Maeder, On the nucleation and growth of {1122} twin in commercial purity titanium: *in situ* investigation of the local stress field and dislocation density distribution, Acta Materialia 120 (2016) 292-301

[4] A. J. Wilkinson, T. B. Britton, Strains, planes and EBSD in materials science, Materials Today 15, No. 9 (Sept 2012) 366-376

[5] T. B. Britton and J. L. R. Hickey, Understanding deformation with high angular resolution electron backscatter diffraction (HR-EBSD) - Accepted for Publication – IOP Conference Proceedings - Proceedings of EMAS-2017/IUMAS-7

[6] C. Niederberger, W. Mook, X. Maeder, J. Michler, Mater. Sci. Eng. A 527 (2010) 4306

[7] X. Maeder, W. Mook, C. Niederberger, J. Michler, Philos. Mag. 91 (2011) 1097

[8] A. J. Wilkinson, G. Meaden, D. J. Dingley, Ultramicroscopy 106 (2006) 307

[9] A. J. Wilkinson, D. Randman, Philos. Mag. 90 (2010) 1159

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