Application Note

In situ micromechanical testing combined with high resolution electron backscatter diffraction (HR-EBSD) to investigate cracking in tungsten microcantilevers)

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]. High resolution electron backscatter diffraction (HR-EBSD) [2] 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.

The application of HR-EBSD to in situ micro-compression has been reported on Ga As [4, 5] and tungsten [1] cantilevers, showing how the combined technique can be applied to different material systems, loading geometries and specimen geometries. This Applications Note summarizes some tests done on tungsten microcantilevers [3].

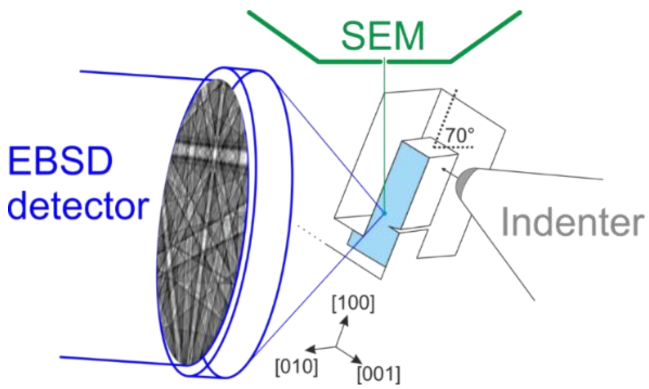

Figure 1. Typical experimental arrangement for nanoindentation on a prefabricated notched cantilever where the EBSD detector can acquire diffraction patterns in the {100} plane. EBSD scans are commonly acquired with an acceleration voltage of 20 kV and a working distance of ~ 15 mm. The entire setup is placed inside the Scanning Electron Microscope (SEM).

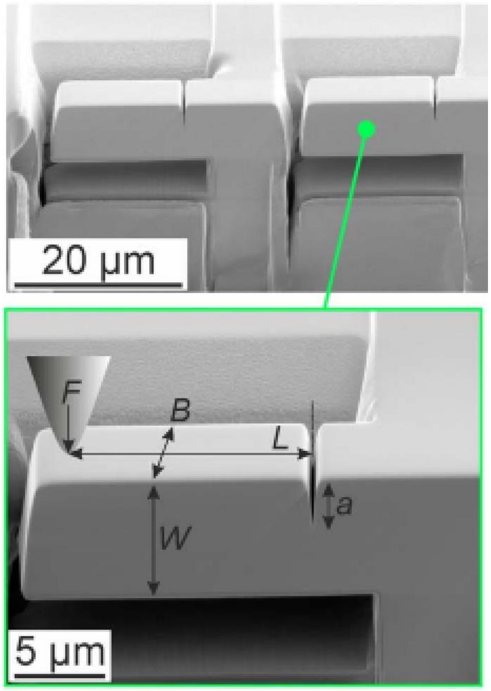

Figure 2. Example of a typical notched tungsten microcantilever specimen with B = 5 µm, W = 7 µm and L = 20 µm.

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, as shown in Fig. 2. A notch was introduced to each cantilever beam by FIB to produce sharp crack tips with a length of ~ 2 µm (radii of the order of 30 nm). Mechanical tests were performed using an Alemnis nanoindentation system (Alemnis SA, Switzerland) mounted with a cono-spherical tip of diameter ~1 µm.

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.

Application Note

Results and Discussion

An example of a typical bend test performed on a single-crystal tungsten micro-cantilever is shown in Fig. 3 where five points have been defined for EBSD analysis. Crack initiation is characterized by a sudden drop in the stress intensity factor (uncorrected data). For the "corrected" data set, the effective crack length was measured from SEM images after testing and used to correct the geometry factor. More information about the fracture process was obtained by analyzing the EBSD measurements shown in Fig. 4. It can be seen that the crack does not propagate through the sample because the released elastically stored energy is not high enough and the lower free surface is in compression. When first paused (2) just after the initiated crack, high tensile stresses in the order of 4 GPa are present at the crack tip, due to activation of {110} slip planes. With further loading (3-4), the tensile stresses increase to ~6 GPa in the vicinity of the crack.

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.

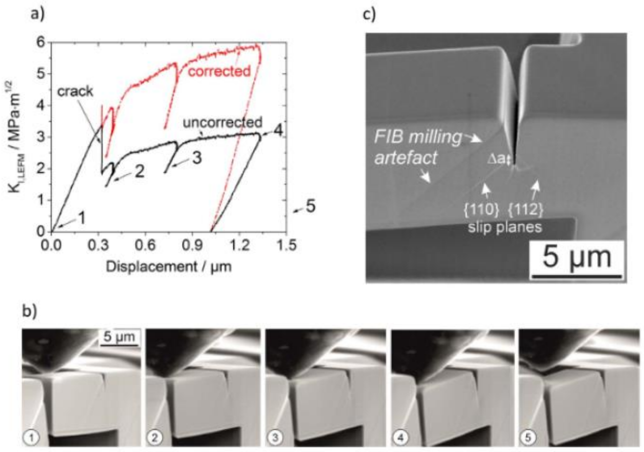

Figure 3. Typical plot (a) of stress intensity factor as a function of indenter displacement with corresponding images (b) of the 5 points where the experiment was paused for EBSD data acquisition and detail (c) of the cantilever after testing (45° tilt)

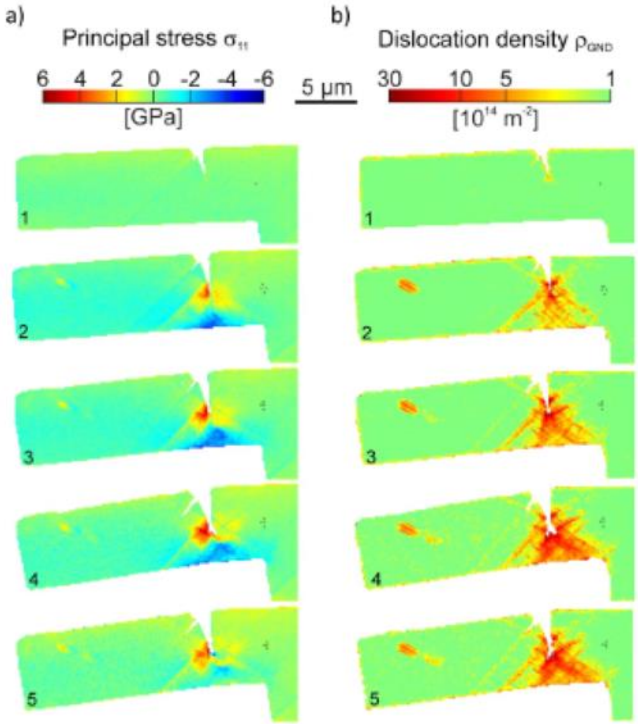

Figure 4. Sequence of EBSD analyses corresponding to the 5 points defined in Fig. 3. The principal stress along the long axis of the beam is shown in (a) and the density of the GNDs in (b).

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

[3] J. Ast, M. N. Polyakov, G. Mohanty, J. Michler, X. Maeder, Mater. Sci. Eng. A 710 (2018) 400-412

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

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

This application note is based on the paper in Ref. [3]:

https://doi.org/10.1016/j.msea.2017.10.096

For further information about the Alemnis range of mechanical testing instruments, please contact:

Dr. Nicholas Randall, nicholas.randall@alemnis.ch