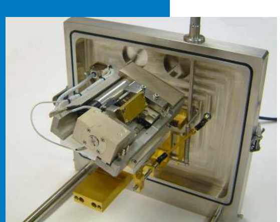
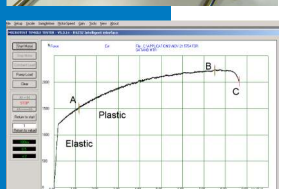
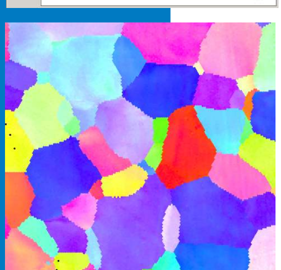
GATAN APPLICATIONS NOTE

Gatan Microtest Tensile Stage used for Grain Rotation Studies in the SEM







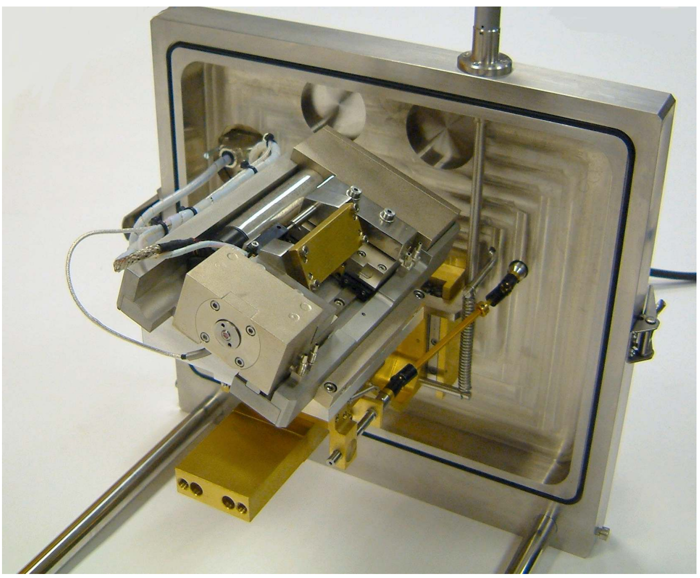
Introduction

In most alloys, materials' properties such as strength and formability are strongly affected by the development of texture. The Scanning Electron Microscope (SEM) fitted with Electron Back Scattered Diffraction (EBSD) instrumentation is widely used to study texture. The current example shows the power of adding a tensile stage *in-situ* with the SEM / EBSD to study microstructural and texture development during forming. In this configuration, the Microtest 5000N tensile stage allows tensile testing dynamically in the SEM, enabling investigations of the relation of crystallographic rotations of grains with traditional stress-strain analysis.

Gatan Microtest Tensile Stage used for Grain Rotation Studies in the SEM

Tensile Stage

The Gatan Microtest range of modules offers dynamic tensile, compression and bending tests of specimens in the SEM. Microstructural changes at the region of interest can be examined with the large depth of field offered by the SEM, while software simultaneously controls the experiment and records stress-strain information.



These modules are optimised for working in the SEM environment, for example by allowing short working distance, and are manufactured using non-magnetic metals. They can also be used on the bench top or under a light microscope. When configured for the vacuum chamber of the SEM, Gatan also offers the option to heat or cool the specimen *in-situ* over a wide temperature range.

In this study, a room temperature module with a maximum load of 5000N was fitted on a replacement SEM door and X,Y, Z translation stage specially configured to fit the stringent criteria of specimen tilt, working distance and diffraction pattern illumination for *in-situ* EBSD studies.

The EBSD camera is inserted once the stage is positioned and an IR chamber scope is helpful in this regard. A single connector using a vacuum feed-through provides stress and strain control and information to the computer.

The photograph shows the 5000N tensile module and the replacement SEM stage and door. In this example the microscope pole piece and tensile module geometry precluded 70 degree tilting of the stage, so 70 degree tilt of the specimen was achieved by partial tilting of the module and of the specimen using specially designed grips. This design allows reverse tilted grips to mount specimens normal to the electron beam for non EBSD dynamic testing studies, or standard micro-characterisation techniques.

Experimental

An alloy specimen (Aluminium A5754-O) was polished to obtain a surface suitable for EBSD analysis.

The initial EBSD grain map prior to deformation is shown in Figure 1 (a). The sample was then subjected to *in-situ* tensile loading to Point A in Figure 2. At this point, the tensile experiment was paused.

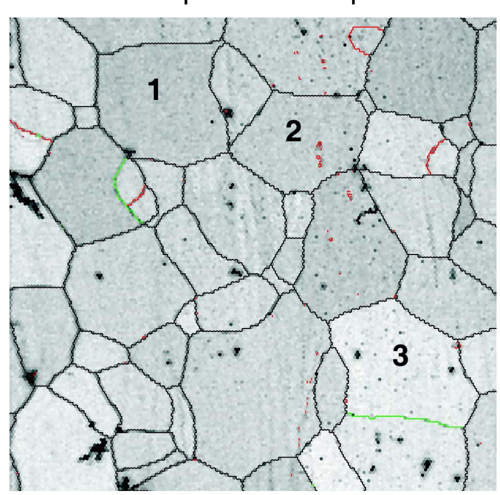


Figure 1(a) *105.0 μm = 35 steps*
Boundary levels: 5° 10° 2° IQ 22.557...134.438

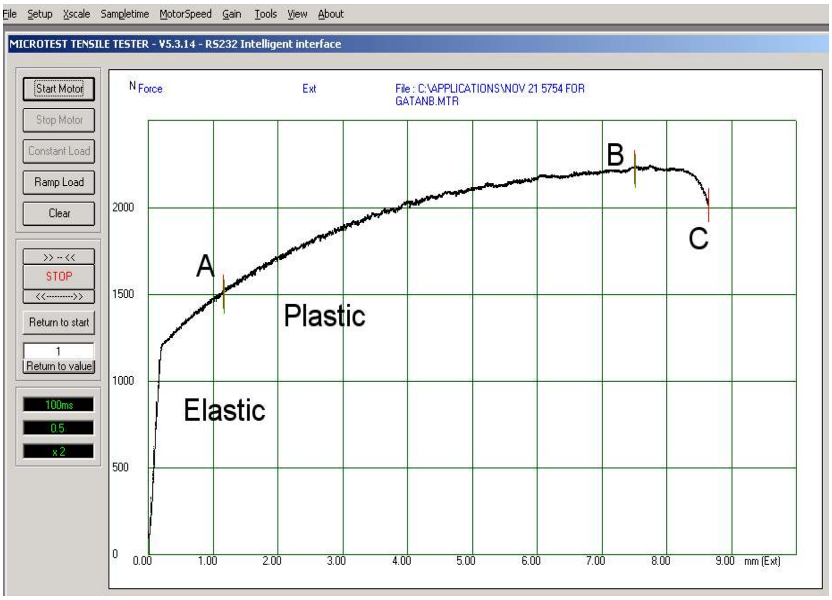


Figure 2: Stress-strain curve showing points A, B and C where the test was interrupted to acquire EBSD data.

The EBSD map collected at 1500N load is shown in Figure 1 (b) from the same area as in Figure 1(a). The straining was resumed to points B and C in Figure 2, stopping at each point to collect matching EBSD data.

Note, for other types of specimens where strain rate is important, the Microtest software provides a choice of strain speeds and allows relaxation to be studied by providing options of continuing data acquisition while extension is stopped, or by controlling extension to keep a constant load. In addition thresholds of load, extension or time can be set as shown in figure 3, thus providing useful dynamic control of the experiment.

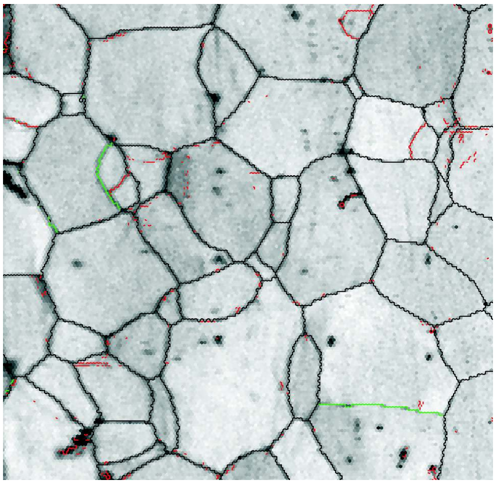


Figure 1(b) *105.0 μm = 35 steps*
Boundary levels: 5° 10° 2° IQ 21.959...130.809

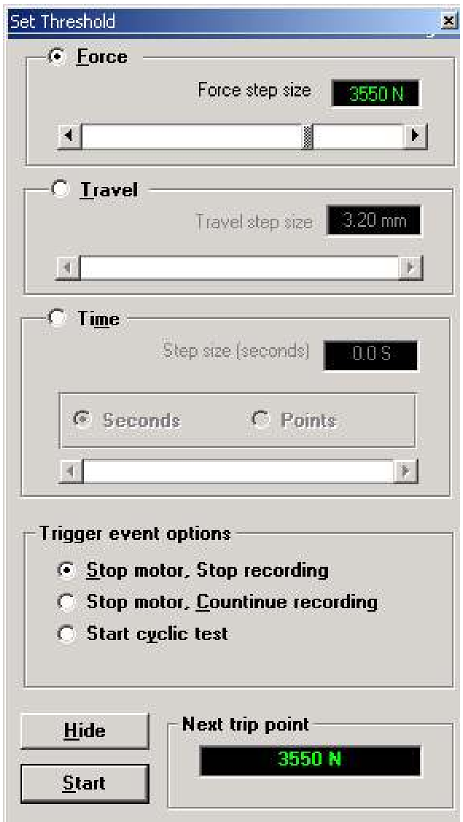


Figure 3. Microtest software "Set Threshold" function provides flexibility in dynamic control.

The changing microstructure under plastic deformation as measured using EBSD can be displayed in many ways to quantify texture, grain size and boundary orientation.

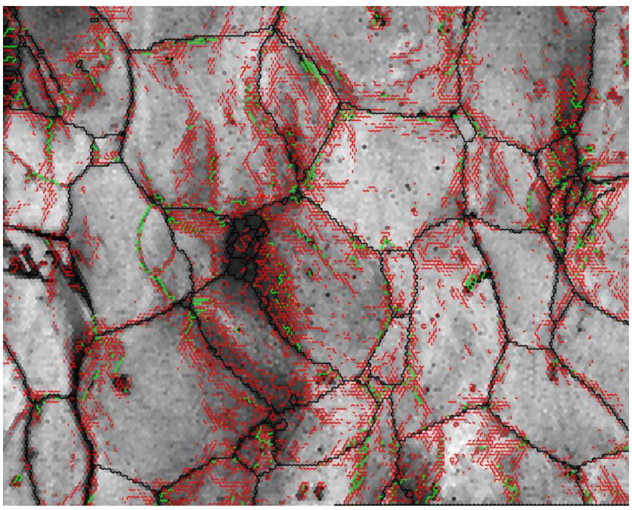
Table 1 lists the measured values of grain rotation and orientation spreads for three typical grains marked 1, 2 and 3 in Figure 1. These values can be quantitatively correlated with strain distribution inside the material.

Grain	Rotation (deg)	Spread (deg) before	Spread (deg) after
1	3.5	0.6	7.2
2	7.4	0.6	5.4
3	11.5	0.7	5.7

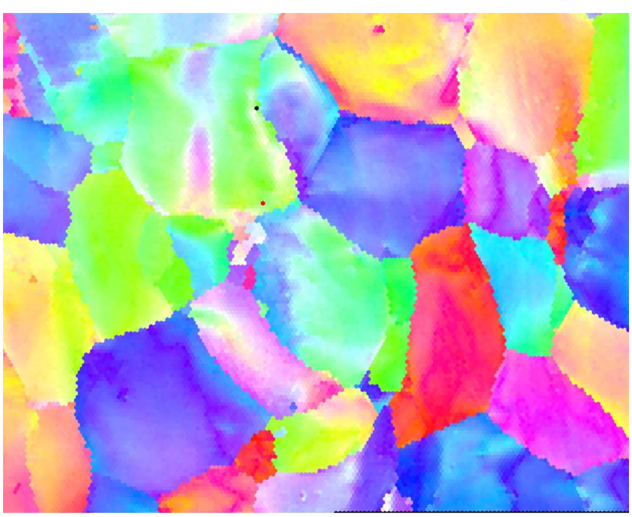
Table 1

Figure 1 (a) & (b): EBSD maps of the sample area before and after applying tensile load to 1500N. Comparison between (a) and (b) where some plastic yield has occurred, shows the formation of localized rotation boundaries (marked red for rotations between 1 and 5 degrees and green for rotations between 5 and 10 degrees).

Figure 4 shows grain rotation and orientation maps of the specimen after loading to 2225N which corresponds to 22% strain. The density of low angle grain boundaries inside grains is noticeably higher in Figure 4(a) and the corresponding grain orientation figure shows colour variations corresponding to orientation spreads. The dark region in Figure 4(a) corresponds to gross topographic variation on the surface.



105.0 μm = 35 steps *Boundary levels: 5° 10° 2°* *IQ 21.442...129.211*



105.0 μm = 35 steps *IPF [001]*

Figure 4 (a) EBSD map showing grain rotation and (b) matching grain orientation from same area but after loading to 2225N. Note the large increase in the number of 1-5 degree grain boundaries. Very dark areas indicate poor diffraction due to the lattice distortion and topographic changes. Changes in local rotation are responsible for colour variation inside grains in (b).

As the material deforms, grains undergo lattice rotation. The amount of rotation depends on the amount of slip in individual grains and their relative orientation and location with respect to the tensile axis. The dislocation networks inside the grains increase the spread of local orientations.

Summary

This work has documented the ability of the Microtest tensile stage configured for EBSD analysis, to show microstructural changes in an aluminium alloy sample subjected to *in-situ* tensile deformation. Increase in lattice mis-orientation in the grains and lattice rotations can be directly measured in samples deformed to large strains using this combination of equipment. Resulting measurements attest to the positional and mechanical stability of the stage in this application.

Acknowledgements:

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