Quantitative estimation of spatial stress gradients from dislocation pile-up at grain boundaries in commercially pure titanium

I. Basu*, V. Ocelík, J.Th.M De Hosson

Materials Science Group, Zernike Institute of Advanced Materials and Materials Innovation Institute, University of Groningen, 9747AG Groningen, The Netherlands

*Corresponding author: i.basu@rug.nl

Abstract

The current work introduces a site specific method utilizing electron back scattered diffraction (EBSD) and FIB-DIC slit milling to accurately determine spatially resolved stress profiles in the vicinity of grain boundaries in commercially pure titanium. Correlations with the GND dislocation density and slip transfer parameter are drawn to validate the stress measurements. Stress profiles within few hundred to thousand nanometers near the grain boundary display a local minimum, followed by a typical Hall-Petch type variation of "one over square root of distance". The observed trends allude to local stress relaxation mechanisms active near grain boundaries.

Introduction

Quantitative estimation of spatially dependent local stress states is crucial in predicting macroscopic fracture mechanisms in structural materials. Spatially resolved internal stress measurements can be made using high resolution electron back scatter diffraction (HR-EBSD) wherein Kikuchi patterns from reference (un-deformed) and deformed states are cross-correlated to measure the residual elastic strains, and subsequently calculate the local elastic stress state [1]. However, the method is limited to 2-dimensional investigations wherein only surface information is obtained. On the other hand, residual stress measurements using focused ion beam – digital image correlation (FIB–DIC) technique allows simultaneous sub-micron lateral and depth resolution in a semi-automated and robust way, thereby accounting for both surface and bulk deformation contribution on internal stress build up.

Experimental Methods

Commercially available grade II titanium was subjected to room temperature in-situ four point bending test inside a Tescan Lyra dual beam (FEG-SEM/FIB) microscope. Microstructural characterization was performed by means of EBSD, thereby extracting both topographical and orientation information of individual grains. The acquired raw EBSD data was subsequently analyzed using Edax TSL OIM Analysis software and MTEX Matlab toolbox [2]. All observations were made on the tensile surface of the bent specimen, with the direction of viewing parallel to the surface normal hereinafter referred to as A3 sample axis.

Local stresses near grain boundaries were experimentally determined by the FIB-DIC slit milling technique described in [3]. Linear slits, oriented normal to the grain boundary trace, of a fixed width 0.5 μm, depth 2.5 μm, and lengths varying from 15-25 μm (depending on the grain size), were milled inside individual grains showing pile up in kernel average misorientation OIM maps. Measurements were performed on multiple grains with different degrees of pile up. For each slit, multiple SEM images of resolution 768x768 pixels were acquired at high magnifications (field of view of 10 – 15 μm) to ensure a high spatial resolution of measured displacement field. DIC was performed using commercial software GOM Correlate 2016. Facet size of 19 x 19 pixels with a step width of 16 pixels was chosen in order to obtain statistically sufficient data points. Yttria-stabilized Zirconia (YSZ) nano-particles were used for surface decoration to obtain optimum image contrast for high accuracy DIC analysis. A multiple fitting approach, wherein a stress value is obtained for displacements corresponding to each row, was implemented to incorporate measurement of spatially heterogeneous stress states.

Results and Discussions

Figure 1 illustrates a representative case of a grain associated with strong pile up, labelled as *grain 2*. Figure 1a represents the kernel average misorientation (KAM) map. KAM values were calculated for the 2nd nearest neighbor with a threshold value of 2°. High angle grain boundaries, classified as larger than 15°, are highlighted in white in Fig. 1a. Fig. 1b shows the variation of local average misorientation (LAM) and GND density with respect to distance from the grain boundary (refer to highlighted line AB in Fig. 1a). The excellent agreement between the LAM and GND values is unsurprising since both values are derived from local misorientation. Interestingly both trends reveal a local minimum close to the grain boundary highlighted by the shaded region in Fig. 1b.

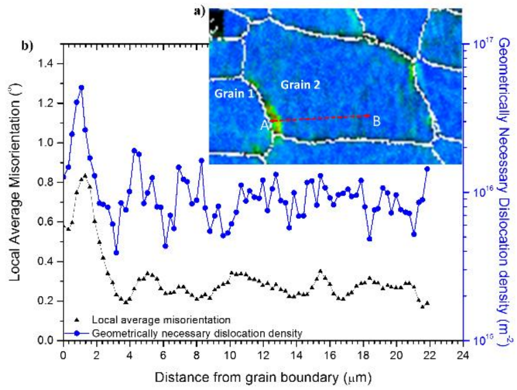

Figure 1 (a) KAM map and (b) GND density and LAM profile between points A and B

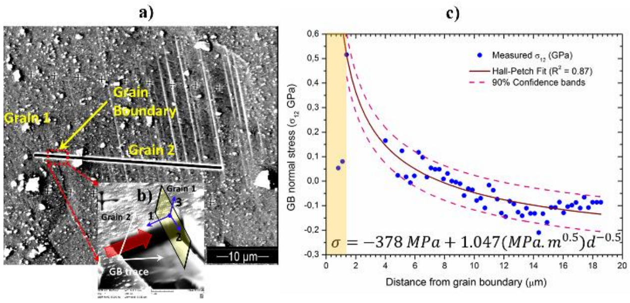

Figure 2 (a) SEM image showing slit and (b) grain boundary plane orientation; (c) corresponding stress profile from points A to B

Fig. 2 represents the FIB-DIC analysis and corresponding stress measurements for the region shown in Fig. 1a. Figs. 2a shows the spatial orientation of the milled slit with respect to the grain boundary. *Grain 2* shows profuse slip traces that were identified as $(10\bar{1}0)$ prismatic plane traces. Fig. 2b represents the orientation of the grain boundary plane with respect to the longitudinal direction of the slit indicated by the red arrow. Fig. 2c displays the measured stress profile as a function of normal distance from the grain boundary plane. The measured stress component σ_{12} describes the stress acting parallel to the grain boundary plane and normal to the length of the slit. The stress values within a distance of 1.3 μm from the grain boundary show a local minimum, which is succeeded by monotonic decrement in the stress values with increasing distance from the grain boundary. On comparing the stress gradient with Fig. 1b the agreement seems excellent, thereby indicating that the observed stress fluctuations indeed confer to the actual stress state near the grain boundary. The data points corresponding to distances greater than 1.3 μm show a good fit to the theoretically known 'one over square root of distance' Hall-Petch variation (c.f. Fig. 2c).

The initial stress minimum observed in Fig. 2c indicates activation of local stress relaxation mechanisms, wherein the dislocation spacing seems to equilibrate in regions very close to the boundary i.e. pile up effect is abated locally. The plausible mechanisms contributing to such effects could be,

a) stress relaxation due to back stresses during unloading and the presence of free surface that relaxes the imposed compatibility constraints on deformation.
b) influence of local hardening on further dislocation pile up
c) change in stress state near the grain boundary due to strain compatibility constraints

Summary

In summary, the proposed correlative technique using EBSD and FIB-DIC approach can successfully provide local stress information at sub-microscopic resolution. The applications of the suggested approach are manifold in terms of understanding stress gradients near grain and phase boundaries and their corresponding impact upon material failure.

Acknowledgement

This research was carried out under project number T61.1.14545 in the framework of the Research Program of the Materials innovation institute (M2i) (www.m2i.nl).

References

[1] T. B. Britton and A. J. Wilkinson, *Ultramicroscopy*, vol. 111, no. 8, pp. 1395–1404, Jul. 2011.
[2] R. Hielscher and H. Schaeben, *J. Appl. Crystallogr.*, vol. 41, no. 6, pp. 1024–1037, Dec. 2008.
[3] C. Mansilla, D. Martínez-Martínez, V. Ocelík, and J. T. M. De Hosson, *J. Mater. Sci.*, vol. 50, no. 10, pp. 3646–3655, 2015.