

Regular article

Probing elementary dislocation mechanisms of local plastic deformation by the advanced acoustic emission technique

A. Vinogradov^{a,b,*}, A.V. Danyuk^b, D.L. Merson^b, I.S. Yasnikov^b

^a Department of Mechanical and Industrial Engineering, Norwegian University of Science and Technology – NTNU, Trondheim N-7491, Norway

^b Institute of Advanced Technologies, Togliatti State University, Togliatti 445020, Russia

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ABSTRACT

To gain a deeper insight into fundamental processes of localised plastic deformation, we propose a new strategy for micro-mechanical testing combining the micro-scale scratching and analysis of low amplitude acoustic emissions (AE) accompanying dynamic dislocation processes in solids. The analysis of the AE spectral density reveals its strong dependence on crystallographic orientations of the grains along the indenter path, thus reflecting the finest features of the local dislocation activity. Adding a temporal dimension with a sub-microsecond resolution, the proposed methodology substantially enhances the existing capacity of micro-mechanical testing and can be used in a wide range of testing schemes.

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Recently, a family of advanced micro-mechanical testing methods [1] emerged as significant tools for characterising the elastic-plastic mechanical response of materials on small scales. Providing an excellent reproducibility and accuracy of mechanical data, the instrumented indentation and scratch testing has become the primary technique to examine the local properties and to investigate the underlying local deformation and fracture mechanisms for a wide range of materials. A dislocation behaviour on the micro-scale is commonly characterised by the posttest analysis of slip patterns arising near the indenter footprint, while the local materials response is assessed by the load-displacement diagram or the coefficient of friction evaluated during a scratch test. However, the temporal resolution of these measurements is insufficient to reveal the details of the underlying dislocation dynamics. Real-time information on the elementary processes of local plastic deformation and fracture can substantially advance the capability of micro-mechanical testing. Providing this information with an unprecedented temporal resolution, the modern acoustic emission (AE) technique becomes increasingly recognised in micro-testing, e.g. during indentation [2] or scratch testing of ductile, brittle [3] and coated materials [4,5]. The main challenge for the AE technique is to assess a very low amplitude signal generated in small, micrometres scale, deforming volumes. Because of the extremely low signal-to-noise ratio (SNR), the amplitude threshold-based methods fail to detect a signal from indentation or scratch testing of ductile materials. Another challenge arises when a link is sought between low amplitude AE signals and elementary

deformation processes. To address these issues we have developed a versatile threshold-less AE signal recording and analysing procedure and applied it to the instrumented micro-scratching of copper polycrystals with the well-characterised initial microstructure.

The samples of 3 N polycrystalline copper with dimensions of $20 \times 20 \times 2 \text{ mm}^3$ were annealed in vacuum at 1170 K for 1 h and electrolytically polished to a mirror-like finish. The microstructure was investigated by electron backscattered diffraction (EBSD) measurements in a field-emission scanning electron microscope (SEM) ZEISS-SIGMA equipped with the EDAX/TSL EBSD detector. Scratch tests were carried out using the Nanovea tester. To reduce the effect of interfacial friction between stylus and specimen [6–8], the Berkovich diamond indenter in the edge-forward orientation was moved linearly at relatively high constant dragging speed $V_s = 12$ and 24 mm/min until the scratch reaches 2 mm length. The normal load F during the test was set constant at the minimum stable controllable value 1 N. The piezoelectric sensor AE-900S-WB (NF-Electronics, Japan) with a frequency band of 100–1000 kHz was attached to the samples through vacuum oil with a rubber band. The signals were amplified by 60 dB by the low-noise pre-amplifier and acquired by the 16-bit PCI-2 board (MISTRAS, USA) operated in a continuous threshold-less mode at the sampling rate of 5MSamples/s.

During signal processing (i) the continuously streamed data were sectioned into consecutive individual realisations of 8192 readings; (ii) a power spectral density (PSD) function $G(f)$ was calculated using a Welch technique; (iii) the average (per realisation) power $P_{AE} = \int_{f_{\min}}^{f_{\max}} G(f) df$, and the median frequency f_m of the PSD function defined by the implicit equation $\int_{f_{\min}}^{f_m} G(f) df = \int_{f_m}^{f_{\max}} G(f) df$ were introduced (see [9] for details); (iv) the time-series of these variables were

* Corresponding author at: Department of Mechanical and Industrial Engineering, Norwegian University of Science and Technology – NTNU, Trondheim N-7491, Norway.
E-mail address: alexei.vinogradov@ntnu.no (A. Vinogradov).

further smoothed by low-pass Butterworth filtering with the cut-off frequency of 19 Hz. Both P_{AE} and f_m were obtained from $G(f)$ after subtraction of the mean PSD of the laboratory noise pre-recorded before the start of loading during each test.

The specimen grain structure revealed by the EBSD technique is shown in Fig. 1a in the inverse pole figures (IPF) colours with the superimposed view of the scratch. The rigid stylus moved along several grains with different orientations. As the size of the indenter footprint is usually smaller than the grain size, one can consider that the indenter ploughs a micro-groove in single crystals separated by grain boundaries. Slip systems with the largest Schmid factor are activated under indenter in each grain. The crystallographic elastic and plastic anisotropy of the grains causes a direction dependent variation in the dislocation behaviour. Near the grain boundaries, the morphology of the scratch has visible facets signifying changes in the materials resistance to the indenter motion due to the interaction between the boundary and the lattice dislocations generated in the plastic zone ahead of the indenter as illustrated in Fig. 1c.

A typical example of the recorded raw AE signal is shown in Fig. 1b. AE is a transient phenomenon resulting from a rapid stress drop in a local region of the deforming solid [10]. Nonetheless, the AE waveform during scratch testing appears as a continuous noise-like signal with a very low amplitude, cf. Fig. 1b, which is typically observed during uniform plastic deformation mediated by dislocations in pure metals [11]. No microcracks were observed by SEM within the scratch imprint. It is, therefore, plausible to suppose that the primary source of AE is associated with plastic deformation. The SNR of the signal shown in Fig. 1 is far too low to enable any conventional threshold-based AE analysis, which is commonly used for detection of transient signals. However, considering the stationarity of the recorded AE stream in a wide sense, the Fourier spectral representation provides a convenient means to characterise the signal and to reveal its fine features depending on the dislocation activity in differently oriented grains. Using an original signal categorization technique (see Appendix A), it was demonstrated that the AE signal generated throughout scratching is uniform, i.e. it is produced by the sources belonging to the same parent population. Fig. 2 shows the behaviour of the AE spectral parameters P_{AE} and f_m during scratch testing with different velocities. One can notice that a

combination of these variables reflects the changes in the grain micro-structure along the scratch path. When the indenter crosses a grain boundary, new slip systems are activated resulting in the change of the hardening rate controlled by the dislocation storage rate in different slip systems. The AE behaviour changes accordingly, although these changes are not necessarily very sharp since the dimensions of the indenter are much greater than the grain boundary thickness.

A strong AE dependence on the crystallographic orientation of single crystals tested in tension has been convincingly demonstrated in many early studies [12,13]. Particularly, the relation exists between the AE parameters - P_{AE} and f_m - and the strain hardening rate resulting from activation of different slip systems in differently oriented crystals [9,11,14]. Despite the very low SNR and the fluctuating behaviour of P_{AE} and f_m in Fig. 2a,b, the strong AE dependence on the crystallographic orientation of the grains under the indenter is particularly evident on the scatter-plot P_{AE} vs. f_m , Fig. 2c,d. The parameters characterising AE from different grains form compact clusters, Fig. 2a,b, with remarkably different centroids (f_m, P_{AE}). Their positions and shapes depend on the crystallographic orientation of the grain, i.e. on the specific dislocation behaviour and hardening in different grains. The green circles “connecting” the dense clusters in Fig. 2c,d are not indexed as independent clusters. Rather, they represent reasonably the transition AE behaviour of the indenter passing through the grain boundary. It is important to notice that the average P_{AE} value increased by a factor of two from 0.015 to 0.03 a.u. with the twofold increase in V_S . It is important to notice here that the scratching velocity exerts opposite effects on adhesive friction and AE: interfacial friction reduces [15,16] while AE increases with the increasing velocity. The observed behaviour corroborates the assumption that the effect of plastic deformation overrides the possible effect of interfacial friction. On the other hand, the proportionality between the AE power and plastic strain rate has long been recognised as the essential feature of continuous AE accompanying plastic deformation of metals [17]. Taking into account that the mean hardness did not change appreciably between these two tests and that for the scratching test the shear strain rate is defined as $\dot{\gamma} = V_S/W_S$ (with W_S - the width of the scratch) [18], one can conclude that the same proportionality holds in scratch testing. It is also good to notice that $\dot{\gamma}$ during scratching is usually much higher than in conventional tensile tests

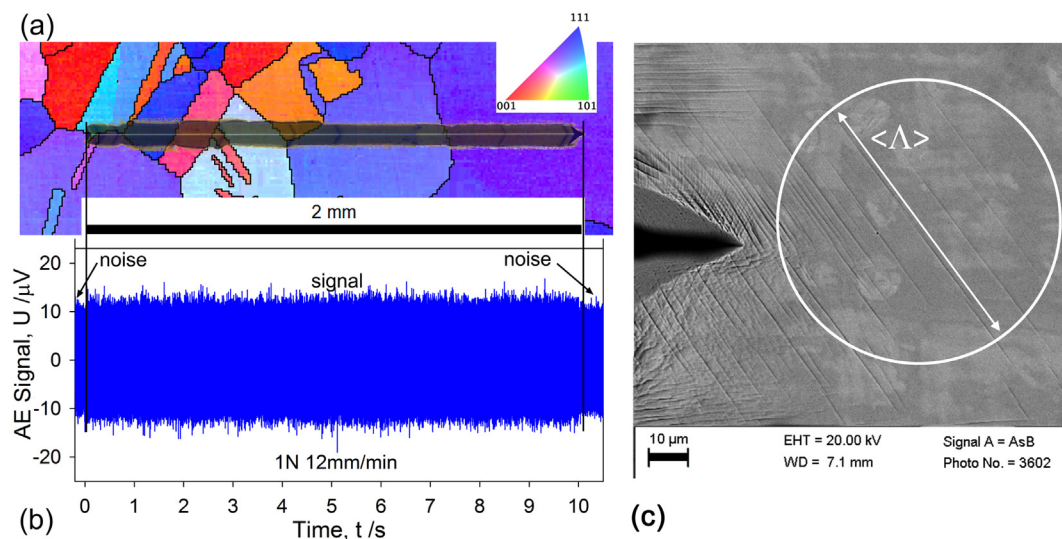


Fig. 1. EBSD orientation map of the surface of pure Cu polycrystal with the superimposed optical microscopy image of the scratch (the map is coded in the inverse pole figures colours corresponding to the standard triangle shown in the inset) (a); corresponding raw AE stream (b), vertical lines indicate the beginning and the end of the scratching process; background noise is seen before 0 s and after 10s; SEM micrograph showing the dislocation slip pattern ahead of the indenter tip (c); the white circle indicates schematically the plastic zone where the active sources of AE are seen as individual slip lines in the primary slip system (other slip systems are visible in the severely deformed region in the proximity to the indenter tip).

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