NEW PROSPECTS TO USE ACOUSTIC EMISSION DURING SCRATCH TESTING FOR PROBING FUNDAMENTAL MECHANISMS OF PLASTIC DEFORMATION

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ABSTRACT

The acoustic emission technique has been widely used during indentation or scratch testing. A common problem is that, when the elementary modes of deformation are of concern, the testing has to be performed locally. Such a testing model implies inevitably in a very small (microscopic) volume involved into plastic deformation under the indenter tip. Therefore, the AE generated during the test has a quite small amplitude hardly distinguishable on a background of the electric noise if the traditional threshold-based acquisition is utilized. With the specific aim at identifying and characterizing fundamental mechanisms of plastic deformation through analysis of AE signals, the present work seeks to offer new strategies in AE acquisition and processing during scratch testing of material to get a better insight on the elementary processes of local plastic deformation. The experiments have been performed on the model pure copper which has been well characterized microstructurally in the local area along the indented path using a scanning electron microscopy. The AE technique appears a viable and powerful tool for investigations of local deformation mechanisms in situ.

Key words: scratch test, deformation mechanisms, acoustic emission, microscopy.

1. Introduction

A broad variety of local destructive or non-destructive methods is applied for routine nondestructive testing as well as for innovative academic research, which benefit strongly from development of modern nano-indentation and kinetic/dynamic micro-indentation and scratching techniques enabling precise characterization of the mechanical behavior of materials and coatings. These techniques have been enjoying booming popularity due to excellent reproducibility of results, high accuracy and capacity to measure the properties of geometrically small objects or local properties in large samples otherwise not assessable in conventional mechanical testing in the laboratory. Overall, these features of local loading techniques has ensured their outstanding reputation and reliability in NDT applications where a rapid and cost-effective evaluation of mechanical properties is required in small objects such as individual grains, grain or phase boundaries. Plenty of valuable information pertinent to the analysis of deformation processes is offered by the post-factum analysis of the morphology of the footprint of the indenter. The real-time information concerning the local behavior of the materials during testing is provided by the kinetic indentation diagram or by the measurements of the friction
coefficient during a scratch-test. However, the temporal resolution of these measurements is not very high to resolve elementary processes of plastic deformation and fracture. To compensate the obvious deficit of the real-time data and provide a new means for characterization of local plastic deformation and fracture, the acoustic emission (AE) technique opens a wide avenue for deriving real time information from a locally deforming object during non-destructive materials evaluation and quantitative characterization of materials properties. The thorough AE analysis enables discrimination between different types of deformation mechanisms and estimation the intensity of AE sources in dependence on the microstructural features and loading conditions. In this way, to turn a modern AE technique to a real quantitative tool for characterization of deformation mechanisms and local properties, it is essential to establish robust relationships between the AE signal and elementary deformation processes. To accomplish this fundamental goal we have chosen a well annealed copper polycrystal with the initial microstructure characterized by orientation image microscopy (OIM) and scanning electron microscopy (SEM). The obvious problem which most researchers have faced while applying the AE measurements to the scratch (indentation) test, is that due to a small deforming volume the cumulative AE signal is small and hardly distinguishable from a background noise if elementary dislocation reactions are of concern. The appealing opportunity is however that local loading allows us to follow the deformation process in its finest details with less interference between different sources in the AE signal. It is therefore a challenging task for the present work to discriminate the features of dislocation behavior under indenter from a low amplitude AE.

2. Experimental procedure and AE methodology during scratch testing

The samples of technical purity 99.9% polycrystalline copper with dimentions 20x20x2 mm were cut by spark erosion. They were mechanically polished by emery paper to the grade of #2000 and then annealed in vacuum at 1170K for 1 h and furnace cooled. Finally, the front surface of the samples was electrolytically polished for microscopic observations prior to and after the test.

The microstructure was investigated by electron backscattered diffraction (EBSD) measurements in high-resolution FE-SEM ZEISS SIGMA microscope with field emission gun and EDAX/TSL Hikari-5 OIM detector.

To correlate the AE signal with the deformation structure along the scratch path, the crystallographic orientation of grains was determined before test by the EBSD technique, Fig. 1 where the orientation of the grains is inked according to the standard inverse pole figure coding showed in the inset. Depending on the crystallographic orientation of grains, the slip systems with the highest Schmidt factor are activated, i.e. the local texture controls the anisotropy of mechanical properties of the crystallites and the appearance of different slip systems on the surface.

Scratch tests were performed under controlled conditions on the Nanovea Scratch Tester with a Berkovich diamond indenter moving linearly along the sample at constant speed of 12 mm/min. For better quantification of the AE signal during indentation/scratch testing one needs to ensure as high signal-to-noise ratio (SNR) as high as possible. However, as mentioned in the Introduction, scribing of ductile materials such as well-annealed pure fcc metals results typically in low level AE. The measures have to be taken to increase the SNR by proper tuning of the recording rig. The tester was adjusted to minimize the mechanical and electrical noise in the AE channels so that the feedback parameters of the mechanical actuator were tuned precisely to a monotonic smooth scratch test. The normal load during the test was chosen below 1 N to minimize the mechanical friction in the vicinity of the indenter tip according to recommendations given in ref. [1]. The final length of the groove was about 2 mm.

The record the AE from dislocation sources operating in a small deforming volume the following conditions imposed onto the sensor and amplifier have to be met:
- minimum internal electrical noise to provide an acceptable SNR;
- maximum sensitivity in the reasonably wide frequency band to enable spectral analysis for source recognition. Assuming that the AE sources during scratching of pure copper would be dislocation segments emerging with a high velocity on a free surface under action of the image force, this type source can be represented simply as a delta-type force function with a wide power spectral density.

The 10 mm diameter piezoelectric sensor AE-9000-S-WB (NF Electronics, Japan), with a frequency band of 100–1000 kHz, was attached with coupling oil on the side of the samples. The signals were passed through low-noise 2/4/6/ pre-amplifier with 60 dB gain and were finally acquired by means of the PIC-2 board (Physical Acoustics Corporation, USA) operated in a continuous threshold-less mode at the sampling rate of 5MSPS. The on-board filters were set in the widest possible range of 20kHz to 2MHz and the additional +6 dB gain was pre-set. The AE waveforms were continuously recorded and synchronized with with the scratch path.

3. Experimental results

The inverse pole figure colored OIM of the surface of copper polycrystal and the view of the scratch across the grain structure is shown in Fig. 1. One can clearly see that during testing the scratch passes through several grains with different orientations, overcomes grain boundaries and triple junctions which well suites the goals of the present work.

![Fig. 1: The inverse pole figure colored OIM of the surface of the annealed copper polycrystal and the view of the scratch across the grain structure.](image)

A typical example of the AE signal recorded during scratching is shown in Figure 2. Although the SNR of this signal is quite low for conventional AE testing assuming a threshold based acquisition, it can be considered “reasonably high” for typical scratch testing of a ductile copper sample. In what follows, we shall demonstrate that using appropriate filtering helps to improve the SNR and to reveal some important fine futures of AE sources in the rectified signal.

The AE signal processing workflow includes the following steps:
1 A continuous raw stream was divided into frames of 8192 readings.
2 For each frame two parameters were calculated in a time domain - the peak Upeak and root-mean-square voltage Urms, respectively.
3 For each frame the FFT denoising procedure involving forward and inverses FFT was applied, and two parameters were calculated in the frequency domain - Energy (power), E, as the integral over the power spectral density and the Median frequency, F_{median}, as the frequency which separates the low and high frequency spectral domains with equal Energy (power).
4 The obtained time series of respective variables (Upeak, Urms, Energy or F_{median}) were smoothed by a low-pass Butterworth filtering procedure with the cut-off frequency of 19 Hz.
Fig. 2: Continuously recorded raw AE data stream acquired during the scratch test with the view of the scratch groove on the surface superimposed. The indenter moved from left to right under 1 N applied force.

Figure 3 (left hand side) shows the behavior of the AE spectral parameters - Median frequency and Energy as well as Upeak and Urms as defined from the original stream. The low-pass filtering applied to these time-series filters out the high frequency components and reveals the trends, which are shown in Fig 3 (right hand side). The use of this filtering with the cut-off frequency as low as 19 Hz is justified because the grain structure of the annealed specimen is coarse and the indenter propagates a reasonably long time within a homogeneous area within the same grain with the mean size around 0.2 - 0.8 mm. Importantly is that the AE signal reflects well the alternations in the microstructure encountered by the indenter tip when it crosses the grain boundaries. When the transition from one grain to the other occurs, the new slip systems are activated resulting in different hardening, the rate of which depends primarily on the dislocation interaction in the intersecting slip systems. The most pronounced changes in the AE signal are seen in the Energy behavior.

The non-surprising anisotropy of mechanical properties during the scratch test of a copper single crystal has long been recognized [2]. Several early studies highlighted a strong AE dependence on the crystallographic orientation of single crystals [3, 4] and the relation of the AE spectral parameters to the activation of different slip systems [5]. The strong dependence of AE parameters on the crystallographic orientation of the grains during the scratch test can be vividly expressed in a scatter-plot showing the distribution of AE descriptive variables such as Energy and Median frequency, Fig. 4. The clustered pattern corresponding to AE, which is observed in several selected grains (see the inset OIM image), is evident. In this diagram, one can see a set of compact well separated clusters with higher or lower energy and/or median frequency. The position of centroids and the shapes of the clusters depend on the crystallographic orientation, i.e. on the specific dislocation behavior and hardening in different grains. The blue points correspond to AE observed during the indenter propagation through the rest of the specimen.
Fig. 3: Evolution AE parameters during scratching: unfiltered data on the left and low-pass filtered data on the right.

Fig. 4: AE Energy - $F_{\text{median}}$ bi-variate distributions highlighting the changes in the AE properties with indenter transition from one grain to the other. The grains are numerically labeled on the OIM map and the corresponding $Energy-F_{\text{median}}$ distributions are colored as shown in legend.
The important feature of the AE waveforms, which are recorded typically during plastic deformation mediated by dislocation slip in pure metals, is that they commonly manifest themselves as a continuous noise-like signal, Fig.2. The continuous signal, such as that shown in Fig. 2, is associated with a huge number of dislocation segments generated here and there within the loaded solid and moving nearly concurrently across the grains. However, on a microscopic scale plastic deformation of metals is spatially heterogeneous and temporally intermittent by nature due to discrete origin of carriers of plasticity - lattice dislocations [6-8] creating clearly separated fine slip lines on the surface during deformation, cf. Fig.5. This discrete character of dislocations is the root cause of local fluctuations in stress and strain giving rise to measurable AE. The detectability of AE is commonly supposed to be quite limited [9]. The detectability threshold is of course dependent strongly on the noise level and the capacity of the AE system to discriminate the signals on a background of the electric noise. The latter in turn is temperature dependent. The modern signal processing techniques can substantially improve the SNR making use the key property of the electric noise – its stationarity. The AE signal and noise are additive. Therefore, the stationary spectral characteristics of the noise can be judiciously subtracted from the signal by a variety of methods called commonly “denoising”. Perhaps, the simples workflow aimed at noise subtraction is based on a Fourier analysis and can be implemented for the continuously streaming AE record as follows:
1 The signal is digitally amplified by +14 to +20 dB.
2 The noise is identified on the AE stream (usually at the beginning of the end of the stream when the testing rig is off).
3 The noise Fourier spectral density is calculated, averaged and used as a baseline for subtraction from the entire data stream.
4 Repeat step 1: the denoised signal is amplified again by +40 to +50 dB.
4 Repeat steps 2 and 3: evaluation of noise average spectrum and use at again subtract the noise from data.

![Fig. 5: SEM micrograph showing the dislocation slip pattern ahead of indenter tip. The rectangle indicates approximately the plastic zone where the active sources of AE are seen as individual slip lines in two highlighted slip systems – primary and secondary.](image)

This kind of rectification procedure was applied to the noise-like continuously looking signal shown in Fig. 2. The shape the “denoised” waveform is shown in Fig. 6. We should note that
even a superficial juxtaposition of the raw and filtered signals shown in Figs. 2 and 6, respectively, reveals an obvious differences between these two: (i) the SNR increased spectacularly, revealing fine temporal features in AE, which correspond well to the position of the tip of the indenter during motion, and (ii) the magnified outlook of the waveform (cf. the inset) reveals a discrete nature of the AE time-series at a smaller time scale. The latter observation is particularly important in view of the above discussion on the intermittency of the dislocation glide. The observed small-scale frequent AE bursts. This poses a new challenge for quantification of a real detectability threshold in terms of the number and properties of dislocation segments which can be detected by the AE technique. We do believe that the commonly accepted estimates of the number of cooperatively moving dislocation segments needed for detection of AE signals, which amount to several thousand, are significantly overestimated. The relevant experimental studies are currently in progress and the results will be reported elsewhere.

Fig. 6: Stream of AE signal after FT denoising.

The dislocations emerging at a free surface have long been recognized as a very powerful source of AE [10]. The slip lines ahead of the indenter tip form a clear plastic zone. The size of the plastic zone and the elastic stress distribution in the material under the indenter has been evaluated by many authors is reviewed [11, 12]. Regardless of details of models and calculations, most of them merge at a point that in the first order approximation the plastic zone can be considered semi-spherical with tensile stresses ahead the indenter tip. The radius of the plastic zone is evaluated roughly to be 1.5 – 2 of the width of the scratch. The size of the plastic area was estimated in the SEM as shown in Figure 5. The white rectangle encompasses approximately the elastic-plastic area with easy slip lines it is area of "strongest" sources of acoustic emission signal. In circles shown exit direction of shear bands of different slip systems.

Finally, the analysis of the data shown in Figs. 3 and 4 allows us to conclude that most powerful AE (in terms of Urms or Energy) corresponds to the motion of the indenter through the grains with the crystallographic ordinations favoring multiple slip and highest strain hardening rate in fair agreement with former observations performed during tensile deformation of conventional massive single crystals [5]. To summarize this part observing, the sources of AE signal move ahead relative indenter tip at a distance like stress field.
4. Conclusions

Using the annealed model copper specimens with carefully characterized microstructure and post-mortem observations of the dislocation slip traces in the local area along the path of the indenter during the scratch test we demonstrate that the modern AE technique appears as a viable and powerful tool for investigations of local deformation mechanisms in situ. The capacity of the AE technique to detect low amplitude events from elementary dislocation slip can be substantially improved if a threshold-less continuous data acquisition and appropriate filtering is utilized. The optimal “denoising” strategy has yet to be found to quantify the real detectability limit of the AE technique.

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5. References