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## TWINNING AND SHEAR BAND FORMATION IN CHANNEL-DIE COMPRESSED SILVER SINGLE CRYSTALS IDENTIFIED BY ACOUSTIC EMISSION METHOD

## IDENTYFIKACJA PROCESÓW BLIŹNIAKOWANIA I TWORZENIA PASM ŚCINANIA METODĄ EMISJI AKUSTYCZNEJ PODCZAS NIESWOBODNEGO ŚCISKANIA MONOKRYSZTAŁÓW SREBRA

In the paper there are presented the results of investigations of the relations between acoustic emission (AE) and the mechanisms of twinning as well as shear band formation and propagation in silver single crystals of  $\{112\}<111>$  orientation, subjected to channel-die compression tests at ambient temperature. The results were obtained using an AE analyzer of a new generation, installed recently at the Institute of Metallurgy and Materials Science, and were compared with those obtained also for single Ag crystals of the same orientation, tested at the room and at liquid nitrogen (-196°C) temperatures, but registered applying an AE analyzer of older generation, used until now. The results of AE measurements obtained using the new analyzer allowed to plot the acoustograms obtained with the application of Windowed Fast Fourier Transform (FFT) method for analysis of large experimental data sets (4 X 132 Msamples). This allowed the authors to attack the problem of identification of the strain mechanisms leading to formation of different dislocation systems.

Keywords: acoustic emission (AE), silver single crystals, channel-die compression, twinning, shear bands, strain mechanisms, dislocations

W artykule zaprezentowano zależność pomiędzy natężeniem generowanego sygnału emisji akustycznej (EA), a mechanizmami bliźniakowania i tworzenia oraz rozwoju pasm ścinania w monokryształach srebra o orientacji {112}<111>, poddanych nieswobodnemu ściskaniu w komorze kanalikowej w temperaturze otoczenia. Badania prowadzono przy pomocy analizatora EA nowej generacji zainstalowanego w Instytucie Metalurgii i Inżynierii Materiałowej PAN. Otrzymane rezultaty porównano z wynikami testów ściskania monokryształów srebra o tej samej orientacji prowadzonych przy temperaturze pokojowej oraz przy temperaturze cieklego azotu (-196°C) przy zastosowaniu analizatora EA starszej generacji. Wyniki badań nowszym analizatorem zostały przedstawione w postaci akustogramów uzyskanych za pomocą algorytmu Okienkowej Szybkiej Transformaty Fouriera, zaprojektowanego do obróbki obszernych zbiorów danych eksperymentalnych ( $4 \times 132 \times 10^6$  próbkowań). Zastosowanie metody akustogramów pozwoliło autorom artykułu na identyfikację mechanizmów odkształcenia w badanym materiale, prowadzących do uformowania się różnych systemów dyslokacji.

### 1. Introduction

The data available in literature [1-9,11] do not definitely reveal which of the dislocation processes actually determine the causes and the operation mechanisms of acoustic emission (AE) sources within the scope of not very large plastic deformation. However, the method of AE has been applied for a number of years in the investigations of the dislocation processes in metals subjected to the channel-die compression [5,6]. These investigations have the character of basic research, and their main purpose is to study the causes and the mechanisms of the EA phenomenon accompanying the microstructure evolution, associated, in the first place, with the localization of plastic strain occurring chiefly as the result of twinning and formation of shear bands, generally in fccmetals, and particularly in silver single crystals examined in this work.

The results of our earlier experimental investigations with reference both to the shear bands (e.g.[3]) and to the AE [5-8], allowed to establish the correlations between the mechanisms of plastic deformation and the observed behaviour of AE in polycrystals and monocrystals of fcc metals, whose occurrence were visible especially for single crystals compressed at low temperature [5,6]. The aim of the present study is to attempt to describe the

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correlations between the AE activity and the microstructure evolution related to transition from twinning to shear band formation in silver single crystals of  $\{112\}<111>$ orientation subjected to channel-die compression at ambient and at the liquid nitrogen (-196°C) temperatures.

### 2. Methods

Channel-die compression tests were carried out using INSTRON-6025 tensile testing machine, additionally equipped with a specially constructed channel-die which enables to plastic flow only in the compression direction (normal direction - ND) and in the direction parallel to the channel axis (elongation direction - ED). In this way the plane state of strain is realized, since in the direction perpendicular to the channel walls (transverse direction – TD) no deformation takes place. The velocity of the traverse of the testing machine was 0.05 mm/min. The investigations were carried out on samples of silver single crystals which were obtained by the Bridgeman's method and all samples had the initial shape of a cube of 10 mm edge, and were subjected to the compression tests in three or four steps in order to obtain the appropriate magnitude of the final and the intermediate reductions. At each step the reduction was increased about 15 percent.

Simultaneously with the registration of the external force F, there were measured the AE descriptors: AE events rate, labelled as  $dN_z/dt$ , in the case of the use of equipment of the old generation and the current value of AE signal, sampled at the rate of 44.1 kHz, in the case of the use of the new equipment. AE events rate was recorded in the time interval  $\Delta t = 6s$ . A broad band piezoelectric sensor enabled to register acoustic pulses in the frequency range from 5 kHz to 1 MHz was used but it was proved experimentally that twinning effects generated signals at low (ca. 10 kHz) frequency. During the measurements made with the new analyser 132 Megasamples of AE signal, (i.e. a measuring operation every 27 microseconds) were registered at every course of compression. To present the AE events occurring in such a large data set a method of Windowed Fast Fourier Transform was chosen. Each time window was formed of a data registered during the period of 0.2 second. To improve the selectivity of digital Fourier Transform algorithm the Hann's Window was applied in the signal processing procedure, similarly as in [10]. The number of required calculations was reduced about of 0.25 using elements of FFT method. To present clearly high- and low energy acoustic emission impulses the resulting data set was later processed by computer into the form of an 10-colour acoustograms. Another software allowed for precise AE event detection and evaluation. The contact between the sensor with the sample was maintained by means of a steel rod used as a pad in the channel-die. The total amplification of the acoustic signals was 80 dB, and the corresponding optimal threshold voltage was 1.19V. In order to eliminate or to reduce the undesired effects of friction against the channel walls each sample was covered with Teflon foil. After each compression test microstructural observations were carried out by means of the conventional technique of light microscopy. In this way for each acoustic and mechanical characteristics there are shown the respective micrographs, illustrating the most essential elements of the microstructure evolution.

### 3. Results and discussion

The most convincing results illustrating the correlations between AE behaviour and the evolution of microstructures and strain localization have been obtained for silver single crystals with  $\{112\} < 111 >$  orientation, compressed at ambient (Fig.1) and at low (Fig.2) temperature (-196°C). It is clearly seen (compare the microstructure presented in Fig.1) that the evolution of microstructure during the compression consists first in a transition from the process of twinning to the formation of primary family of shear microbands. The transition from twinning to shear bands is still more distinctly visible in the case of silver monocrystals of the same orientation {112}<111> compressed at liquid nitrogen temperature (Fig.2a and 2b). The sequence of the microstructure images in these figures and the behaviour of AE indicate clearly that scheme of the microstructure evolution is as follows: the predominance of twinning, the decay of twinning and the beginning of the formation of a primary family of shear band (Fig.2a) and next the predominance of the formation of shear band of the secondary family (Fig.2b). AE events evoked by the described phenomena at room temperature have smaller energies than those investigated at -196°C. This was a reason of improving of the AE registration technique.



Fig. 1. The rate of AE events registered every 4 seconds, external force and corresponding microstructure (at the right: deformations twins - T and shear bands - SB) of Ag single crystals during channel-die compression (T = 293K, reduction z = 31.8%, orientation {112}<111> at image magnification x200)

Figures 3 and 4 present the results of channel-die compression test made at room temperature and registered with a new AE analyser. Application of the high-speed data sampling technique allowed the authors to construct the acoustogram of the registered data (Fig.4a). Analysis of the force vs. time (Fig.3) and the acoustogram (Fig.4a) shows clearly the existence of two ranges of the twinning. The first is related to a smaller frequency, whereas the second one to the greater frequency of the appearing of twins. This effect occurring of two rates of AE signals related to twinning processes is most probably connected, with the fact that in the first range the twinning occurs in the central part of a sample where the time of twin plates formation is markedly longer than in the second range where the twins are formed in the peripheral parts of the specimens.



Fig. 2. AE events registered every 6 seconds, external force and microstructure of Ag single crystals of  $\{112\}<111>$  orientation during low temperature compression in channel-die: (a) – reduction z = 33.0%, and (b) – reduction z = 63.4%, at image magnification x200



Fig. 3. External force, registered at channel-die compression of single Ag crystal with the existence of two ranges of the twinning



Fig. 4. Acustograms, registered at channel-die compression of single Ag crystal with the application of the new AE analyser – with improved resolution of measured data. Applied force is shown in Fig.3. The upper part presents high energy twinning process, the lower part presents low energetic shear band formation

The effect of shear band formation was also registered in the specimen presented in Fig.4b after exceeding 40% of reduction of specimen dimensions. AE energy of that effect was ca. two orders of magnitude weaker than the signal registered at twinning and also its spectral characteristic was of another type. The effect of shear band formation generates a narrow-band AE signal at frequency of 16 kHz. There is also no remarkable sign of shear band formation to trace at the force curve – in time interval presented in Fig.4b, that is, at 1700 to 2300 s. Its worth of mention that AE signal generated during twinning phase has remarkably greater amplitude when compared to the other one related to the shear band formation what can be useful to distinguish the both processes.

# 4. Conclusions

Acoustic emission signals registered during channel-die compression tests in silver single crystals are generated by twinning and shear band formation processes. The first mentioned phenomenon releases greater amounts of energy then the second one. The application of Windowed Fourier Transform and mapping using acoustograms of registered AE signal can be treated as a tool for precise capturing the occurred AE events and determining of their energies. The authors of present study are going to apply the described investigation method for monitoring the similar processes in other crystals.

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