

INFLUENCE OF THE STRAIN RATE ON THE PLC EFFECT AND ACOUSTIC EMISSION IN SINGLE CRYSTALS OF THE CuZn30 ALLOY COMPRESSED AT AN ELEVATED TEMPERATURE

VPLIV HITROSTI DEFORMACIJE NA POJAV PLC IN AKUSTIČNO EMISIJO MONOKRISTALOV ZLITINE CuZn30, STISKANE PRI POVIŠANI TEMPERATURI

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The purpose of these investigations was to determine the effect of the strain rate on the phenomenon of a heterogeneous plastic deformation of the Portevin–Le Chatelier type while testing free compression of CuZn30 single crystals with a crystallographic orientation of [139] at 300 °C. Moreover, the relations between the work-hardening curve $\sigma - \epsilon$ displaying the PLC effect and the characteristics of the signals of the acoustic emission generated in the uniaxial-compression test were determined. It was found that the process of plastic deformation of the tested single crystals in the analyzed range of the frequencies up to 35 kHz generates differentiated sources of acoustic-energy emission, mainly the impulsive emission generated by signal events, correlated with the oscillations of the stresses on the work-hardening curves $\sigma - \epsilon$. The strain rate mainly causes the changes in the intensity of the oscillation typical for the PLC effect.

Keywords: plastic strain, Portevin–Le Chatelier effect (PLC), single crystals, copper alloys, compression test, acoustic emission (AE)

Namen raziskav je bil ugotoviti vpliv hitrosti deformiranja pri preizkusu stiskanja monokristalov CuZn30 s kristalografsko orientacijo [139] pri 300 °C na pojav heterogene plastične deformacije vrste Portevin-Le Chatelier. Poleg tega so bile ugotovljene še odvisnosti med krivuljo deformacijskega utrjevanja $\sigma - \epsilon$, ki kaže pojav PLC, in značilnostmi signalov akustične emisije pri enoosnem tlačnem preizkusu. Ugotovljeno je, da proces plastične deformacije preizkušanih kristalov v analiziranem področju frekvenc do 35 kHz proizvaja diferencirane vire emisije akustične energije, predvsem impulzivne emisije signalov, ki se skladajo z oscilacijo napetosti na krivulji deformacijskega utrjevanja $\sigma - \epsilon$. Hitrost deformacije se kaže predvsem v spremembi intenzitete oscilacij, značilnih za PLC-pojav.

Ključne besede: plastična deformacija, Portevin-Le Chatelierov pojav (PLC), monokristali, zlitine bakra, tlačni preizkus, akustična emisija (AE)

1 INTRODUCTION

In order to determine the kinetic relations and structural changes, conditioning the mechanisms of plastic deformation it is necessary to know the process and understand the essence of various phenomena occurring in the course of plastic deformations. In many alloys a heterogeneous plastic deformation in the form of irregularities on the work-hardening curve is observed during tensile (or compression) tests. The earliest investigations concerning this phenomenon in medium-carbon steel and aluminium were published by Portevin and Le Chatelier in 1923, hence, it is called the PLC effect. Although the effect of the instability of plastic deformations of the PLC type has been known and investigated for nearly one hundred years, it is still not fully recognized and explained.¹⁻⁷ Generally, this effect was investigated on the basis of material factors, taking into account the microstructural conditions of an initiation of a localized plastic deformation and the rheological factors connected

with the mechanics of plastic deformations at various thermodynamic and physico-chemical conditions.

One of the more recent methods of analyzing the phenomena occurring in the course of a plastic deformation is the acoustic emission (AE). This method consists of a detection and analysis of the acoustic signal emitted by the material while it is being mechanically loaded. The acoustic signal is a result of the propagation of elastic waves generated in the material due to a fast release of the energy accumulated in this material. The shape of an AE signal is affected by many factors such as the chemical composition and macrostructure of the tested material, the kind of the applied heat treatment,^{8,9} the temperature and strain rate ($\dot{\epsilon}$),¹⁰ the grain size,¹¹ the texture and the state of precipitations.^{12,13} The investigations based on AE measurements are characterized by non-invasiveness and an incomparably high sensitivity in recording physical phenomena in comparison with the other methods of investigations.

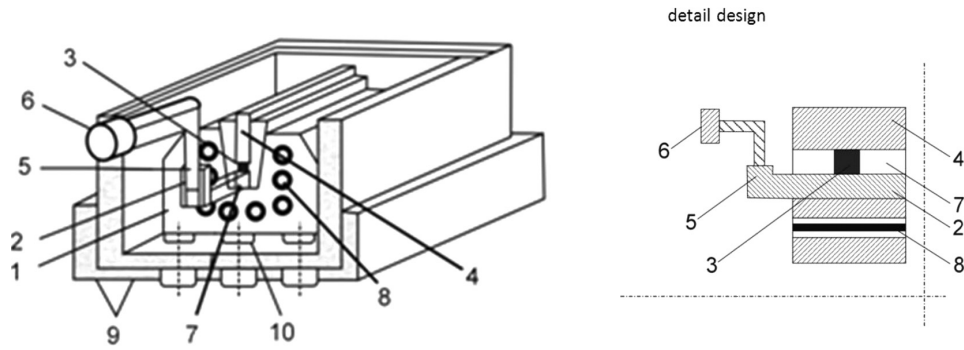


Figure 1: Duct-die block for deforming the samples at an elevated temperature with an AE probe: 1 – die-block body, 2 – guide bar, 3 – sample, 4 – punch, 5 – wave guide, 6 – AE probe, 7 – duct, 8 – heating elements, 9 – foamed polystyrene, 10 – tack bolt

Slika 1: Orodje s kanalom za deformiranje vzorcev pri povišanih temperaturah z uporabo AE-sonde: 1 – telo orodja, 2 – paličasto vodilo, 3 – vzorec, 4 – tlačilna palica, 5 – valovni vodnik, 6 – AE-sonda, 7 – vodilni kanal, 8 – grelni element, 9 – penjeni polistiren, 10 – spenjalni vijak

Table 1: Chemical composition of the monocrystalline alloy applied in the investigations

Tabela 1: Kemijska sestava monokristalne zlitine, uporabljene pri preiskavah

No.	Determination of the alloy and the kind of the applied analysis of CuZn30	Chemical composition, mass fractions, w/%						
		Zn	Fe	Al	Ni	Sn	Pb	Cu
1	smelting analysis of CuZn30 ingot	30.3	0.024	0.039	0.024	0.003	0.01	bal.
2	CuZn30 PN-EN 12163:2002	28.3–30.3	max 0.05	max 0.02	max 0.3	max 0.1	max 0.05	bal.

The aim of this paper is to determine the influence of the strain rate in the range of 10^{-5} – 10^{-1} s⁻¹ on a non-homogeneous plastic deformation, the so-called Portevin-Le Chatelier effect, in a single-crystalline CuZn30 alloy with the crystallographic orientation of $[\bar{1}139]$, compressed at a temperature of 300 °C. The additional aim is to determine the relation between the behaviour of the signals of acoustic emissions generated in the course of a plastic deformation at elevated temperature and the shape of the work-hardening curves $\sigma - \epsilon$ in the range of the PLC effect.

2 EXPERIMENTAL PROCEDURE

The investigated material was a single-crystalline CuZn30 alloy in the form of a rod with a diameter of 3.8 mm, a length of about 200 mm and crystallographic orientation of $[\bar{1}139]$, the chemical composition of which is shown in **Table 1**. Single crystals of this alloy were obtained with the Bridgman’s method in a vertical, electrical tubular heating furnace by displacing the temperature gradient of the zone in the furnace in

relation to the crucible (a quartz tube), with a charge in an atmosphere of inert gas.

In order to realize the purpose of these investigations the following procedures had to be carried out:

- mechanical tests of free compression of single crystals at 300 °C and a strain rate of 10^{-5} – 10^{-1} s⁻¹,
- investigations concerning the plastic deformation of single crystals by means of acoustic emission.

Compression tests of single crystals were performed at a temperature of 300 °C and at $\dot{\epsilon}$ amounting to 10^{-5} – 10^{-1} s⁻¹ on a universal testing machine INSTRON 3382 equipped with a duct die including heating elements and a quartz outlet of the waveguide with an AE sensor (**Figure 1**). For the AE measurements the following setting was used: the acoustic-emission wideband sensor of type WD (PAC) provided by a company called Physical Acoustic Corp., a low-noise amplifier set to 66 dB of gain, background-noise discrimination at 0.75 V and 12-bit digital-to-analogue conversion at a rate of samples $88.2 \cdot 10^3$ s⁻¹. The settings used for the AE acquisition had been adjusted experimentally during the

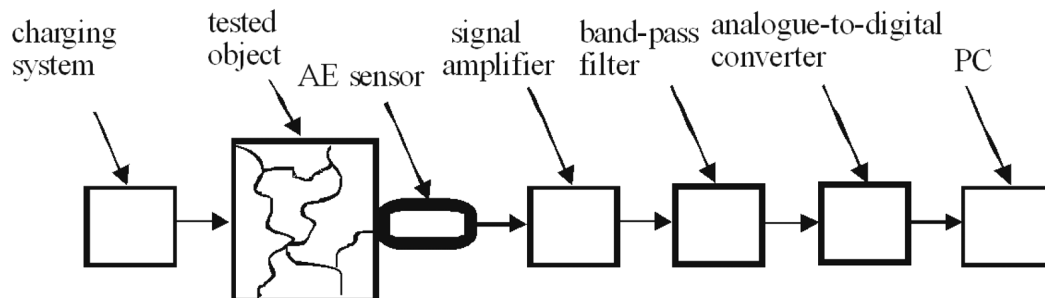


Figure 2: Simplified block diagram of the measurement and AE recording system

Slika 2: Poenostavljeni sestav merilnega in zapisovalnega sistema AE

previous tests dealing with the tension and compression of metal samples.^{14,15} The final deformation of the sample after the compression test amounted to about 50 %. The values of the forces within the entire range of the measurements were recorded with an accuracy of up to 0.5 %. The acoustic emission was measured during the compression tests of microcrystalline samples. In order to reduce the coefficient of friction between the butting face of the compressed sample and the steel punch, the samples were coated with a strip of Teflon.

The block diagram of the measurement, the system and the recording of AE are illustrated in **Figure 2**. The AE measurement system was connected with the recording system of the testing machine. The tests were carried out in the Accredited Laboratory of the Strength of Materials, the Polish Academy of Science, Cracow.

3 RESULTS AND DISCUSSION

The influence of the strain rate in the range of 10^{-5} – 10^{-1} s⁻¹ on the mechanical characteristics $\sigma - \epsilon$ of the CuZn30 single crystals with the initial crystallographic orientation of [139] at the constant temperature of the compression amounting to 300 °C is presented in **Figure 3** and **Table 2**. It was found that the strain rate does not cause any changes in the general shape of the work-hardening curves of the investigated single crystals, but causing a considerable effect on the level of the stresses at the yield point and intensive oscillation of the stresses on these curves. When the strain rate grows, a tendency toward decreased values of the actual stresses could be observed, particularly during the initial stage of the deformation (15–20 %). Moreover, a tendency toward decreased oscillation of the stresses on the analytical work-hardening curves was observed.

At the investigated temperature of the compression, the strain rate considerably influences the intensity of the oscillation of the stresses characterized by an occurrence of the PLC effect (**Figure 3**). The conditions of the plastic instability of the deformed single crystals are distinct in the case of low values of $\dot{\epsilon}$ (10^{-5} – 10^{-4} s⁻¹) but they fade at a medium strain rate of about 10^{-3} s⁻¹ and do

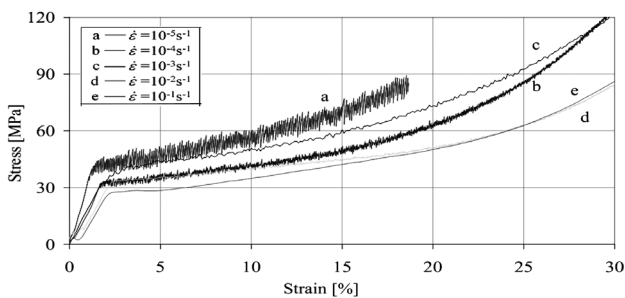


Figure 3: Influence of the strain rate on the shape of curves $\sigma - \epsilon$ of CuZn30 single crystals with the initial orientation of [139], compressed at 300 °C

Slika 3: Vpliv stopnje deformacije na obliko krivulje $\sigma - \epsilon$ monokristalov CuZn30 z začetno orientacijo [139], stisnjenih pri 300 °C

not occur when $\dot{\epsilon} > 10^{-2}$ – 10^{-1} s⁻¹. It was also found that the strain rate only slightly affects the type of the oscillation of the stress in the described conditions of deformation.

According to the classification by Brindley and Worthington^{16,17} three fundamental types of the oscillation of stresses are to be distinguished. Type A occurs at a low temperature of a tensile test, characterized by an increase, followed by a sudden drop in the force, such as the oscillation of the forces occurring periodically. At elevated temperatures type-B deformation oscillations occur that are smaller and irregular. These oscillations are symmetrical in relation to the level of the work-hardening curve. Type C occurs at the highest temperature of a plastic deformation. These oscillations are characterized by a decrease of the force in relation to the level of the work-hardening curve and they are often separated from the area of homogeneous plastic deformation.

According to this system, on the compression curves ($\sigma - \epsilon$) of the tested single crystals in the range of deformation ϵ (5–15 %), type-B oscillations were detected. The initiation of the PLC effect, conditioned by the critical strain (ϵ_c), was observed on the work-hardening curves in the range of ϵ (1.2–2 %). In most cases the initiation of the oscillations on the curves of the compression coincides with the yield point of the material. The value of ϵ_c proved to be rather independent of the strain rate. In order to quantitatively describe the serration on the stress-strain curves, the analytic parameters used in quantitative fractography were applied.¹¹ One of them, the so-called coefficient of development of the R_L line, was derived from the relation of $R_L = L/L'$, where L is the length of a given segment of the $\sigma - \epsilon$ line and L' is the length of its projection. An approximately normal projection on the stress-strain curves was used.

The geometrical analysis also indicates that for the serration occurring with a particular frequency (f), R_L can be calculated with equation $R_L \approx (\bar{A}/f)^{-1}$, where \bar{A} is the average amplitude of serration. The parameters calculated in compliance with the MATLAB software are gathered in **Table 2**. It was found that the higher the values of coefficient R_L (at a higher amplitude and

Table 2: Specification of the coefficients of the shape of $\sigma - \epsilon$ curves in CuZn30 single crystals with the orientation of [139] compressed in the range of deformations (5–15 %)

Tabela 2: Pregled koeficientov oblike krivulje $\sigma - \epsilon$ v monokristalih CuZn30 z orientacijo [139], stisnjenih v območju deformacij (5–15 %)

No.	Temperature of deformation (°C)	$\dot{\epsilon}/s^{-1}$	$\epsilon_c/\%$	Coefficient of the shape of $\sigma - \epsilon$ curves with the PLC effect		
				R_L	A	f
1	300	10^{-2}	2.7	6.0	1.5	9.2
2		10^{-3}	2.6	4.1	2.9	12.2
3		10^{-4}	1.6	43.7	3.8	166.2
4		10^{-5}	1.2	71.6	5.5	395.2

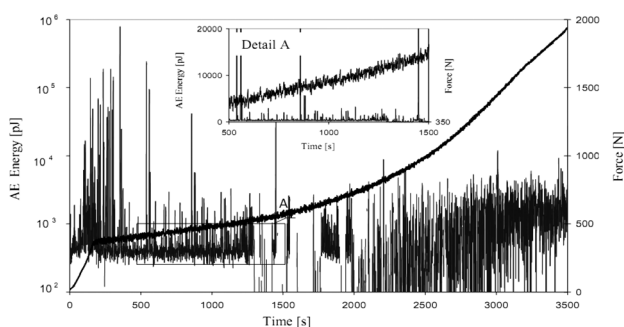


Figure 4: Dependence of the AE energy on the PLC effect recorded on the work-hardening curve during the compression testing of mono-crystalline CuZn30 alloy at 300 °C and $\dot{\epsilon}$ up to about 10^{-4} s^{-1}

Slika 4: Odvisnost energije AE od PLC-učinka, zabeleženega na krivulji preoblikovalnega utrjevanja pri tlačnem preizkusu monokristala zlitine CuZn30 pri 300 °C in $\dot{\epsilon}$ do 10^{-4} s^{-1}

frequency of serration), the larger is the instability of the PLC plastic deformation (**Table 2**). The highest values of coefficient R_L (about 72) in the range of deformations ϵ (5–15 %) as well as the highest value of the amplitude of stresses (calculated as $2 \cdot A$) amounting to about 11 MPa were determined for the samples compressed at the temperature of 300 °C and with $\dot{\epsilon}$ amounting to about 10^{-5} s^{-1} . The obtained values confirm the qualitative description of the compression curves.

The obtained results allow us to maintain that in the tested single crystals the PLC effect is a result of dynamic strain aging (DSA), which is an interaction between the sliding dislocations and free atoms. The sources of these interactions, complying with the dynamic-dislocation model of the PLC effect, are the multiplications of dislocations in the course of being affected by Frank-Read sources. The occurrence of DSA is conditioned by the rate of the migration of foreign atoms constituting the Cottrell atmosphere. The effects of the atoms of the alloy and the impurities with dislocations are responsible for retarding the dislocations, and thus also for hardening the alloy. If, therefore, a given strain rate is "imposed", we must also apply a stress that exceeds the resistance to the motion of dislocations, including the resistance resulting from the effect of the atoms of the alloy with dislocations. In order to explain, in a simple way, the qualitative model of the changes in the stress in the hardening diagram, we must assume that the break-away of a dislocation from the alloy atoms reduces the stress. This is indispensable for a further displacement of the dislocation by the value of this effect. The moment of a break-off of a dislocation from the blocking atoms involves a sudden drop in the force exciting the deformation.^{18–20}

According to the dislocation-dynamic model of the PLC effect, every local drop in the loading force recorded on the $\sigma - \epsilon$ curves is connected with unlocking the dislocation sources in a certain localized area of the sample. If there is a high concentration of internal stresses, the adjacent sources of dislocation will, successively, become unlocked due to these stresses. Therefore,

the unlocked sources of dislocation operate in a state of being considerably overcharged, so that the dynamically generated dislocation increases. Consequently, this involves a formation of sliding bands, which then propagate until the time of waiting (t_w) again reaches the value of the time of aging (t_a). Then, all the sources of dislocation are effectively locked by the Cottrell atmosphere,²¹ after which the process of unlocking starts again.

The results of the measurements of the acoustic emission (AE) during the compression testing of CuZn30 single crystals with the orientation of $[139]$ at the temperature of 300 °C and the strain rate of 10^{-5} – 10^{-1} s^{-1} are gathered in **Figures 4 to 6** and **Table 3**. In the investigations of AE the descriptors of the recorded signals are usually added, such as:

- the count rate over the preset noise-discrimination level
- the signal amplitude within an AE event
- the energy of an AE event, understood as a half of the product of the AE-event duration and the squared amplitude of the AE event.

Table 3: Specification of the descriptors of the AE signals in the R_e range concerning the investigated parameters of compression

Tabela 3: Pregled opisovalcev AE-signalov v območju R_e preiskovanih parametrov pri tlačnem preizkusu

No.	Temperature of deformation (°C)	Strain rate $\dot{\epsilon}/\text{s}^{-1}$	Sum of AE events in the R_e range	Average energy of the AE events in the R_e range (pJ)
1	300	10^{-4}	14539	20.3
2		10^{-3}	8903	42
3		10^{-2}	6714	68
4		10^{-1}	816	30

The last parameter was applied in the presented research because the squared signal amplitude combined with the event duration applied in the formula for data processing distinguishes well the signals caused by material effects from the unwanted signals caused by the drive of the loading machine and other system noises. This is because the unwanted AE sources have different spectral characteristics than the desired ones.

In most compression tests of the investigated single crystals a distinct growth of the activity of the AE energy was recorded during the initial stage of their compression and in the area of passing from the elastic to the plastic range (**Figures 4 and 5**). This growth depends on the value of $\dot{\epsilon}$ when deformations ϵ amount to about 2–5 %.

An increase in AE is in both cases characterized by a more or less wide maximum change in the energy of the signal, after which AE reaches its minimum. The observed increase in the energy of the AE activity in the initial stages of the $\sigma - \epsilon$ curves may, however, be caused, among others, by mechanical factors due to the friction and the matching of the sample to the compress-

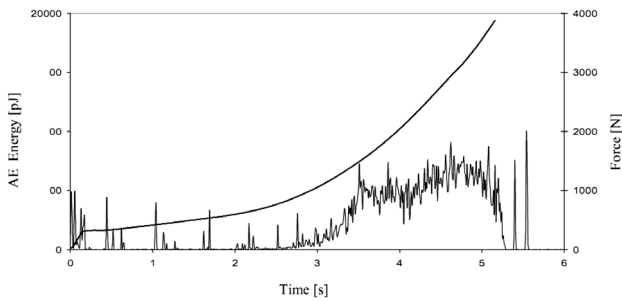


Figure 5: Dependence of the AE energy on the shape of the work-hardening curve during the compression testing of a CuZn single crystal at 300 °C and $\dot{\epsilon}$ amounting to about 10^{-1} s^{-1}

Slika 5: Odvisnost energije AE od oblike krivulje napetostnega utrjevanja med tlačnim preizkusom monokristala CuZn pri 300 °C in vrednostjo $\dot{\epsilon}$ do 10^{-1} s^{-1}

ing punch of the testing machine. In the range of the yield point, however, where AE displays a merely physical aspect, the increasing AE activity is undoubtedly connected with the processes of dislocation. The AE level in this stage of the hardening of the alloy was found to be much more intensive than in the advanced stages of deformation occurring in the cases of greater drafts.

The sum and mean energy of AE depending on the strain rate during the compression test are gathered in **Table 3**. It was found that in the $\dot{\epsilon}$ range from about 10^{-4} s^{-1} to about 10^{-2} s^{-1} the sum of events in the range of the yield point (R_e) increases with the slowing down of the strain rate. The opposite is the case when the energy of a single event reaches its mean value. A higher average value of the energy of the event, except for $\dot{\epsilon}$ amounting to about 10^{-1} s^{-1} , corresponds to a smaller sum of events. The minimum sum of events of about 816 also corresponds to a low value (30 pJ) of the average energy of AE events. The highest sum of events occurred in the case of the single crystals deformed at the temperature of 300 °C with $\dot{\epsilon}$ amounting to about 10^{-4} s^{-1} and the mean value of the energy of event of about 20 pJ.

It was found that in the first stage of the hardening (the stage of easy sliding) of the single crystals that do not display any PLC effect (**Figure 5**), the AE level is lower, growing with the increase in the rate of deformation, whereas in the single crystals displaying the PLC effect, the AE level is higher and its characteristics are more complex. A change in the strain rate at a given temperature does not involve any essential changes in the level of the frequency of AE. In the second stage of the hardening within the $\dot{\epsilon}$ range of (10^{-5} – 10^{-1} s^{-1}) the AE activity is greater, particularly during the initial stage. These are mostly single samples of the AE signal characterized by differentiated energy or a continuously increasing level of the AE energy, which grows with the increasing rate of deformation, independent of the occurrence of the PLC effect in the course of the compression of single crystals.

When a work-hardening curve is of a parabolic character (the third stage of hardening), AE appears in the

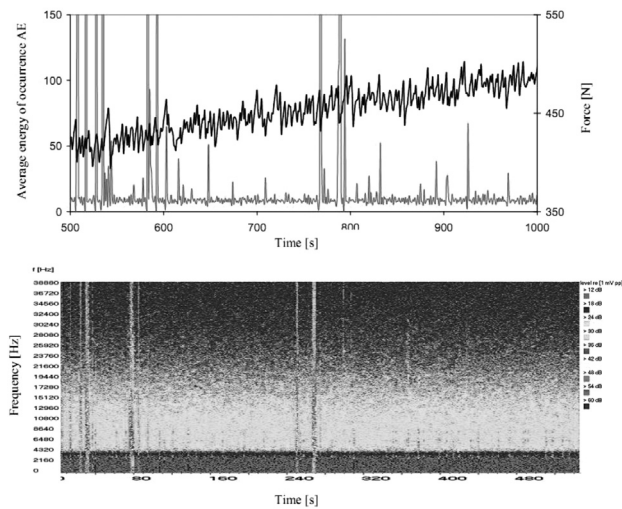


Figure 6: a) Dependence of the AE energy on the serration along the stress-strain curve (within the fragment of the diagram presented in **Figure 4**) of a CuZn30 single crystal with the orientation of [139] at 300 °C and $\dot{\epsilon}$ up to about 10^{-4} s^{-1} and b) an acoustogram corresponding to this set of EA events

Slika 6: a) Odvisnost energije AE od nazobčanosti vzdolž krivulje napetost – deformacija (z izrezkom iz diagrama, predstavljenega na **sliki 4**) pri CuZn30-monokristalu z orientacijo [139] pri 300 °C in $\dot{\epsilon}$ do 10^{-4} s^{-1} in b) akustogram, ki ustreza temu sklopu AE-dogodkov

form of strong cumulative maximum values of the energy changes in the signal in time, particularly at the beginning. Irrespective of the appearance of the PLC effect in the course of the compression testing of single crystals, it was found that in the final stage of the deformation there is also a range, in which the AE energy is intensified, although less intensive with respect to the number of pulses than in the R_e range. The observed correlations between the AE behaviour and the exerted compressive force and the evolution of the microstructure may be satisfactorily explained with the qualitative level based on the dynamic processes of dislocation connected with the motion of dislocation.^{8–10,19} The processes of the formation of sliding lines in the range of the yield point were proved to be acoustically most effective. Every collective dislocation motion in the sliding systems probably leads to a release of elastic energy, generating the recorded signals.

In most compression tests of the CuZn30 single crystals displaying the PLC effect sudden reductions of the compressive forces recorded on the work-hardening curve were found to be distinctly correlated with the AE peaks (**Figure 6**). It is supposed that every increase in the oscillation of the stress on the $\sigma - \epsilon$ curves is connected with a locking of dislocations, and that every violent reduction represents a motion of a large group of dislocations (avalanches of dislocations). Similarly, it is assumed that only regular oscillations of the stresses of certain given types are connected with the generation of slip bands of the PLC effect.

4 CONCLUSIONS

The investigations dealt with in this paper lead to the following conclusions:

- The phenomenon of instability of the plastic deformation of the Portevin-Le Chatelier type occurs in the tested single crystals of alloy CuZn30 in the course of free compression at 300 °C within a limited range of the strain rate from 10^{-5} s^{-1} to 10^{-2} s^{-1} .
- In the conditions of free compression applied in the investigations, CuZn30 single crystals display characteristic oscillations of the stresses on the work-hardening curves, corresponding to the oscillations classified in the literature as the B type.
- The increasing strain rate in the compression tests leads to a distinct decrease in the stress-oscillation intensity, typical for the PLC effect.
- The plastic deformation of the investigated single crystals at elevated temperature generates in the analyzed range of frequencies (up to 35 kHz) diversified source of AE energy, mainly an impulsing emission from single events, i.e., pulsating acoustic signals with a high energy in the frequency band from 4 kHz to 8 kHz.
- The correlation between the PLC and AE effects in the tested single crystals can be explicitly explained on the basis of a dislocation-dynamic model of the PLC effect.
- In the process of plastic deformation of the tested single crystals, the applied AE method exhibits the dependence of the activity of acoustic emissions on the given stage of the hardening of the analyzed alloy.
- The intensity of AE increases mainly in the range of the yield point (R_e) on the $\sigma - \varepsilon$ curves, and also in the case of considerable deformations. When the maximum intensity of the AE signal has been reached, it fades, in most cases, to the minimum value.

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