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THE PORTEVIN – LE CHATELIER EFFECT AND ACOUSTIC EMISSION OF PLASTIC DEFORMATION CuZn30 MONOCRYSTALS

EMISJA AKUSTYCZNA W BADANIU EFEKTU PORTEVIN-LE CHATELIER W MONOKRYSTALICZNYM STOPIE CuZn30

The paper presents the investigation of the relation between the acoustic emission (AE) and instability of plastic deformation type Portevin-Le Chatelier (PLC) of single-phase brass CuZn30 monocrystals with crystallographical orientation [13 9]. The monocrystals have been investigated applying the method of free compression at a constant strain rate and the temperature within the range from 200°C to 400°C, simultaneously recording PLC phenomenon by means of acoustic emission. During hot axial compression tests the correlation between work-hardening curves σ - ε , which display PLC effect and characteristic of acoustic emission signals has been found. Moreover, it was proved that in the range of the PLC effect, the acoustic signal is an impulse a character of cyclic repeatability, distinctly correlated qualitatively with the stress oscillations on the curves σ - ε . The analysis of the obtained results leads to the conclusion that in the tested monocrystals the effect PLC is probably controlled by complex processes similar to the phenomenon of dynamic strain ageing (DSA), which are described by diffusion models.

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

W pracy badano zależność między emisją akustyczną (EA), a niestabilnością odkształcenia plastycznego typu Portevin Le Chatelier monokryształów jednofazowego mosiądzu CuZn30 o orientacji krystalograficznej [13 9]. Monokryształy poddano próbie swobodnego ściskania przy stałej prędkości odkształcenia w zakresie temperatury od 200°C do 400°C stosując jednocześnie pomiar emisji akustycznej. Określono zależność pomiędzy przebiegiem krzywych umocnienia σ - ε wykazujących efekt PLC, a charakterystyką sygnałów emisji akustycznej generowanych w badanej próbie ściskania jednoosiowego. Stwierdzono, że w zakresie występowania efektu PLC podczas próby ściskania, sygnał EA ma charakter impulsu cyklicznego, wyraźnie skorelowany jakościowo z oscylacjami naprężeń na krzywych σ - ε . Analiza uzyskanych wyników pozwala przypuszczać, że w badanych monokryształach efekt PLC jest kontrolowany prawdopodobnie przez złożone procesy podobne do zjawisk starzenia dynamicznego po zgnioście (DSA), które są opisane modelami dyfuzyjnymi.

1. Introduction

Plastic deformation of crystalline solid bodies is a complex process, mostly heterogeneous, due to the simultaneous effect of several mechanisms of deformation. The mechanism of deformation, which dominates depends on the properties of the material and also on external coefficients, viz. temperature, stress and strain rate. As metallic materials are practically widely applied, the knowledge of the structural processes taking place in any given conditions of plastic deformation is of essential importance.

Many metallic materials exhibit serrations on their global stress/strain tensile curves in a given range of temperature and applied strain rate. The occurrence of such serrations is called the Portevin-Le Chatelier effect (PLC) known in literature as the effect of plastic instability, often synonymically defined as "jerky flow" or "serration". For the first time this effect was

detected in iron at the temperature of deformation 110÷300°C [1]. It often occurs also in many alloys of non-ferrous metals, e.g. copper, aluminium, titanium and nickel, based superalloys and also in some aluminium-ceramic composites materials, in the course of their deformation at elevated temperatures [2÷6]. Characteristic stress oscillations seen on the work-hardening curve in the range of plastic flow differ from each other both in shape and size, which depends among others, on the temperature and the strain rate ($\dot{\varepsilon}$). Brindley and Worthington [7] systematized the character of these oscillations, distinguishing three basic types of them, which are seen in Fig. 1 [8]. Type A occurs at a low temperature of the tensile test. It is characterized by a growth and then a sudden drop of the force; such oscillations of the force occur periodically. The heterogeneous stress arises above the curve σ - ε after the occurrence of the critical stress. Type B occurs in the case of an elevated temperature of the tensile tests. It is usually observed after

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oscillations of the type A. Oscillations of the flow stresses occur symmetrically in the range of the curve σ - ε . Type C occurs at the highest temperature of plastic deformation. It is characterized by a heterogeneous range of plastic deformation and a decreased stress below the level of the curve σ - ε . The presented classification of the PLC effect is generally applied, although in some papers this way of classification has been modified [9].

The phenomenon of heterogeneous plastic strain Portevin- Le Chatelier type is in some conditions of plastic working an essential factor determining the possibility of application in some metals and alloys. In the literature for many years there were efforts to explain the causes of this phenomenon, but until now they were explained univocally. Mechanical and structural models of the PLC effect have been elaborated by means of macro-and microparameters of plastic deformation, such as: sensitivity of stress to the strain rate or strain – hardening coefficient.

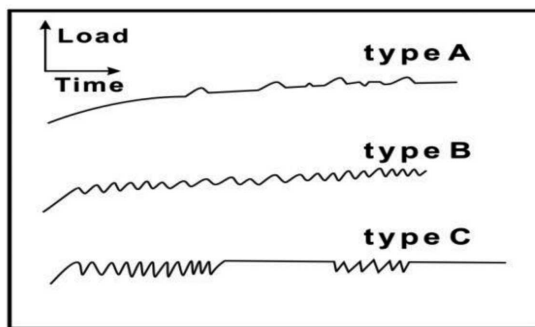


Fig. 1. Types of oscillation of the stress characteristics for the PLC effect [8]

For the purpose of investigating the PLC effect of the instability of deformation usually conventional mechanical tests of uniaxial tension or compression are applied, but also modern methods are used, e.g. the digital correlation of the image or acoustic emission (AE) [10÷18]. Investigations making use of acoustic emission consist in the detection and analysis of acoustic signals emitted by the material during mechanical loading. The acoustic signal results from the propagation of elastic waves generated in the material due to the rapid release of the energy accumulated in this material. The release of elastic strain energy in some materials is connected with the formation of instantaneous local unstable states, which may result from processes accompanying miscellaneous phenomena, viz. from those occurring in the submicroscopic scale, e.g. diffusion of atoms into adjoining positions of the crystalline

lattice, to phenomena in the macroscopic scale, e.g. cracking of the material. The plastic deformation of metals and alloys is directly connected with the dislocation movement depending on the micro-structure of the material. Acoustic emission is applied in investigations concerning the PLC effect as a highly sensitive method of measuring elastic waves arising in the material due to the dynamic local reconstruction of the structure. Comparing the obtained AE results with the diagrams of tensile tests, we can analyze the effect of various parameters of deformation on the PLC phenomenon. This method is often used while investigating the precipitation of hardened alloys, because during this process two mechanisms frequently turn up, conditioning the PLC effect, viz. the effect of foreign atoms with dislocations (Cottrell's model) and the shearing of precipitations [19÷21]. Acoustic emission measurements are characterized by non-invasive testing and an incomparably high sensitivity in recording the physical phenomena in comparison with other methods of investigations [22÷24].

The aim of the present paper is to determine the effect of temperature in the free compression test of the monocrystalline alloy CuZn30 with a crystallographic orientation $[\bar{1}3\ 9]$ within the range of 200°C to 400°C, with a strain rate to about 10^{-4}s^{-1} upon the phenomenon of heterogeneous plastic deformation, the so-called Portevin-Le Chatelier effect, as well as to determine the dependence of the descriptors of the signals of acoustic emissions, generated in the course of testing on the shape of the curves of work-hardening σ - ε .

2. Investigated material

The metallic charge for the production of monocrystals was single-phase brass of the type CuZn30, assayed as CW506L, in compliance with PN-EN [25], whose chemical composition is shown in Table 1. The alloy CuZn30 is monocrystallized by means of the method of crystallization with a controlled temperature gradient in laboratory conditions making use of an electrical tubular furnace permitting a displacement of the zone of the temperature gradient of the furnace towards the crucible containing the charge [26].

In order to determine the temperature effect of compression on the mechanism of the Portevin-Le Chatelier phenomenon in monocrystalline CuZn30 the following tasks had to be undertaken:

- mechanical free compression tests of monocrystals with a crystallographic orientation $[\bar{1}3\ 9]$ at elevated temperature,
- investigations concerning the process of plastic deformation of monocrystals by means of acoustic emission.

TABLE 1

Chemical composition of the alloy for the production of monocrystals

No.	Denotation of the alloy and the type of analysis	Chemical composition, % by weight						
		Zn	Fe	Al	Ni	Sn	Pb	Cu
1	CuZn30 ingot analysis of smelting	30.3	0.024	0.039	0.024	0.003	0.01	rest.
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	rest.

Compression tests of monocrystals were performed within the temperature range of 200°C to 400°C at strain rate ($\dot{\epsilon}$) to about 10^{-4}s^{-1} , using a testing machine INSTRON 3382, equipped with a duct die, containing heating elements and an quartz outlet of the waveguide and AE sensors (Fig. 2). In the uniaxial free compression test the duct-die block (matrix) played merely the role of the holder of the sample exerting pressure of the external force, whereas its bottom plate is also applied as a natural metallic conductor for signals of acoustic emission generated in the tested monocrystalline sample. In this way an original solution was attained concerning the problem of the contact between the tested sample and the sensor of acoustic emission. These investigations were carried out at the Accredited Laboratory of Strength of Materials, Polish Academy of Sciences, in Cracow. The final deformation of the sample after the compressive test was about 50% and the accuracy of the recorded force within the entire range of measurements up to 0.5%. The acoustic emission was measured during the tests of free compression of monocrystalline samples in the form of a rod with the diameter of 3.8 mm and initial length of about 6 mm. For these investigations selected were monocrystals, whose surfaces displayed the least porosity. In order to reduce the coefficient of friction between butting face of the compressed sample and the steel punch an interlayer of thin (75 μm) teflon (PTFE) foil was applied, the advantage of which is that it does not introduce an additional source of acoustic emission. The block diagram of the measuring and recording system AE has been presented in Fig. 3. The measuring system AE is connected with the system recording the results of the testing machine.

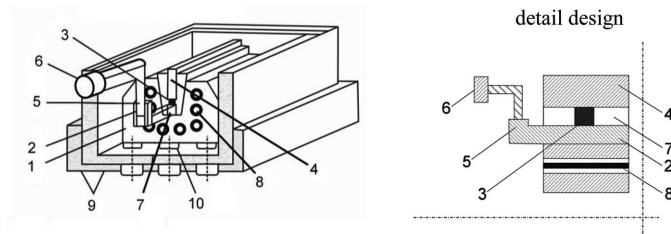


Fig. 2. Duct die block for the deformation of samples at elevated temperature by means of an AE probe: 1-body die block, 2-quake bar, 3-sample, 4-punch, 5-wave-guide, 6-AE probe, 7-duct, 8-heating elements, 9-foamed polystyrene, 10-tackbolt

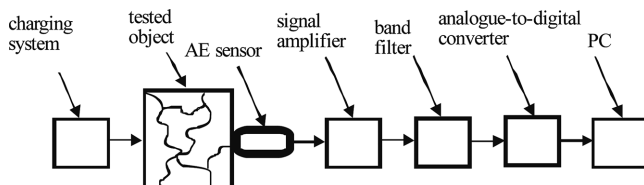


Fig. 3. Simplified block diagram measuring and recording system of the AE

3. Results of investigations and discussion

The diagram presented in Fig.4 illustrates the temperature influence of deformation on the mechanical characteristics in

compression tests of monocrystals alloy type CuZn30 with an initial crystallographic orientation [13 9].

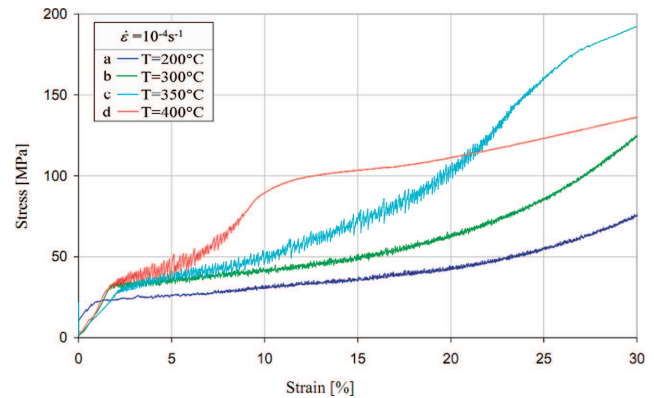


Fig. 4. The effect of the temperature of deformation on the shape of the σ - ϵ curves of CuZn30 monocrystals with an initial orientation [13 9], compressed in the range of temperature from 200°C to 400°C at a strain rate up to about 10^{-4}s^{-1}

Compression curves obtained in the range of temperature from 200°C to 400°C at a constant strain rate $\dot{\epsilon}$, of about 10^{-4}s^{-1} display a differentiated character of strain hardening in its respective stages. Although all of them display distinct oscillations of the force, which indicates the occurrence of the PLC phenomenon. The PLC effect starts in the analyzed curves in the range of critical deformation (ϵ_c) up to about $1.5\div 2\%$. The starting of oscillations on the compression curves coincides mostly with the range of the yield point of the material. It has been found that the quantity ϵ_c does not depend actually on temperature of deformation, whereas values of amplitude of stress oscillation $\Delta\sigma$ and the oscillation character depend on it. In the case of samples compressed at 200÷350°C oscillations of the stress on the curves are observed in all stages of strain-hardening of monocrystals. Temperature increase of the compression test up to 400°C resulted in an apparent shortening of deformation range in which oscillations of stresses are evident, characterizing PLC phenomena. During the initial stages of strain-hardening the mean values of the flow stress do not differ a lot. In the stage of linear strain-hardening, particularly in the range of parabolic hardening, increased maximum stresses are observed at higher temperatures of plastic deformation. This is due to a higher intensity of thermally activated events per unit of deformation. The higher mobility of dislocations shifts the state of equilibrium of generating the sources of dislocations towards a larger density of dislocation. Therefore, in the case of higher values of strain in the first and second stage of hardening, the intensity of plastic instability of the deformed monocrystal increases.

An analysis of the curves σ - ϵ leads to the assumption that in the investigated monocrystals the PLC effect results from the dynamic strain ageing (DSA), connected with the interaction of mobile dislocations and point defects or in compliance with the model of the dynamic-dislocational PLC effect - multiplication of dislocations during the activity of the Frank-Read source. The DSA effect is the result of presence in the material atoms of dissolved substance, as is the case at a temperature conditioning a sufficient mobility of atoms dissolved in a solid solution permitting the formation of atmospheres on the dislo-

cations. As the average rates of diffusion are smaller than the real rates of dislocation, the mutual effect of foreign atoms and dislocation is more perspicuous at medium temperatures and a slow strain rate [19,21,27]. In order to motivate the qualitative model of changing stresses in the strain-hardening diagram, we must assume that the separation of dislocation from the atom of the alloy reduces stresses which are indispensable for a further displacement of the dislocation by the quantity of the previously mentioned effect. The moment of the separation of the dislocation from the blocking atoms is marked by a violent drop of the stress exciting the deformation. According to the dynamic - dislocating PLC effect, each local decrease of the load recorded on the curves σ - ε is connected with the unlocking of the source of dislocation in some localized area of the sample, where simultaneously a high concentration of internal stresses has turned up. The transmission of these stresses unlocks successively the sources of dislocations. The unlocked sources of dislocations are, however, active, due to being overcharged, and this involves increased dynamics of the generated dislocations, resulting in the formation of slide stripes, propagating up to the moment when the time of incubation has reached again the value of ageing time [28].

Selected results of measurements concerning the energy of acoustic emissions in the compression test of investigated monocrystals have been gathered in the diagrams (Fig. 5 and 6) and in Table 2.

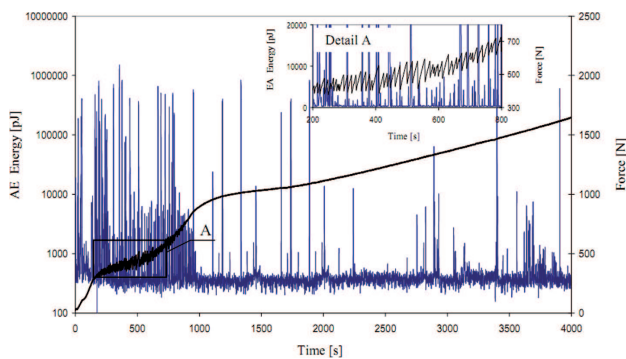


Fig. 5. The energy of acoustic emission and the external load during the compression test of the monocrystalline alloy CuZn30 at 400°C with $\dot{\varepsilon}$ to about 10^{-4}s^{-1}

In most cases of compression tests a distinct increase AE energy activity was recorded during the initial stage of compression and in the transition zone from the elastic to the plastic range (Fig. 5). The rising activity of the energy AE is in both cases characterized by a more or less wide maximum of the energy of the signal, after which the energy AE reaches its minimum. The observed increased AE energy activity in the initial stage of the curves σ - ε may, however, be caused, among others, by mechanical factors due to friction in the course of matching the sample to the pressure of the punch in the testing machine. In the range of the yield point, the increased AE energy activity is connected without any doubt with the dislocation processes. The level of the AE energy in this phase of strain-hardening has been found to be much higher than in the case of larger strains. Table 2 provides a comparison of the sum and average energy of occurrences depending on the temperature of deformation in the compression test. It has been found that the sum of events of AE in the

range of the yield point (R_e) grows together with the temperature, similarly as the average energy of a single occurrence. The highest sum of the number of events was attained in the case of deformed monocrystals with $\dot{\varepsilon}$ to about 10^{-4}s^{-1} at a temperature of 400°C and a mean value of energy (25 pJ) and the lowest in the case of samples compressed at 200°C, the mean value of the energy of its to about 12 pJ.

TABLE 2

Results of the analysis AE signal of monocrystals compressed at a strain rate of about 10^{-4}s^{-1} depending on the temperature of the test

No.	Temperature of deformation [°C]	Sum of occurrences of AE in the range of the yield point (R_e)	Average energy of a single occurrence AE [pJ]
1	200	9990	12
2	300	14539	20
3	350	16566	17
4	400	34253	25

In order to analyze in detail the behaviour of AE and to determine the correlation with the effect of the mechanical oscillation of stresses on the curves σ - ε in the course of compressing the investigated monocrystals at elevated temperature, the obtained signals were subjected to successive digital processing, applying software concerning acoustic analyses, in this particular case concerning sounds which are audible for the human ear. In order to facilitate an acoustic interpretation of these sounds, their amplitude was amplified by about 500% and in some cases slowed down tenfold. Representative characteristics of AE have been presented in the diagram and acoustogram (Fig. 6). The spectral characteristics of the recorded sonic signals were analyzed, as well as the time dependence in the range of 5% deformation of the sample after the appearance of the PLC effect on the curve. The intensity of the spectral density function is marked on the acoustogram by a blue code.

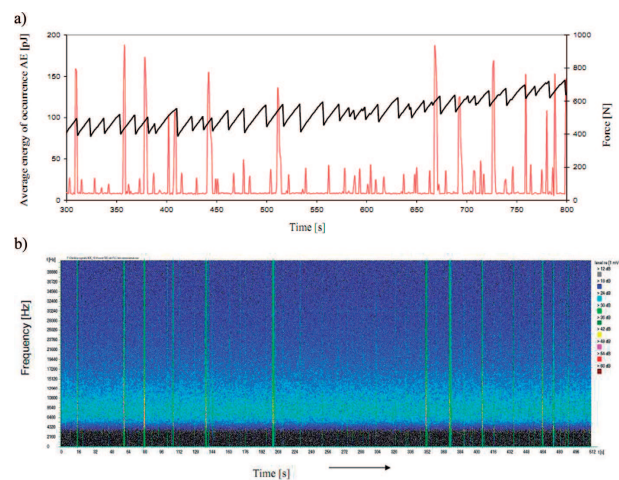


Fig. 6. Fragment of the diagram concerning the dependence of AE and the external force on the duration of the compression test (from 300 sec. to 800 sec.) of the monocrystal CuZn30 with the orientation $[1\bar{3}9]$ at the temperature of 400°C with $\dot{\varepsilon} = 10^{-4}\text{s}^{-1}$: a) average energy of occurrences AE; b) acoustogram of the set of occurrences AE presented in Fig. 6a

It has been found that in most cases of compression tests of monocrystals displaying the PLC effect, the local rapid drops of the load recorded on the force-time curve are distinctly correlated with the AE peaks. According to Cottrell's theory this may be due to avalanche separation of the dislocations from the atmospheres of foreign atoms which have been blocking them. The most evident correlation of these effects was recorded in the case of samples compressed at 400°C (Fig. 6). The latter one is a fragment of Fig. 5, in which the unit of time has been expanded. Practically, about 500 seconds of AE have been recorded during the compression test, with special regard to the correlation: load – AE. The compression test generates and propagates the highest sounds with an intensity of about 54 dB within the frequency range from 4320 Hz to 8640 Hz.

4. Conclusions

Basing on the investigations the following conclusions may be derived:

1. Monocrystals of the alloy CuZn30 subjected to special conditions of free compression tests at elevated temperatures display the phenomenon of unstable plastic deformation, the so-called PLC effect, with characteristic oscillations of stresses, in literature classified as type B.
2. The amplitude of oscillations stresses and acoustic emission energy in the investigated alloys increases with the temperature of compression.
3. The intensity of oscillations of the stresses and acoustic emission in the monocrystalline alloy CuZn30, appearing on the strain-hardening curves and recorded during the stage of easy glide, as well as in rather limited cases of parabolic strain-hardening, depends mainly on the temperature of compression.
4. The AE method applied in the process of plastic deformation of monocrystals of the alloy CuZn30 displays also a dependence of the activity of acoustic emissions on the stage of strain-hardening of the investigated alloy.
5. In the range of the occurrence of the PLC effect during the compression test of the investigated monocrystals the signal AE displays a cyclic character, distinctly correlated qualitatively with the oscillations of stresses on the curve σ - ϵ .

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REFERENCES

- [1] A. Portevin, F. Le Chatelier, Sur un phénomène observé lors de l'essai de traction d'alliages en cours de transformation, Comptes Rendus de l'Académie des Sciences Paris **176**, 507-510 (1923).
- [2] A.H. Cottrell, A note on the Portevin–Le Chatelier effect, Philos. Mag. **44**, 829-832 (1953).
- [3] M. Maziere, S. Forest, J. Besson, H. Wang, C. Berdin, Numerical simulation of the Portevin – Le Chatelier effect in various material and at different scales, Materials Science Forum **638-642**, 2670-2675 (2010).
- [4] V. Scott, F. Franklin, F. Mertens, M. Marder, Portevin Le Chatelier effect, Physical Review E **62**, 8195-8206 (2000).
- [5] W. Ozgovicz, B. Opeldus, The influence of the chemical composition and temperature of plastic deformation on the PLC effect in tin bronzes, Journal of Achievements in Materials and Manufacturing Engineering **17**, 129-132 (2006).
- [6] W. Ozgovicz, B. Grzegorzczak, E. Kalinowska-Ozgovicz, The structure and mechanical properties of the alloy CuZn30 after recrystallizing annealing, Journal of Achievements in Materials and Manufacturing Engineering **40**, 1, 15-24 (2010).
- [7] B.J. Brindley, P.J. Worthington, Serrated yielding in Aluminium-3% Magnesium, Acta Metallurgica **17**, 11, 1357-1361 (1969).
- [8] A. Wijler, J. Schade van Westrum, Strain rate experiments and the Portevin – Le Chatelier effect in Au (14 at% Cu), Scripta Metallurgica **5**, 531-536 (1971).
- [9] P. Rodriguez, Serrated plastic flow, Bull. Mater. Sci. **6**, 653-663 (1984).
- [10] A. Pawełek, J. Kusnierz, Z. Ranachowski, Z. Jasienski, Anisotropy of acoustic emission and-Portevin-Le Chatelier phenomena in polycrystalline aluminum alloys subjected to tensile tests, Archives of Acoustics **31**, 4, 102-122 (2006).
- [11] A. Pawełek, J. Kuśnierz, J. Bogucka, Z. Ranachowski, P. Ranachowski, F. Rejmund, T. Dębowski, Acoustic emission and the Portevin – Le Chatelier effect in tensile tested Al alloys processed by ARB technique, Archives of Acoustics **32**, 955-962 (2007).
- [12] R. Pascual, Acoustic emission and dislocation multiplication during serrated flow of an aluminum alloy, Scripta Metallurgica **8**, 1461-1466 (1974).
- [13] S. Mintzer, R. Pascual, R.M. Volpi, Acoustic emission and grain size in plastic deformation of metals, Scripta Metallurgica **12**, 6, 531-534 (1978).
- [14] F. Zeides, I. Roman, Study of serrated flow in two Al-Li-Cu-Mg base alloys with acoustic emission technique, Scripta Metallurgica et Materialia **24**, 10, 1919-1922 (1990).
- [15] Z. Jiang, Q. Zhang, H. Jiang, Z. Chen, X. Wu, Spatial characteristics of the Portevin-Le Chatelier deformation bands in Al-4at%Cu polycrystals, Materials Science and Engineering, 154-164 (2005).
- [16] M. Golec, Z. Golec, Application of the frequency analysis of acoustic emission in the study of the metal alloy solidification, Archives of Acoustics **25**, 1, , 43-50 (2000).
- [17] E. Le Clézio, T. Delaunay, M. Lam, G. Feuillard, Piezoelectric material characterization by acoustic methods, Archives of Acoustics **4**, 33, 603-608 (2008).
- [18] L. Majkut, Acoustical diagnostics of cracks in beam like structures, Archives of Acoustics **1**, 31, 17-28 (2006).
- [19] L.P. Kubin, Y. Estrin, Evolution of dislocation densities and the critical condition for the Portevin – Le Chatelier effect, Acta Metall. **38**, 697-708 (1990).
- [20] Y. Estrin, L.P. Kubin, Plastic instabilities: phenomenology and theory, Materials Science and Engineering A **137**, 125-134 (1991).
- [21] A. Korbel, The analyses of the non-uniform deformation in the substitutional solid solutions, Scientific Bulletins of the S. Staszic, University of Mining and Metallurgy, 474, Cracow, 1974.

- [22] I. Malecki, J. Ranachowski, Emisja Akustyczna Źródła, Metody, Zastosowania. PASCAL Publications – Komitet Badań Naukowych, Warszawa (1994).
- [23] Z. Ranachowski, Metody pomiaru i analiza sygnału emisji akustycznej, Prace IPPT PAN, 7/1997, Wyd. IPPT PAN, Warszawa (1997).
- [24] P. Ranachowski, F. Rejmund, Z. Ranachowski, A. Pawełek, A. Piątkowski, Mechanoacoustic and microscopic study of aluminous porcelain resistance to structural degradation, Archives of Metallurgy and Materials **56**, 4, 227-1233 (2011).
- [25] PN-EN 12163:2011 Miedź i stopy miedzi – Pręty ogólnego przeznaczenia (Copper and copper alloys — Rod for general purposes).
- [26] B. Grzegorzczak, The Portevin Le Chatelier effect in monocrystalline alloy Cu-Zn deformation at elevated temperatures, (in Polish) Ph.D. Thesis, Silesian University of Technology, Gliwice (2010).
- [27] A. Van den Beukel, Theory of the effect of dynamic strain ageing on mechanical properties. Phys. Stat. Sol. (a) **30**, 197-206 (1975).
- [28] A. Pawełek, Dyslokacyjne aspekty emisji akustycznej w procesach odkształcenia plastycznego metali, Polska Akademia Nauk, Instytut Metalurgii i Inżynierii Materiałowej w Krakowie, Wyd. OREKOP S.C., Kraków, (2006).

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