Relation between the plastic instability and fracture of tensile tested Cu-Sn alloys investigated with the application of acoustic emission technique

W. Ozgowicz, B.Grzegorczyk
Silesian University of Technology, Poland
wojciech.ozgowicz@polsl.pl

A. Pawełek, W. Wajda, W.Skuza. A.Piątkowski
Institute of Metallurgy and Materials Science of Polish Academy of Sciences, Poland
a.pawelek@imim.pl

Z. Ranachowski
Institute of Fundamental Technological Research of the Polish Academy of Sciences, Poland
zranach@ippt.pan.pl

ABSTRACT. The work concerns the application of the acoustic emission (AE) method in testing the mechanical properties of continuously cast industrial tin bronze CuSn6P, which reveals tendencies to instable plastic flow connected particularly with the Portevin-Le Chatelier (PLC) effect. The relations between the jerky flow connected with the PLC effect, AE intensity and the evolution of a fracture of the investigated alloy subjected to the tensile test at a strain rate ($\dot{\varepsilon}$) of about $1.2 \cdot 10^{-3} s^{-1}$ in the range of temperatures (20÷400°C) has been analyzed. It has been found that the highest intensity of the oscillation of stresses, corresponding to the instability of plastic deformation PLC occurred at 200°C, whereas the maximum of the AE activity is at about 200÷250°C. The brittle intergranular fracture starts in the range of equicohersive temperature (T_E) of about 200°C. Plastic deformation of the investigated alloy in the range of the temperature of minimum plasticity, amounting to about 400°C, results in intercrystalline fractures on the entire surface of the stretched samples.

KEYWORDS. Copper alloy; Portevin-Le Chatelier phenomenon; Tensile test; Acoustic emission; SEM; Intercrystalline fracture.

INTRODUCTION

Plastic deformation of copper alloys, particularly those with a small energy of the stacking fault (SFE), for instance tin bronzes and brasses at elevated temperature (about 0.4 to 0.6 T_t) and a strain rate ($\dot{\varepsilon}$) amounting to about $10^{-5}s^{-1}$ ÷ ok. $10^{-2}s^{-1}$ is a complex process, occurring most often in a heterogeneous way, due to the simultaneous effect of several mechanisms of deformation. The knowledge of these mechanisms and structural processes encountered in the given conditions of plastic deformation is indispensable for an optimal formation of the structure and properties of the investigated alloys and for programming the technology of the industrial plastic working of the products [1÷3].

Many alloys of iron and nonferrous metals indicate the phenomenon of heterogeneous deformation (Cu, Al, Ti, Ni) at elevated or high temperature in the course of tensile or compression tests in the form of irregularities on the work-hardening curve. This effect of plastic instability is often determined as "jerky flow" or "serration" and in literature it is called Portevin – Le Chatelier effect (PLC) [4, 5]. The characteristic oscillations of the stress on the work – hardening curve in the range of the plastic flow differ in their shape and size, depending mainly on the temperature and strain rate [6]. The PLC effect has been know for many years, although so far it has not been fully explained [7]. This effect is mainly investigated from the viewpoint of material factors, taking into account the microstructural conditions of the initiation of the localized plastic deformation resulting from the formation and propagation of the shearing bands and rheological factors, connected with the mechanics of plastic deformation in various thermodynamic and physico-chemical conditions.

Investigations concerning or the PLC effect base both on traditional methods and mechanical tests of uniaxial stretching or compression, and also on modern methods , as for instance the digital correlation of the image or acoustic emission AE [8,9]. The method AE belongs to the group of passive methods, because the apparatus of AE does not emit signals, nor does it affect the physical state of the tested objects. It depends on the detection of automatically occurring effects in the monitored object and analyzes this acoustic signal, resulting from the propagation of elastic waves generated in the mechanically loaded material due to the fast release of the energy accumulated in them. The release of elastic energy is connected with the formation of instantaneous local metastable states caused by various phenomena is a sub-microscoping scale, as for instance the diffusion of atoms in the crystallographic lattice, or in macroscopic scale – the formation of twins of deformation or nucleation of cracking. The shape of the signal AE is influenced by many factors, namely the chemical composition and microstructure of the investigated alloys, the size of the grains, heat treatment, the temperature of the process and the strain rate as well as the state of precipitations and the texture of the material. AE measurements are in comparison with other methods, characterized by a high sensitivity in recording the physical phenomena [10].

The aim of the present paper is to apply methods of acoustic emission in mechanical tests of uni-axial stretching of tin bronze of the type CuSn6P from industrial smelting, indicating distinctly a tendency to instability of plastic flow at elevated temperature of deformation due to the effect PLC. The integral purpose of these investigations is to determine the relation existing between the PLC effect and the AE generation and the phenomena of intercrystalline cracking of the investigated alloy at an elevated temperature of the tensile test.

EXPERIMENTAL PROCEDURE

The investigated material was standardized tin bronze CuSn6P provided from industrial smelting, in the form of a rod cast continuously (Wertli's process) with a diameter of 11.6 mm and the chemical composition presented in Tab. 1.

No.	Denomination of the alloy and kind of analysis	Chemical composition in % of mass								
		Sn	P	Bi	Pb	Sb	As	S	Fe	Cu
1.	CuSn6P ladle analysis	6.70	0.42	0.010	0.080	0.010	0.025	0.003	0.018	bal.
2.	CuSn6P PN-EN 1982:2008	5.5÷7	0.01÷0.4	-	0.02	-	-	-	0.1	bal

Table 1: Chemical composition of the bronze used (mass %).

Static tensile tests were carried out at an elevated temperature and a strain rate ($\dot{\varepsilon}$) amounting to about $1.2\cdot10^{-3}s^{-1}$, applying for this purpose the testing machine ZWICK Z 1200 in the range of loading up to 100 kN, making use of the digital recording of the tensile curves. The values of the force was recorded in the entire range of measurements with an accuracy of 0.5 %. The samples were preheated within the range of the temperature of stretching (20÷400°C) in a MAYTEC furnace, recording the temperature with an accuracy of ±4°C. The temperature chamber permits to run the tests at a temperature from 123 K to 873 K. For testing spherical samples were used 4 mm in diameter and 62 mm long, with threaded heads. The deformation was recorded on a length of measurements amounting to l_o = 27 mm. Measurements and AE recording were accomplished in the course of tensile testing, by means of a piezoelectric sensor, used for recording the acoustic impulses. The measurement system AE was connected with a devise for fastening the samples by mechanic clamps, from which the AE signal if emitted by a quartz waveguide and an AE sensor. For measuring the AE a WD sensor produced by the Physical Acoustic Corporation and an amplifier of the authors' own production were used. This system was provided with special filters permitting the elimination of frequencies corresponding to the range of free vibrations of the strength machine. The arrangement of the blocking system, as well as the recording of AE have been presented in Fig. 1.

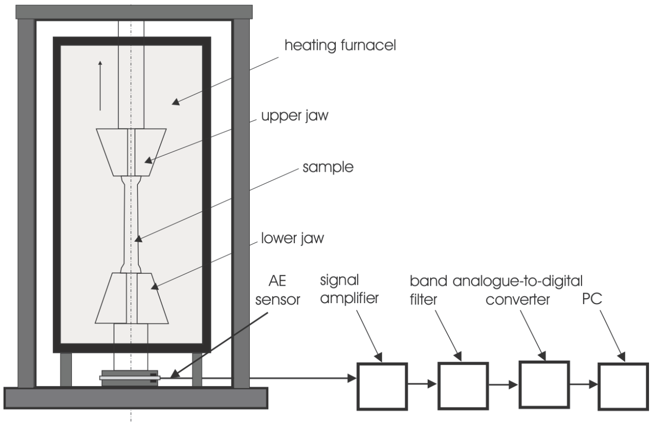

Figure 1: Simplified block diagram measuring and recording system of the AE

Metallographic tests were carried out on longitudinal microsections of statically stretched samples. The preparation of the microsections consisted in standard operations of immarging the samples in chemohardening resin, grinding and mechanical polishing on the machine Struers Labo Pol-21, and etching them in a 2% solution of hydrofluoric acid in water. The structure was observed on a scanning light microscope OLYMPUS GX71F with magnifications within the range from 200 to 1000 times.

For fractographic tests fractures of torn-off samples after their decohesion in tensile tests were applied, using for this purpose the scanning microscope (SEM) ZEISS SUPRA 25 with the electron part GEMINI of voltage 20 kV at magnitudes of 60 to 2000 times.

RESULTS

The results of mechanical tests of the PLC effect of the bronze CuSn6P in the tensile test at a temperature of 20÷400°C and a strain rate amounting to about $1.2 \cdot 10^{-3} sec^{-1}$ are to be seen on the curves σ-ε (Fig. 2 and 3) and have been gathered in Tab. 2. After continuous casting and homogenizing annealing of the investigated alloys their work- hardening curves display a qualitatively similar course of oscillation of stresses, mainly a mixed one (A+B+C), starting as soon as the stresses corresponding to the yield strength ($R_{p0.2}$) has been reached and disappear in the zone of unstable flow at the moment of the rupture of the sample. The temperature of the tensile test has been found to influence considerably the range of occurrence of the PLC effect and in a less distinct way the shape and intensity of the recorded oscillation of the stresses. The PLC effect does not appear in samples stretched at room temperature of up to about 150°C and above 300°C. In the investigated alloys the PLC effect is initiated similarly, in the case of values of critical deformations (ε_C) in the range from 0.5 to 1% of deformation, coinciding with yield point of the material. The highest intensity of the oscillation of stresses, and thus also of the unstability of plastic deformation PLC has been found in samples tested at a temperature of amounting to 250°C (Tab. 2), basing on the quantitative description of the PLC effect, applied in stereology, making use of the calculation software MACLAB. For the sake of a more complete description of serration recorded on the curves σ-ε, the coefficient of expansion of the line (R_L) was determined, as well as the average of the stress of oscillation in the given range of deformation (A) and the frequency of their occurrence (f) with this interval, applying the following two Eqs. (1) and (2):

$$R_L = \frac{L}{L'} \qquad (1)$$

where: L – the length of development of the line, L' - length of the projection of the line.

$$R_L = \frac{f}{A} \tag{2}$$

where:
A – mean value of oscillation in the given interval of deformation,
f – the frequency of the occurrence of oscillation.

The coefficients R_L, A and f were calculated by analyzing fragments of the curves σ-ε in the range of deformation (ε_x) of about 10% preceding the rupture of the sample. It has been found that the higher the values of the coefficient R_L and therefore also the amplitude (A) and frequency of serration (f), the higher is the instability of plastic deformation PLC. The application of this method for the purpose of assessing quantitatively the PLC effect in tensile tests at elevated temperature is, however, restricted due to the insufficient precision of measurements of the recorded quantities , mainly the values of the load and the deformation of the tested samples.

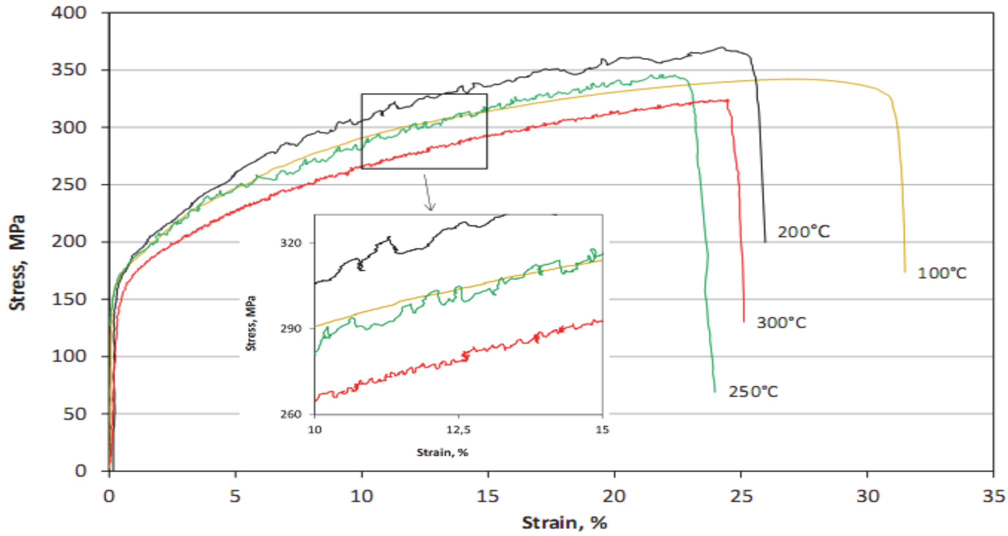

Figure 2: Curves of stretching of tin bronze CuSn6P after continuous casting.

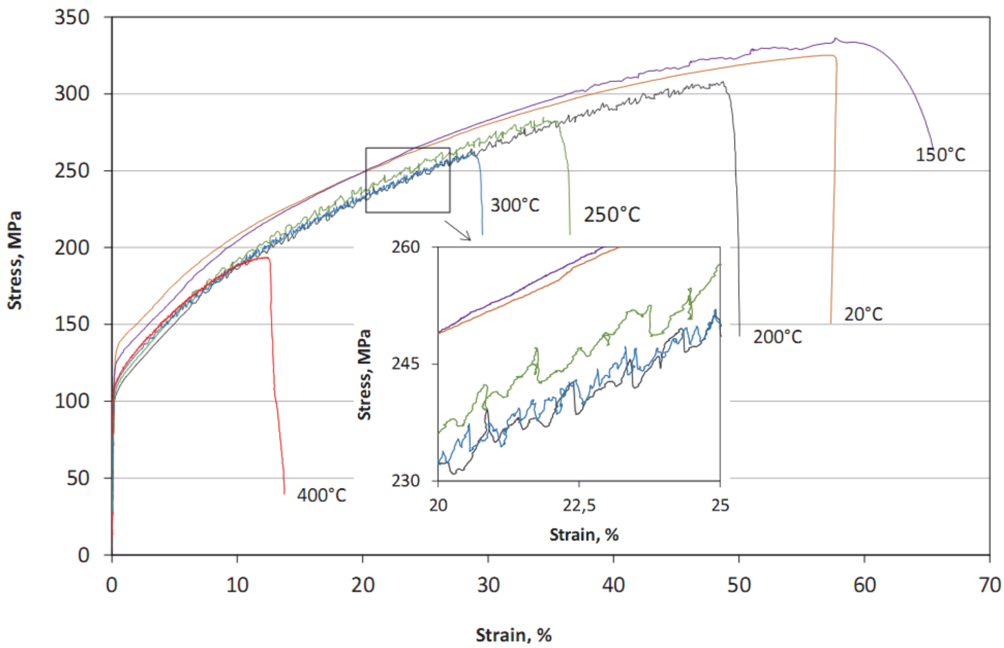

Figure 3: Curves of stretching of tin bronze CuSn6P in the annealed state.

No.	Temperature of deformation	ε_c [%]	Coefficients of the shape of curves σ-ε in the range (ε_{max} -10% < ε < ε $_{max}$)			
			R_L	A	f	Oscillation type
CuSn6P after continuous casting						
1	150	17	1.67	1.97	3.29	A+B
2	200	5	2.68	3.37	9.05	B+C
3	250	1	4.92	3.02	14.87	B
4	300	1.5	5.74	2.36	13.56	B+C
CuSn6P in the annealed state						
5	150	34	2.35	3.57	8.41	A+B,
6	200	1	5.93	3.12	18.52	A+B, B, C
7	250	1.5	6.00	3.87	23.23	A+B, B
8	300	2	5.70	1.73	9.88	B+C

Table 2: Contractual coefficients of the shape of the curves σ-ε with the effect PLC concerning the CuSn6P alloy.

The temperature of tensile tests determines also the fundamental characteristics of mechanical properties of the investigated alloy. With the rise of temperature the strength of the alloy distinctly decreases, slightly also a monotonous drop of the value of the yield point can be observed (Fig. 4). Tensile tests also proved that the plastic properties of the investigated bronze become distinctly worse (Fig.5) in the range of equicohesive temperature (T_E), which determines the transition of the alloy from the ductile state to brittleness , independent of the state in which the samples were provided for the purpose of being tested. The mean values of the reduction of area (Z) by about 70% in the range of the temperature of deformation (20÷400°C) decreases to about 10% at about 300°C. The shape of the function Z = f(T_{def}) indicates a distinct relation between thermally activated phenomena and the intensity of the instability of the PLC-type of plastic deformation, as well as the phenomena which control the brittleness of the alloy in the temperature range T_E.

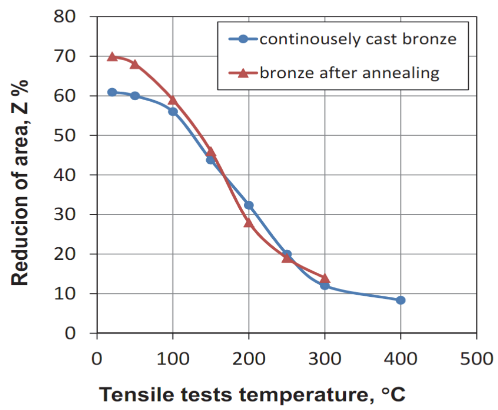

Figure 4: The dependence of the reduction in area on the temperature of stretching of the CuSn6P alloy in varying states of delivery.

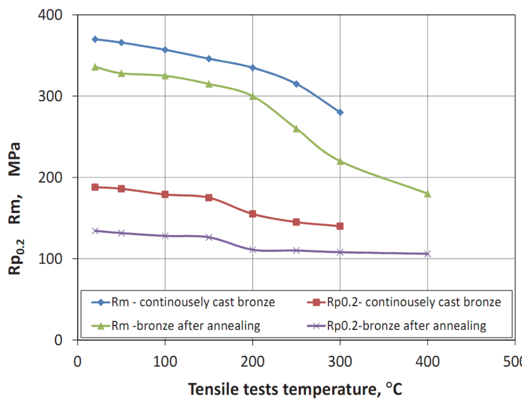

Figure 5: The influence of the temperature on the tensile properties of industrial bronze CuSn6P in various metallurgical states

The results of measurements of the acoustic emission (AE) in tensile tests of the bronze CuSn6P within the range of temperature from 20°C to 400°C with a strain rate of $1.2 \cdot 10^{-3} sec^{-1}$ have been presented in the diagrams (Fig.6 to 8) and Tab. 3. In most tensile tests the activity AE increased distinctly in the elastic range of curves σ-ε and also in the range of the transition from the elastic to the plastic state (Fig.6). The growth of the activity AE is in these cases characterized by a more or less wide maximum of changes of the energy of the signals, after which AE is at its minimum. This growth may be caused by such mechanical factors as friction and the matching of the sample in the clamps of the strength machine. In the range of the yield point, however, the observed growth of activity AE of the signal displays a physical character, connected with processes of the motion of dislocation. It has been found that the level of AE of the investigated bronze stretched at the temperature of 250°C is after continuous casting (Fig.6) much higher than after homogenizing annealing (Fig.7). The activity of the AE signal has also been observed to increase with elevated temperature of deformation (Fig.8). Nevertheless, the frequency of oscillation of the stresses recorded on the curves σ-ε and the frequency of events do not indicate any explicit correlation in the investigated experimental conditions. The highest number of events (or mean energy of the events of AE) was recorded in the case of the CuSn6P alloy cast continuously (or homogenizingly annealed) at the temperature of stretching, amounting in the range 250÷350°C with mean value of the energy of the events of about 66 nJ.

The results of metallographic observation of the investigated bronze being deformed at the temperature of 150÷250°C have been presented in the microphotographs (Fig.9). It has been found that up to the temperature T_E of about 250°C, bronze displays in the annealed state axially elongated grains of the solution-α with typical effect of cold work-hardening and heterogeneous plastic deformation (Fig. 9a). A distinct initiation of the process of cracking at the grain boundaries mostly in the point of contact of three grains was detected in sample stretched at a temperature of 250°C (Fig. 9b). An increase of the temperature of deformation to about 400°C, corresponding to the minimum temperature of plasticity (TMP), results in an increase of the number of intercrystalline cracks independent of the state of the investigated bronze.

No.	Temperature of deformation	Sum of AE events	Average energy of the AE events [nJ]
	CuSn6P after continuous casting		
1	50	547	76
2	100	322	56
3	150	441	55
4	200	216	24
5	250	743	66
6	300	130	120
	CuSn6P in the annealed state		
1	RT	971	125
2	100	692	111
3	150	1254	140
4	200	8261	57
5	250	306	105
6	300	63	30

Table 3: Descriptors of the analysis of the AE signals for the CuSn6P alloy.

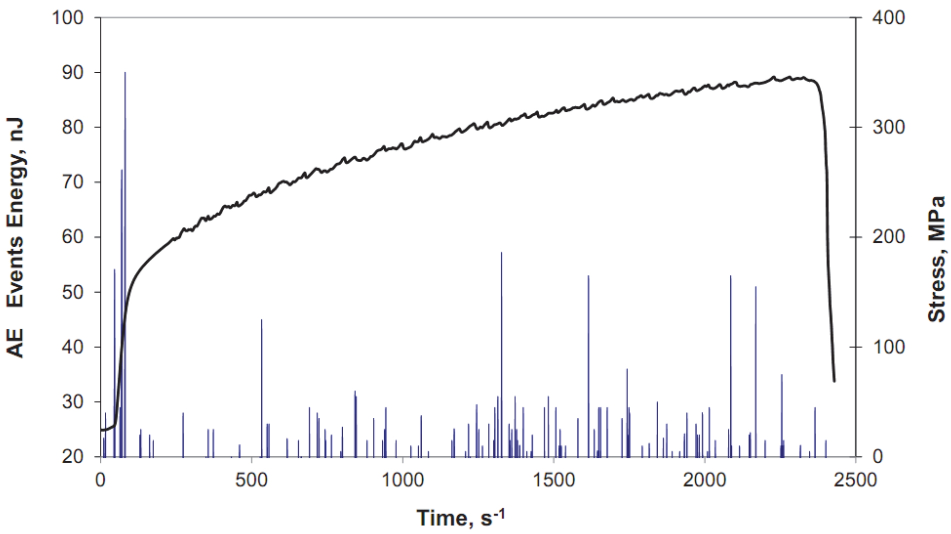

Figure 6: Acoustic emission and stress during the deformation of CuSn6P alloy at 250°C after continuous casting.

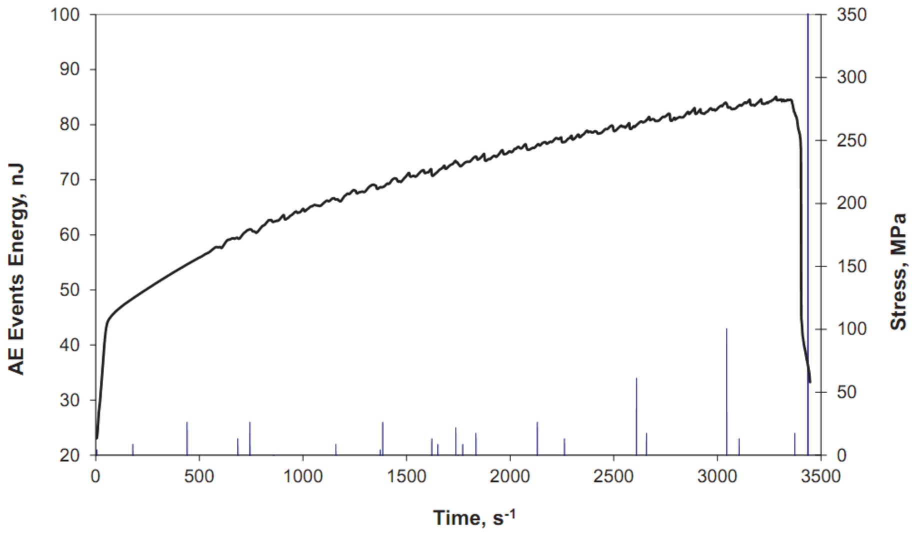

Figure 7: Acoustic emission and stress during the deformation of CuSn6P alloy at 250°C after annealing.

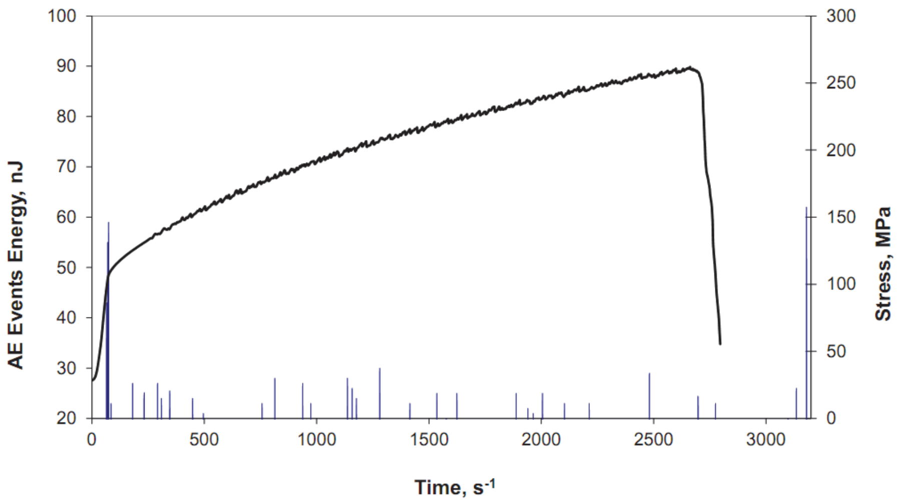

Figure 8: Acoustic emission and stress during the deformation of CuSn6P alloy at 300°C after annealing.

Fractures of the tested samples of tin bronzes after tensile tests in the temperature range (T_E÷TMP) have been presented in the microphotographs (Fig. 10 to 13). The temperature of deformation has been found to be the main factor determining the fractographs of the surfaces of the tested fractures of tin bronzes. Annealed and continuously cast bronze CuSn6P displays in the range of the temperature of transition from ductility to brittleness (T_E) a mixed fracture with a considerable share of smooth cleavage surfaces (Fig. 10), characteristic for brittle intercrystalline cracking. The share of ductile surfaces in the fracture (Fig.11) drops considerably with the rise of the temperature of deformation. In the range T_E on intercrystalline surfaces there occur distinct traces of plastic deformation and cavitation (Fig.10 and 11). It has been found that deformation in the range of TMP (about 400°C) leads to the formation of a fracture on the fully intercrystalline brittle surface (Fig.12, 13). Intercrystalline cracking encountered on boundary surfaces at the contacts of two or three grains (Fig. 13) usually runs across the pores of cavitations, which is typical for cast structures. Moreover, it has been found that intercrystalline fissures mostly nucleate in the vicinity of microvoids at the grain boundaries and on intersections twins or bands of deformations and the grain boundaries.

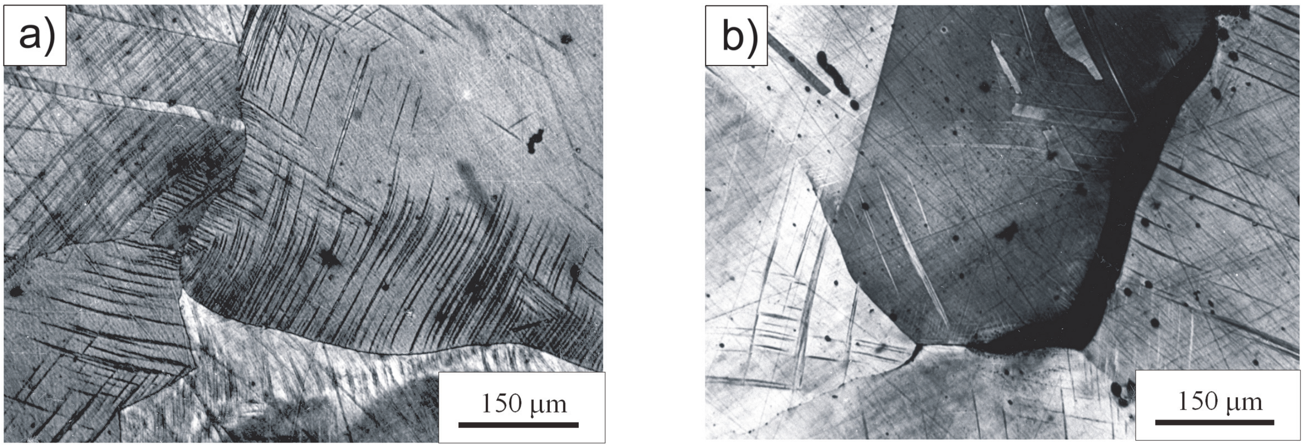

Figure 9: The structure of industrial bronze CuSn6P in the annealed state with a grain size about 500 μm after stretching ($\dot{\varepsilon}$ = 1,2·10^{-3} s^{-1}) at the temperature: a) 150°C; b) 250°C [3].

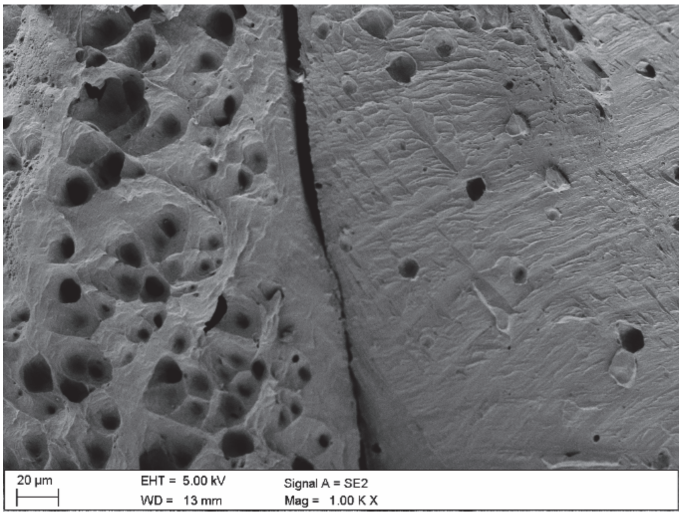

Figure 10: Transcrystalline mixed fracture of CuSn6P alloy after stretching at temperature of 250°C.

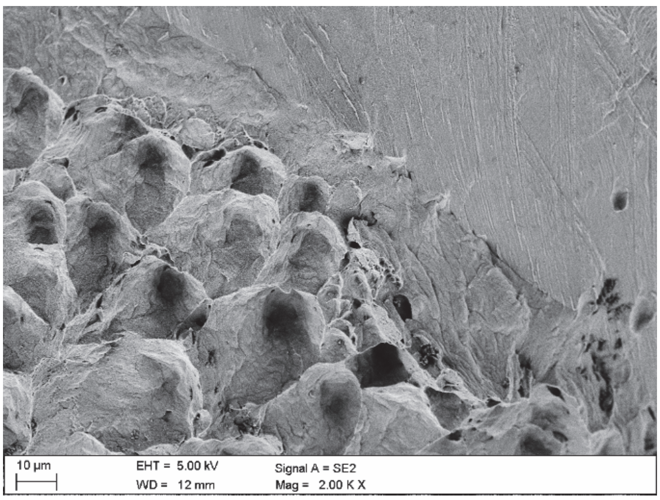

Figure 11: Transcrystalline mixed fracture of CuSn6P alloy after stretching at temperature of 300°C.

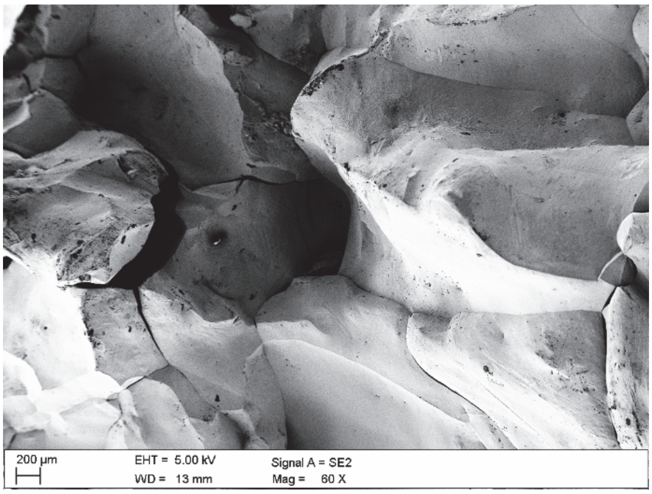

Figure 12: Intercrystalline brittle fractures of the CuSn6P alloy after stretching at the temperature of 400°C.

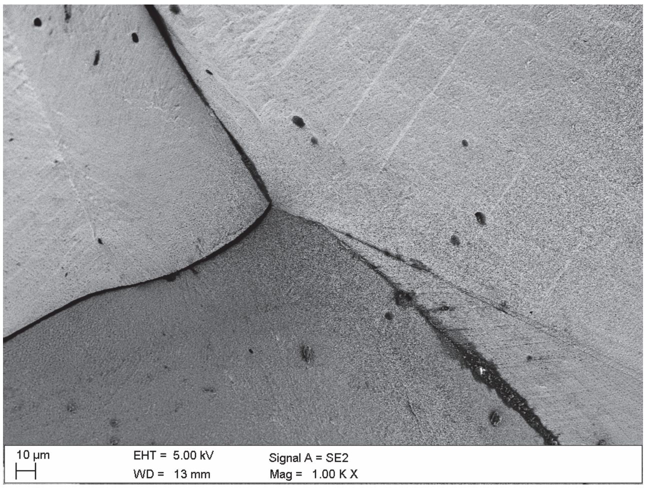

Figure 13: Intercrystalline brittle fissures at the contact point of three grains of the CuSn6P alloy after stretching at the temperature of 400°C.

CONCLUSIONS

The obtained results of investigations concerning the mechanical properties of industrial tin bronze CuSn6P at elevated temperature of deformation, applying the AE method and the performed structural and fractographic, analysis of the influence of temperature of deformation on the PLC effect and the intercrystalline cracking of the alloy permit to draw the following conclusions:

1. The PLC effect, which is characteristic for the phenomenon of heterogeneous plastic deformation of the investigated alloy turne up in the course of tensile tests at the equicohesive temperature (T_E) of about 250°C up to the minimum temperature of plasticity (TMP) amount to about 400°C.
2. The effect of the instability of plastic deformation PLC of the investigated alloy depends considerably on the temperature of deformation and its maximum is attained at the temperature of stretching amounting to about 250°C.
3. Within the range of temperature of the tensile test (20 to 400°C) the strength of the alloy (R_m) and the value of the reduction in area (Z) decrease much, (R_m) from about 350 MPa to about 180 MPa and (Z) from about 70% to about 10%, respectively, independently of the state in which the alloy has been delivered.

4. The activity AE of the recorded signals increases with the rise of the temperature of deformation, reaching its maximum at the temperature of stretching of about 250°C.
5. In the given conditions of plastic deformation no explicit correlation was found between the frequency of oscillations of stresses on the curves σ-ε indicating the PLC effect with the frequency of AE events.
6. The intercrystalline brittleness of the investigated tin bronze initiates in the temperature range (T_E), independently of the state of the alloy at its delivery, by the nucleation of microfissures, mainly of the type of wedges at the grain boundaries, privileged with respect to the direction of stretching stresses, mostly at the contact points of three grains, in which concentration of stresses attains it maximum.
7. The deformation of the investigated alloy in the temperature range of minimum plasticity (TMP), being 400°C, leads to the typical effect of intercrystalline brittle cracking of the entire surface of analyzed fractures of the stretched samples.

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