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# Analysis of the Portevin - Le Chatelier effect in tin bronzes at elevated temperatures

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# Materials

# <u>ABSTRACT</u>

**Purpose:** The aim of the present paper is the determination of the effect of the chemical composition and temperature of deformation of standardized tin bronzes and bronze modified with zirconium on the Portevin – Le Chatelier (PLC) phenomenon, mainly basing on the shape of stress-strain curves within the temperature range of 100-300°C and observations of their structure. The reasons of the occurrence of such en effect are so far no fully known and explained and the opinions concerning is physical basis vary.

**Design/methodology/approach:** Of essential design in this research is determination of the dependence of PLC effect on the chemical composition, temperature and strain rate and the preliminary heat treatment and grain size. The main method used in this investigation is tensile test at elevated temperature.

**Findings:** The main conclusions are following: the PLC effect on tin bronzes with a micro-addition of zirconium in an amount of 0.01-0.05% depends the temperature of deformation in the tensile test in the range of 100-300°C and the chemical composition of the alloys; the type of servation revealed on the  $\sigma$ - $\varepsilon$  curve depend mainly on the temperature of deformation and can be differ during the respective stages of the analyzed curves.

Practical implications: In this paper implications for practice are not taken into consideration.

**Originality/value:** In this paper an additive type of serration observed on the curves  $\sigma$ - $\epsilon$ , denoted by the symbol D, is new.

Keywords: Metallic alloys; Plastic instability; Portevin - Le Chatelier effect; Tensile test at elevated temperature

# 1. Introduction

2In solid solutions of many metallic alloys, both interstitial and substitutional ones, deformed at elevated temperature the phenomenon of heterogenic deformation occurs, generally called Portevin – Le Chatelier effect (PLC). The most characteristic feature of this phenomenon is the presence of irregularities on the tensile curves (the so-called teeth or steps - Fig. 1) which occur in a definite range of temperature and strain rate ( $\hat{\epsilon}$ ) depending of the kind of the investigated alloys. There are several publications [1-5, 10, 11, 13-15] concerning this phenomenon, but there is still no explicit explanation of the reasons of its occurrence in some definite conditions of plastic deformation. This phenomenon is still the topic of numerous investigations, verifying the actual theories of the phenomenon PLC, and concerning the aspect of modeling it and its computer simulation [6-9, 12].

Up-to-date theories concerning the PLC effect assume a prior the influence of alloy atoms with dislocation which hampers the dislocation movement, thus preventing the plastic deformation of alloys. In the case of a defined strain rate (É), therefore, such a stress is required which is higher than the resistance of the dislocation movement and the effect of the alloy atoms with dislocation. The break away of the dislocation from the alloy atom results in a drop of the stress indispensable for the further dislocation movement by the magnitude of this effect. The model suggested by Cottrell [1, 3-5, 10, 13, 15] motivates qualitatively the cyclic changes of stresses recorded during the tensile test. The moment of dislodging of the dislocation from the blocking atoms is displayed by a violent drop of the stresses determining the deformation. Experimentally confirmed oscillations of stresses (serrations of stresses) in the course of the heterogeneous deformation usually depend on the kind of the applied strength machine [9-11] in most cases, irregularities along the curve are to be observed when a "soft" machine is use, the construction of which does not permit to measure sudden changes of the load. The most reliable image of those changes of the load is warranted by "rigid" machines; deprived of the inertion of the system of measurements, displaying only minimum elastic deformation in the machine. It has been found that the magnitude and character, particularly the repeatability of the stress serrations on the  $\sigma$ - $\epsilon$ curve, depends also on the degree of dislodging of the dislocation from the presence of blocking atoms.



Fig. 1 Types of "teeth" characteristic for the heterogeneous deformation of the Portevin – Le Chatelier phenomenon [10]

An analysis on of the PLC effect requires, therefore, the consideration of not only the conditions of dislodging of

#### Table 1.

Chemical composition of the studied alloys

dislocations from the atmosphere of alloy atoms, but also of the renewed flow of atoms towards the dislocation, which is a diffusive process. The occurrence of the PLC effect is, in the case of the assumed model, conditioned by the equalization of the dislocation rate and the velocity of diffusion of the alloy atoms [2, 3, 10]. As the average rates of diffusion are small if compared with normal dislocation rates, the effect of interdependence of alloy atoms and dislocation becomes more evident when the strain rate is small [1, 15]. A detailed analysis of this problem requires, therefore, the determination of the dependence of the dislocation rate and the rate of the migration of atom alloys on the stress, temperature, strain rate and structure of the alloys. Studies of references [1, 2, 4, 5, 10-13, 15] concerning the PLC effect indicate that in many cases the fundamental model of this phenomenon is not satisfactory and does not explain the experimental results. There are also only few publications verifying the assumed model from the viewpoint of the structure.

The aim of the investigations was to analyze preliminarily the PLC effect, revealed in the course of the static stretching of tin bronzes containing about 6-7% Sn from industrial and laboratory smelting with a comparable concentration of alloy elements and modified with a micro-addition of zirconium in an amount of up to 0.1% in the temperature range from 100 to 300°C and a constant strain rate of about  $10^{-3}$ s<sup>-1</sup>.

### 2. Experimental procedure

Experiments were carried out on tin bronze samples, mainly of the type CuSn7 resulting from industrial and laboratory casting shaped as bars with a diameter of about 30 mm and plates about 20 mm thick, with a chemical composition as shown in Table 1.

In this investigations also model bronzes were used, modified with microddition of zirconium within the range of 0.01-0.1%. The tensile tests were carried out within the temperature range of  $100\div300^{\circ}$ C at strain rate of  $1.19\cdot10^{-3}$  s<sup>-1</sup> on a strength machine INSTRON (4405) with a loading range up to 100kN with computer control of the tensile test and numeral recording of the results. The dimensions of the stretched sample are to be seen in Fig. 2.

Kind of the alloy _		Chemical composition, wt %							
		Sn	Р	Zr	Bi	Pb	Si	Zn	Cu
1	CuSn7	6.07	0.0037	-	0.0003	0.0052	0.001	0.0130	res.
2	CuSn7Zr0.01	6.86	0.0006	0.020	0.00004	0.0049	0.002	0.0130	res.
3	CuSn7Zr0.05	6.8	-	0.001	-	-	-	-	res.
4	CuSn7Zr0.1	7.16	0.0001	0.0005	0.0008	0.004	0.012	0.0220	res.
5	CuZr0.01	0.0001	0.0008	0.015	0.0001	0.0004	-	0.0001	res.
6	CuZr0.05	0.0006	0.0001	0.0665	0.0001	0.0003	-	0.0013	res.

Tabl	le 2.

Parameters analyzed on tensile curve of the investigated alloys displaying the PLC effect

Allov	Deform.	Туре	of $\Delta F_+$	ΔF.	Δx	$\tau_{\Delta x}$	ΔL	$ au_{\Delta L}$
- J	[°C]	serration	[N]	[N]	[mm]	[s]	[mm]	[s]
	150	А	168	184	0.144	4.33	0.732	21.97
CuSn7	250	В	298	255	0.033	1.01	-	-
(1)	300	С	163	243	0.040	1.27	0.084	1.50
		D	131	016	0.055	1.65	-	-
	150	Α	448	216	0.160	4.87	0.770	23.11
		В	200	199	0.084	2.54	-	-
CuSn7Zr0.01	200	В	230	205	0.039	1.18	-	-
(2)	200	D	326	189	0.080	2.39	-	-
	280	В	283	568	0.033	0.99	-	-
	200	С	384	384	0.077	2.04	0.612	18.36
	) 200	A+B	506	383	0.126	3.79	0.424	12.71
CuSn7Zr0.05 (3)		В	288	256	0.044	1.32	-	-
	300	С	171	177	0.045	1.36	0.122	3.67
	150	Α	410	502	0.105	3.17	0.692	20.78
CuSn7Zr0.1		A+B	325	351	0.074	2.23	0.692	20.78
(4)		В	241	201	0.044	1.30	-	-
	250	В	371	330	0.051	1.52	-	-
CuZr0.01	200	Α	420	29	0.091	2.75	0.133	3.99
(5)	200	В	299	275	0.041	1.23	-	-
CuZr0.05 (6)	250	В	355	335	0.051	1.54	-	-

 $\Delta x$  – quantity of deformation during a single cycle;

 $\Delta L$  - quantity of deformation in repeated cycles;

 $\tau_{\Delta L}-$  duration of single cycle;

 $\tau_{\Delta L}$  – duration of repeated cycles;



Fig. 2. Sample of tensile test at elevated temperatures

Metallographic investigations on a light microscope were carried out longitudinal polished sections of samples stretched at elevated temperature, prepared in the conventional way and etched in an agent based on  $K_2Cr_2O_7$  diluted with water. The phenomenon of heterogeneous plastic deformation was analyzed basing on  $\sigma$ - $\epsilon$  curves, classifying the kind of serration, the amplitude of the stress ( $\Delta$ F±), the range of homogeneous strain

 $(\varepsilon_s)$  and the characteristic periods of oscillation of stress  $(\Delta_x, \Delta_L)$  during the unstable plastic flow of material in the range of stable deformations  $(\Delta \varepsilon)$ . Stress were measured with on accuracy of 0.001kN, the deformation with an accuracy of 1µm.

### **3. Experimental results**

The results of the analysis of tensile curves of the investigated model copper alloys within the temperature range of the PLC effect have been gathered in Table 2 and in the diagrams presented in Figs. 3, 5, 6 and 8-14. Mainly two-compositional tin bronzes (Cu-Sn) were analyzed with a concentration of Sn about 7% (alloy 1) and three-elemental alloys (Cu-Sn-Zr) modified by a microaddition of zirconium with a concentration of about 0.01-0.1% (alloy 2-4), as well as for the sake of comparison alloys of pure copper (0FHC) with a microaddition of Zr with a concentration of 0.01% (alloy 5) and 0.05% (alloy 6). The characteristics of typical parameter of tensile curves displaying



Fig. 3. Tensile stress-strain curve for CuSn7 alloy at 150°C

the PLC effect comprised the increase of forces  $\Delta F_+$  and their violent drop  $\Delta F_-$  concerning the subsequent oscillations  $(\Delta x, \tau_{\Delta x})$  or periods of repeated oscillations  $(\Delta L, \tau_{\Delta L})$ . It has been found that the shape of the curves  $\sigma{-}\varepsilon$  in the tested range of temperatures (100-300°C) at a constant strain rate  $\acute{\varepsilon}{=}1.19{\cdot}10^{-3}~s^{-1}$  depends essentially on the temperature of deformation and chemical composition of the tested alloys. The heterogeneous stage of deformation PLC, occurring in the form of serration on the tensile curves in usually preceded by an preliminary range of homogeneous plastic deformation  $\epsilon_s$ .

The model alloy CuSn7 (alloy 1) displays the first signs of heterogeneous deformation in steady-state of he curve  $\delta$ - $\epsilon$  at a temperature of 100°C. The PLC effect is however insignificant and the periodical character of the teeth approximates the shape of the "teeth" type A suggested in the literature [10]. A distinct PLC effect has been recorded only at a temperature of 150°C (Fig. 3), with a characteristic arrangement of the teeth corresponding exactly to the standard A (Fig. 1). It has been found that in the case of this type serration the repeatability of the periods of the cycle of stress changes ( $\Delta$ L,  $\tau_{\Delta L} \approx 22s$ ) is considerable and the increase of the force and violent drops are comparable (Table 2).

The observed shape of the analyzed curve  $\sigma$ - $\varepsilon$  at a temperature of 150°C confirms, among others, the theorem of A. Korbel [10] saying that at lower temperatures of deformation after the homogeneous deformation ( $\varepsilon_s$ ) teeth of type A are to be observed. After deformation at such a temperature the structure displays a large number of slip lines and deformation bands (Fig. 4) confirming the hypothesis that teeth of type A are mainly connected with the nucleation of deformation bands, which analogically to Lűder's bands in the case of the yield points of soft carbon steel are shifted across the entire length of the sample. It has been found that every periodical drop of forces along the curve  $\sigma$ - $\varepsilon$  corresponds to the activation of any single deformation band. An increase of the temperature of deformation to about 250°C involves a modification of the shape of the curve  $\sigma$ - $\varepsilon$ 

revealing regular teeth of type B (Fig. 5) or at a temperature of  $350^{\circ}$ C a complex character of serration of type C and D (Fig. 6).



Fig. 4. Single – phase structure of bronze CuSn7 with sliding lines and deformation bands; tensile temp.150°C, deformation zone of the sample; 500x

Serration type B and C is characterized by the fact that the rising part of the curve of strengthening displays a characteristic slope due to the elastic behaviour of the system. It is to be supposed that in the case of maximum stresses glide bands or shear bands are nucleated (Fig. 7) accompanied by a violent drop of forces. Plastic deformation changes, therefore , from the elastic to the plastic one. This is connected with a large number of local deformation band which do not necessarily are dislocated across the whole sample. On the  $\sigma$ - $\epsilon$  there are also local oscillations of the forces, frequently not homogeneous in their character (Fig. 5), sometimes divided by ranges of homogeneous deformation (Fig. 6). The PLC effect in this alloy disappears above the temperature of deformation 310°C.

Materials



Fig. 5. Tensile stress-strain curve for CuSn7 alloy at 250°C



Fig. 6. Tensile stress-strain curve for CuSn7 alloy at 300°C

The modification of the basic bronze with concentration 0.01% Zr (alloy 2) does not effect any essential changes in the occurrence of the PLC effect, if compared with alloy 1. It has been found, however, that at the tested temperatures a complex character of serration is to be observed, viz. at 150°C the teeth type A and B (Fig. 8) and at 200°C teeth of type A, B, D and at 280°C teeth of type B and C (Fig. 9). Moreover, it has been found that at lower temperatures of deformation and at lower values of  $\varepsilon$  usually teeth of type A do occur, whereas at higher temperatures and higher value of  $\varepsilon$  teeth of type B and C.

An increased concentration of the addition of Zr in this basic bronze (up to 0.05% - alloy 3) and to about 0.1% (alloy 4) does not result is essential changes in the temperature range of the PLC effect, and the type of characteristic serration along the curve  $\sigma$ - $\epsilon$  (Figs. 10-12). It has been found, however, that in alloy 3 no serration occurred which would correspond the standard A (Figs. 10, 11), and in alloy 4 no serration was detected classified as type C (Fig. 12).

The PLC effect of the investigated pure copper modified by Zr with a concentration of 0.01% (alloy 5) was detected within a narrow tensile temperature range of about 200°C. It has been found that the curve  $\sigma$ - $\varepsilon$  displays periodically ( $\Delta$ L) teeth of the general type A locally modulated by B teeth, occurring cyclically ( $\Delta x$ ) in the periods  $\tau_{AI}$  (Fig. 13). Moreover, the cyclically occurring increase of the force exceeding the level of the approximated tensile curve, followed by violent drop below this level. The PLC effect of pure copper containing 0.05% Zr (alloy 6) could be observed at higher tensile temperature of about 250-300°C. This denotes a higher energy of activation for the diffusion of Zr atoms in  $\alpha$  -solution. The stress-strain curves recorded at 300°C display characteristic teeth of type B, localized merely in the initial stage of the strengthening curve (Fig. 14). The revealed complex character of serration type A+B in pure copper modified with Zr is similar to the serration of tensile curves of the alloy 3 and 4 within the temperature range of 150-200 °C (Figs. 10, 12).



Fig. 7. Single - phase structure of bronze CuSn7 with the shear bands; tensile temp. 250°C, break-off zone of the sample; 500x



Fig. 8. Tensile stress-strain curve for CuSn7Zr0.01% alloy at 150°C



Fig. 9. Tensile stress-stain curve for CuSn7Zr0.01% alloy at 280°C

Materials









Fig. 11. Tensile stress-strain for CuSn7Zr0.05% alloy at 300  $^{\circ}\mathrm{C}$ 



Fig. 12. Tensile stress-strain curve forCuSn7Zr0.1% alloy at 150°C



Fig. 13. Tensile stress-strain curve for CuZr0.01% alloy at 200°C



Fig. 14. Tensile stress-strain curve for CuZr0.05% alloy at 250°C

An analysis of the parameters of the curves  $\sigma - \varepsilon$  of the investigated alloys with a PLC effect has shown that in the case of teeth type A the repeatability of the cycle a heterogeneous deformation ( $\Delta L$ ,  $\tau_{\Delta L}$ ) at a constant strain rate and test temperature of 150°C is considerable, independent of the chemical composition of the alloy (Table 2). The amplitude of the drop of forces ( $\Delta F_{-}$ ) increases, however, with the growing content of zirconium in the alloy Nos.1, 2 and 4. In the case of teeth type B it has been noticed that the magnitude of the respective cycles ( $\Delta x$ ) decreases with the temperature of deformation and does not depend on the chemical composition of the investigated alloys. The repeatability of the cycle  $\Delta x$  is also considerable and indicates a dependance on the temperature of the PLC effect. Teeth of type C at a comparible tensile temperature (about 300°C) are characterized by a shorter cycle of heterogeneous deformation  $(\tau_{\Delta L})$  than teeth of type A and a symmetrical distribution of the quantities  $\Delta F_{+}$  and  $\Delta F_{-}$ . The influence of the content of zirconium on the shape of the strengthening curve within the range of elastic strain in the respective cycles of the teeth type C is similar to

those of type B. The analysis of the parameters of the curves  $\sigma$ - $\epsilon$  displayed also the occurrence of an additive type of serration A+B (Figs. 12, 13) and the presence of teeth described as type D (Fig. 6). The quantities of the individual cycles  $\Delta x$  in teeth type D approach the value determined for teeth type B. The shape of the strengthening curve is also similar within the range of elastic strain in a single cycle of stress changes. Different, however, is the shape of the curve of decreasing forces, suggesting changes of the kinetics of the progress of deformation after the break–away of dislocation from the zirconium atoms blocking, the dislocation in the  $\alpha$  – solution.

#### 4.Conclusions

The investigations permit to draw the following conclusions: Polycrystalline tin bronzes type CuSn7 display a heterogeneous plastic deformation, the so-called PLC effect, in

# Materials

the temperature range of stretching amounting to about 100-300°C and strain rate  $1.2 \cdot 10^{-3} s^{-1}$ .

The heterogeneity appearing on the curves  $\sigma$ - $\varepsilon$  depend mainly on the temperature of plastic deformation and chemical composition of the tested alloys.

The type and parameters of the "teeth" on the curves  $\sigma$ - $\epsilon$  may differ in the respective stages of the analyzed diagrams.

The investigated alloys display on the curves  $\sigma$ - $\epsilon$  a distinct

range of homogeneous deformation ( $\varepsilon_s$ ) preceding the characteristic PLC effect.

The degree of deformation  $\mathcal{E}_s$  decreases distinctly with the rising temperature of plastic deformation.

The amplitude of cyclic load changes ( $\Delta F_{+.}$ ) on the curves  $\sigma$ - $\epsilon$  of the investigated alloys grows distinctly with the increasing temperature of plastic deformation.

The rising concentration of the alloy elements (Sn, Zr) in the investigated bronzes leads to an intensive PLC effect within a wider range of the temperature of plastic deformation.

Model bronzes with a microaddition of zirconium is characterized by an additive type of "teeth" on the curves  $\sigma$ - $\epsilon$ , type A+B, as well as of the "teeth" denoted by the symbol D.

The investigated bronzes deformed at elevated temperature display in  $\alpha$ -solution structure a large number of deformation and shearing bands.

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