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The relationship between hot ductility and intergranular fracture in an CuSn6P alloy at elevated temperatures

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Abstract: The hot ductility of tin α-bronzes has been studied by hot tensile and hot torsion tests, microscopic observations of grain boundary sliding, fracture surface observations and Auger's spectrometric technique. It has been found that the minimum plasticity of the tested alloy observed as a function of deformation temperature is directly connected with grain boundary sliding and the segregation of tin atoms and impurities, mainly sulphur, at the grain boundary. The complex effect of these mechanisms determines the intergranular fracture of CuSn6P grade bronze within the temperature range of minimum plasticity.

Keywords: Ductility minimum temperature, Intergranular fracture, Tin α-bronzes, Grain boundary sliding, Auger's spectrometry

1. INTRODUCTION

The macro-measured minimum plasticity of many metals and alloys as a function of the temperature of deformation, generally known as the effect of minimum plasticity (DMT), occurs both in copper and its alloys [1÷4], in steel [5] and in other metals and polycrystalline alloys [6]. This effect has been known for a long time and explained in literature, but up to now no equivocal interpretation has been given concerning the reason of the behaviour of such materials, particularly in the case of the variated composition of the alloy and grain size and the wide range of the strain rate. Both the variety of tested materials and the frequent lack of an equivocal determination of the preliminary structures in the tests, as well as the applied conditions of deformation and various methods of investigations seem to be the main reason hampering a generalization and explanation of the DMT effect.

An analysis of literature concerning this effect (also called intermediate temperature embrittlement - ITE) shows that it depends on the effect of many physico-chemical, structural and mechanical factors connected implicite both with the chemical composition and the structure of the alloys and the conditions of hot deformation. It has been found that the observed embrittlement of copper and its alloys is the result of the effect of intergranular cavitation, induced by grain-boundary sliding (GBS) [7, 8]. The loss of plasticity in the DMT range may also be conditioned by the intergranular segregation of alloying components or impurities [9]. The comparatively least investigated mechanisms of the intergranular fracture of copper alloys within the DMT range are GBS and the segregation of alloying component atoms on the grain boundary.

The aim of the investigations is to find out the relation between the level of hot plasticity of the tested bronze and the final effects of micromechanisms, such as: grain boundary sliding and intergranular segregation in the course of plastic deformation at elevated temperature within the DMT range.

2. MATERIALS AND EXPERIMENTAL PROCEDURES

In the investigations standard bronzes were used of the type CuSn6P and CuSn6 with a chemical composition complying with NF and PN from industrial casts obtained in the French firm Trefimetaux, and from Huta Warszawa, Poland (Table 1).

Table 1.
Chemical composition of the investigated tin bronzes-α

No	Material		Chemical composition, in mass %											
	Designation	Casts	Sn	P	Cu	Impurities								
						Bi	Pb	Sb	As	S	Fe	Zn	Ni	O_2
1	CuSn6P	Trefimetaux	6.70	0.42	92.1	0.0100	0.0800	0.0100	0.0250	0.0026	0.0180	0.046	0.030	-
2	CuSn6	H. Wa-wa	5.25	0.17	rest	0.0018	0.0120	0.0150	0.0200	0.0020	0.0080	0.010	0.200	-

Experiments of static tension at elevated temperature were carried out at a strain rate of $1.2 \cdot 10^{-5} s^{-1}$ to $1.2 \cdot 10^{-1} s^{-1}$ on the an INSTRON-machine in a protective atmosphere, and experiments of hot-torsion tests on a torsional plastometer at a strain rate of $1.1 \cdot 10^{-1} s^{-1}$ to $10 s^{-1}$.

Grain boundary sliding was investigated basing on the analysis of mechanical and electronic surface gauges on the surface of samples stretched at elevated temperature. Spectrometric investigations were carried out applying a RIBER spectrometer. Selected samples of bronze were also subjected to ion bombardement by Ar^+ in order to determine the gradient of the chemical composition as a function of the depth of ion bombardement from the grain boundary.

3. RESULTS

3.1. Hot tensile and torsion tests

The results of investigations on the plasticity of industrial bronze type CuSn6 in the temperature range of 20÷800°C have been gathered for tensile tests in Fig. 1 and for torsional tests in Fig. 2.

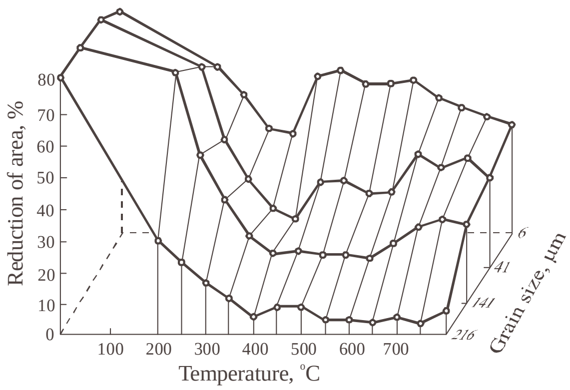

Figure 1. Reduction of area versus the temperature of deformation and grain size of industrial bronze CuSn6 stretched with a strain rate of $1{,}19 \cdot 10^{-3} s^{-1}$

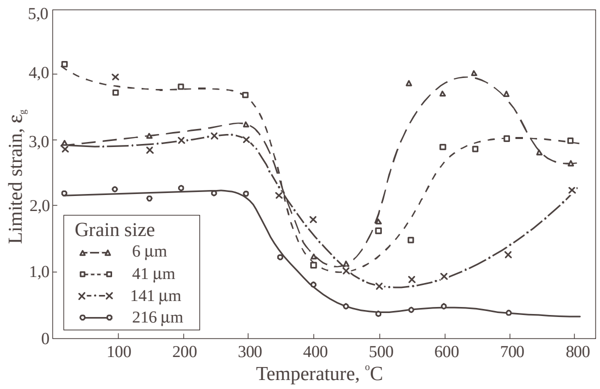

Figure 2. Dependence of the critical strain on the torsion temperature and grain size of industrial bronze CuSn6; strain rate: $1{,}1 \cdot 10^{-1} s^{-1}$

3.2. Fracture surface observations

Fractures of the investigated bronze samples after hot tensile and torsion tests have been presented by microphotos (Fig. 3). It has been found that the essential factors affecting the fractography of the analysed fractures comprise deformation temperature and grain size of the tested alloys.

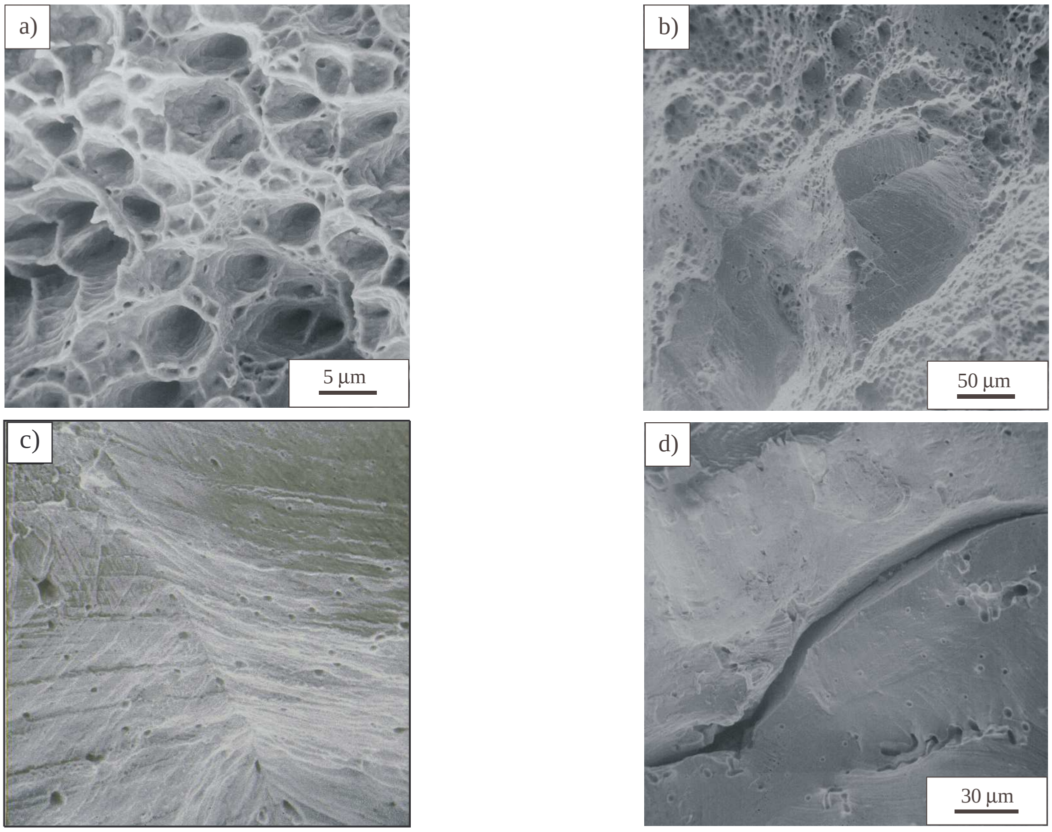

Figure 3. Fractography of industrial bronze CuSn6P with a grain size of about 500μm after tensile tests in the temperature range (20 ÷ 200)°C: (a) ductile fracture (20°C), (b) mixed fracture (150°C), (c) higher magnification of the intergranular surface shown in (b), (d) intergranular fracture

3.3. Metallographic observations

Bronze CuSn6P with an initial grain size of 500μm, stretched at elevated temperatures, displays a distinct initiation of the process of fracture at the grain boundary, most often at the contact of three grains at $\dot{\varepsilon}=1.2\cdot10^{-3}s^{-1}$ and 250°C (Fig. 4) and at about 500°C at $\dot{\varepsilon}=1.2\cdot10^{-1}s^{-1}$. The structures of industrial bronze after high-temperature torsion are generally similar to structures obtained after hot-tensile tests, at a comparable strain rate.

Intercrystalline slots and displacements of the mechanical and electronic gauges, due to the slide at the grain boundary, have been analyzed in detail in the course of metallographic observations, applying SEM. After streching the samples with $\dot{\varepsilon}=3.3\cdot10^{-5}s^{-1}$ at 400°C mainly wedge-type slots and cavitation-type were observed, as well as displacements of continuities of mechanical and electronic gauges at a distance of several μm. It has been found that intercrystalline fracture takes place together with GBS and an increase of the pores on the grain boundary.

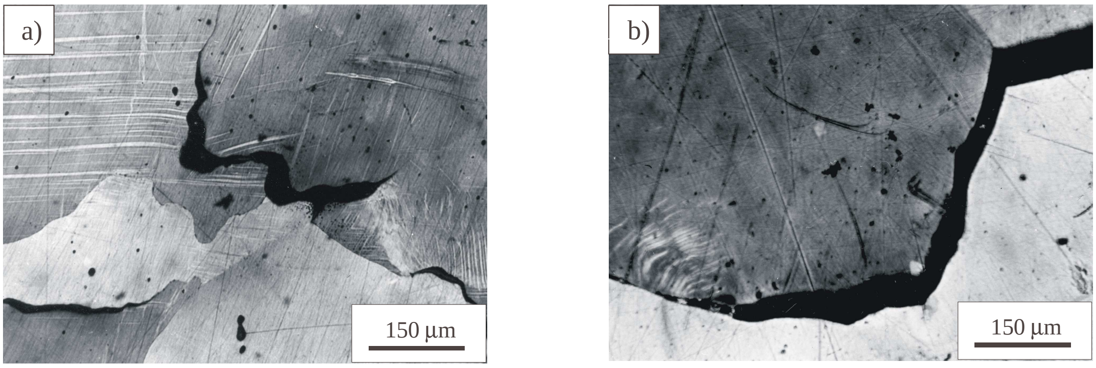

Figure 4. Structure of industrial bronze CuSn6P with a grain size of about 500 μm stretched at a strain rate of $1,2\cdot10^{-3}$ s^{-1} at a temperature of 300°C (a) and $1,2\cdot10^{-1}$ s^{-1} at a temperature of 500°C (b)

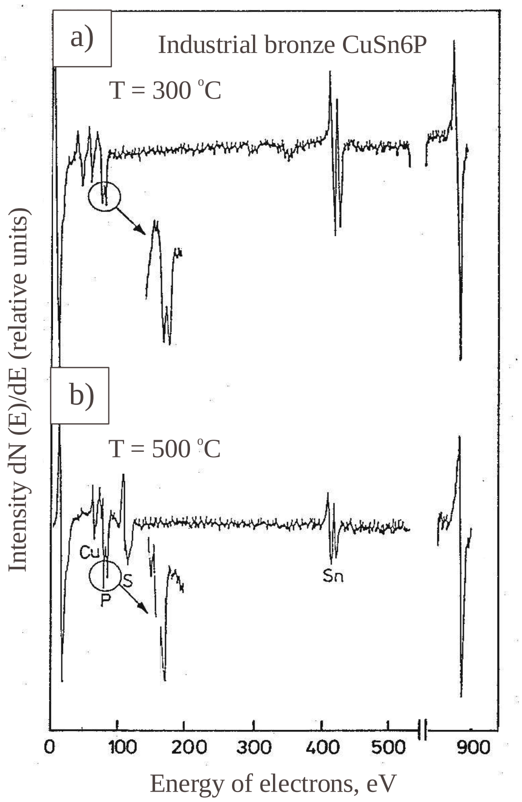

Figure 5. Auger's spectrum of the intercrystalline fracture surface of industrial bronze CuSn6P (a, b) after tensile tests at (a) 300°C and (b) 500°C

Auger's emissive spectra recorded on the crystalline surface of CuSn6P bronze in the DMT range display besides the reference lines (Cu) distinct lines from Sn and S and a double line of P (Fig. 5, 6). The untypical shape of the phosphorus spectrum results probably from a special atomic bond Cu-P. A doublet of this kind has been found experimentally also in the spectrum of the Cu_3P phase [10, 11]. It has also been found that the number of both the spectral lines and their intensity depends essentially on the deformation temperature.

Ion bombardement (Ar^+) of the intercrystalline faces of bronze CuSn6P after stretching in the DMT range (~500°C) displayed that the concentrations of P and S drops distinctly during the bombardement to about half an hour. The observed changes of the concentration of alloying components (Sn and P) and sulphur as a function of the time of bombardement indicate distinctly the occurrence of segregation of the atoms of these elements, probably grouped in monolayers or in the nanometrical external layer on the surface of the grain boundaries.

4. DISCUSSION

An analysis of the result of tensile and torsion tests indicates that DMT occurs in some range of temperature, in which bronze type CuSn6P or CuSn6 is characterized by a lower plasticity. The range of temperatures DMT and its localization and the average level of plasticity depend essentially on the strain rate, the grain-size and the chemical composition, particularly at the grain boundaries. The occurrence of the DMT effect in a large range of strain rates indicates that it occurs in regions of various effects and conditioned micromechanisms of fracturing. The measuring analysis of sliding at the grain boundaries as well as the results of fractographic analysis prove precisely the occurrence of an essential interdependence of brittleness of the investigated bronzes in the DMT range and the mechanism GBS and the phenomenon of the segregation of alloying component and impurities at the grain boundaries. It has been found that the share of GBS in the total deformation of the samples to rupture depends on temperature and the strain rate. Thus it has been proved that the maximum share of GBS occurs in the range DMT of the tested alloys. Metallographic observations have shown that the grain size is the principal structural factor conditioning the average level of the diminished ductility in the DMT range. The temperature range of intercrystalline fracture in CuSn6P and CuSn6 bronzes corresponds exactly to the range of equicohesive temperature (Fig. 1).

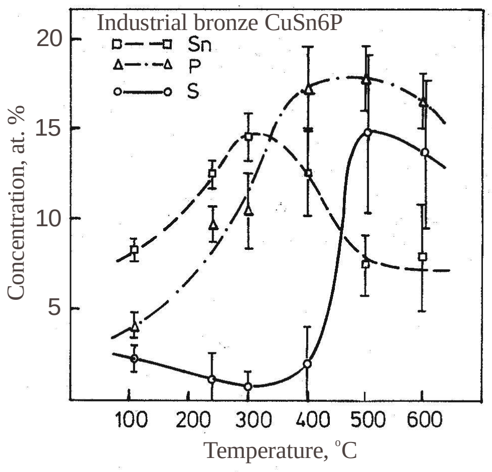

Figure 6. Dependence of the atom concentration Sn, P and S from the intercrystalline fracture surface on the temperature of tensile tests: industrial bronze CuSn6P

Spectrometric investigations of tin bronzes have proved principally the role of sulphur, suggested in literature, concerning the strong tendency to intercrystalline segregation leading to a decrease of ductility at elevated temperature of many metals and their alloys. AES analysis has also shown that the atoms of the components of alloys (Sn and P) indicate a distinct tendency to segregation at the grain boundaries.

The results of investigations concerning the structure and mechanical properties of CuSn6P and CuSn6 bronzes at elevated temperatures permit to determine the essential mechanisms of the decohesion at the grain boundary in the course of plastic deformation at the strain rate $\dot{\varepsilon} \geq 1.2 \cdot 10^{-5} s^{-1}$. The rupture of intercrystalline α-tin bronzes is due mainly to the mechanism GBS causing the localization of nucleation of microcracking, mainly wedge-type (Fig. 4) at privileged grain boundaries versus tensile stresses mostly at the contact of two or three grains, where the stresses reach their maximum. This mechanism is intensified by the synergetic effect of the segregation of Sn, P and S atoms at the grain boundaries. The presence of such segregations leads to an acceleration of GBS, the growth of potential intergranular cavitation, the decohesion of bronze on the grain boundary in the macroscopic scale and the formation of intercrystalline-brittle fractures in the case of coarse-grained structures of bronze or fractures with a relative small share of the surface deformation in the case of a fine-grained structure.

5. CONCLUSIONS

1. The intercrystalline brittleness of investigated α-tin bronzes deformed in hot tensile and torsion tests is determined mainly by the chemical composition of the matrix, particularly the purity of the grain boundary and the mean size of the grains.

2. Industrial bronzes CuSn6P and CuSn6 with a coarse grain structure have an intercrystalline embrittlement within the DMT range (300÷700°C), independently of the way and strain rate in the range $\dot{\varepsilon} \geq 1.2 \cdot 10^{-5} s^{-1}$.
3. The refinement of grains to about 10μm ensures a satisfactory ductility of the bronzes (Z~40%) in a limited DMT range (~300÷500°C) and a higher ductility (Z~60%) at a deformation temperature of 400°C to 500°C.
4. α-tin bronzes fail mainly in a brittle intercrystalline way with a varying degree of the share of plastically deformed surfaces depending on the grain size.
5. α-tin bronzes deformed beyond the range of DMP fail typically in a transcrystalline ductile way.
6. Auger's spectrometric analysis of intercrystalline fractures of CuSn6P bronze *in situ* indicated a segregation of Sn, P and S atoms at various intensities depending on the deformation temperature.
7. The intercrystalline brittleness of the investigated α-tin bronzes is mainly due to the GBS mechanism, generating a concentration of tensile stresses at the grain boundary, which - depending on the chemical composition of the grain boundaries - activate the nucleation of microcracking at the contact of two or three grain boundaries. The synergetic effect of these micromechanisms is the direct reason of the formation of macroscopically smooth (brittle) or cavitationally-ductile intercrystalline surfaces, detected fractographically.

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