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## Superplastic deformation behaviour of aluminium containing brasses



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#### ABSTRACT

Superplastic behaviour of binary two-phase brass and brasses with aluminium addition are compared. Indicators of the superplasticity are considered at a temperature range of  $525-600\,^{\circ}\text{C}$  and at constant strain rates of  $5\cdot 10^{-4}\,\text{s}^{-1}$  and  $1\cdot 10^{-3}\,\text{s}^{-1}$ . The effect of aluminium addition on the superplastic deformation indicators is studied. Improvement of superplasticity and significant decreasing of cavitation are identified due to alloying by aluminium. Surface relief of the samples after superplastic deformation, and grain boundary sliding contribution are analysed, and deformation mechanisms are discussed.

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#### 1. Introduction

Superplasticity is an ability of fine-grained materials (with grain size less than 10  $\mu m$ ) to show large neck-free elongations during deformation at high temperatures (usually T=0.5  $T_{melt}$ ) prior to failure due to high strain rate sensitivity of flow stress [1–3]. It is known that fine grain structure is an important element of superplasticity; it is vital to control the grain growth during superplastic deformation to provide a stable flow. Optimal superplastic temperature arrangement of volume fraction of  $\alpha/\beta$  phase is about 50/50 for duplex type Ti and Cu based alloys due to high grain stability [3]. Grain growth retardation is a direct function of high elongation at superplastic deformation [4]. Elongation is 500–640% in Cu–40Zn at the test with low initials strain rates of about  $10^{-4}\, s^{-1}$  [5]. Severe plastic deformation allows increasing strain rates to  $10^{-2}\, s^{-1}$  with the same elongation due to significant grain refinement [5].

Mechanisms of superplastic deformation are the same as creep mechanisms: grain boundary sliding (GBS), dislocation slip (creep) and diffusion creep. GBS is the predominant mechanism responsible for high strain rate sensitivity of many alloys. Contribution of GBS can be 90% in Zn–22Al alloy and in similar alloys with a eutectoid or eutectic structure [6,7]. It was established that the main mechanism in binary brass Cu–40Zn is GBS [8,9]. Superplastic deformation of Al Brass (Cu–Zn–Al–Fe–Mn system) was reported in [10] and the dominant mechanism of deformation was also GBS at  $1\times 10^{-3}~\text{s}^{-1}$ , and deformation process was supported by dynamic recrystallisation at  $1\times 10^{-2}~\text{s}^{-1}$  strain rate. In two-

phase materials with equal volume fractions of phases, the interphase boundaries occupy about 80% of the boundaries. Therefore, the sliding nature of interphase boundaries is more dominant at superplastic deformation [11]. Superplastic deformation in brass appears under friction in boundary lubrication and it is attributed to intergranular sliding inside  $\alpha$ -grains [12]. Temperature has an enormous effect on superplasticity by subsequently improving diffusion processes and grain boundary sliding phenomena.

The main problem of superplastic forming of Cu-Zn alloys is high susceptibility to cavitations [13-17]. A cavitation phenomenon is an important factor that decreases elongation of brass, which occurs during straining leading to early necking [18,19]. Moreover, cavities decrease the mechanical properties of asformed parts and applicability of superplastic forming of brass. It is well known that the level of cavitation increases as strain, strainrate and grain size increase and decrease as the deformation temperature increases [8,14]. GBS influences the cavity growth rate and can be the main cause of cavitation. Pores are formed in triple grain boundaries and ledges are formed on grain boundaries due to an increase of stress in these places [4,19]. Some studies find that the amount of cavities depends on  $\alpha/\beta$  ratio; thus, the cavitation is low for  $\alpha/\beta$  brass with high-volume fraction of  $\beta$ phase [18]. For  $\alpha/\beta$  brass alloys with high-volume fraction of  $\alpha$ phase, when the strains of  $\beta$  phase is too high, coalescence of cavities occurs, which results in early fracture without external necking [20]. Superplasticity of binary brass has been studied carefully [5,8,9,14], and only a few works [8,9] have discussed the superplastic deformation of alloyed brasses. There is no analysis of the alloying elements effect on superplastic behaviour and deformation mechanisms of alloyed brasses. In the present study, the effect of alloying by Al in  $\alpha/\beta$  brass on superplasticity, role of GBS and cavitation during superplastic deformation are analysed.

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#### 2. Experimental methods

#### 2.1. Materials and processing

Cu–Zn based alloys with a duplex structure were studied. The alloys were cast out into water-cooled mould, hot rolled at the temperature of  $750\pm10$  °C, and subsequently cold rolled in 75% to a final sheet thickness of 1.2 mm. Alloy compositions in as-cold worked condition are given in Table 1. Binary Cu–Zn alloy and tree alloys with different amount of Al were analysed. Solidus temperature ( $T_s$ ) was determined by a differential thermal analysis using "Setaram Labsys DSC 1600" at a heating rate of 5 K/min. Solidus temperature is non-significantly changed with an increase of Al concentration (Table 1).

#### 2.2. Microstructures

Microstructure characterizations were carried out by means of an optical Axiovert 200MMAT Carl Zeiss" (Germany) and scanning electron Tescan-VEGA3 LMH (Czech republic) microscopes. Specimens were prepared by mechanical grinding and polishing in colloidal silica (1  $\mu m$  dispersion) using Struers LaboPol-5 machine (Denmark). Electro polishing was performed in orthophosphoric acid with a density of 1.2 g/cm³ for about 10 s at 3 V. The cathode was a corrosion-resistant steel. Chemical etching in 3% solution of FeCl₃ in 10% HCl during 2–3 s was used to identify phases and grain boundaries.

The average grain size was determined by a random secant method, using more than 300 measures along the rolling and tensile direction. Volume fraction of cavities and phases were analysed of 5 – 7 SEM pictures by AxioVision programme software version 4.5. Error bars were determined experimentally using a standard deviation of values and a confidence probability of 95%.

#### 2.3. Tensile tests

Specimens with a gauge section size of  $F_o=6\times 1.2~\mathrm{mm}^2$  and length of  $I_o=14~\mathrm{mm}$  were cut parallel the rolling direction to determine the superplasticity indicators. A Walter Bay LFM – 100 test machine (Switzerland) with a programme service Dion-Pro for the control of the traverse motion was used. Two different types of uniaxial tensile tests were applied: (1) tests with a step-by-step increase of the stretching rate in 1.5 times and (2) tests at the constant strain rates. The tests with step-by-step increasing the stretching rate were performed at a temperatures range of 525°C to 600°C with a step of 25 °C, which corresponds to 0.68–0.74T $_s$ ; where  $T_s$  is a melting (solidus) temperature of the alloys. The strain rate sensitivity index m was determined via increases in the strain rate according to the calculation of the slope of the logarithmic stress-strain curve.

Constant strain rates tests were carried out at  $5 \times 10^{-4} \, \text{s}^{-1}$  and  $1 \times 10^{-3} \, \text{s}^{-1}$ , and at temperatures of 550 °C and 600 °C. Traverse speed was increased with an increase in the length of the testing sample to maintain a constant strain rate; the accuracy of the constant strain rate was not less than 97%.

 $\label{eq:Table 1} \textbf{Table 1} \\ \text{The compositions (in wt\%) and solidus temperature } (T_s) \text{ of the alloys via chemical analysis.}$ 

№	Alloy	Cu	Zn	Al	$T_{s,}{}^{\circ}C$
1	CuZn	59.2	40.8	-	899
2	CuZn0.5Al	59.1	40.5	0.5	897
3	CuZn1Al	61.2	37.8	1.0	903
4	CuZn2Al	64.5	33.3	2.2	909

#### 2.4. Constitutive modelling

The constitutive models of hot deformation behaviour were constructed using II-region data (liner part of ln(stress) – ln(strain rate) curves). It allows making a quantitative analysis of the alloying elements influence on the superplastic deformation behaviour. Zener-Holomon parameter (Z) (Eq. (1)) [21] for the flow stress in terms of deformation temperature and strain rate was used to calculate the effective activation energy of superplastic deformation:

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right),\tag{1}$$

where  $\dot{\varepsilon}$  is the strain rate (s<sup>-1</sup>); T the deformation temperature (K); Q the effective activation energy of deformation (kJ/mol); and R the universal gas constant (8.314 kJ mol<sup>-1</sup> K<sup>-1</sup>). The relationship between Z and the flow stress ( $\sigma$ ) is expressed by different empirical equations, such as the power function (Eq. (2)), the exponential function (Eq. (3)), and the hyperbolic sine function (Eq. (4)) [22].

$$Z = A_1 \sigma^{n_1} \tag{2}$$

$$Z = A_2 \exp(\beta \sigma) \tag{3}$$

$$Z = A_3 \left[ \sin h(\alpha \sigma) \right]^{n_2} \tag{4}$$

where  $A_1$ ,  $A_2$ ,  $A_3$ ,  $n_1$ ,  $n_2$ ,  $\beta$ , and  $\alpha$  are material constants;  $\alpha$  is a stress multiplier and a constant that adjusts the predicted values into the right range;  $\alpha$  can be described as:

$$\alpha \approx \frac{\beta}{n_1}$$
 (5)

#### 2.5. Study of the surface relief and contribution of GBS

The surface of the tensile specimens was prepared before the tests by grinding them on SiC papers and a colloidal silica suspension. Deformation at the microstructural scale was studied using scratches oriented in  $90^{\circ}$  to the tensile direction on the polished surfaces of specimens. The cross scratches were produced by a  $3 \, \mu m$  diamond paste. The specimens were tested at  $1 \times 10^{-3} \, s^{-1}$  constant strain rate at 550 °C in an Ar atmosphere to prevent substantial oxidation. The contribution of GBS was assessed based on the offsets of scratches; the experimental method was discussed in detail in [23]. Focused ion beam (FIB) milling was employed, using a STRATA FIB –  $205.^1$  The FIB method was used for milling trenches in the deformed surfaces to investigate the structure beneath the surface [24].

#### 3. Results

#### 3.1. Initial grain structures and phase composition analyses

The studied alloys belong to  $\alpha+\beta$  field at a temperature range from 525 °C to 600 °C as calculated by ThermoCalc (see equilibrium isothermal sections for 550 °C and 600 °C in Fig. 1). Transformation  $\beta\to\beta'$  occurs at 454 °C but we write  $\beta$  in all cases, which means that  $\beta'$ -phase exists at temperatures lower than 454 °C and

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