



Analysis of cracking of low-alloy copper stretched at elevated temperature

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ABSTRACT. This paper presents both mechanical and structural aspects of micro-cracking of CuNi2Si copper alloy in CNCS grade revealed during the static tensile test in the temperature range between 20°C and 800°C.

The purpose of this paper is to determine the impact of plastic deformation temperature and structural condition of the tested alloy on the type and mechanism of its cracking under specified deformation conditions. Therefore, the subject of the detailed metallographic analysis in a microscopic scale is the location of initiation and propagation of cracking, morphology of precipitations and their impact on the cracking process, nature and type of the fractures formed during material decohesion as well as microstructural analysis of the alloy by electron diffraction method. The obtained results allow the determination of the impact of analysed factors on cracking mechanism in the tested alloy as well as the specification of effective methods for limitation of the effects of cracking and thus the improvement in plasticity of alloy and workability of its products.

KEYWORDS. Low-alloy copper; Plastic deformation; Structure; Mechanical properties; Brittleness; Process of cracking.

INTRODUCTION

Low-alloy copper alloys are used in various ways. Most of them are applied in the electrical engineering and electronics. They are also used in the production of welding electrodes, elements of bearings, non-sparking tools and chemical apparatus. An important technological problem is the occurrence of brittleness in these alloys during hot plastic deformation. This phenomenon is a result of reduced plasticity at a specific deformation temperature, called the ductility minimum temperature (DMT). The copper alloy cracking mechanisms, which have not been fully explained yet, depend on many physicochemical, structural and mechanical factors related to the chemical composition (liquid films and segregations at the grain boundaries as well as inclusions and precipitations), structure of the alloy (grain size, crystallisation defects, non-uniform deformation) and deformation parameters (deformation temperature and strain rate, specimen surface condition, type of mechanical test) [1÷4].



In this paper, the impact of deformation temperature on the mechanical properties and structure of low-alloy copper containing nickel, silicon and chromium of the CuNi2Si type [5] was investigated. In particular, its temperature range of reduced plasticity as well as the impact of deformation temperature and condition of the structure of the alloy on the mechanism of its cracking was determined.

MATERIALS AND METHODS

The investigations were carried out on copper alloy CuNi2Si with grade designation mark CNCS (Hovadur). The alloy was delivered as a rod of 30mm in diameter and 200mm in length. The $\phi 30$ rod was forged into specimens of 14mm in diameter at 850°C with air cooling. After forging, the tested alloy showed tensile strength $R_m=450\text{MPa}$, yield point $R_{p0.2}=365\text{MPa}$, elongation $A=25\%$, reduction of area $Z=48\%$ and hardness $HV10=110$. The chemical composition of the tested alloy is presented in Tab. 1.

Alloy designation CuNi2Si (CNCS) acc. EN-Norm	Concentration of elements, % wt				
	Ni	Si	Cr	others	Cu
acc. to check analysis	1.99	0.59	0.43	0.3	remainder
acc. to PN- EN 12163	1.6 - 2.5	0.4 – 0.8	0.40	0.3	remainder

Table 1: Chemical composition of CuNi2Si alloy.

After forging, the tested alloy was supersaturated in water from 850°C, 940°C and 1000°C. The supersaturation temperature was determined according to the analysis of binary systems of phase equilibrium of copper with nickel, silicon and chromium [6,7]. Based on the results of initial metallographic observations and hardness measurements, the optimum supersaturation temperature of 940°C was selected. The average grain size in α solution after supersaturation from that temperature was approx. 30 μm . For static hot tensile tests the specimens were supersaturated from 940°C. The operations of heating and soaking for supersaturation were carried out in Thermolyne chamber furnace of 4.4 kW equipped with a controller to ensure temperature measurements with accuracy of $\pm 1^\circ$. The total heating and soaking time in supersaturation process was 60 min. After supersaturation, the mechanical properties of the CNCS alloy were as follows: $R_m=271\text{MPa}$, $A=66\%$, $Z=83\%$ and $HV10=55$.

In order to accomplish the purpose of this paper, the investigations of chemical composition by spectrographic method, testing of mechanical properties in the range of temperatures between 20°C and 800°C, metallographic investigations with light microscope, scanning electron microscope (SEM) and transmission electron microscope (TEM), and hardness measurements were performed.

Mechanical properties of the CuNi2Si alloy were tested on a universal testing machine INSTRON 4505 using specimens with a threaded grip of 6mm in gauge diameter and 26mm in length. The hot tensile tests were carried out in the temperature range between 20°C and 800°C at a tensile rate (v_t) of 2 mm/min, corresponding to the strain rate of $\dot{\epsilon} = 1.2 \cdot 10^3 \text{ s}^{-1}$, in the protective atmosphere of argon. The operations of heating and soaking of specimens to be stretched were performed in a chamber furnace with zone-controlled temperature measured with a Pt-PtRh thermocouple. The microprocessor furnace control system ensured temperature measurement accuracy of $\pm 2^\circ\text{C}$. Hardness was measured by Vickers method with a hardness testing machine HASUSER by applying the load of 10N to specimens in an as-forged condition and after supersaturation. Micro-hardness was measured on microsections of the CNCS alloy after tension at 200°C, 400°C, 550°C and 800°C using a micro-hardness testing machine PMT3 by applying the load of 50G for 15s.

Metallographic investigations of the CuNi2Si alloy were carried out on longitudinal microsections after hot forging, supersaturation and hot tensile tests in the temperature range between 20°C and 800°C. In order to display their structure, the microsections were etched in a reagent containing 5g of ferric chloride (FeCl_3), 10cm³ of hydrochloric acid (HCl) and 90cm³ of ethyl alcohol ($\text{C}_2\text{H}_5\text{OH}$). The metallographic observations were performed using a light microscope Leica

MEF4A with a magnifying power from 100x up to 1000x. For determination of the microstructure and phase identification of precipitations in the tested alloy the observations were performed under a scanning/transmission electron microscope S/TEM Titan80-300(FEI) at 245÷300kV in classic TEM operation system with image resolution below 0.1 μ m. The preparations for observations were made in the form of foils using mechanical thinning and ion polishing. Fractographic tests of specimen fractures after hot tensile test were performed using a scanning electron microscope with resolution of 2 μ m at accelerating voltage of 20kV. For micro-analysis of precipitations revealed on fractures of the tested specimens an X-ray micro-analyser EDX was used. The tests performed on a scanning microscope allowed recording the topographic images of fractures with a magnifying power from 200x up to 15000x.

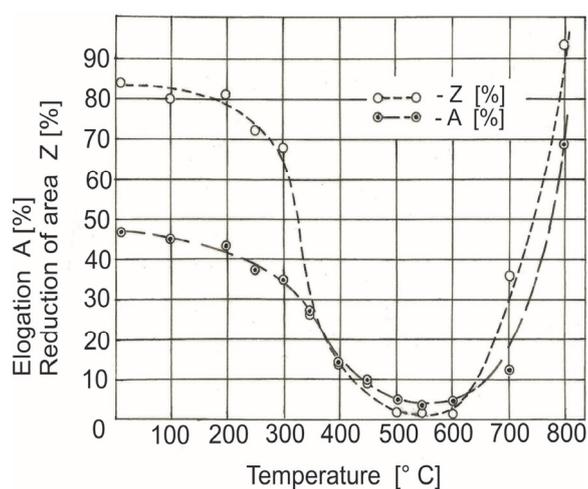


Figure 1: Strength (R_m) vs. CuNi2Si alloy tension temperature.

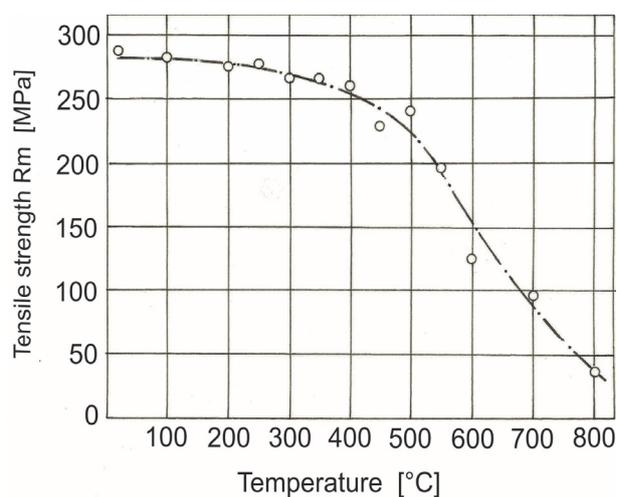


Figure 2: Elongation (A) and reduction of area (Z) vs. CuNi2Si alloy tension temperature.

RESULTS AND DISCUSSION

The results of the static hot tensile test in the examined range of temperature and at the strain rate $v_r = 2\text{mm/min}$ have enabled the effect of temperature on the mechanical properties of the CuNi2Si alloy to be determined. The obtained results of elongation and reduction of area allowed the determination of the range of temperature in which reduced plasticity occurs. The results of investigations of mechanical properties after hot tensile tests of the tested alloy are presented in diagrams (Fig. 1, 2). The rise of temperature from 100°C up to 500°C has been found to result in insignificant reduction in R_m from 282 MPa to 239 MPa, while the rise of deformation temperature from 550°C to 800°C results in sudden decrease in strength from 189 MPa to 36 MPa (Fig.1).

The effect of deformation temperature on the value of elongation and reduction of area of the tested alloy is presented in the diagram (Fig. 2). The curves of elongation (A) and reduction of area (Z) show the range of temperature in which reduced plasticity of the test alloy occurs. The minimum value of elongation and reduction of area is shown the CNCS alloy during deformation at 550°C. The course of the elongation vs. tension temperature curve is variable. With increase in the tension temperature from 350 to 500°C, the elongation reduces suddenly to reach its minimum at 550°C. A further rise of the deformation temperature affects the increase in plasticity of the tested alloy (Fig. 2). The dependences of the reduction of area and elongation on temperature have been found to be similar (Fig.2). The increase in deformation temperature from 350°C to 450°C results in significant decrease in the reduction of area from approx. 27% to approx. 9%. In the temperature range between 550 and 600°C, the minimum reduction of area of the tested alloy equal to approx. 1% is observed. The rise of alloy deformation temperature in the range of (600 ÷ 800°C) results in increase in the reduction of area. After tension at 800°C, the CuNi2Si alloy reaches its maximum elongation of 92.5%.

The results of metallographic investigations have enabled the impact of structure of the tested alloy in an as hot-forged state and after supersaturation from 940°C as well as after hot deformation on the mechanism of initiation and propagation of its cracking to be assessed. The results of observations are shown in microphotographs (Fig. 3÷8).

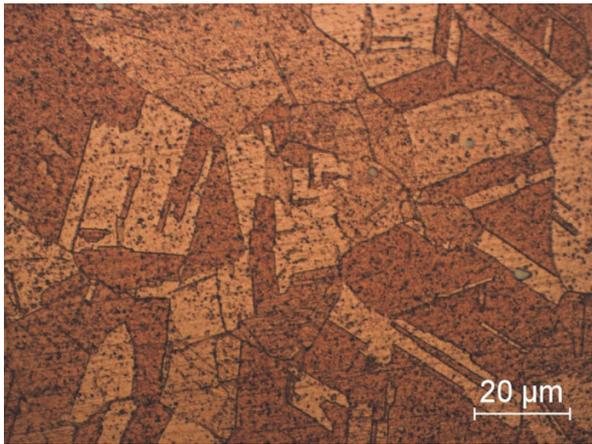


Figure 3: Grains of α phase in CNCS alloy after supersaturation from 940°C.

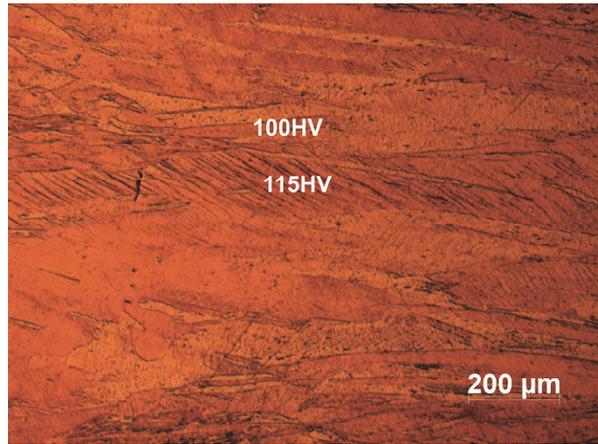


Figure 4: Elongated grains of α phase after tension at 200°C

The structure of the tested alloy after hot forging is characterised by fine grains of α solution, elongated towards plastic deformation, with average diameter of approx. 30 μ m. After supersaturation from 940°C, the grains of α solution with twins of 10÷30 μ m were revealed in structure of the CuNi2Si alloy (Fig.3).

In structure of the tested alloy stretched at 200°C, the elongated grains of α phase with numerous sliding bands have been revealed (Fig.4). Micro-hardness of the area with sliding bands has been found to be approx. 115 HV05, while hardness measured within the area of the twin is approx. 100 HV05 (Fig.4). After tension at 400°C, the areas of grains, varying in their size, with micro-cracks at their boundaries occur in structure of CNCS alloy (Fig. 5). In addition to large grains of approx. 200 μ m and micro-hardness of approx. 90 HV05, there are also recrystallised grains (HV05=85) with average diameter of approx. 10 μ m (Fig.6). After tension at 550°C, micro-hardness of non-recrystallised grains increases up to 151HV05 and of crystallised ones to120HV05 (Fig.7). Micro-cracks and diverse grain size in the tested alloy after tension at 550°C have been found to result in a sudden decrease in plasticity during plastic deformation. These effects are the result of slide at the grain boundaries and intergranular precipitation during hot deformation.

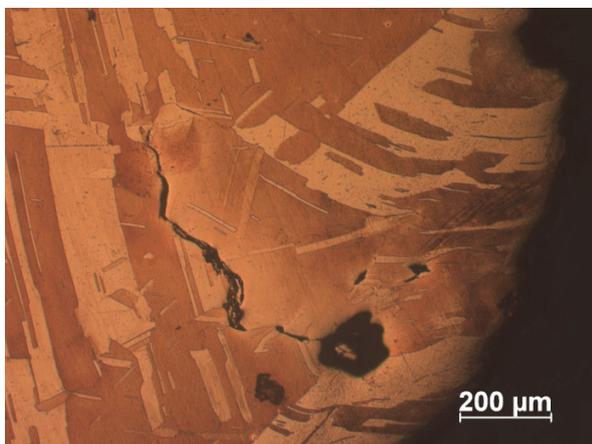


Figure 5: Micro-cracks at the boundaries of α grains in CNCS alloy after deformation at 400°C

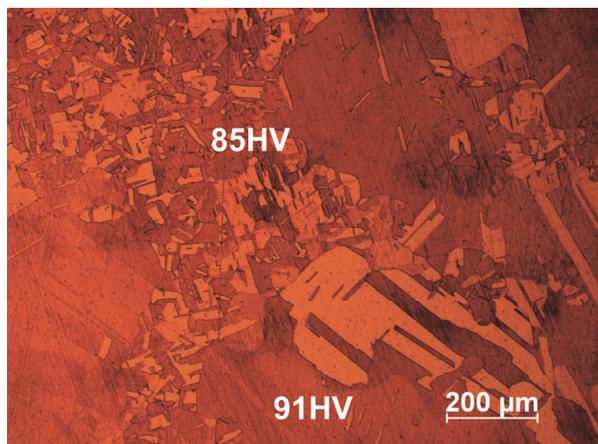


Figure 6: Grains of α phase varying in their size in structure of CNCS alloy after deformation at 400°C

In the specimen rupture zone there are areas of grains with average diameter of approx. 10 μ m and micro-hardness of approx. 120HV05 as well as micro-cracks along the grain boundaries (Fig. 7), while in the central zone there are large non-recrystallised grains of α phase of approx. 250 μ m with annealing twins and micro-cracks at the end face of the twins (Fig. 8).

After tension at 800°C, fine recrystallised grains of α phase with mean diameter of approx. 10 μ m as well as cracks and micropores were revealed in structure of the tested alloy within the specimen rupture zone. Micro-hardness of these areas

is approx. 95HV05. In the central part of the specimen, there are partially recrystallised grains varying in their size with deformed twins and voids.

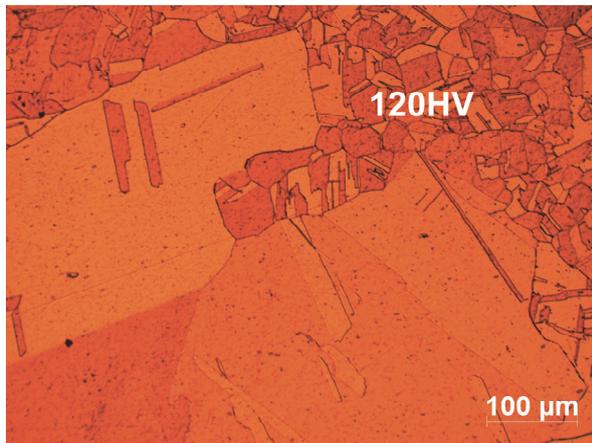


Figure 7: Fine recrystallized grains at the boundaries of large α grains in structure of CNCS alloy after tension at 550°C

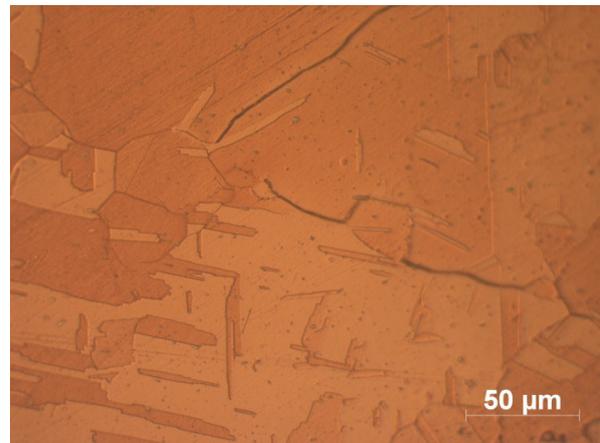


Figure 8: Cracks at the boundaries of α phase grains in structure of CNCS alloy after tension at 550°C

The fractographic investigations have allowed the nature of fractures of the CuNi2Si alloy in an as-forged state, after heat treatment and after deformation in the hot tensile test in the temperature range of 200–800°C to be determined. The results of fracture observations and EDX microanalysis of identified precipitations occurring in the CNCS alloy are shown in microphotographs (Fig. 9–11). The occurrence of a typical ductile fracture with numerous pits and craters with precipitations at their bottoms has been found in an as-forged state. The concentration of copper (93.21%), nickel (3.55%) and chromium (1.0%) has been revealed in their chemical composition. After supersaturation from 940°C, the nature of the CuNi2Si alloy fracture is similar to that in an as-forged state. The existence of precipitations in fracture of the supersaturated alloy indicates their primary nature.

After the tensile test at 200°C, there is a transcrystalline ductile fracture in the tested alloy, with numerous characteristic pits and craters in which the presence of fine precipitations (Fig. 9) with small concentration of copper, nickel, chromium, silicon and manganese as well as sulphur contamination has been found. The ductile nature of the fracture mainly confirms high plasticity of the tested alloy (Fig. 2).

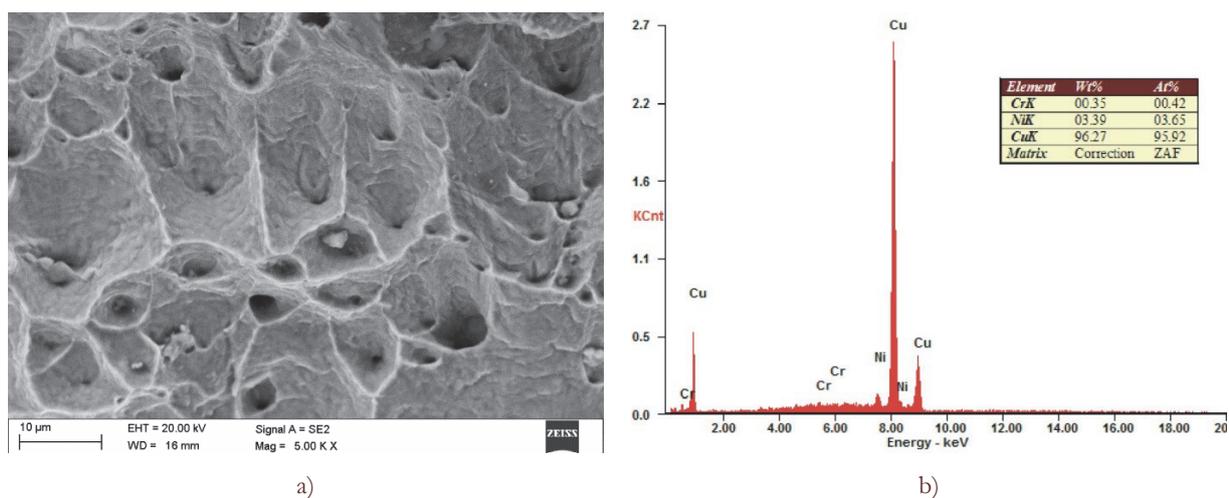


Figure 9: a) Ductile transcrystalline fracture with precipitations in craters, b) microanalysis of chemical composition of precipitation in CuNi2Si alloy after tension at 200°C



At the deformation temperature of 400°C, mixed fracture is observed. In addition to small areas showing typical plastic deformations, there are areas characteristic of brittle intergranular cracking. Trace effects of plastic deformation are also observed on the planes of cracking at the grain boundaries (Fig. 10).

After tension of the alloy at 550°C ($A=3.2\%$, $Z=0.7\%$), the brittle intergranular fracture with small plastically deformed areas is observed (Fig. 10).

Micro-cracks occurring due to slide at the grain boundary are observed on the fractures as an effect of one of the probable intergranular brittleness mechanisms [2]. The characteristic “catchment areas” and faults formed during relocation of the grain boundaries and numerous precipitations are observed on the planes of brittle cleavage cracking (Fig. 11).

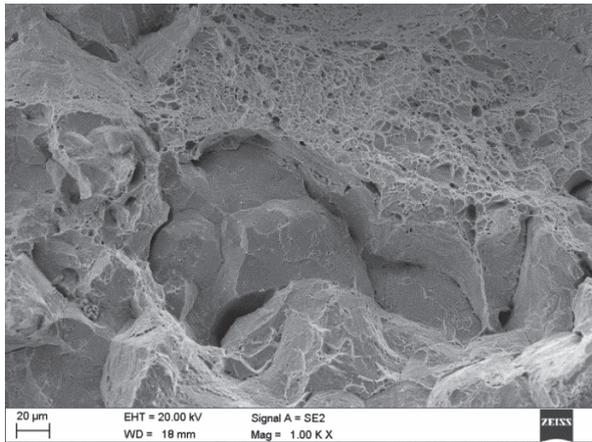


Figure 10: Mixed fracture after deformation of CNCS alloy at 400°C

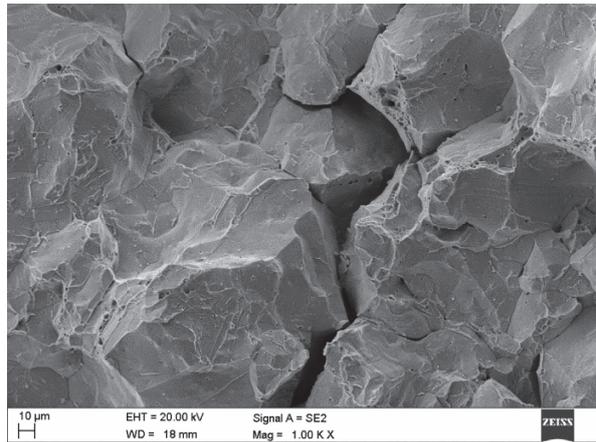


Figure 11: Brittle intergranular fracture of CNCS alloy after deformation at 550°C

The following were found in the chemical composition: Cu, Ni, Cr, Si and Mn as well as S as contamination. The concentration of these elements is 50%Cu, 31.69%Cr, 1.79%Ni and 0.22%Si, respectively. The observed alloy brittleness is above all the effect of intergranular precipitation that occurs during heating and deformation of the alloy at 550°C, resulting in the occurrence of numerous non-coherent coagulated precipitations on the cracking planes. After tension at 800°C, there is a fracture of typical ductile nature in the tested alloy showing high plasticity ($A=68.7\%$, $Z=92.5\%$).

The example of precipitation at the α solution grain boundary as well as the distribution of elements comprising this precipitation, revealed by means of a transmission electron microscope, is shown in the microphotograph (Fig. 12).

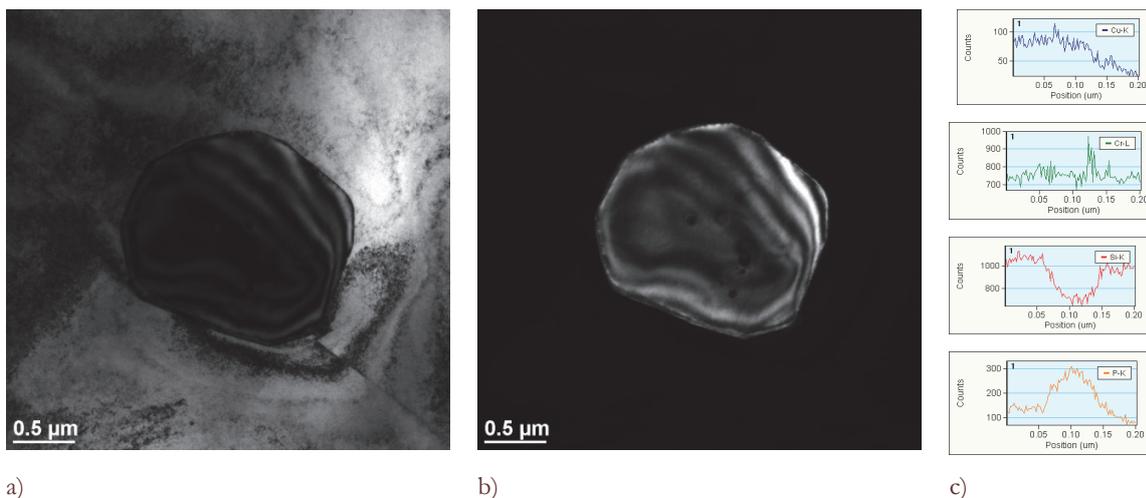


Figure 12: a) Precipitation at the boundary of α phase grains, b) dark field of the precipitation, c) linear distribution of elements in the precipitation.



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CONCLUSION

The performed investigations of the CuNi2Si alloy and the analysis of obtained results allowed the following conclusions to be drawn:

1. The CuNi2Si alloy after supersaturation from the optimum temperature of 940°C shows homogeneous α solution grains of approx. 30 μ m with annealing twins and hardness of approx. 55HV.
2. The increase in deformation temperature of the tested alloy from 20°C to 800°C results in monotonic reduction in strength properties and non-monotonic change in plastic properties when the so-called ductility minimum temperature (DMT) occurs.
3. The minimum plastic properties of the tested alloy at DMT of approx. 550°C are as follows: elongation (A) – approx. 3% and reduction of area (Z) – approx. 1%.
4. In structure of the CuNi2Si alloy deformed in the range of DMT, the α solution grains with precipitations and micro-cracks at the boundaries of grains and twins have been revealed. The revealed effects confirm that the main cracking mechanism in the tested alloy at elevated deformation temperature is intergranular precipitation and slide at the grain boundary.
5. The tested alloy deformed in the range of DMT is characterised by brittle intergranular fracture with traces of plastic surface deformation.

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