

Volume 50 Issue 2 August 2011 Pages 98-109 International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

Changes of microstructure in CuNi25 alloy deformed at elevated temperature

P. Sakiewicz*, R. Nowosielski, S. Griner, R. Babilas

Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

* Corresponding author: E-mail address: piotr.sakiewicz@polsl.pl

Received 29.05.2011; published in revised form 01.08.2011

ABSTRACT

Purpose: The aim of this paper was to present behaviour of CuNi25 alloy during elevated temperature tensile tests and describe changes of microstructure of material after deformation at the range of the Ductility Reduced Area (DRA) in which the phenomenon of Ductility Minimum Temperature (DMT) is situated.

Design/methodology/approach: Numerous techniques were used to characterize properties of material: high temperature tensile tests, transmission electron microscopy (TEM), HRTEM, FFT.

Findings: During the experimental studies the course of elongation and reduction of area curves has been determined. Morphology of material after deformation at elevated temperature was analysed.

Research limitations/implications: Further studies should be undertaken in order to correlate effects, processes and mechanism existing and superimpose in material in range of Ductility Minimum Temperature phenomenon, it should help us understand high temperature properties of mentioned material.

Practical implications: Knowledge about material properties during high temperature deformation leads to selection of the appropriate production parameters. Misapplication of parameters leads to multiplication of costs and often destruction of material during production or operating. Correct selection of technical and economical parameters of material production processes gives us supremacy in economic and technological competition.

Originality/value: Investigations of this CuNi25 alloy complete knowledge about mechanical properties and help us develop correct parameters for more effective technologies for material production.

Keywords: Metalls; Metallic alloys; Properties; Ductility Minimum Temperature (DMT); Elevated Temperature Ductility Reduced Area (DRA); Copper alloys; CuNi25; TEM; Changes of microstructure

Reference to this paper should be given in the following way:

P. Sakiewicz, R. Nowosielski, S. Griner, R. Babilas, Changes of microstructure in CuNi25 alloy deformed at elevated temperature, Archives of Materials Science and Engineering 50/2 (2011) 98-109.

PROPERTIES

1. Introduction

The phenomenon of Ductility Minimum Temperature (DMT) is one of the unexplained features of metals and their alloys, observed as the effect of intermediate temperature ductility decrease during high-temperature plastic deformation. This work is dedicated to the investigation of changes of microstructure observed in TEM of CuNi25 alloy (grain size 50 μ m and 150 μ m

and as cast alloy) deformed at the range of 0.3-0.7 T_m (melting point temperature). Based upon literature studies, observation and analysis of plastic deformation processes and their influence on range and level of DMT phenomenon, in CuNi25 alloy can be accepted, as in brass, hypothesis of non-uniform deformation [1].

In literature the phenomenon of Ductility Minimum Temperature is widely described [2-9], scientific research shows that this effect is a common attribute of many polycrystalline metals and alloys for example copper and its alloys, steel, titan. The explanation of the cause of the DMT effect is difficult because of the variety of studied materials, investigative methods, experiment conditions and limited possibilities of identical repetition of the experiment each time. In different metals and alloys, it is possible to identify heterogeneous mechanisms responsible for the loss of ductility and crack formation resulting in destruction of the material [8].

Conditions of hot deformation at intermediate temperature in many metals and their alloys depend on many factors, selection of appropriate conditions requires understanding of processes and theirs causes. Identification of one mechanism responsible for ductility through observed during intermediate temperatures hot working processes is difficult because of many correlations between inter alia: different grain boundary/bulk diffusivity ratios, different internal oxidation course, kind and morphology of grain boundaries and grain junctions, grain boundary serrations, rate of deformation, non-uniformity of chemical composition and process of deformation, segregation of impurities on grain boundaries, geometrical heterogeneity in each scale, shape and size of grains, the grain boundary character distribution, Strain Induced Grain Boundary Premelting, character of the grain boundary connectivity, thermal activated internal dynamic transmutation, temperature and its local changes, type of environment, differences in quantity and type of crystalline building defects, cavitations, Diffusion Induced Grain Boundary Migration, different kind, rate, and localization of stress relaxation processes and many others. While temperature rises, thermal activated processes take place in more areas, after reaching suitably high temperature, almost in whole volume of sample, provoking stress relaxation leading to increase of material ductility [8].

The critical level of non-homogeneity, caused by factors mentioned above leads to concentration of stress in the material in both macro and microscopic scale causing decrease of ductility. As the temperature rises, thermally activated processes take place in more areas, after reaching suitably high temperature, almost in whole volume of sample provoking stress relaxation which lead to increase of material ductility. The non-uniformed course of plastic deformation process at mixed areas caused by various forms, conditions, and type of deformation in micro scale correlated with a number of factors affecting DMT phenomena cause different but always reduced level of ductility and temperature range of the Ductility Reduced Area [1].

Therefore, the main problem with DMT clarification is explanation of the connections between proceeding micro mechanisms and the decreasing level of ductility, which as a result, leads to described macroscopic behaviour and destruction of material. We can make an assumption that DMT is a result of specific, different for each case, combinations of mechanisms accompanying the process of deformation at 0.3-0.7 T_m and none of them specifies mechanisms responsible for DMT phenomenon [1].

In metals, we can identify three temperature areas of ductile deformation: low-temperature, high temperature, and intermediate also called transitional, located in the range of 0.3-0.7 homologous temperature (T_H). The Ductility Reduced Area (DRA) around the DMT (Fig. 1) deserves special attention because there the low and high-temperature plastic deformation mechanisms overlap one another, and as a demarcation point between them recrystallization temperature was set. The DRA is also a range of occurring and accompanying various combinations of mechanisms, processes and many phenomenon which

affect the dynamic changes of structure and they are located heterogeneously in different areas and run unhomogenously with different intensity.

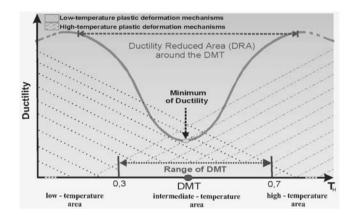


Fig. 1. The theoretical range of the Ductility Reduced Area (DRA) and scope of Ductility Minimum Temperature phenomenon

However, it should be remembered that DMT effect is only a quantitative indication of temperature which is characterized with the lowest level of ductility in DRA. From the material and technological engineering point of view, most interesting is the answer to the question about the reason for occurrence of reduced ductility area. This will allow for optimization of technological processes and financial savings associated with the processes of production and exploitation of products.

The course of plastic deformation is inextricably linked to the macroscopic growth, and locally to the concentration of stresses and their relaxation. Local differences in the level of stress may reach values up to hundredfold. The course of the formation and stress relaxation in the DMT is in its nature a heterogeneous process, the factors that determine it include local structure and grain size, chemical composition and velocity of the processes [1, 9, 10].

A special meaning of the grain boundaries for the process of deformation and strengthening is connected with the fact that they are the areas on which creation and relaxations of local concentration of stress occur [11]. Such concentrations may be of geometrical character which results from differences in shape and size of co-interacting grains. High concentration values are obtained for example in conditions of occurring grain boundaries sliding [12]. Yet another kind of concentration - usually more intense - appears as a result of heterogeneity of ductile deformation of grains and the interaction between dislocations and grain boundaries, so it is a microstructural process [13]. Stress relaxation on the grains boundaries may take place through generation and movement of dislocations or point defects, which may be accompanied by the phenomenon of annihilation of dislocations concentrated at grain boundaries. In extreme cases, relaxation may occur as a result of the transformation of grain boundary structure or be the result of formation of voids at grain boundaries, or intergranular cracking [8-14].

Stress relaxation is a phenomenon consisting in reducing the stress with time, while maintaining the total deformation caused by this stress. Stress relaxation, preventing nucleation and spreading of cracks by local removal of stress. Effect of stress relaxation on the ductility was studied in works of Zaren's [15], Xaio and Bai's [16] and Virtanen's [17]. In the work [1] it was found that at a certain critical level of heterogeneity in stress concentration occurs in the material at the macro and microscopic scale, influencing the course and location of deformation. The higher the temperature, the larger number of areas which are influenced by thermally activated processes, enabling stress relaxation in almost entire volume of the sample, contributing to the increase of ductility [18].

Research [19] carried out on thin films of Cu57Ni42Mn1 alloy showed that stress relaxation process occurred throughout the temperature range 200-550°C. It was also found that with increasing temperature the time needed for the occurrence of the relaxation process was shorter. Microscopic examination [20] of AlZn6Mg2.5Cu1.8 alloy revealed the occurrence of one of the mechanisms responsible for stress relaxation – grain boundary sliding, which contributes to the increase of ductility at elevated temperatures. In the work [16] it was shown that twinning together with the grain boundary sliding are responsible for stress relaxation at this range of temperature.

Diffusion processes in the border areas run faster and easier, and are often induced by stress [21]. Transport of matter flows along the grain boundaries and through the grains in the direction of the grains contact areas. This was proved by Palumbo [22] in a study carried out on nickel. Diffusive stress relaxation in the areas of grain boundaries is a special property occurring, inter alia, through loss of ability to transfer shear stress across the boundaries, especially at high temperatures. This property is related to the annihilation of dislocations at grain boundaries. The disappearance of strange grain boundaries dislocations is the mechanism by which grain boundaries can act as microstructural channels for dislocation network of different misorientation between crystallites in a polycrystalline material, so the disappearance of them is one of recovery mechanisms [11].

In polycristals there are always boundaries with different relaxation properties. The boundaries of the special properties are distinguished by the fact that – due to the limited capacity for stress relaxation – under given conditions the deformations constitute stiffer element of microstructure than the stronger-relaxing, soft general boundaries. The increase of participation in the microstructure of the boundaries with special properties is caused by technological factors or conditions of deformation and should therefore have an impact on the increase of the boundary of metals ductility as well as emergence of a tendency to homogeneous plastic flow [1, 11].

The occurrence of different phases and local differences in chemical composition of micro areas during the changes of metal temperatures due to the different values of the coefficient of thermal expansion may generate stress on the boundaries of this phases. This is the result of no concurrent changes of dimensions, which result in the fact that phases with a greater coefficient change in volume more rapidly, consequently causing compressive stress space.

In this article authors have focused on changes of microstructure of CuNi25 alloy deformed at elevated temperature and observed in TEM and HRTEM.

2. Material and experimental procedure

The investigations conducted on copper alloys [23-26] confirm occurrence of the DMT phenomenon throughout function

of temperature. The single-phase copper alloys seems to be an ideal material for investigations on DMT effect. Two kinds of singlephase copper-nickel alloy CuNi25 were used in the test. The first one, called in the work the alloy A, is the basic material for experimental studies. In order to check the influence of chemical composition on the scale of the DMT phenomenon, an industrial copper-nickel alloy CuNi25 was taken from Mill "Dziedzice", in this paper it was called alloy B. The chemical composition of investigated alloy has been shown in Table 1. The A alloy was delivered in the form of ingots with measurements of 250x400x600 mm. It was hot forged and cut, after this the material has been dragged to rods onto the diameter of 15 mm and then cut into sections of 300 mm. In order to diversify the structure, this material has been divided into two series and subjected to annealing. After heat treatment the structure of the material was homogeneous, and the average grain size of the material was 50 µm (called A50 series) and 150 µm (called A150 series). The results of measuring the size of the grain, were made by light microscopy image analyser. Plates of B alloy from continuous casting process with measurements of 250 x 500 x 15 mm were cut into rods of square section and has dendrite structure. From both types of CuNi25 alloy identical samples were made.

T 1	1	1
1.21	ne	
1 uu	510	1.

Chemical composition of CuNi25 (A) alloy, %

	CuNi25 (A)	CuNi25 (B)
Cu	rest	rest
Ni	25.1	24.43
Mn	0.3	0.22
Pb	0.005	0.01
Fe	0.3	0.1
Si	-	0.033
Zn	0.3	0.1
Sn	-	0.011
С	0.05	0.016
S	0.01	0.006
Al.	-	0.001
As	-	0.003
Р	-	0.012
Cd	_	0.002

Static tensile test was carried out at elevated temperatures and it was conducted on the testing machine INSTRON 1195. The proper tensile temperature was ensured by the electronically controlled resistance furnace equipped with a thermocouple Pt-PtRh13 and electronic temperature controller with an accuracy of \pm 2°C. After the static tensile test, the samples for the preservation of their structure were immediately cooled in water. After determining the scope of occurrence of the DMT effect, the temperature interval was set on 25°C in order to indicate accurately course of reduction of area and elongation in DRA. At each measuring point, 5 samples were deformed.

In order to investigate the microstructure of deformed samples, the study in transmission electron microscope on thin foils was conducted. The study was performed on two JEOL electron microscopes: transmission electron microscope (TEM) JEM 200 and the high-resolution transmission electron microscope (HRTEM) JEM-3010 made by JEOL. The thin foils were made from samples spanning the temperature range of deformation 300 to 800°C. They were ionic, prepared by polishing with polishing ion PIPS, Gatan model 691, and polisher providing nitrogen cooling during polishing. Image obtained in the microscope was recorded by the use of CCD camera.

3. Results and discussion

High temperature tensile test results revealed that variation of elongation and reduction of area for all analysed materials shows strong dependence on the test temperature, and for all cases Ductility Minimum Temperature phenomenon was found (Figs. 2-4). Results of tests performed for specimens of CuNi25 alloy were very similar, and showed existence of minimum of DMT phenomenon between 450-500°C for A alloy and between 525-675°C for B alloy. The area of reduced ductility around the DMT was detected in all cases between 350-650°C.

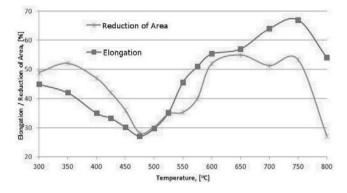


Fig. 2. Elongation and reduction of area versus test temperature for CuNi25 (A) 50 μ m, after deformation during high temperature tensile tests with strain rate $4.2 \cdot 10^{-3} \cdot s^{-1}$

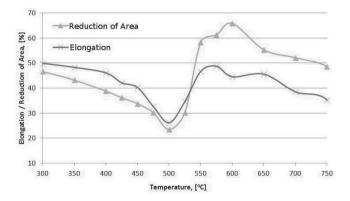


Fig. 3. Elongation and reduction of area versus test temperature for CuNi25 type (A) 150 μ m, after deformation during high temperature tensile tests with strain rate $4.2 \cdot 10^{-3} \cdot s^{-1}$

The curves of elongation show that the minimum of DMT effect for CuNi25 (A) 50 μ m alloy deformed with strain rate $4.2 \cdot 10^{-3} \cdot s^{-1}$ obtained at 475°C level of 27% for elongation and value 28% for reduction of area. Ductility minimum level for CuNi25 (A) 150 μ m series deformed with strain rate $4.2 \cdot 10^{-3} \cdot s^{-1}$ for elongation and reduction of area at 500 °C get value of 23% and 26% respectively. Minimum value of elongation and reduction

of area for as cast CuNi25 (B) alloy, after deformation with strain rate $2.7 \cdot 10^{-1} \cdot s^{-1}$ has been identified at 14% for both (RoA=550°C, and A=525°C).

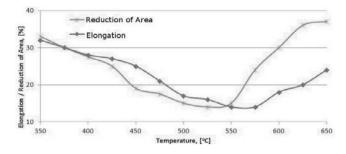


Fig. 4. Elongation and reduction of area versus test temperature for as cast CuNi25 (B) alloy, after deformation during high temperature tensile tests with strain rate $2.7 \cdot 10^{-1} \cdot s^{-1}$

The research of the structure in transmission electron microscope was performed for samples obtained from both cupronickel alloys series. Thin foils for the series CuNi25 (A) 50 and (A) 150 alloy and as cast alloy, were made from specimens deformed rate of $4.2 \cdot 10^{-3} \cdot s^{-1}$ in the temperature range of 300-800°C. The observations of CuNi25 alloy (B) were performed on preparations obtained from the samples deformed with strain rate of $2.7 \cdot 10^{-1} \cdot s^{-1}$ in the range 350 - 650 °C. The results of the studies conducted in the transmission electron microscope and high-resolution transmission electron microscope, complement each other, therefore, the results of the study were collected and described collectively for all the initial structures of the material. For the analysis, the sample was divided into three temperature ranges: below, on the level of and above occurrence of DMT.

The studies of thin films made of specimens of both cupronickels after deformation in the range of 300-400°C which is below the minimum DMT, reveal the presence of poorly formed cellular dislocation structure (Figs. 5 and 6) with a high density of dislocations in cell walls and the presence of areas with the occurrence of local dislocations cumulation. Near the grain boundaries there are dislocations cells of variable size and shape (Figs. 6 and 7), and microtwins of variable lengths (Figs. 5 and 8). The presence of areas characterized by different density of defects in the crystalline structure occurring in the vicinity of less deformed areas can be also found.

The samples CuNi25 alloy of A and B series after being deformed above 400°C, so in DRA, show in solid solution CuNi grains area local dislocation agglomeration, partially in the cellular system, characterized by different levels of density of dislocations (Figs. 9-13). Segregation and dispersed spherical emission on dislocations are most probably visible. In all of the studied materials, the presence of border zone of complex dislocation structure characterized by the occurrence of the areas of different density of defects of crystalline structure (Figs. 9-12). The zones of different saturation of dislocations were observed within grains and also the border areas. In the vicinity of wide angle grain boundaries the deformation structure is characterized with high heterogeneity in terms of density, shape and size of dislocation cells. In these zones the presence of elongated deformation cells was often observed.

The samples of CuNi25 alloy of A and B series after being deformed above 400°C, so in DRA, show in solid solution CuNi

grains area local dislocation agglomeration, partially in the cellular system, characterized by different levels of dislocations density (Figs. 9-13). Segregation and dispersed spherical emission on dislocations are most probably visible. In all of the studied materials, the presence of border zone of complex dislocation structure characterized by the occurrence of the areas of different density

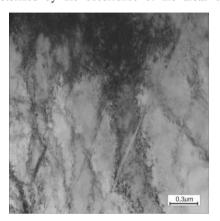


Fig. 5. The structure of CuNi25 (A) 50 after deformation at 300°C (TEM)

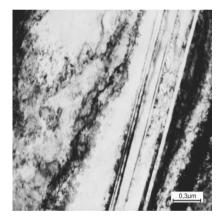


Fig. 8. The structure of CuNi25 (B) after deformation at 400° C (TEM)

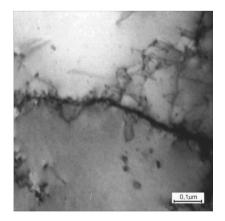


Fig. 11. The structure of CuNi25 (B) after deformation at 475° C (TEM)

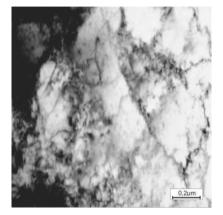


Fig. 6. The structure of CuNi25 (A) 50 after deformation at 350°C (TEM)

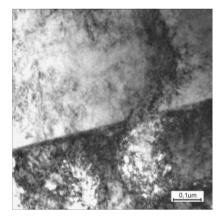


Fig. 9. The structure of CuNi25 (A) 50 after deformation at $450^{\circ}C$ (TEM)

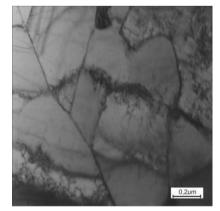


Fig. 12. The structure of CuNi25 (A) 50 after deformation at $450^{^\circ\text{C}}$ (TEM)

of defects of crystalline structure (Figs. 9-12). The zones of different saturation of dislocations were observed within grains and also the border areas. In the vicinity of wide angle grain boundaries the deformation structure is characterized with high heterogeneity in terms of density, shape and size of dislocation cells. In these zones the presence of elongated deformation cells was often observed.

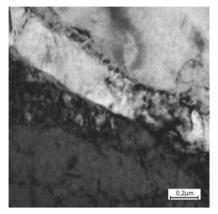


Fig. 7. The structure of CuNi25 (A) 150 after deformation at 400°C (TEM)

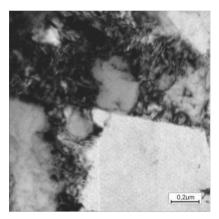


Fig. 10. The structure of CuNi25 (A) 150 after deformation at 500°C (TEM)



Fig. 13. The structure of CuNi25 (A) 150 after deformation at 475°C (TEM)

In the samples deformed in the range of 450-500°C within individual grains large differences in the density of defects of crystal structure was noticed, as well as the characteristic boundaries with "serrations" and areas with a dislocation cellular structure with visible "flexure" of grain boundaries and accumulation of dislocations in the border areas (Figs. 11-13). Also noticeable are the low-angle deformed subgrain boundaries with visible accumulation of dislocations in border areas. In samples after deformation in the range of 450-550°C, and therefore the range of DRA in both (A) 50 and (A) 150 series of CuNi25 alloy there are visible areas of increased density of defects in the crystalline building occurring on the junction point of the three and more grains (TJ) (Fig. 13) Observations conducted in high resolution transmission electron microscopy on thin foils obtained from the samples of CuNi25 (A) 50 deformed at 450°C revealed the sporadic presence of an almost linear grain boundaries, and individual dislocations and their agglomeration (Fig. 14) often occurring in the vicinity. The analysis performed in this areas using Fast Fourier Transformation revealed the occurrence of various density of defects of crystal structure in the areas lying on the opposite sides of the border.

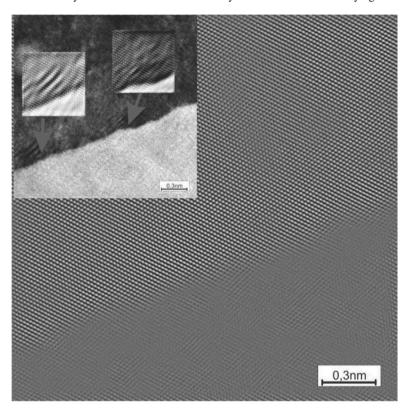


Fig. 14. The structure of CuNi25 (A) 50 after deformation at 450°C (FFT, HRTEM)

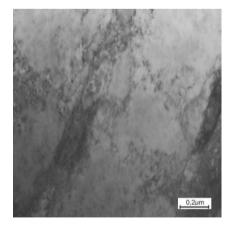


Fig. 15. The structure of CuNi25 (A) 150 after deformation at $475^{\circ}C$ (TEM)

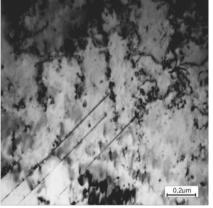


Fig. 16. The structure of CuNi25 (B) after deformation at $475^{\circ}C$ (TEM)

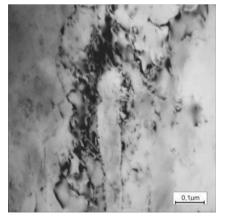


Fig. 17. The structure of CuNi25 (A) 150 after deformation at 550° C (TEM)

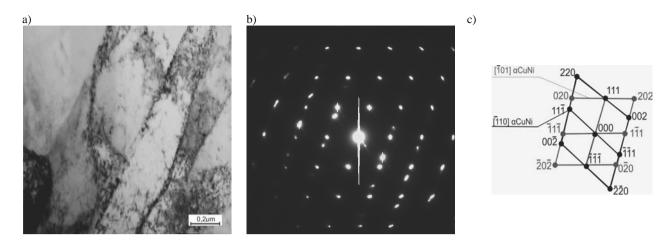


Fig. 18. a) The structure of CuNi25 (A) 150 after deformation at 550°C (TEM), b) diffraction pattern from Fig. 18a, c) Solution of diffraction pattern from Fig. 18b (thin foils)

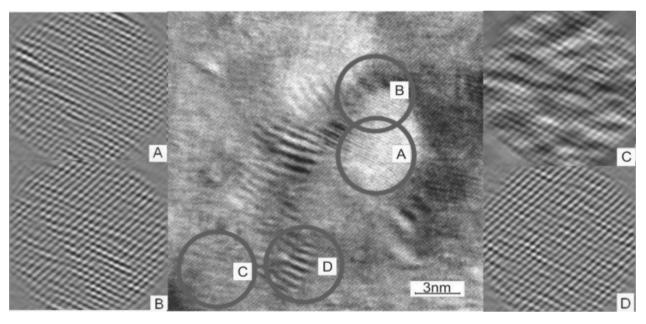


Fig. 19. The structure of CuNi25 (A) 50 after deformation at 550°C (FFT, HRTEM)

In the range of the Ductility Minimum Temperature phenomenon, it was also noticed that the low stacking fault energy deformation twins have often heterogeneous dislocation structure and also visible arched, curved twin grain boundaries. This may be observed in the photographs of microstructures obtained from thin foils of samples deformed in the range of 475-550°C (Figs. 15-18). The vicinity of borders of twins formed during plastic deformation is often characterized by various density of linear defects in the crystalline structure. Within the grains of α CuNi there are deformation twins visible with a distorted structure and uneven distribution of dislocations (Figs. 15, 18). In the thin film obtained from the samples deformed in the DMT there are recrystallized areas (Fig. 17). Also, the areas of low defects level - adjacent to the high density of defects of crystalline structure may be noticed (Figs. 16 and 17).

As a result of using High Resolution Transmission Electron Microscopy and the Fast Fourier Transformation for example in the structure of CuNi25 (A) 50 after deformation at 550°C, the heterogeneous degree of crystalline lattice deformation was revealed (Fig. 19). In the analysed area, there are also clear crystalline lattice areas and different density dislocations regions, different degree of deformation is observed, depending on the observed area.

In all of the analysed samples of the alloy CuNi25 A and B series can be determined the presence of oxide inclusions, which were identified as Cu₂O, NiO, and SiO₂ (Figs. 20-23). In both CuNi25 A and B it was revealed that there is a presence of areas of varying density of inclusions, and the density of their occurrence, depending on the area, is different and it is difficult to find a regularity in their location. Sporadic inclusions occurrence and their concentrations on the grain boundaries as well as in grain interior were observed.

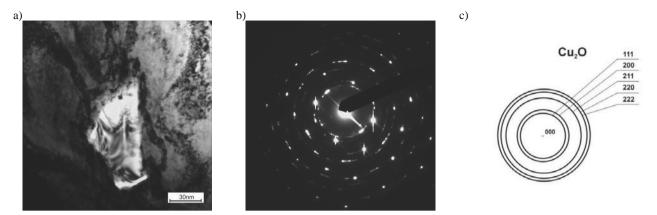


Fig. 20. a) The structure of CuNi25 (B) after deformation at 550°C (TEM), b) diffraction pattern from Fig. 20a, c) solution of diffraction pattern from Fig. 20b

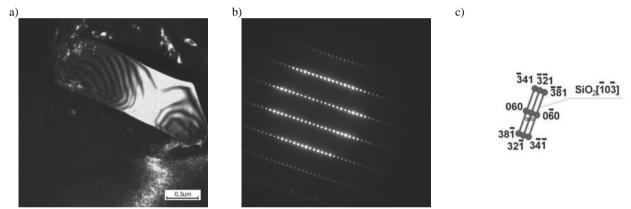


Fig. 21. a) The structure of CuNi25 B after deformation at 550°C, visible SiO₂, inclusions, dark field micrograph (TEM); b) diffraction pattern from crystalline phase of SiO₂ from Fig. 21a; c) solution of diffraction pattern from Fig. 21b

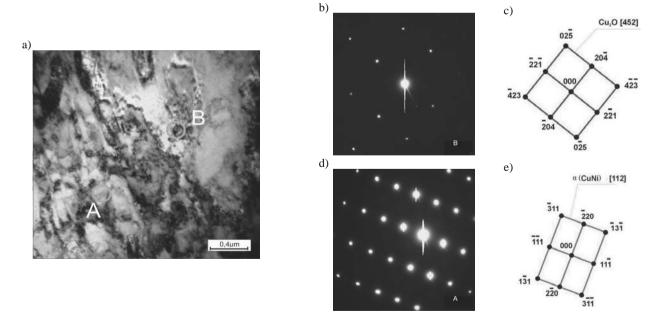


Fig. 22. a) The structure of CuNi25 (A) 150 after deformation at 550°C, b) diffraction pattern from the B point Cu₂O phase from Fig. 22a, c) diffraction pattern from Fig. 22b, d) diffraction pattern from the A point, CuNi matrix, e) diffraction pattern from Fig. 22d

The occurrence of inclusions cause escalation of the structural heterogeneity of the material and influences the increase of the defects density of crystal structure in their neighbourhood. In the border area around the Cu_2O oxide inclusions as well as in the area of solid grain, for example the endogenous inclusions

of SiO_2 cause concentration of dislocations and probably cause formation of cracks during deformation (Figs. 20 and 21). We can also find areas with different dislocation densities and the radial spreading of group of dislocations generated by the inclusions.

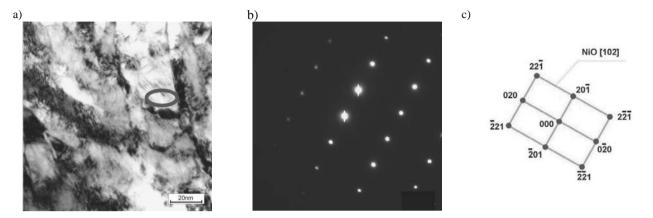


Fig. 23. a) The structure of CuNi25 (B) after deformation at 550°C (TEM), b) diffraction pattern from Fig. 23a, c) solution of diffraction pattern from Fig. 23b

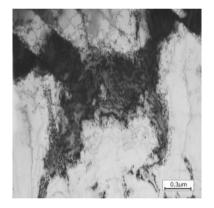


Fig. 24. The structure of CuNi25 (A) 50 after deformation at 550°C (TEM)



Fig. 25. The structure of CuNi25 (A) 150 after deformation at 550°C (TEM)

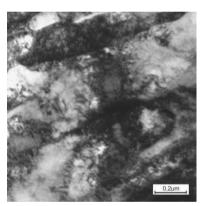
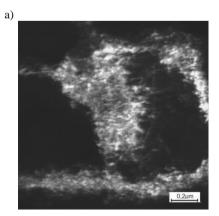
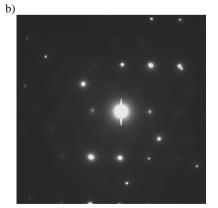


Fig. 26. The structure of CuNi25 (A) 50 after deformation at 550°C (TEM)

c)





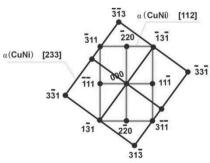


Fig. 27. a) The structure of CuNi25 (A) 50 after deformation at 550°C, b) dark field micrograph (TEM), Diffraction pattern from Fig. 27a, c) solution of diffraction pattern from Fig. 27b

In the samples deformed in the range of 450-550°C also the deformation twins are present and slight deflection of high-angle grain boundaries are visible. In this temperature range the following has been noticed: at the junction point of the three and more grains with twin boundaries there is a presence of very high density of defects in the crystalline structure, often locally differentiated (Figs. 24 and 25). There was also a concentration of dislocations located in the border areas, side by side there are areas with a disorder in crystalline building arrangement (Figs. 26, 27).

In the thin films obtained from the samples deformed above DMT, at 600°C for CuNi25 (A) 150 and 650°C for CuNi25 (B) respectively, there is an increasing number of recrystallized areas. We can notice that areas of low density of dislocations are contiguous to the areas with high density of crystalline structure defects. In some cases, mainly near low-angle grain boundaries, similar areas are formed, they may be new small grains. Small spherical grains next to the larger grains were also observed (Figs. 28, 29).

The TEM observations of samples deformed above 600°C reveal a larger participation of recrystallized structure. It was also noted that the twin grain boundaries tend to bend. In the areas adjacent to TJ of grains accumulation of crystalline structure defects was noticed. The existence of different sizes of dislocations cells with different density of dislocations (Figs. 30, 31) was also observed. It was also observed around the twins low and high defected areas, with high density of dislocations located alternately

to undefected zones, areas with different densities of defects are located within the border zones on both sides of the twin boundaries (Figs. 32, 33).

TEM and HRTEM observations on thin films made of all series of samples deformed in the range of RDA show similar regularity. Places having a tendency to accumulation of point and linear defects are inclusions and especially privileged areas are grain boundaries, particularly the TJ. These sites generate or accumulate dislocations leading to increase of local stress. One of the probable mechanisms contributing to the local stress relaxation in RDA is twinning, it may be indicated by the presence of microtwins. A similar mechanism was noted in the work of Bruckner in another species of cupronickel alloy [18, 19]. With increasing temperature above DMT in samples after deformation the processes of rebuilding the microstructure, probably dynamic recovery and dynamic recrystallization are observed. This was revealed by the presence of grain boundary serrations and cellular dislocation structure of subgrains and local existence of undefected areas, as well as small new grains. Traces of dynamic recrystallization in the alloy samples CuNi25 are relatively less visible than in brasses and copper due to the higher value of stacking fault energy and the experiment procedure. Samples for the preservation of its structure, immediately after the deformation, were cooled in water, which probably caused the freezing of recrystallization nucleation.

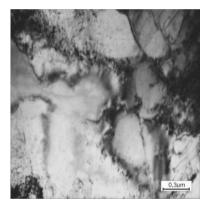


Fig. 28. The structure of CuNi25 (A) 150 after deformation at 600°C (TEM)

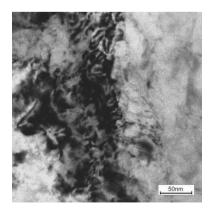


Fig. 31. The structure of CuNi25 (A) 50 after deformation at 600°C (TEM)

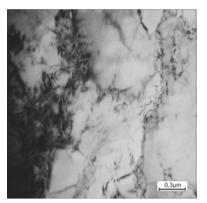


Fig. 29. The structure of CuNi25 (B) after deformation at 650°C (TEM)

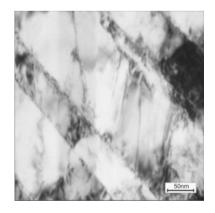


Fig. 32. The structure of CuNi25 (A) 50 after deformation at 750°C (TEM)

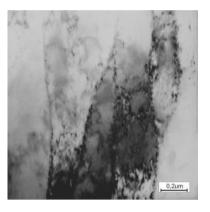


Fig. 30. The structure of CuNi25 (B) after deformation at 650°C (TEM)

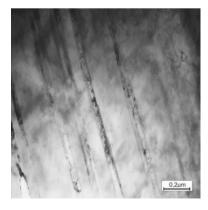


Fig. 33. The structure of CuNi25 (A) 150 after deformation at 650°C (TEM)

4. Summary

On the basis of high temperature ductility tests in CuNi25 alloy and TEM observation a relation between microstructure, and DMT phenomenon has been found. The studies of CuNi25 alloy samples show a clear relationship with the deformation curves. Significant differences in the course of structural changes are noticeable in the analysis of the same series of samples deformed at different temperatures. While making a qualitative assessment, it can be concluded that the structure is associated with the deformation temperature. The greatest number of microstructural heterogeneity is observed in samples deformed at a temperature corresponding to the DMT existence.

The non-homogeneous character of microstructure composition concentrating in areas of grain boundaries and cracks at high temperature has been investigated. This analysis shows that local areas of non-equilibrium formation concentrate at this places. The deformation in temperature approximating to beginning of thermal activated processes provokes superimposing of many "inhomogeneities" causing local changes of physical, mechanical and chemical material proprieties. We can assume that in microscale we have to deal with materials locally with different properties. The process of deformation is located in the space privileged to easier deformation in this conditions. The critical level of nonhomogeneity is causing nucleation and growth of cracks. The critical level of stress concentration in the whole sample volume causes decrease of ductility and destruction of material.

Based upon literature studies, observation and analysis of plastic deformation processes and their influence on range and level of DMT phenomenon, in CuNi25 alloy can be accepted, as in brass, hypothesis of non-uniform deformation [24]. Confirmation of this hypothesis can be model of "soft" and "hard" places. The location of deformation process in small volume of heterogeneous material causes the formation of stress between "soft" and "hard" areas. The critical level of non-homogeneity is causing concentration of stress in whole volume of material causing nucleation and growth of cracks. Resulting in further influence of strength, to reduced surface cause growth of stress provoking lowered ductility and destruction of material. The differences of nickel and copper's local concentration, material geometrical structures, size and shape of grains in this case are responsible for dimension of DMT effect. "Inhomogeneities" leading to perturbation in physical and chemical equilibrium provoke lower ductility of material. The "soft" and "hard" places model, based on difficult to measure and define concept of heterogeneous deformation reflects in macro scale the process of plastic deformation in the range of the DMT effect [1]. Therefore, quantity description of this phenomenon in structural scope is very difficult and explanation of DMT has a character of the hypothesis.

References

 R. Nowosielski, Ductility minimum temperature in selected mono-phase, binary brasses, Journal of Materials Processing Technology 109 (2001) 142-153.

- [2] M. Vedani, D. Ripamonti, A. Mannucci, D. Dellasega, Hot Ductility of Microalloyed Steels, La Metallurgia Italiana (2008) 19-24.
- [3] J. Kömi, Hot ductility of austenitic and duplex stainless steels under hot rolling conditions, Department of Mechanical Engineering University of Oulu, Oulu, 2001.
- [4] A. Lis, J. Lis, C. Kolan, M. Knapiński, Effect of strain rate on hot ductility of C-Mn-B steel, Journal of Achievements in Materials and Manufacturing Engineering 41 (2010) 26-33.
- [5] S.A. Gavin, J. Billingham, J.P. Chubb, P. Hancock, Effect of trace impurities on hot ductility of as-cast cupronickel alloys, Metals Technology 5/11 (1978) 397-401.
- [6] W. Ozgowicz, The relationship between hot ductility and intergranular fracture in a CuSn6P alloy at elevated temperatures, Journal of Materials Processing Technology 162-163 (2005) 392-401.
- [7] R. Nowosielski, P. Sakiewicz, J. Mazurkiewicz, Ductility Minimum Temperature phenomenon in as cast CuNi25 alloy, Journal of Achievements in Materials and Manufacturing Engineering 17 (2006) 193-196.
- [8] R. Nowosielski, P. Sakiewicz, P. Gramatyka, The effect of ductility minimum temperature in CuNi25 alloy, Journal of Materials Processing Technology 162-163 (2005) 379-384.
- [9] J. Dobrzański, A. Zieliński, M. Sroka, Microstructure, properties investigations and methodology of the state evaluation of T23 (2.25Cr-0.3Mo-1.6W-V-Nb) steel in boilers application, Journal of Achievements in Materials and Manufacturing Engineering 32/2 (2009) 142-153.
- [10] R.A. Varin, K.J. Kurzydłowski, K. Tangri, The effects of nitrogen content and twin boundaries on the yield strength of various commercial heats of type 316 austenitic stainless steel, Materials Science and Engineering 101 (1988) 221-226.
- [11] M.W. Grabski, Mechanical properties of Internal Interfacest, Journal de Physique I 46 (1985) 567.
- [12] K. Konopka, J.W. Wyrzykowski, The effect of the twin boundaries on the yield stress of a material, Journal of Materials Processing Technology 64/1-3 (1997) 223-230.
- [13] C. Devadas, I.V. Samarasekera, E B. Hawbolt, The thermal and metallurgical state of steel strip during hot rolling, Microstructural Evolution 22 (1991) 335-349.
- [14] T. Watanabe, S. Tsurekawa, Toughening of brittle materials by grain boundary engineering, Materials Science and Engineering A 387-389 (2004) 447-455.
- [15] A. Zeren, M. Zeren, Stress relaxation properties of prestressed steel wires, Journal of Materials Processing Technology 141 (2003) 86-92.
- [16] L. Xaio, J.L. Bai, Stress relaxation properties and microscopic deformation structure of H68 and QSn6.5-0.1 copper alloys at 353 K, Materials Science and Engineering A 244 (1998) 250-256.
- [17] P. Virtanen, T. Tiainen, Stress relaxation behavior in bending of high strength copper alloys in the Cu–Ni–Sn system, Materials Science and Engineering A 238/2 (1997) 407-410.
- [18] W. Bruckner, S. Baunack, Stress and oxidation in CuNi thin films, Thin Solid Films 355-356 (1999) 316-321.
- [19] W. Bruckner, V. Weihnacht, Stress relaxation in CuNi thin films, Journal of Applied Physics 85 (1999) 3602-3608.

- [20] W. Ozgowicz, Physico-chemical, structural and mechanical factors of intergranular brittleness of alpha-bronzes at elevated temperature, Silesian University of Technology Publishing House, Gliwice, 2004 (in Polish).
- [21] B. Druyanov, I. Roman, A continuum model for grain junctions in polycrystalline aggregate, Mechanism of Materials 30 (1998) 31-40.
- [22] G. Palumbo, D.M. Doyle, A.M. El-Sherik, U. Erb, K.T. Aust, Intercrystalline hydrogen transport in nanocrystalline nickel, Scripta Metallurgica et Materialia 25/3 (1991) 679-684.
- [23] W. Ozgowicz, B. Grzegorczyk, The influence of the temperature of plastic deformation on the structure and mechanical properties of copper alloys CuCo2Be and

CuCo1Ni1Be, Archives of Materials Science and Engineering 39/1 (2009) 5-12.

- [24] R. Nowosielski, Explication of minimum plasticity effect of mono-phase Brassens, Silesian University of Technology Publishing House, Gliwice, 2000 (in Polish).
- [25] V. Laporte, A. Mortensen, Intermediate temperature embrittlement of copper alloys, International Materials Reviews 54/2 (2009) 94-116.
- [26] W. Ozgowicz, E. Kalinowska-Ozgowicz, B. Grzegorczyk, The influence of the temperature of tensile test on the structure and plastic properties of copper alloy type CuCr1Zr, Journal of Achievements in Materials and Manufacturing Engineering 29/2 (2008) 123-136.