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The Influence of Heat Treatment on Microstructure and Phase Transformation Temperatures of Cu-Al-Ni Shape Memory Alloy

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Abstract

This paper presents the results of thermal and microstructural analysis of Cu-Al-Ni shape memory alloy before and after heat treatment. After casting, a bar of Cu-12.8 Al-4.1 Ni (wt.%) alloy, obtained by the vertical continuous casting technique, was subjected to a certain heat treatment procedure. Solution annealing was performed at 850 °C for 60 min, followed by water quenching. Tempering was then performed at four different temperatures (150 °C, 200 °C, 250 °C and 300 °C). The microstructural results were obtained by optical and scanning electron microscopy. Thermodynamic calculation of ternary Cu-Al-Ni system under equilibrium was performed using Thermo-Calc 5 software. Phase transformation temperatures were determined by differential scanning calorimetry (DSC). The DSC results show the highest values of transformation temperatures in as-cast state. After solution annealing and tempering, the transformation temperatures show lower values with exceptional stability of M_s temperature (martensite start temperature).

Keywords

Shape memory alloy, Cu-Al-Ni, heat treatment, phase transformation, microstructure

1 Introduction

Today there are a large number of known shape memory alloys (SMAs); nickel-based shape memory alloys, copper-based shape memory alloys, ferrous-based shape memory alloys, noble metal-based shape memory alloys, *etc.* The term shape memory alloys is applied to a group of metallic materials, which show the ability to return to their previously defined shape or size during a special heat treatment procedure. Shape change is a consequence of austenitic to martensitic transformation, which is characterised by the following temperatures: A_s – austenite start temperatures, A_f – austenite finish temperatures, M_s – martensite start temperature and M_f – martensite finish temperature.[1–7]

Compared to Ni-Ti SMAs that are generally considered to be superior to Cu-based alloys, Cu-Al-Ni alloys also offer some considerable advantages. Not only is the material cost 15–30 % of that for Ni-Ti, but the melting, composition control, and casting are less difficult, they exhibit higher Young's modulus, better machinability, and better work/cost ratio. In addition, the stability of the two-way shape memory effect is better, which is very important when designing the actuators.[8]

Phase diagrams are very important for designing and development of a material due to its functional properties, microstructure and phase stability under specific conditions. Thermodynamic modelling offers valuable information for equilibrium thermodynamics, modelling of diffusion processes and grain growth. It represents the advantage according to expensive and time-consuming experimental investigations.[9] Reliable thermodynamic databases, with optimised parameters, are crucial for thermodynamic calculations of Gibbs energy of all phases existing in an investigated system and accuracy of calculated phase equilibria. Thermodynamic descriptions of binary systems Cu-Al, Al-Mn, and Cu-Mn are given in a number of references, but there is a lack of relevant experimental and optimised thermodynamic data for ternary Cu-Al-Ni alloy.[9,10,11] Considering the wide application of Cu-Al-Ni alloys, it seems very interesting to analyse the thermodynamic properties of this ternary system.

This paper studies the influence of heat treatment procedure on microstructure and phase transformation temperatures of the alloy. The results obtained after heat treatment are compared with the results obtained on the sample in as-cast state.

2 Experimental

Thermodynamic calculation for equilibrate conditions was performed with Thermo-Calc 5 software, using database SSOL 6. Calculations of Gibbs energy were performed according to binary sub-systems Cu-Al, Cu-Ni and Al-Ni.[10–13]

Cu-12.8 Al-4.1 Ni (wt.%) shape memory alloy was produced by vertical continuous casting procedure in a vacuum induction furnace connected to the device for vertical continuous casting. The alloy, in the shape of a bar of

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Table 1 – Samples with heat treatment conditions
Tablica 1 – Popis uzoraka s uvjetima toplinske obrade

Samples Uzorci	Heat treatment parameters Parametri toplinske obrade
L	As-cast state of Cu-Al-Ni SMA Lijevano stanje Cu-Al-Ni slitine s prisjetljivošću oblika
K-1	Solution annealed at 850 °C/60′/WQ Kaljenje na 850 °C/60′/voda
K-1-1	Solution annealed at 850 °C/60′/WQ and tempered at 150 °C/60′/WQ Kaljenje na 850 °C/60′/voda i popuštanje na 150 °C/60′/voda
K-1-2	Solution annealed at 850 °C/60′/WQ and tempered at 200 °C/60′/WQ Kaljenje na 850 °C/60′/voda i popuštanje na 200 °C/60′/voda
K-1-3	Solution annealed at 850 °C/60′/WQ and tempered at 250 °C/60′/WQ Kaljenje na 850 °C/60′/voda i popuštanje na 250 °C/60′/voda
K-1-4	Solution annealed at 850 °C/60′/WQ and tempered at 300 °C/60′/WQ Kaljenje na 850 °C/60′/voda i popuštanje na 300 °C/60′/voda

WQ – water quenching

8 mm in diameter, solidified in a crystalliser and came out passing between two rolls rotating in opposite directions. The alloys' casting temperature was 1240 °C and casting speed was 320 mm min^{-1}. After casting, the heat treatment procedure was performed as shown in Table 1.

For microstructural observation, the samples were metallographically prepared by grinding, polishing, and etching. The detailed metallographic preparation of the samples is explained elsewhere.[14] The samples were investigated with optical microscope (OM) and scanning electron microscope (SEM). To determine phase transformation temperatures, differential scanning calorimetry (DSC) was performed. The samples were heated and cooled in one cycle from room temperature to 400 °C by heating/cooling speed of 10 K min^{-1}.

Results and discussion

3.1 Results of thermodynamic calculation of Cu-Al-Ni ternary phase diagram

Thermodynamic calculation was performed by CALPHAD method, based on minimisation of the free Gibbs energy of system.[10,15] The Figs. 1 and 2 show the vertical section of calculated phase diagram for Cu-4.1Ni-Al.

Figs. 1 and 2 show precipitation of austenite, parent β-phase in B2 crystal structure at 1237 °C, under equilibrate conditions. Solidus temperature was observed at 1031 °C, and under this temperature, the β-phase exists as two crystal structures, B2 and A2. The γ-phase starts to precipitate at 611 °C. At 567 °C, the β-phase in A2 crystal structure decomposes to α-phase, and at room tempera-

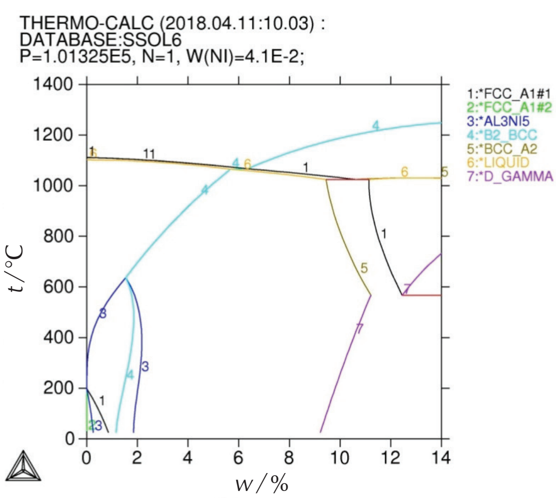

Fig. 1 – Vertical section of calculated phase diagram for Cu-4.1Ni-Al
Slika 1 – Vertikalni presjek izračunatog faznog dijagrama za Cu-4.1Ni-Al

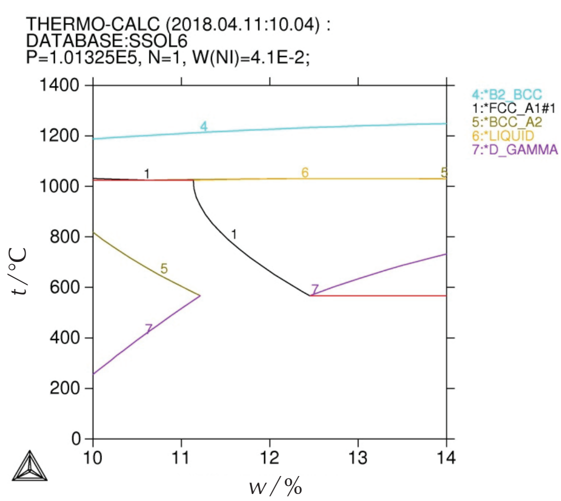

Fig. 2 – Vertical section of phase diagram for Cu-4.1Ni-Al (narrowed section)
Slika 2 – Vertikalni presjek faznog dijagrama za Cu-4.1Ni-Al (suženi dio)

ture, the coexistence of α-, β-(B2) and γ-phase is obvious. Decomposition of parent β-phase can be suppressed by fast cooling or quenching in water, which causes formation of martensitic structure.

During heat treatment, microstructural changes occur, affecting the phase transformation temperatures. Such behaviour of Cu-Al-Ni alloys, obtained by melt-spinning process, was observed by *Morawiec et al.*[16] The change in phase transformation temperatures can also be attributed to the effect of internal strains caused by different grain size in the microstructure, as confirmed by *Pelegrina and Romero* on Cu-Al-Zn SMA.[17]

3.2 Optical, scanning electron microscopy, and DSC results

Cu-Al-Ni shape memory alloys from the initial austenitic β phase, if casting conditions (*e.g.* sufficient cooling speed) were satisfied, form a martensitic microstructure, which is responsible for the shape memory effect. However, during the casting, due to unfavourable cooling conditions, equilibrium low temperature phases can be created according to the equilibrium phase diagram of Cu-Al-Ni alloy.[6,7]

In order to avoid the possible forming of residual low temperature phases during casting, a heat treatment process in the austenitic β phase area was performed. According to the literature[18], copper-based SMAs are alloys in which heat treatment process cannot be avoided.

In the microstructure of the investigated Cu-Al-Ni alloy, the existence of a completely martensitic microstructure (Figs. 3 and 4) was found. It can be noticed that the microstructure consists of self-accommodating needle-like shaped martensite. In addition, the orientation of the crystal was different.

The Cu-Al-Ni alloy microstructure can change depending on the heat treatment procedure. Optical micrographs of solution annealing and tempering are presented in Figs. 3b–3d. The grain boundaries are clearly visible, and the microstructure after heat treatment was completely martensitic. Martensite needles had different orientation within each grain, which could be explained by the nucleation of groups of martensitic plates in numerous places within the grain, and creation of local strain within the grain, which allowed the formation of several groups of differently oriented plates.

Changes in microstructure influence changes in the alloy's properties, which occur due to thermal processing. Solution annealing of the Cu-Al-Ni shape memory alloy must be performed in order to achieve a fully martensitic microstructure. Along with the martensitic phase which is formed from the initial austenitic (β) phase, the change in grain size varies depending on the process conditions of heat treatment (heat treatment temperature, holding time at the selected temperature, or cooling agents).[19]

The martensitic microstructure was confirmed on all samples by scanning electron microscopy (Fig. 4). The resulting microstructure appeared by transformation of β phase to martensite below M_s temperature. Martensite originated primarily as a needle-like martensite. On some of the samples, after solution annealing and tempering (Figs. 4b–4d), the V-shape of the martensite can be noticed. The morphology of the resulting martensitic microstructure is a typical self-accommodating zig-zag morphology, which is

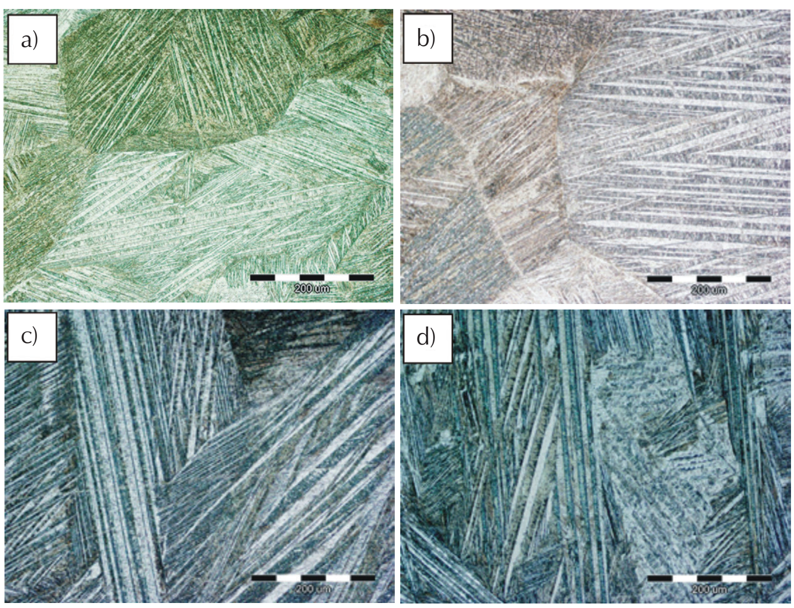

Fig. 3 – Optical micrographs of Cu-Al-Ni shape memory alloy in as-cast state (a), solution annealed at 850 °C/60′/WQ (b), tempered at 150 °C/60′/WQ (c), and 300 °C/60′/WQ (d)

Slika 3 – Optičke mikrografije Cu-Al-Ni slitine s prisjetljivošću u lijevanom stanju (a), kaljenom na 850 °C/60′/voda (b), popuštenom na 150 °C/60′/H_2O (c) i 300 °C/60′/H_2O (d)

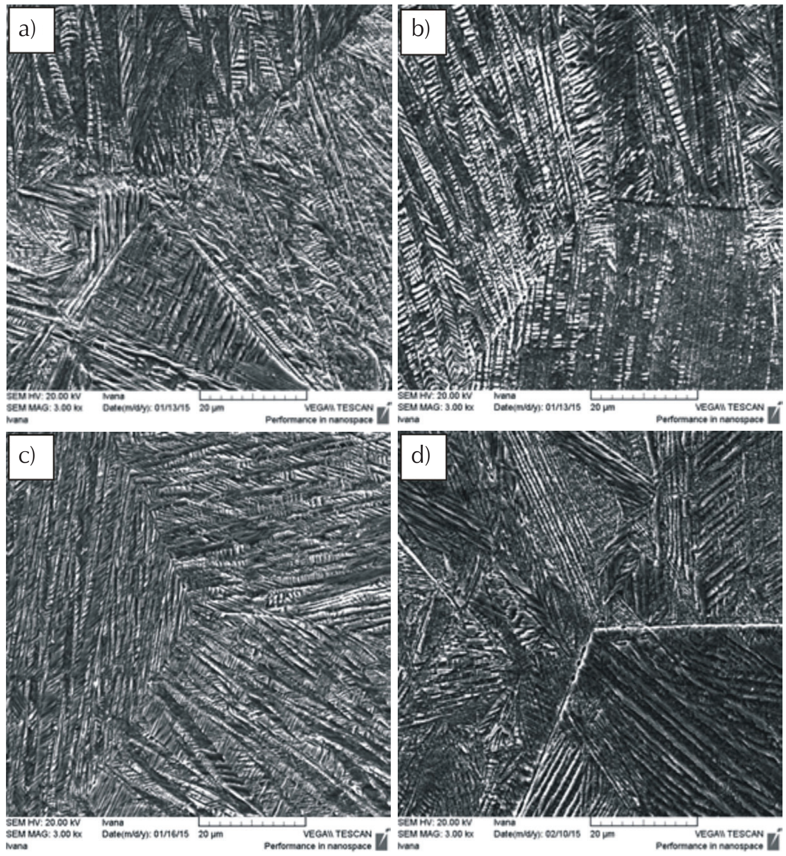

Fig. 4 – SEM micrographs of Cu-Al-Ni shape memory alloy in as-cast state (a), solution annealed at 850 °C/60′/WQ (b), tempered at 150 °C/60′/WQ (c), and 300 °C/60′/WQ (d)

Slika 4 – SEM mikrografije Cu-Al-Ni slitine s prisjetljivošću u lijevanom stanju (a), kaljenom na 850 °C/60′/voda (b), popuštenom na 150 °C/60′/H_2O (c) i 300 °C/60′/H_2O (d)

primarily characteristic for β_1' martensite in Cu-Al-Ni shape memory alloys.[20,21]

The changes in microstructure can be seen in samples after tempering. OM and SEM micrographs (Figs. 3c, 3d, 4c, and 4d) reveal a different type of martensite plate characteristic for γ_1' martensite. According to the literature,[6,22] the fine needles and V-shaped laths are typical morphologies of β'_1 and γ'_1 martensites, respectively. Alloys with aluminium content of 11–13 wt. % in the microstructure, transform into 18R (β_1') martensite from the β parent phase. Higher aluminium content (>13 wt. %) follows the formation of 2H (γ_1') martensite. If the chemical composition is on the boundary between both martensites, then both can coexist in the microstructure. Which of them will appear in the microstructure depends on the chemical composition, temperature conditions, and stress conditions.[18,21,23,24]

The appearance of martensite in the microstructure can be described by the transformation mechanism of austenitic β phase through the transformation $\beta \rightarrow \beta_1'$. Only after heat treatment, another type of martensite in the microstructure appeared. It can be assumed that γ_1' martensite appears in the microstructure. The appearance of γ_1' martensite can be described by the following transformation mechanism from $\beta \rightarrow \gamma_1'$. Also, not only is one type of martensite present in the microstructure, but both types of martensite, which can be described by the transformation $\beta \rightarrow \beta_1' + \gamma_1'$.

It was observed that changes in temperature of phase transformations occur due to changes in temperature of heat treatment process (Table 2 and Figs. 5–7). Figs. 5 and 6 present DSC curves for alloy in as-cast state and solution annealed, respectively. The phase transformation temperatures (M_s, M_f, A_s, and A_f) were determined by tangent method and marked in Figs. 5 and 6.

Fig. 7 shows the effect of heat treatment temperature on the phase transformation temperatures before and after heat treatment. It can be noticed that the temperature of the phase transformation is the highest for as-cast sample. The reason for this may be a large amount of internal strain and imperfection in the microstructure, as a result of casting and solidification.

Since the heat treatment procedure (solution annealing and tempering) was carried out in order to achieve order in the alloy's structure and stabilisation of the phase transformation temperatures, the characteristic behaviour

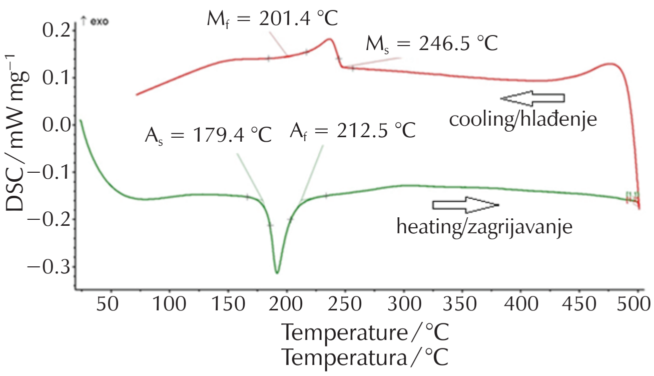

Fig. 5 – DSC curves of Cu-Al-Ni shape memory alloy after casting
Slika 5 – DSC krivulja Cu-Al-Ni slitine s prisjetljivošću oblika nakon lijevanja

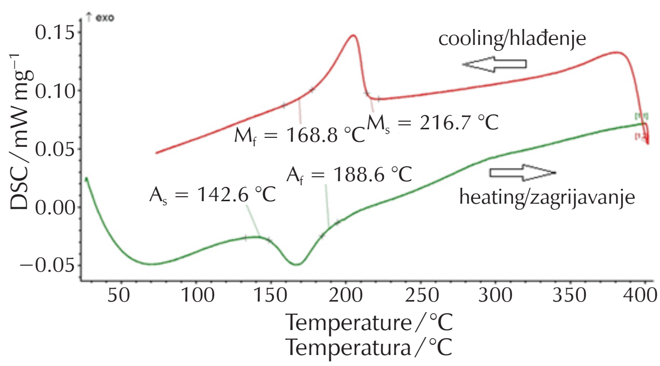

Fig. 6 – DSC curves of Cu-Al-Ni shape memory alloy after solution annealing at 850 °C/60′/WQ
Slika 6 – DSC krivulje Cu-Al-Ni slitine s prisjetljivošću oblika nakon kaljenja na 850 °C/60′/voda

Table 2 – Temperatures of austenitic and martensitic transformation in Cu-Al-Ni shape memory alloy, °C
Tablica 2 – Temperature austenitne i martenzitne transformacije Cu-Al-Ni slitine s prisjetljivošću oblika, °C

Samples Uzorci	A_s	A_f	M_s	M_f
	(°C)			
L (as-cast state) L (lijevano stanje)	179.4	212.5	246.5	201.4
K-1 (solution annealed at 850 °C/60′/WQ) K-1 (kaljeno na 850 °C/60′/voda)	142.6	188.6	216.7	168.8
K-1-1 (tempered at 150 °C/60′/WQ) K-1-1 (popušteno na 150 °C/60′/voda)	150.4	180.5	214.0	176.4
K-1-2 (tempered at 200 °C/60′/WQ) K-1-2 (popušteno na 200 °C/60′/voda)	152.5	182.0	213.7	181.4
K-1-3 (tempered at 250 °C/60′/WQ) K-1-3 (popušteno na 250 °C/60′/voda)	163.4	192.0	216.8	181.4
K-1-4 (tempered at 300 °C/60′/WQ) K-1-4 (popušteno na 300 °C/60′/voda)	180.3	205.5	213.1	183.3

of M_s temperature was observed after solution annealing and tempering (Fig. 7, Table 2). The graph shows the M_s temperature matching in all cases after heat treatment. The values of M_s temperature only slightly differ, ranging from 213.1 °C to 216.8 °C.

4 Conclusion

The influence of heat treatment on microstructure and phase transformation temperatures was investigated. The results of thermal and microstructural analysis of Cu-Al-Ni shape memory alloy before and after heat treatment suggest the following conclusions:

– Microstructural analysis in this study confirmed the complete transformation of β phase into martensite phase in the as-cast and heat treated samples at all investigated temperatures. A detailed analysis of the results, suggests that, in the as-cast state, the only micro-constituent is a β_1' martensite, which arises from the mechanism of austenitic β phase through the transformation $\beta \rightarrow \beta_1'$. After heat treatment, another type of martensite in the microstructure appears (γ_1' martensite), which can be described by the following transformation mechanism from $\beta \rightarrow \gamma_1'$. The presence of both types of martensite in the microstructure can be described by the transformation $\beta \rightarrow \beta_1' + \gamma_1'$.
– Thermodynamic calculation shows firstly precipitation of parent β-phase under equilibrate conditions. At solidus temperature (1031 °C), β-phase exists as two crystal structures. Also, the precipitation of γ-phase starts at 611 °C. Decomposition of β-phase to α-phase is at 567 °C. The α-, β-(B2) and γ-phases were obvious at room temperature.
– The DSC analysis show the highest values of phase transformation temperatures for the as-cast sample. After heat treatment, the characteristic behaviour of M_s temperature was observed. The stabilisation of M_s temperature was accomplished by heat treatment, and varied only slightly in the range from 213.1 °C to 216.8 °C.

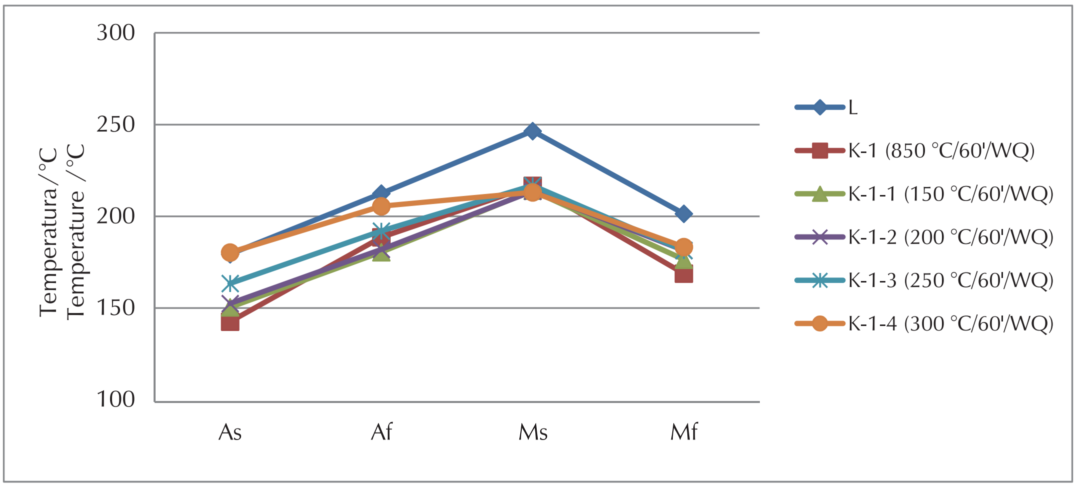

Fig. 7 – Influence of heat treatment procedure on phase transformation temperatures of the CuAlNi shape memory alloy
Slika 7 – Utjecaj toplinske obrade na temperature fazne transformacije CuAlNi slitine s prisjetljivošću oblika

ACKNOWLEDGEMENTS

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Popis kratica o simbola

A_s – austenite start temperature, °C
– temperatura početka austenitne transformacije, °C

A_f – austenite finish temperature, °C
– temperatura završetka austenitne transformacije, °C

A2 – form of crystal structure
– oblik kristalne strukture

bcc – body-centred cubic crystal structure
– volumno-centrirana kubična kristalna rešetka

B2 – form of crystal structure
– oblik kristalne strukture

DSC – differential scanning calorimetry
– diferencijalna pretražna kalorimetrija

fcc – face-centred cubic crystal structure
– plošno-centrirana kubična kristalna rešetka, PCK

M_f – martensite finish temperature, °C
– temperatura završetka martenzitne transformacije, °C

M_s – martensite start temperature, °C
– temperatura početka martenzitne transformacije, °C

OM – optical microscopy
– optička mikroskopija

SEM – scanning electron microscopy
– pretražna elektronska mikroskopija

SMAs – shape memory alloys
– slitine s prisjetljivošću oblika

2H – form of crystal structure
– oblik kristalne strukture

18R – form of crystal structure
– oblik kristalne strukture

α – phase, primary solid solution of Al and Ni in copper, fcc crystal structure
– faza, primarna čvrsta otopina Al i Ni u bakru, PCK strukture

β – austenite phase
– austenitna faza

β_1' – martensite phase
– martenzitna faza

γ – equilibrium phase
– ravnotežna faza

γ_1' – martensite phase
– martenzitna faza

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SAŽETAK

Utjecaj toplinske obrade na mikrostrukturu i temperature faznih transformacija Cu-Al-Ni slitine s prisjetljivošću oblika

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U radu su prikazani rezultati toplinske i mikrostrukturne analize Cu-Al-Ni slitine s prisjetljivosti oblika prije i nakon toplinske obrade. Nakon lijevanja, šipka slitine Cu-12.8 Al-4.1 Ni (mas. %) dobivena tehnikom vertikalnog kontinuiranog lijevanja, podvrgnuta je određenom postupku toplinske obrade. Provedeno je homogenizacijsko žarenje pri 850 °C, zadržavanje na toj temperaturi 60 min i hlađenje u vodi. Nakon toga je provedeno popuštanje na četiri različite temperature (150 °C, 200 °C, 250 °C i 300 °C). Mikrostrukturna analiza provedena je optičkim i pretražnim elektronskim mikroskopom. Termodinamički proračun ravnotežnog ternarnog Cu-Al-Ni sustava proveden je pomoću Thermo-Calc 5 softvera. Temperature fazne transformacije određene su diferencijalnom pretražnom kalorimetrijom (DSC). Rezultati DSC-a pokazuju najveće vrijednosti temperatura fazne transformacije u lijevanom stanju. Nakon homogenizacijskog žarenja i popuštanja temperature fazne transformacije pokazuju niže vrijednosti s iznimnom stabilnošću M_s temperature (temperatura početka nastajanja martenzita).

Ključne riječi
Legura s prisjetljivošću oblika, Cu-Al-Ni, toplinska obrada, fazna transformacija, mikrostruktura

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