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# EVALUATION OF THE MICROSTRUCTURE AND PHASE TRANSITION TEMPERATURES OF THE Cu-9%Al-8%Mn SHAPE MEMORY ALLOY

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**Keywords:** Shape memory alloy, Cu-Al-Mn alloy, Microstructure, Martensitic transformation

## ABSTRACT
*Shape memory alloys (SMAs) belong to the group of advanced functional materials for numerous technical applications. Among many known Cu-based shape memory alloys, Cu–Al–Mn ternary alloys are characterized by a combination of good shape memory properties and excellent ductility which makes them commercially attractive.*
*In this work the Cu-9%Al-8%Mn alloy was prepared by induction melting of pure metals. Microstructure of the alloy was investigated in the as-prepared state, after homogenization annealing at 850 °C and slow cooling, and after quenching from 850 °C using SEM-EDS technique. Transformation temperatures of the quenched alloy were investigated using DSC technique.*

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## 1. INTRODUCTION

Cu-based shape memory alloys (SMAs) show good shape memory properties, high electrical and thermal conductivity, are easier to produce and process and have lower production cost comparing to Ni-Ti-based SMAs [1, 2, 3].
The shape memory effect is based on martensitic transformation (MT) which represents a diffusion less and reversible solid state phase transformation which occurs between the high-temperature austenite phase and the low-temperature martensite phase [2, 3, 4, 5, 6].
During cooling, the martensitic transformation (MT) starts at a temperature Ms (martensite start) and continues to evolve until a temperature $M_f$ (martensite finish) is reached. Similarly, during the heating cycle, the reverse transformation (martensite-to-austenite) begins at the temperature $A_s$ (austenite start), and ends at $A_f$ (austenite finish) when the material is fully austenite [2, 3].
In this work the Cu-9%Al-8%Mn alloy was prepared by induction melting of pure metals. Microstructure of the alloy was investigated using scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) in the as-prepared state, after homogenization annealing followed by slow cooling and after quenching. Differential scanning calorimetry (DSC) was used for determination of martensitic transformation temperatures.

## 2. EXPERIMENTAL PROCEDURE

The investigated alloy with designed composition Cu-9%Al-8%Mn was prepared by induction melting of calculated quantities of pure copper (99.99%), aluminium (99.97%) and manganese (99.95%) in the graphite crucible under a charcoal cover. The cylindrical shaped ingot (20 mm diameter and 40 mm length) was produced. The sample was mechanically grinded and polished. Etching of sample after different heat treatments was done using a solution containing 2.5 g $FeCl_3$x $6H_2O$ and 1 ml HCl in 48 ml methanol. After microstructure analysis in the as-prepared condition, sample was annealed at 850 °C for five hours, slowly cooled inside the furnace and again subjected to the microstructure analysis. Finally, sample was annealed at 850 °C for 1 hour, quenched in the ice water and analyzed.
Microstructure investigation of the prepared alloy sample was performed on TESCAN VEGA3 scanning electron microscope with energy dispersive spectroscopy(EDS) (Oxford Instruments X-act) and the measurements were carried out at 20 kV.
Martensitic transformation temperatures were studied on DSC analyzer Mettler Toledo 822e. Measurement was performed in inert atmosphere, through 1 heating/cooling cycle from -50 to 120 °C with heating/cooling rate 10 °C/min.

## 3. RESULTS AND DISCUSSION

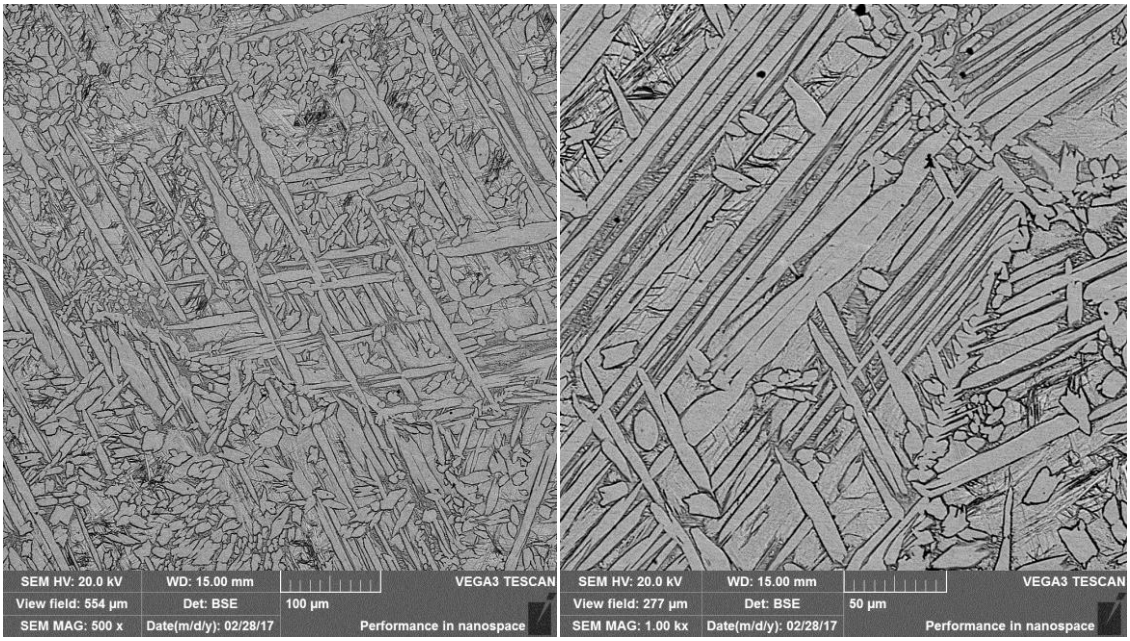
### 3.1. Microstructure of the Cu–9%Al–8%Mn alloy after induction melting

Microstructure of the Cu–9%Al–8%Mn alloy after induction melting was investigated using SEM-EDS. Overall chemical composition of the investigated alloy was checked using EDS area analysis. Designed and average overall chemical compositions of the investigated sample obtained by EDS analysis are given in Table 1.

*Table 1. Designed and experimentally determined overall composition of the investigated alloy*

| Sample | Designed composition (wt.%) | | | Experimentally determined composition with calculated standard uncertainties (wt.%) | | |
|---|---|---|---|---|---|---|
| | Cu | Al | Mn | Cu | Al | Mn |
| 1 | 83 | 9 | 8 | 83.1±0.4 | 9.0±0.2 | 7.9±0.1 |

As it can be seen from Table 1 designed and experimentally determined overall composition of the investigated alloy are in very good mutual agreement.
Characteristic SEM micrographs under different magnifications of the investigated bulk alloy after induction melting (as-prepared condition) are shown in Fig. 1.




(a) (b)

*Figure 1. SEM micrographs of the investigated Cu–9%Al–8%Mn bulk alloy after induction melting: (a) magnification 500x, (b) magnification 1000x.*

Average chemical compositions of identified phases determined by EDS analysis and shown in Table 2.

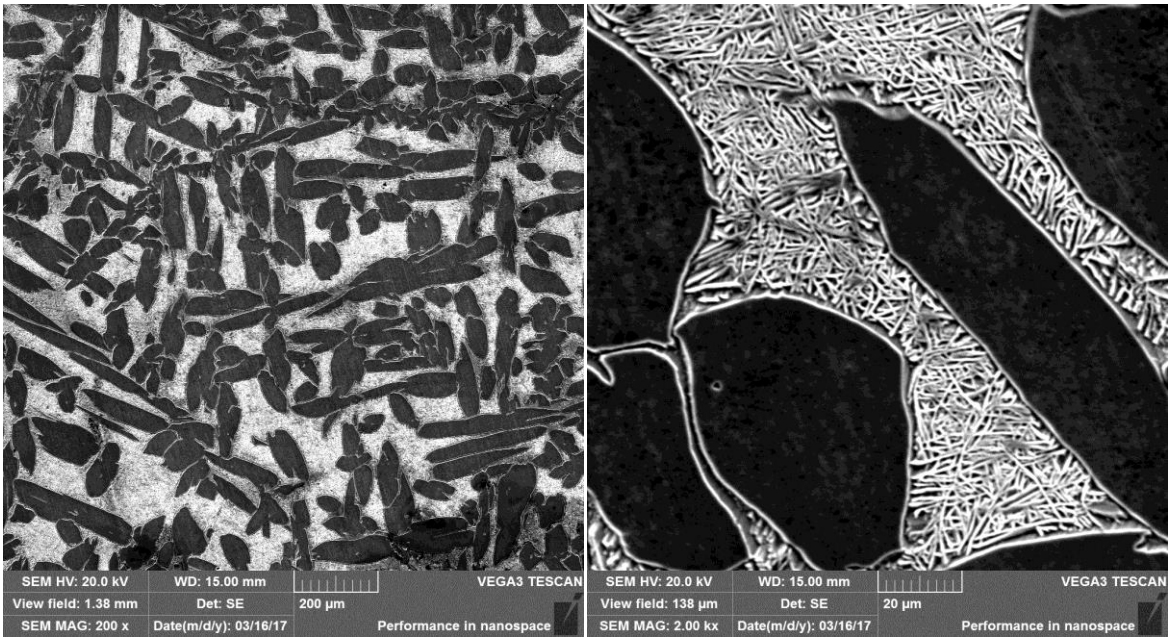
*Table 2. Chemical compositions of co-existing phases after induction melting determined by EDS analysis*

| Alloy | Phase | Cu (wt.%) | Al (wt.%) | Mn (wt.%) |
|---|---|---|---|---|
| Cu–9%Al–8%Mn | Dark phase | 80.6±0.3 | 10.4±0.3 | 9.0±0.1 |
| | Light phase | 84.1±0.4 | 8.2±0.2 | 7.7±0.1 |

It can be concluded that light phase represents Cu-rich α phase and dark matrix phase represents β phase.

### 3.2. Microstructure of the Cu–9%Al–8%Mn alloy after homogenization annealing at 850 °C and slow cooling

SEM micrographs of the investigated Cu–9%Al–8%Mn bulk alloy after homogenization annealing at 850 °C for five hours and cooling inside the furnace are given in Fig. 2.
Two co-existing phases can clearly be noticed: light matrix phase with characteristic lath structure and dark grains of precipitate phase.




(a) (b)

*Figure 2. SEM micrographs of the investigated Cu–9%Al–8%Mn bulk alloy after homogenization annealing at 850 °C for 5 hours and slow cooling: (a) magnification 200x, (b) magnification 2000x.*

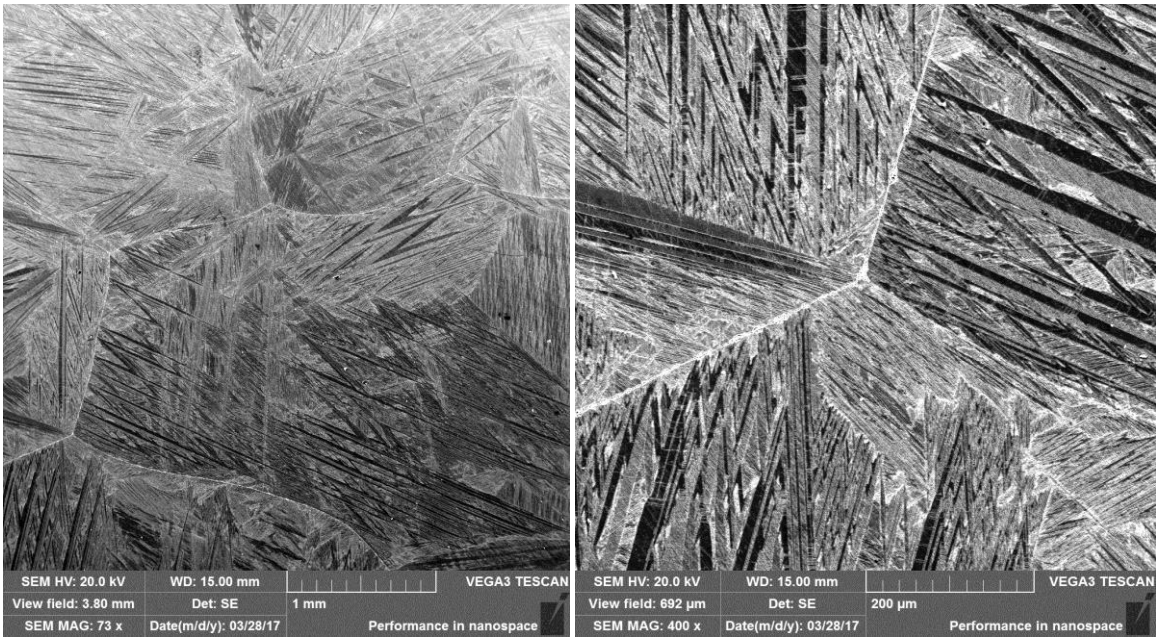
Average chemical compositions of identified phases were determined using EDS analysis and shown in Table 3. The dark phase has higher amount of copper and lower amount of aluminium and manganese than the light matrix phase. Chemical composition of the dark phase is almost identical to the composition of the light phase in the as-prepared sample. Thus, it can be concluded that dark phase represents Cu-rich α phase. The exact identification of the lath-type matrix phase should be done using additional experimental techniques such as XRD and TEM.

*Table 3. Chemical compositions of co-existing phases after homogenization annealing at 850 °C for 5 hour and slow cooling determined by EDS analysis*

| Alloy | Phase | Cu (wt.%) | Al (wt.%) | Mn (wt.%) |
|---|---|---|---|---|
| Cu–9%Al–8%Mn | Dark phase | 84.5±0.3 | 8.1±0.2 | 7.4±0.1 |
| | Light phase | 82.7±0.3 | 9.2±0.2 | 8.1±0.2 |

### 3.3. Microstructure of the Cu–9%Al–8%Mn alloy after quenching

Annealing of the investigated Cu–9%Al–8%Mn alloy at 850 °C in the β region followed by quenching in the ice water induced characteristic martensitic microstructure (Fig. 3). Bright and dark regions observable in the secondary electron (SE) mode of scanning electron microscopy do not represent different phases. They are caused by morphology and surface topography of the studied sample. The fine plate- or spear-like martensitic groups (most probably $\beta_1$' martensite) are observed in the microstructure of the quenched alloy.




(a) (b)

*Figure 3. SEM micrographs of investigated Cu–9%Al–8%Mn bulk alloy after annealing at 850 °C for 60 minutes and quenching in the ice water showing martensitic structure: (a) magnification 73x, (b) magnification 400x.*

### 3.4. Experimental investigation of transformation temperatures for the as-quenched alloys

DSC technique was used for investigation of martensite and reverse martensite transformation temperatures for the Cu–9%Al–8%Mn as-quenched alloy in the temperature range from -50 to 120 °C. Figure 4 shows DSC heating and cooling curves obtained for the Cu–9%Al–8%Mn alloy. Martensite start temperature ($M_s$) was determined as the temperature of the extrapolated peak onset while the martensite finish temperature ($M_f$) was determined as the extrapolated peak endset temperature on cooling [3]. One exothermic peak was detected during cooling run. The peak onset was at 28.9 °C($M_s$) and extrapolated endset of the peak was 10.4 °C ($M_f$). However, on heating related endothermic peak for reverse martensite transformation (martensite to austenite transformation) was not identified. The reason could be martensite stabilization. The stabilization of martensite in many copper-based shape-memory alloysis recognized effect resulting in an increase of the reverse martensitic transformation (As, Af) temperatures [3,7,8].

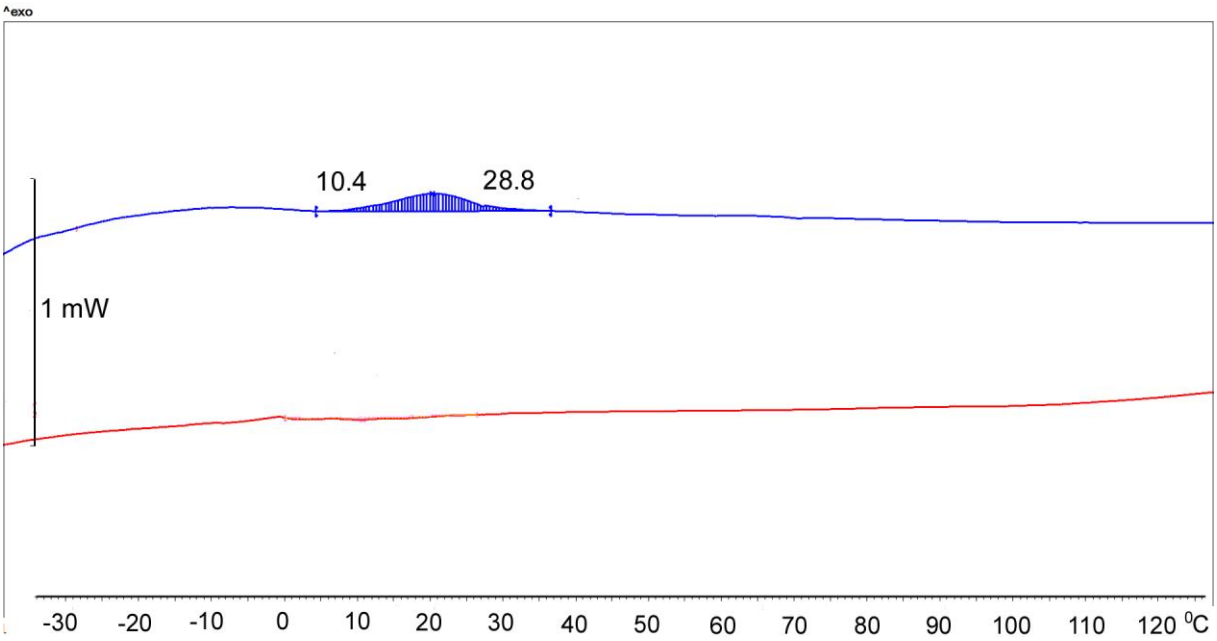



*Figure 4. DSC heating and cooling curves for the as-quenched Cu–9%Al–8%Mn alloy*

## 4. CONCLUSION

Microstructure and martensitic phase transformation temperatures of Cu–9%Al–8%Mn bulk alloy were investigated in this work. Microstructure of prepared bulk alloy was investigated in the as-prepared state, after homogenization annealing and after direct quenching into the ice water.

Based on the obtained results following conclusions can be made:

1) Microstructure of the as-prepared Cu–9%Al–8%Mn alloy includes α and β phases.
2) After homogenization annealing at 850 °C and slow cooling microstructure of the investigated alloy includes the lath-type phase in the matrix and large amount of irregularly distributed α grains.
3) Direct quenching from the 850 °C into the ice water produced martensitic structure.
4) Using DSC heating and cooling runs martensitic start ($M_s$) and finish ($M_f$) temperatures were determined to be 28.9 and 10.4 °C.

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