# Effect of Silver Addition on Cu-based Shape Memory Alloys

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Shape memory alloys (SMAs) are smart materials with unique properties of superelasticity and shape memory effect. These properties are the consequence of thermoelastic martensitic transformation, which can occur under thermal or mechanical deformation. Cu-Al-Ag alloys have high temperatures of martensite transformation, unlike other SMAs, which makes them suitable for use in specific applications. In this paper, Cu-10Al-1Ag alloy was prepared by melting pure metals in an electric arc furnace and casting the melt in a cylindrical mold. The microstructure of the as-cast and quenched material was determined by optical microscopy (OM) and scanning electron microscopy (SEM) equipped with energy dispersive analysis (EDS). X-ray diffraction (XRD) analysis was performed to identify crystal phases in the microstructure, while transformation temperatures were determined by differential scanning calorimetry (DSC). The hardness of Cu-Al-Ag SMA was also determined with a microhardness tester. The results showed partially formed martensite in the as-cast state, and fully formed martensite structure, 18R-type, in the quenched Cu-10Al-1Ag alloy.

### Keywords

shape memory alloys, Cu-Al-Ag alloys, microstructure, phase transformations, martensite

# Introduction

Cu-based shape memory alloys (SMAs) show unique properties of superelasticity and shape memory effect, as a consequence of a difusionless reversible martensitic transformation.<sup>1-5</sup> Cu-SMAs are inexpensive and easily processed, so consequently they have a wide range of application, from medical to electronic and electric devices, in the robotics, aerospace industries, etc.6-11 Both basic Cu-SMAs, Cu-Zn and Cu-Al based, can achieve a thermoelastic martensitic transformation from the high-temperature  $\beta$ -parent phase, austenite phase, by fast cooling or quenching in water. β-Phase decomposes through the eutectoid reaction to  $\alpha + \gamma_1$  phase, in equilibrium conditions or by slow cooling.<sup>12–15</sup>

Recently, ternary Cu-Al-Mn SMAs have attracted great interest since the addition of manganese to binary Cu-Al alloy stabilizes and extends the area of  $\beta$ -phase towards the lower aluminum content.<sup>16–18</sup> Therefore,  $\beta$ -phase becomes more stable with respect to diffusional decomposition.<sup>19</sup> During cooling,  $\beta$ -phase undergoes disorder-order transformations  $\beta(A2) \rightarrow \beta_2(B2) \rightarrow \beta_1(L2_1)$ .<sup>20–25</sup> The body cubic (b.c.c.) superlattice structures can be of the Fe<sub>2</sub>Al type (DO<sub>2</sub>), CsCl type (B2), and Cu<sub>2</sub>MnAl (L2<sub>1</sub>) type. At low temperatures the spinodal decomposition can occur between DO<sub>2</sub> (Cu<sub>2</sub>Al) and L2, (Cu,AlMn) phase.<sup>26</sup> The ordering transformation from A2 to  $L2_1$  cannot be suppressed at high aluminum concentrations, so L2, phase, the Heusler alloy (Cu<sub>2</sub>AlMn), transforms in a metastable way to 6M martensite. At low aluminum contents, usually below 16 at.%, A2 transforms to A1 (disordered face crystal cubic structure f.c.c.) or 2M structure.<sup>19</sup> Different types of martensite structures can be observed in Cu-based SMAs, depending on the chemical composition: a hexagonal structure 2H and rhombohedral structures 9R, 18R, 6R, 3R.<sup>27-29</sup>

The other most important ternary Cu-based SMAs are Cu-Zn-Al, Cu-Al-Ni, Cu-Al-Be and Cu-Al-Ag.<sup>30-34</sup> The addition of nickel to binary Cu-Al alloys enhances the shape memory effect (SME) and increases the martensite transformation temperature, but the alloy tends to brittle intergranular cracking and shows low cold workability.

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Cu-Al-Be SMAs are very cheap for processing, and as low as 0.5–0.56 wt.% of beryllium is enough to achieve SE properties. They show good mechanical properties, high corrosion resistance, and good damping properties.<sup>35</sup> By additional microalloying of SMA, it is possible to obtain better microstructural properties and reduced grain size, especially with the addition of Ti, B, and Si.<sup>36–38</sup> Furthermore, the addition of other elements can improve SME and other functional properties, as well as adjust the temperature of martensitic transformation, which is the key factor for the area of application. Previous investigations of Cu-SMAs were done with microalloying elements such as Cr, Si, Mg, Fe, etc.<sup>39–42</sup>

So far, there has been a limited investigation on the addition of silver to binary Cu-Al SMAs and especially ternary Cu-SMAs. Previous reports have shown that the addition of silver to binary Cu-Al alloys improves the corrosion resistance, increases the martensitic transformation temperature, and enhances the microhardness.<sup>43-46</sup> Cu-Al-Ag SMAs are specific among Cu-based SMAs due to the highest temperature of martensitic transformation, mostly from 200–600 °C, in dependence on the aluminum and silver content, and therefore high-temperature Cu-Al-Ag SMAs have an application in the specific areas.<sup>47,48</sup>

So far, different silver contents in Cu-Al SMAs have been investigated, mostly higher than 5 wt.%. The present study investigated the influence of 1 wt.% silver addition to binary Cu-Al alloys on the microstructure and stability of martensitic transformation.

# Materials and methods

## **Materials**

Cu-Al-Ag alloy with 10 wt.% Al and 1 wt.% Ag was prepared by melting in an electric arc furnace, in an argon atmosphere. The pure raw materials were melted: copper (Cu), purity of 99.9 %; aluminum (Al), purity of 99.5 %, and silver (Ag), purity of 99.99 %. The alloy was melted four times for better homogenization, and then cast into a cylindrical mold with a diameter of 8 mm and length of 12 mm. Heat treatment was performed at 900 °C for 30 minutes, followed by quenching in water.

# Characterization

## Optical and SEM analysis

Samples for microstructure investigations were ground with 600#, 800# and 1200# SiC abrasives, and then polished with 3  $\mu$ m and 1  $\mu$ m diamond

paste on a grinding-polishing machine TERGAM-IN-30 (Struers, Germany). To reveal microstructure, the prepared specimens were etched using a solution of 2.5 g FeCl<sub>3</sub>/48 mL CH<sub>3</sub>OH/10 mL H<sub>2</sub>O. The microstructure of SMA was observed using an Axio Vert A1 light microscope (Zeiss, Germany), as well as with a FED QUANTA 250 SEM FEI scanning electron microscope, equipped with an energy dispersive X-ray spectroscopy (EDS) detector (U.K., Oxford).

#### Hardness

Microhardness of investigated alloys was determined using FM-ARS-9000 fully automatic microhardness measurement system, applying a load of 100 g for a dwell time of 15 seconds. Before the micro-hardness measurements, each sample surface underwent a meticulous preparation process, which included careful cleaning, grinding, and polishing to ensure the accuracy of results. The Vickers micro-hardness values were calculated as the average of five individual measurements taken from each sample.

## Differential scanning calorimetry (DSC)

Transformation temperatures were determined by a Mettler-Toledo 822e modulated differential scanning calorimeter. Dynamic measurements were performed in two heating/cooling cycles from -50 °C to 350 °C, in a nitrogen atmosphere, with heating/cooling rate of 10 K min<sup>-1</sup>.

#### XRD analysis

XRD analysis was performed with a Bruker D8 Advance diffractometer with CuK $\alpha$  radiation. The scans were collected in the  $2\theta$  range of  $20-90^{\circ}$ , with a  $2\theta$  step size of  $0.02^{\circ}$  and a counting time of 0.6 s, under the accelerating voltage of 40 kV and current of 25 mA.

# **Results and discussion**

Table 1 shows the experimentally determined average overall composition of the prepared sample as determined by EDS. A negligible difference of the designed and experimental composition of Cu-10Al-1Ag SMA can be observed.

 Table 1 – Average composition of SMA alloy determined by
 SEM-EDS

Composition (wt.%)						
Alloy	Alloy Cu		Ag			
As-cast state	89.46 ±2.46	9.61 ±0.56	0.93 ±0.1			



Fig. 1 – OM micrographs of as-cast Cu-10Al-1Ag shape memory alloy: a) BF, magnification 100x, b) POL, magnification 100x, c) BF, magnification 500x, d) POL, magnification 500x



Fig. 2 – OM micrographs of quenched Cu-10Al-1Ag shape memory alloy: a) BF, magnification 100x, b) POL, magnification 100x, c) BF, magnification 1000x, d) POL, magnification 500x



Fig. 3 – SEM micrograph of Cu-10Al-1Ag shape memory alloy, magnification 2000x: a) as-cast state, b) quenched state, magnification 5000x: c) as-cast state, d) quenched state, and magnification 10000x: e) as-cast state, f) quenched state

Microstructural analysis of the investigated Cu-Al-Ag alloy in the as-cast state and in the quenched state was carried out by optical microscopy (Figs. 1 and 2), and scanning electron microscopy (Fig. 3). Optical microscopy images are collected in the bright field (BF) and with polarized light (POL). Fig. 1 shows the precipitates of  $\beta$ -parent phase in the microstructure of the as-cast state, and just the beginning of martensite structure formation is observed at the grain boundaries. Inside the grains, short needle-like martensite crystals can be observed, probably of  $\beta_1$ -type with monoclinic 18R structure, formed from the DO<sub>3</sub> parent phase. In the polarized OM micrographs, the grain boundaries can be seen clearly, with different martensite crystals orientations inside the grains. After quenching, the completely formed martensite structure is observed (Fig. 2). Only  $\beta_1$  -martensite crystals can be seen, which are formed mostly in the typical zigzag morphology (Figs. 2 and 3).  $\beta_1$ -variants of martensite exhibit thermoelastic behavior due to the controlled growth in self-accommodating zig-zag groups.<sup>49</sup> Figs. 3b), d), and f) show 18R martensite formed as the needle-type and wedge-type crystals.

SEM micrographs of quenched state indicate the existence of only one type of martensite. There

are no visible coarse plates of  $\gamma_1$  martensite, which is the other possible thermally induced martensite in Cu-Al SMAs. The driving force for nucleation of  $\gamma_1$  (2H) is higher than for  $\beta_1$  (18R) martensite. The coexistence of both 18R and 2H structures depends on the alloy composition and the parameters of thermal treatment process.  $\beta_1$  and  $\gamma_1$  have a different morphology due to the different ways of inhomogeneous shear. In the 18R martensite, the inhomogeneous shear occurs by the distributed stacking disorder of the  $\beta$ -parent phase. In 2H martensite, the inhomogeneous shear occurs by twinning on a {1 2 1} plane.<sup>50</sup>

XRD analysis confirmed the existence of only  $\beta_1$ '-martensite phase in the quenched state, and the presence of Cu<sub>3</sub>Al and Cu-rich precipitates in the as-cast state (Fig. 6).

EDS analysis (Fig. 4) showed that, in the ascast Cu-10Al-1Ag alloy, beside  $\beta$ -parent phase and martensite, there were no visible precipitates of pure silver, which were reported previously for samples having higher silver contents.<sup>51</sup> EDS analysis of quenched state is given in Fig. 5.

The quenched Cu-10Al-1Ag alloy shows lower microhardness in relation to the as-cast state (Table



Position 1		Position 2			Position 3		
Element	Wt%	Element	Wt%		Element	Wt%	
Al	8.82 ±0.25	Al	9.87 ±0.46		Al	11.29 ±0.50	
Cu	88.17 ±0.70	Cu	83.39 ±1.30		Cu	88.04 ±0.55	
Ag	1.40 ±0.23	Ag	1.27 ±0.40		Ag	0.00	

Fig. 4 – EDS analysis of as-cast Cu-10Al-1Ag alloy



Position 1			Position 2			
Element	Wt%		Element	Wt%		
Al	10.92 ±0.46		Al	10.90 ±0.51		
Cu	88.00 ±0.59		Cu	88.17 ±0.64		
Ag	1.09 ±0.42		Ag	0.93 ±0.44		

# Fig. 5 – EDS analysis of quenched Cu-10Al-1Ag alloy at different positions



Fig. 6 – XRD diffractogram of Cu-10Al-1Ag SMA alloy

Table 2 - Microhardness of Cu-Al-Ag SMA alloy

Hardness	HV							
Cu10Al1Ag	1	2	3	4	5	Average		
As-cast state	314.94	292.65	284.43	295.89	307.84	297.34		
Quenched state	278.57	284.55	270.88	269.11	280.45	278.00		

2), in agreement with the previous investigations of SMAs.<sup>52</sup> The small difference in hardness between the as-cast and quenched states may be attributed to mechanically induced martensite formation in the stressed regions that occurs under high indenter loading.<sup>53</sup> Despite the microstructural changes that the alloy undergoes during quenching, there is no significant change in hardness. Probably due to the low Ag content in the SMA composition.

The results of DSC analysis are shown in Figs. 7–10. The DSC thermogram for as-cast Cu-Al-Ag alloy shows, after the first cycle, an endothermic transformation with the start temperature of austenite transformation  $A_s = 176$  °C and finish temperature of austenite transformation  $A_f = 225$  °C. After the second heating, transformation is barely detectable (Fig. 7). The martensite transformation is observed in cooling curves with start and finish temperatures of martensite transformation,  $M_s = 182$  °C and  $M_f = 154$  °C (in the first cooling cycle), and 151 °C (in the second cooling cycle) (Fig. 9).



Fig. 7 – DSC thermograms of as-cast Cu-10Al-1Ag shape memory alloy



Fig. 8 – DSC thermograms of quenched Cu-10Al-1Ag shape memory alloy



Fig. 9 – Martensite transformation for as-cast Cu-10Al-1Ag shape memory alloy



Fig. 10 – Martensite transformation for quenched Cu-10Al-1Ag shape memory alloy

After quenching, austenite transformation is not detectable at DSC heating curves, while martensitic transformation shows start martensitic temperature at  $M_{e} = 180$  °C and finish transformation temperature  $M_{\rm f} = 132$  °C. Martensitic transformation temperatures did not change significantly in the first and second cycle (Figs. 8, 10). In relation to as-cast alloy, start martensite temperature is similar, but transformation releases higher heat fusion, and finish transformation temperature is shifted to lower value. DSC results indicate that the formed  $\beta_1$  -martensite is thermally stable, which is very important for the application of SMA. The microstructural and phase analysis, as well as DSC results showed that the shape memory properties of material can be achieved with only 1 wt.% of added silver, with the stable martensite phase and very high transformation temperatures.

# Conclusion

Cu-10Al-1Ag alloy was successfully prepared by smelting pure metals in an electric arc furnace. The results showed partial formation of martensitic structure in the as-cast state, and completely formed martensite phase after quenching in water. Only one type of the martensite structure was found with microstructural investigations, i.e. monoclinic 18R or  $\beta_1$ ` structure. The martensite transformation temperature was found in the range of 180 °C to 130 °C. Therefore, a very small amount of added silver of only 1 wt.% leads to achieving shape memory properties in Cu-Al-Ag alloy.

#### List of abbreviations and symbols:

- SMA shape memory alloy
- OM optical microscope
- SEM scanning electron microscope
- EDS energy dispersive X-ray spectroscopy
- DSC differential scanning calorimetry
- XRD X-ray diffraction
- $M_{\rm s}$  temperature at which the transformation of martensite begins (starts), °C
- $M_{\rm f}$  temperature at which the transformation of martensite is completed (finished), °C
- $A_{\rm s}$  temperature at which the transformation of austenite begins (starts), °C
- $A_{\rm f}$  temperature at which the transformation of austenite is completed (finished), °C
- BF bright field
- POL polarized light

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