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## The effects of thermal procedure on transformation temperature, crystal structure and microstructure of Cu-Al-Co shape memory alloy

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Abstract. The purpose of this study is to investigate the effects of different thermal procedures of the Cu-Al-Co shape memory alloy on its crystal structure, transformation temperature and microstructure. The alloys were subjected to a heat treatment and then cooling was applied at four different conditions. After the thermal process, XRD, DSC, optical microscopy and micro-hardness measurements were carried out. The experimental studies showed that crystal structure, microstructure and transformation temperature of Cu-Al-Co alloy were changed from the cooling conditions.

### **1.Introduction**

There is a significant demand on Cu-based shape memory alloys for high temperature applications as these alloys have martensite transformation temperatures higher than 200 °C [1]. Addition of a third element into the Cu-Al binary alloys represents the regular DO<sub>3</sub> structure in the form of (CuX)<sub>3</sub>Al. Here, Cu atoms are substituted by X atoms, where Cu atoms are dominant. If the amount of X is increased significantly, then the crystal structure transforms from  $DO_3$  to  $L2_1$  of which stoichiometric representation is  $Cu_2XAI$ [2]. Cu-Al-Co, which has been started to investigate recently, represents a group of high temperature shape memory alloys about which the knowledge is limited [3,4]. In the literature, generally, changes in the martensitic transformation of Cu-Al-Co alloy after the production and post-production stages have been investigated crystallographically and thermally [1,3]. However, there is lack of study focusing on the thermal procedures applied to these alloys.

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Martensitic transformations of the most of the Cu-based alloys are affected from quenching and post quenching. More clearly, it can be stated that stabilization temperatures of these alloys depend on the quenching condition. Direct quenching from high temperature is quite effective to achieve full martensitic transformation [5,6]. Quenching is a different heat treatment process applied to the shape memory alloys. Generally, the most of the heat treatment procedures performed on the shape memory alloys are known as the application of a high temperature heat treatment after which the alloy is suddenly cooled by immersing it into a water with ice-salt bath. There are studies mainly concerning about the temperature of the heat treatment temperature of the cooling bath have been limited [5,6;7-11]. For this reason, in this study, the heat treatment temperature was kept constant at 850 °C, while the effects of different cooling media on martensitic transformation temperature, crystal structure, microstructure and micro-hardness of the Cu-Al-Co alloy were investigated.

### 2. Materials and methods

Cu-%at.21Al-%at.1Co shape memory alloy samples were cut from ingot such that they form five different groups. The processes outlined as follow:

a) M0; Without heat treatment

b) M1; Heat treatment at 850 °C for 1 hour and then quenching in water with ice-salt mixture

c) M2; Heat treatment at 850 °C for 1 hour and then quenching in water at room temperature

d) M3; Heat treatment at 850 °C for 1 hour and then quenching in boiling water for 3 min and finally quenching in water with ice-salt mixture

e) M4; Heat treatment at 850 °C for 1 hour and then furnace cooled.

The cooling rate of the alloys could be ranked as M1>M2>M3>M4. These are the results observed in the course of the experimental processes.

DSC measurements of the Cu-Al-Co alloys were conducted between 100 and 450 °C at a cooling rate of 25 °C/min under the nitrogen atmosphere so as to investigate the effects of the cooling rate and cooling conditions on the transformation temperature. Similarly, XRD measurements were performed by Bruker Discover D8 computer X-Ray diffractometer to identify the changes occurred in the crystal structure. Finally, optical microscopy investigation of the samples, which were moulded into polyester, were performed by using etchant made out of 20 ml HCl, 5 g FeCl<sub>3</sub>-H<sub>2</sub>O and 96 ml methanol and Nikon Eclipse MA200 optical microscope and the micro-hardness measurements of the samples were conducted by a micro-hardness test machine (Emco Test DuraScan).

### **3.Results and discussion**

Phase transformations of Cu-Al-Co alloy at different cooling conditions were measured by XRD measurements and shown in Fig. 1. The diffraction peaks were indexed according to the data given in the literature [12,13]. It was detected that as-cast and all other heat treated samples, except for furnace cooled one, possessed  $\beta_1^i$  martensite phase. The furnace cooled sample, however, had both  $\beta_1^i$  and  $\alpha$  phases. It is considered that  $\beta$  phase could transform into both Cu-rich  $\alpha$  fcc phase and Al rich  $\beta_1^i$ -Cu<sub>9</sub>Al<sub>4</sub> phase [12,14]. XRD diffraction analysis showed that this was also  $\alpha$  phase.

Cooling rate yields a significant change in the microstructure of alloys. These morphological changes can be seen in Fig. 2. In Fig. 2 a, optical microscope images for as-cast samples are given. Here, grains can be seen clearly. The reason of the dark and bright appearance of the grains is the different orientation of the martensite plates. V-type and and needle-like martensites were dominant in the microstructure. Martensite plates aligned in parallel to each other.



Figure 1. X ray difraktograms of Cu-Al-Co alloy at different cooling conditions.

The microstructure of the Cu-Al-Co alloy sample quenched in water with ice-salt mixture was similar to that of as-cast one (Fig 2 b.). Similar to as-cast sample, V-type, needle-like and plate martensites oriented in parallel to each other were observed in the microstructure. As seen from Fig. 2 c-d, there are similarities between the microstructure of the water quenched sample, the sample quenched into boiling water and the sample quenched into water with ice-salt mixture. Regularly aligned diamond shaped martensite plates can be observed in the structure. Microstructure of the furnace cooled sample obtained by the optical microscope, see Fig. e, showed that the microstructure had austenitic  $\alpha$  phase that was also detected by XRD measurements. The dominant phase in the microstructure was  $\alpha$  phase, while there was also minor amount of martensite ( $\beta_1$ ) in it. The variation in the transformation temperature of the Cu-Al-Co shape memory alloys, an indicator of the shape memory effect, subjected to different cooling conditions can be seen in Fig. 3. Meanwhile, transformation temperature values are summarized in the Table 1. According to the Table 1, there were significant differences between martensite start temperature of the Cu-Al-Co alloys, cooled at different conditions, while the austenite start temperature of these alloys did not change much.

The martensite start temperature was quite high for the furnace cooled sample. If the heat flow curve is examined for the same sample, it can be stated that DSC curve is broaden. It was considered that this broadening was caused by multiple phase transformation. As it can be revealed form the XRD plots, austenitic  $\alpha$  phase and martensitic  $\beta_1^i$  phase appears simultaneously. This multiphase structure has been also detected in the furnace cooled NiTi alloy by Zhang and coworkers [10]. If the average enthalpy and entropy values are examined, the most striking difference can be observed for the furnace cooled sample. According to DSC measurements, transformation temperature of the alloy was significantly affect from the cooling rate and the presence of  $\alpha$  phase in the furnace cooled sample reduced the transformation temperature



**Figure 2.** Optical micrographs of Cu-Al-Co alloy at different cooling conditions ( $\blacktriangleleft$  is  $\beta_1^1$  phase,  $\blacktriangleright$  is  $\alpha$  phase) a) M0, b) M1, c) M2, d) M3 and e) M4



Figure 3. DSC curves of Cu-Al-Co alloy at different cooling conditions

Table 1. Thase transformation temperatures and micro narchess values of examined Anoys									
Sample Code	As	Ap	$A_{\rm f}$	Ms	$M_p$	$M_{\mathrm{f}}$	$\Delta H_{ave}$	$\Delta S$	Average hardness (HV)
	$(^{\circ}C)$	$(^{\circ}C)$	$(^{\circ}C)$	$(^{\circ}C)$	$(^{\circ}C)$	$(^{\circ}C)$	(J/g)	$(J/g^{o}C)$	
M0	295.3	333.1	357.4	264.9	243.1	217.3	2.76	0.010	263.3
M1	296.4	346.7	368.2	217.2	174.6	151.2	4.13	0.016	396
M2	294.7	346.3	376.7	226.9	173.1	148.5	4.28	0.016	309
M3	289.2	337.9	368.4	213.9	173.5	151.5	4.85	0.019	312
M4	286.5	317.9	343.2	350.1	292.8	239.7	1.32	0.004	257.3

Table 1. Phase transformation temperatures and micro hardness values of examined Alloys

Micro-hardness measurements are important as they give information about the brittleness of the shape memory alloys. Increasing hardness value could be considered as the reduction in the brittleness. Vickers hardness measurement scale is an important tool to understand the relation between microstructure and micro-hardness. The Vickers hardness value can be found by using the following equation, which includes the experimental data from micro-hardness measurement:

$$HV = \frac{2Psin\emptyset/2}{d^2} \tag{1}$$

Here,  $\emptyset$  is the apex angle of the indenter, *P* is the applied load and d is the average length of the diagonals [15].

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For the same heat treatment temperature and duration, the variation of cooling conditions resulted in significant changes in the Vickers hardness value of the Cu-Al-Co alloy (Table 1). The lowest and highest hardness values were measured for the furnace cooled sample and the sample quenched to water with saltice mixture, respectively. Vickers hardness values of the samples quenched in a single step (directly into water) and quenched in two steps (first into boiling water for 3 minutes and then into water) were nearly equal but lower than hardness value of the sample quenched into water with ice-salt mixture. As a result of this, it can be revealed that the hardness value of the Cu-Al-Co alloy decreased with reducing cooling rate. Similar results have been also obtained for AISI 1020, AISI 1040 and AISI 1060 steels. In these studies, steels with four different carbon content has been furnace cooled, quenched in air or water after which the hardness values of the samples have been measured. It has been detected that the hardness values of the samples decreased with reducing cooling rate. This can be attributed to formation of the primary martensite phase upon rapid cooling [15-16].

### 4. Conclusions

It was observed that cooling conditions have significant effects on the transformation temperature, crystal structure, micro-hardness and microstructure of Cu-based shape memory alloys. It was detected that transformation temperature changed with cooling rate. The experiments showed that, with the application of furnace cooling, which was equivalent to the slowest cooling rate, cooling curve broadened. Meanwhile, the crystal analysis of this slowly cooled sample demonstrated the presence of  $\alpha$  phase together with main martensite phase making the transformation more difficult to occur. Additionally, the presence of  $\alpha$  phase was also verified by optical microscope analysis. It was measured that the microhardness value reduced with  $\alpha$  phase.

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