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MICROSTRUCTURAL ANALYSIS OF COLD DRAWN CuAlMn SHAPE MEMORY ALLOY WIRE

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Abstract

The Cu-11.9Al-2.5Mn (wt. %) shape memory alloy was produced by vertically continuous casting technique obtaining bars of 8 mm in diameter which is applicable for plastic deformation. With the process of hot rolling and forging the 4.80 mm bar was produced. Afterwards, the obtained 4.80 mm bar was subjected to cold drawing process. After first run and after fourth run of cold drawing process the wire with diameter of 4.47 mm and 3.22 mm was produced, respectively. Optical microscopy (OM) and scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS) shown the insight in the samples microstructure. The as-cast state sample has two phase (α+β) microstructure. After cold working process the two-phase (martensite+α) microstructure appears. As the result of the cold working process it can be noticed a texture inside the sample depending on cold drawing direction. The microhardness of samples increases as the wires diameter decreases.

Keywords: *CuAlMn wire, shape memory alloy, microstructure, hot working, cold working*

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INTRODUCTION

Cooper based shape memory alloys (SMAs) are very interesting material for industrial application due to its good shape memory effect (SME), relatively low cost, good machinability and damping capacity in comparison to the properties that NiTi alloy obtain.

Its excellent thermal stability and very high martensite transformation temperature makes them rapidly become one of the most promising high temperature shape memory alloys [1,2].
CuZnAl and CuAlNi shape memory alloys have been extensively investigated due to its previously mentioned properties. However, these shape memory alloys are too brittle for cold working because of coarse grain structure and high elastic anisotropy [3]. The large grain size problem can be solved by using grain size refinements (as titanium and boron for example) or production by rapid solidification techniques. This type of production ensures shaping and forming procedures easier [4-6]. Mechanical properties improvement, preferably meaning ductility, can be provided by adding alloying elements and by heat treatment as well [7].
Addition of manganese to binary CuAl alloy can significantly improve alloys properties by stabilizing the bcc phase, widening the single β-phase region to lower temperatures and improving ductility in low Al alloys (less than 18 at. %) [8,9]. The austenitic β-phase arranged to the lower order system β_1 (L21; Cu2AlMn) and martensitic phase stabilization of unstable β'_3 (18R) to stable γ'_3 (2H) ratio can be adjusted by quenching and the amount of aluminium (Al) and manganese (Mn) [10].
The CuAlMn alloys exhibit shape memory effect and superelasticity based on cubic β_1 (L21) to monoclinic β_1' (6M) martensitic transformation. The shape memory characteristics (superelasticity and shape memory effect) of CuAlMn SMAs can be enhanced by the addition of alloying elements and the application of microstructure control achieved by thermomechanical treatment. It has been reported that those characteristics strongly depend on grain size and the development of texture [11]. Some investigations have been demonstrated that low thermal expansion can be obtained in CuAlMn shape memory alloys by controlling stress-induced martensitic transformation due to cold-rolling [8,11].
Due to the fact that CuAlMn shape memory alloy possesses significant ductility the aim of this paper is to produce a ductile CuAlMn shape memory alloy wire for possible industrial application with favorable microstructural properties by hot/cold plastic deformation process.

MATERIALS AND METHODS

Copper-based SMA with the nominal composition of Cu-11.9Al-2.5Mn (wt. %) was prepared in a vacuum induction furnace. The bar of 8 mm in diameter (Figure 1) was obtained and it was used for hot/cold plastic deformation process to obtain wire. Firstly, the bar was subjected to hot working process which was performed by a combination of hot rolling and hot forging. The forging was performed as free forging and forging in a profiled tool (Figure 2). The obtained bar after first run of rolling and forging was presented at Figure 3. The forging and rolling procedures were performed alternately till the cross section of the bar was 4.80 mm. Afterwards, the obtained bars were taken to recrystallization annealing at 580 °C for 60 minutes. After annealing, surface of the samples is brushed for elimination of possible surface defects propagation during cold working process. After first run of cold drawing the wire with diameter of 4.47 mm was obtained. Followed by three more runs the wire with ø 4.02 mm, 3.61 mm and 3.22 mm was produced. Between last two runs (ø 3.61 mm and 3.22 mm) the recrystallization annealing at 580 °C for 60 minutes was performed. The exact procedure of plastic deformation process can be seen in Table 1. Samples selected for investigation were in as-cast state (Figure 1) and after cold working process with dimensions ø 4.47 mm and ø 3.22 mm (Figure 4), marked bold and underlined in the Table 1. Insight into samples microstructure was obtained by optical microscopy (OM) equipped with digital camera and scanning electron microscopy (SEM) along with energy dispersive x-ray spectroscopy (EDS). The alloys hardness was determined by Vickers method.

Figure 1. Photograph of CuAlMn shape memory alloy bar ø 8 mm after casting

Table 1. Procedure of plastic deformation process on CuAlMn SMA bars

Deformation process	**Dimensions (mm)**	**Shematic illustration of crossection**
Sample in as-cast state		
HOT WORKING PROCESS - hot rolling and forging		
Hot rolling (880 – 900 °C) 2 runs	6.10x8.50	
Hot forging (880 – 900 °C) in a profiled tool	Square side length 6.00	
Hot rolling (880 – 900 °C) 1 run	4.50x9.50	
Hot forging (880 – 900 °C) in a profiled tool	Square side length 5.20	
Hot rolling (880 – 900 °C) 1 run	4.00x8.70	
Hot forging (880 – 900 °C) in a profiled tool	Square side length 4.60	
Hot forging (880 – 900 °C) in a profiled tool	Square side length 4.80	
COLD WORKING PROCESS - cold drawing		
Cold drawing	**Ø 4.47**	
Cold drawing	Ø 4.02	
Cold drawing	Ø 3.61	
Annealing at 580 °C/60’		
Cold drawing	**Ø 3.22**	

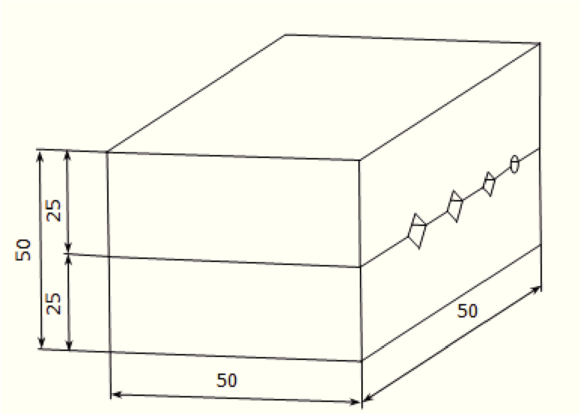

Figure 2. Schematic illustration of a profiled forging tool

Figure 3. Photograph of CuAlMn shape memory alloy bar after first run of hot rolling and forging

Figure 4. Photograph of CuAlMn shape memory alloy wire ø 4.47 mm (a) and ø 3.22 mm (b)

RESULTS AND DISCUSSION

By hot and cold plastic deformation process CuAlMn shape memory alloy wire was successfully produced. The ductility as a useful property of this cooper based shape memory alloy enables exhibition of better pseudoelasticity and therefore better hot or cold workability. It has been reported [8-11] that alloying additions play a very significant role in the properties of Cu-based shape memory alloys and required properties can be achieved by proper designing/selection of the alloying elements.

Figure 5 and 6 shows results of metallographic analysis by OM and SEM method of CuAlMn selected samples.

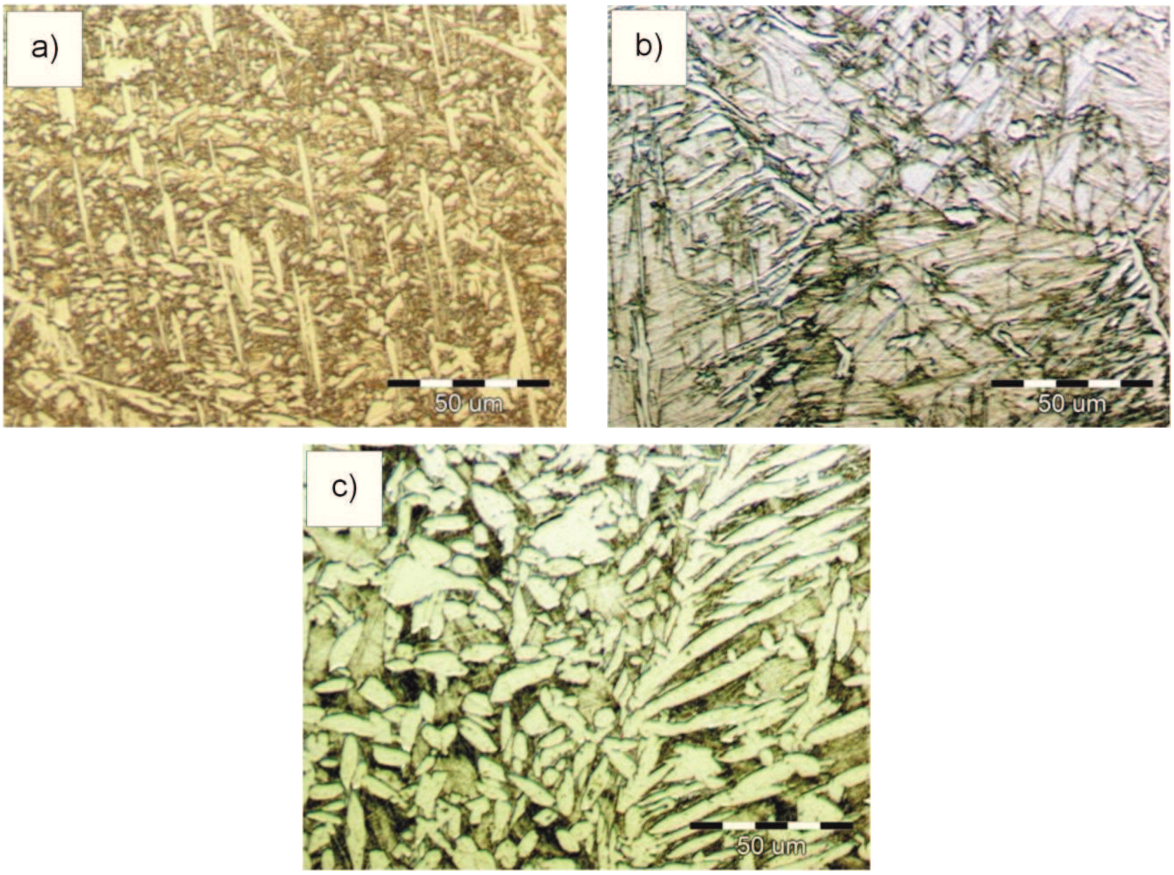

Figure 5. Optical micrographs of CuAlMn SMA in as-cast state (a), after cold drawing: ø 4.47 mm (b), ø 3.22 mm (c)

As can be seen, the CuAlMn as-cast bar has two-phase (α+β) microstructure (Figure 5a and 6a). It is known that in vertical section of phase diagram of Cu-Al-10 at.% Mn the single phase region is broadened by addition of Mn and $\alpha+\beta$ microstructure exists [12]. Grain structure with α+β phases a prerequisite for martensite formation after quenching. This clearly indicates that all the alloys have potential to exhibit the shape memory behaviour [13].
The influence of plastic deformation process can be seen on microstructures at the obtained ø 4.47 mm and ø 3.22 mm wires after cold working (Figure 5b, 5c, 6b and 6c). In both cases after cold working process the martensite appears in some places inside the microstructure. Considering that the β phase exists in the alloy in as-cast state, during plastic deformation process transforms into the martensite, probably came to appearance of stress-induced martensite. The stress induced martensite transformation occurs during loading under the almost constant stress [14].

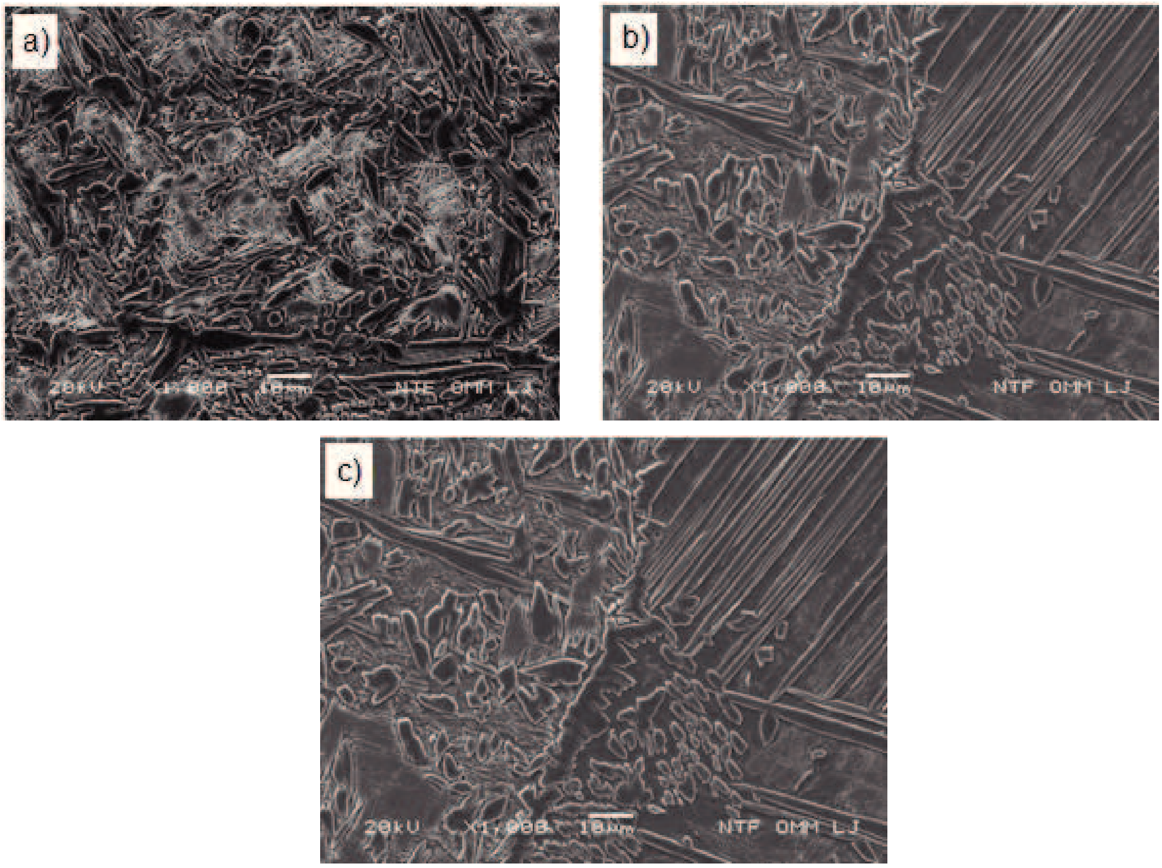

Figure 6. SEM micrographs of CuAlMn SMA in as-cast state (a), after cold drawing: ø 4.47 mm (b), ø 3.22 mm (c)

Figures 7 and 8 shows SEM micrographs along with the marked positions for EDS analysis of CuAlMn wire ø 4.47 mm and ø 3.22 mm, respectively. The results of the EDS analysis for cold drawn wire with diameter ø 4.47 mm show the difference between position 1 which shows higher amount of aluminum (7.45 wt.%) in comparison to positions 2 and 3 with amount of aluminum of (5.30 and 5.03 wt. %), Figure 7 and Table 2. Similar composition difference is noticed for wire with ø 3.22 mm, Figure 8 and Table 3. The difference between positions 1 and 2 has higher amount of aluminum (6.93 and 6.76 wt. %) in comparison to position 3 with amount of aluminum of (4.92 wt. %). It can be assumed that the aluminum enriched phase presents α phase. Sutou et al. [2] reported existence of two-phase microstructure (martensite + α) in Cu-Al-Mn-Ni-Si alloy after heat treatment at 850 °C. Due to the fact that the samples between the cold drawing runs were heat treated at 580 °C/60 minutes, the martensite + α microstructure is possible.

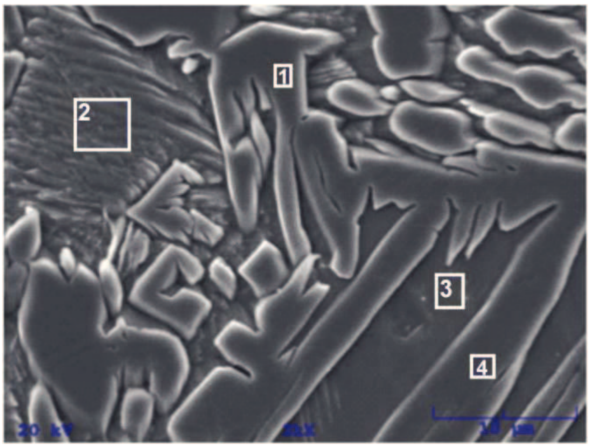

Figure 7. SEM micrograph of CuAlMn SMA wire ø 4.47 mm

Table 2. Chemical composition of CuAlMn SMA wire ø 4.47 mm positions marked at the Fig.7, wt. %

	Cu	Al	Mn
Position 1	82.74	7.45	9.81
Position 2	86.27	5.30	8.43
Position 3	86.47	5.03	8.50
Position 4	86.16	5.17	8.68

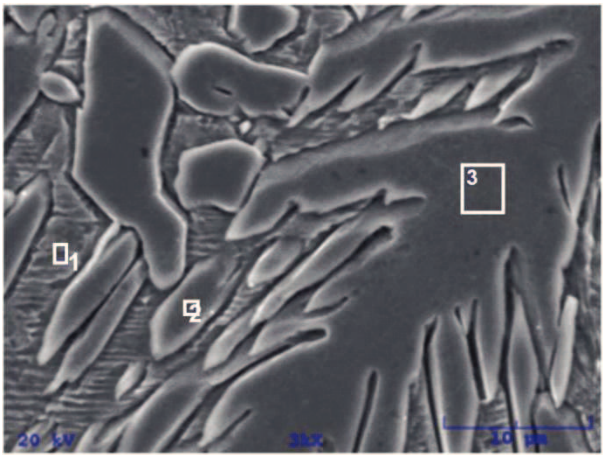

Figure 8. SEM micrograph of CuAlMn SMA wire ø 3.22 mm

Table 3. Chemical composition of CuAlMn SMA wire ø 3.22 mm positions marked at the Figure 8, wt. %

	Cu	Al	Mn
Position 1	82.66	6.93	10.41
Position 2	83.17	6.76	10.09
Position 3	86.63	4.92	8.45

The hardness values of the investigated samples are given as mean value of three measurements and the results can be seen at Figure 9. It is obvious that the hardness of the alloy increases through the process of cold drawing. The hardness of the alloy in as-cast state was 268 HV 0.05. Increase in alloys hardness is noticed for wire with ø 4.47 mm (318 HV 0.05) and for wire with ø 3.22 mm (341 HV 0.05), respectively.

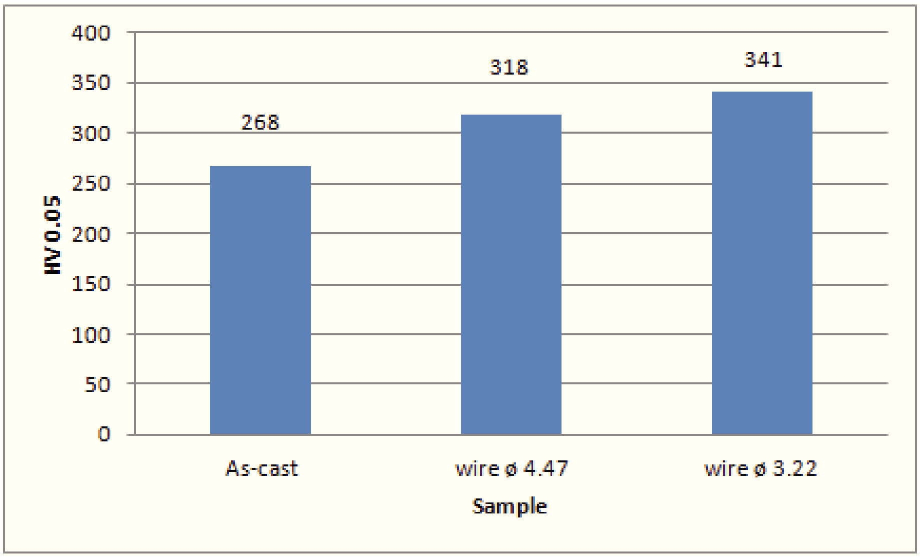

Figure 9. Hardness of CuAlMn shape memory alloy before and after cold working process

CONCLUSIONS

With hot and cold plastic deformation process it was successfully produced the CuAlMn shape memory wire with ø 4.47 mm and ø 3.22 mm in diameter from continuously casted ø 8mm CuAlMn shape memory alloy bar. From the results of microstructural characterization and hardness measurements can be withdrawn following conclusions:

- From the optical and SEM micrographs obtained grain structure with α+β phases is visible in the as-cast condition of the CuAlMn shape memory alloy. This microstructure is a prerequisite for martensite formation after quenching indicating that the alloy has potential to exhibit the shape memory behaviour.

- In some places the martensite formation is observed in CuAlMn alloy wires (ø 4.47 mm and ø 3.22 mm) produced by cold drawing process. During hot and cold working process appear favorable conditions for appearance of stress induced martensite.
- The results of EDS analysis for cold drawn wires with diameter ø 4.47 mm and ø 3.22 mm shows the difference between the positions enriched with aluminum content indicating the existence of the α phase in the microstructure.
- The hardness of the alloy increases through the process of cold drawing from 268 HV 0.05 in as-cast state to 318 HV 0.05 wire with ø 4.47 mm and 341 HV 0.05 for wire with ø 3.22 mm.

REFERENCES

[1] Q. Wang, F. Han, C. Cui, S. Bu, L. Bai, Effect of ageing on the reverse martensitic phase transformation behaviors of a CuAlMn shape memory alloy, Materials Letters 61(2007), pp. 5185-5187.
[2] Y. Sotou, N. Koeda, T. Omori, R. Kainuma, K. Ishida, Effects of aging on stress-induced martensitic transformation in ductile Cu-Al-Mn – based shape memory alloys, Acta Materialia, 57(2009), pp. 5759-5770.
[3] T. Omori, N. Koeda, Y. Sotou, R. Kainuma, K. Ishida, Superplasticity of Cu-Al-Mn-Ni shape memory alloy, Materials Transactions, 48(2007)11, pp. 2914-2918.
[4] C. E. Sobrero, P. La Roca, A. Roatta, R. E. Bolmaro., J. Malarría, Shape memory properties of highly textured Cu-Al-Ni-(Ti) alloys, Materials Science and Engineering A, 536(2012), pp. 207-215.
[5] I. Ivanić, M. Gojić, I. Anžel, S. Kožuh, M. Rimac, O. Beganović, K. Begović, B. Kosec, Microstructure and properties of casted CuAlNi and CuAlNiMn shape memory alloys, Proceedings Book 13th International Foundrymen Conference, (eds. Z. Glavaš, Z. Zovko Brodarac, N. Dolić), University of Zagreb Faculty of Metallurgy, 2013, Sisak, Croatia, pp. 153-162.
[6] Z. Wang,X. F. Liu, J. X. Xie, Effects of solidification parameters on microstructure and mechanical properties of continuous columnar-grained Cu-Al-Ni alloy, Progress in Natural Science: Materials International, 21(2011), pp. 368-374.
[7] U. Sari, T. Kirindi, Effects of deformation on microstructure and mechanical properties of a Cu-Al-Ni shape memory alloy, Materials Characterization, 59(2008), pp. 920-929.
[8] Y. Sutou, T. Omori, R. Kainuma, K. Ishida, Ductile Cu-Al-Mn based shape memory alloys: General properties and applications, Materials Science and Technology, 24(2008)8, pp. 896-901.
[9] U. S. Mallik, V. Sampath, Effect of composition and ageing on damping characteristics of Cu-Al-Mn shape memory alloys, Materials Science Engineering A, 478(2008), pp. 48-55.

[10] U. Arlica, H. Zak, B. Weidenfeller, W. Riehemann, Impact of alloy composition and thermal stabilization on martensitic phase transformation structures in CuAlMn shape memory alloys, Materials Research, 21(2018), pp. 1-6.
[11] Y. Sutou, T. Omori, J.J. Wang, R. Kainuma, K. Ishida, Characteristics of Cu–Al–Mn-based shape memory alloys and their applications, Materials Science and Engineering A, 378(2004), pp. 278-282.
[12] Y. Sutou, T. Omori, R. Kainuma, N. Ono, K. Ihida, Enhancement of superelasticity in Cu-Al-Mn-Ni shape-memory alloys by texture control, Metallurgical and Materials Transactions A, 33(2002), pp. 2817-2824.
[13] M. Reza Rezvani, A. Shokuhfar, Synthesis and characterization of nano structured Cu-Al-Mn shape memory alloy by mechanical alloying, Materials Science and Engineering A, 532(2012), pp. 282-286.
[14] H. Sakamoto, Distinction between thermal and stress-induced martensitic transformations and inhomogeneity in internal stress, Materials Transactions, 43(2002)9, pp. 2249-2255.