Preliminary results on microstructure profile of Cu-based shape memory alloy

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Abstract. Shape memory alloys represent a perfect solution for noiselessly actuation with possibility of natural activation without extra energy consume. Copper-based shape memory alloy represent a cheap solution of classical Nitinol. After melting and heat treatment the experimental alloy was mechanically polished and chemical etched in order to highlight the alloy microstructure in hardened state. In this article results about an experimental alloy microstructure relief are presented. Scanning electron microscopy (SEM, VegaTC demo) and atomic force microscopy (AFM, Nanosurf EasyScan II) were used to analyze the profile of a shape memory alloy based on copper after the annealing heat treatment.

1. Introduction

Shape memory alloys (abbreviated as SMA) have a number of properties that are distinct from conventional metallic materials. Of these, characteristic is the ability to change its geometric shape when switching from low to high [1-5]. Under certain conditions, the shape change can be reversed so that the material can store two geometric shapes, namely both high temperature (warm form) and low temperature (cold form) - these transformations are performed as a result of a memory effect shape (through this effect the material can also perform a mechanical work during the transition from cold to hot form) [6,7].

The effect of shape memory is based on the existence of a reversible phase transformation in solid state called martensitic transformation. A phase symbolized by "β" or "A" (austenitic) under the influence of temperature decrease turns into another symbolized phase "M" (martensite). Transformation β → M is called direct martensitic transformation, and M →β of reverse martensitic transformation. The name martensite was given, over a century ago, to the carbon steel product, in honor of the German metallurgist Adolf von Martens. The Cu-Al, Cu-Al-Ni, Au-Cd, Ni-Al, Cu-Zn, Cu-Zn-Al alloys are also referred as β-phase materials [8-10]. As mentioned in the literature, martensitic transformation occurs only during continuous cooling, with speeds of at least 600 °C/s, by the germination and the growth of new martensite plates (and not by the increase of the old ones), in
time intervals of the order of $10^{-7}$ s [11]. Shape memory characteristics depends on the martensite microstructure type, profile and dimensions [12].

In this article we present and comment some experimental results on sizing the martensite variants plates profile obtained after hardening heat treatment of a copper based shape memory alloy. Microstructure is a critical parameter for the training process of the shape memory alloys in order to enforce the austenite phase to transform in exactly specific martensitic variants (keeping the same macroscopic outer shape every time).

2. Materials and methods

A potential shape memory alloy made of CuZnAl was realized using classical induction furnace using high purity materials (Cu: 99.999%, Zn: 99.995% and Al: 99.9%) under argon atmosphere [13-15]. After pouring the alloy was heat treated by hardening (heating at 700 °C, 900 seconds maintaining and cooling in water+ice). An experimental sample was mechanically polished and after chemical etching with FeCl3 the microstructure relief was analysed using scanning electron microscopy (SEM VegaTescan LMH II, SE detector, 30kV, VegaT software) and atomic force microscopy (AFM, EasyScan II, non-contact mode).

The experiments were performed in accordance with the occupational health and safety laws and regulations in order to eliminate all the risks and dangers which can affect the human resource during the experiment procedures [16-18].

3. Results and discussions

The martensite formation takes place from the monocristalline phase $\beta$ through phase transformation that produces a number of martenstic domains, each with different indices of the usual planes, but crystallographic equivalents, domains that will appear distributed throughout the sample [1]. These martenstic domains are called "variants" and in a series of phase $\beta$ alloys the normals at the habitat are symmetrically grouped around the poles $\{110\}_\beta$ [19], forming 4 variants that develop into a self-supporting group [20]. In this regard, six such groups of 4 variants of martensite plates can be formed, each group corresponding to a family of compact phases of the parent phase $\beta$ [21].

Different possible groupings between the martensite variants have typical configurations on samples prepared metallographically, which allowed the establishment of a morphological classification [22,28]. Although martensite formation takes place from the monocristalline $\beta$ phase transformation produces a number of martensitic domains having each different indices of the usual but crystallographic equivalents, domains that will appear distributed throughout the sample [23]. Cu-based shape memory alloy present different specific relief type for $\beta$ phase like zig-zag, or arrow tip that are also observed in Figure 1.

The microstructural characterization of the alloys in this state refers to the relatively large primers with average lengths of 448.6 μm and widths of 202.7 μm, mediation achieved by measuring 50 plates, with the arrangement of variants of martensite in different directions determined in special grain orientation in which it is found [24-29].

Grain measurement was performed using the VegaTC specialized software provided with the electron scanning microscope (SEM) and the results are given in the order of nanometers tens. In order to follow the evolution of the dimensions of the martensite variants that characterize the microstructure of the shape-memory alloys, their measurements were made in the casting state having an average size (mediated on 50 grains and about 100 martensite variants) of 3.5 μm. In Figure 2 are presented atomic force microscopy images of CuZnAl shape memory alloy in a) 2D profile, b) 3D profile and c) mean fit of plates observed in a) area. In the 2D profile, Figure 2 a), intersection of the grains present the specific orientation of the martensite plates, the profile of the plates and the dimensions. In the 3D scan, Figure 2 b), beside primary plates we observed the secondary plates in the left side of the scan area.
Cu-Zn-Al shape memory alloy martensite plates have an internal defect substructure created by inverse planar shear, the acoustics of the plates in the austenitic matrix are made by maculation to maintain the coherence of the crystalline mesh on the austenite-martensite interface [25]. For this reason it can be appreciated that the macules observed in the microstructure of these alloys are accommodating rather than transforming. In addition, it should be noted that approx. 50% of these macules are type II, as well as Cu-Al-Ni-based alloy memories [26].

Due to the maculation process (macles ordering) the martensite from Cu-Zn-Al SMA has a characteristic superficial relief. The micrograph from Figure 2 a) shows the line limit intersection of the boundaries between the two grains. Primary martensite plates were formed on all grains in, increasing over their entire length, without crossing the boundary between them [23]. The dimensions of the martensite plates are presented in Table 1.

![Figure 1. SEM images of CuZnAl alloy](image)

**Table 1. Dimensions of the martensite plates.**

| Plates (25 measurements) | Widths [µm] | Heights [µm] | Angles between plates [°] |
|--------------------------|-------------|--------------|--------------------------|
| Minimum value            | 3.04        | 2.51         | 14.48                    |
| Maximum value            | 19.97       | 6.14         | 23.5                     |
| Mean Value               | 10.16       | 3.92         | 18.7                     |
| Standard deviation       | 4.46        | 0.85         | 2.45                     |
Figure 2. AFM images of CuZnAl shape memory alloy a) 2D profile, b) 3D profile and c) mean fit of plates observed in a).

The structure is also visible on the secondary plates, as shown in the electronic micrographs of Figures 1 and atomic force microscopy in Figure 2, where a comparison is made between the relief characteristic of the primary plates - which passes through the entire micrograph field and the martensite secondary plates which which grow and clutter between the grains limits and primary plates. The latter (secondary plates) are shorter and finer because they did not have any time or space to increase to the size of the main plates, but martensite secondary boards also have their own relief.

4. Conclusions
Microstructure is a critical parameter for the training process of the shape memory alloys in order to enforce the austenite phase to transform in exactly specific martensitic variants (keeping the same macroscopic outer shape every time). This experimental alloy presents proper structural characteristics in order to exhibit a good shape memory effect.

In this case, after hardening heat treatment, we observe and sized two types of martensite plates like zig-zag and arrows. Atomic force microscopies confirm the martensite plates profile highlighting the secondary plates formed between the primary plates (shorter and finer comparing to primary plates).

5. References
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