



Stress induced martensitic transformations and phases stability in Cu–Al–Be shape-memory single crystals

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ABSTRACT

The stress induced martensitic transformations between different metastable phases in Cu–Al–Be shape-memory alloy single crystals were assessed experimentally. In particular, the successive stress induced transformations from the high temperature austenitic DO₃ structure (β_1) to the 18R and to the 6R martensites were considered. Several distinct features which characterize the stress induced transformations in Cu–Al–Be and distinguish this system from other Cu-based shape-memory alloys are reported in this work. It was found that the 6R phase forms from a distorted 18R and that this stress induced transformation is highly reversible with no plastic deformation accompanying the process. The stress induced transformation from the distorted 18R to the 6R structure exhibits wide hysteresis, comparable with values observed in the NiTi system. A slightly negative temperature dependence of the critical stress to induce the 6R martensite was determined. Finally, a stress–temperature phase transformation diagram involving the metastable β_1 , 18R and 6R phases is proposed.

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1. Introduction

Ternary Cu-based (e.g., Cu–Zn–Al, Cu–Al–Ni, Cu–Al–Be) shape-memory alloys (SMAs) exhibit an interesting behavior related with the solid to solid transitions between different metastable phases [1–4]. They include the high temperature β_1 -phase, usually referred to as austenite, and several low temperature phases, usually referred to as martensites. The most relevant structures are named 18R, 6R and 2H [1,5,6].

The high temperature austenitic phase is obtained from the sufficiently rapid cooling of a disordered bcc structure (A2). Different atomic ordering processes might take place during cooling, depending on the exact composition of the metallic system considered [7–9]. From now on, we will use austenite or β_1 to differentiate the ordered structure obtained after cooling from the disordered one stable at high temperatures. The β_1 phase and the martensitic structure which forms from it are metastable phases. The type of martensite which is thermally or

stress induced from the β_1 austenite depends on the particular composition of the alloy being considered. As an example, in Cu–Zn–Al alloys with electron to atom concentration $e/a=1.48$, 18R martensite is formed. This martensite is characterized by a slightly monoclinic structure with a stacking period of 18 planes. Cu–Zn–Al alloys with higher e/a ratios show, instead, a transformation from β_1 into 2H martensite. The latter structure is described by an orthorhombic structure with a stacking sequence of 2 planes [2,5,6]. Both 18R and 2H martensites might also form in the Cu–Al–Ni system. In this case, the dependence of the type of martensite on the composition has been nicely shown by Recarte et al. [10], where higher Ni content is shown to favor the formation of 18R martensite.

Both 18R and 2H martensites may transform into other martensitic structures when stresses are applied. These martensite to martensite transitions are also diffusionless, solid to solid phase transformations, similar to the β_1 to martensite transition mentioned in the previous paragraph. An example of martensite to martensite transition has been reported by Otsuka et al. in Cu–Al–Ni alloys [4]. These authors have shown that further straining the 2H martensite formed from the austenitic structure results in a martensitic structure referred to as 18R₂. This structure differs from the 18R martensite in the stacking of basal planes [11]. A similar transformation was reported by Arneodo et al. to occur in Cu–Zn–Al alloys with an electron concentration per atom $e/a=1.53$ [12]. In the context of the present work, an important transition is

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the one taking place between the 18R and the 6R martensites. This transformation can be stress induced by tensile deformation of a stress-induced 18R single crystal and it has been reported to occur in Cu–Zn–Al and Cu–Al–Ni alloys [3,4,13–15]. Two characteristics associated with this transition are worth being emphasized here. On the one hand, it has been reported that the critical stress to induce the 6R martensite shows a slight or even null dependence on temperature [1,3,14,16,17]. Additionally, the reported stress hysteresis associated to the 18R–6R transition is wider than the one involved in the β_1 –18R transition. These parameters have been assessed in Cu–Al–Ni [18] and Cu–Zn–Al by several authors [15,19].

A noticeable and interesting result concerning mechanical properties of the 6R martensite in Cu–Zn–Al alloys has been reported by Cuniberti et al. [20]. These authors have shown that the critical stress to deform the 6R martensite plastically is smaller than the critical stress to induce the formation of the 6R structure. This leads to 6R plastic deformation concomitant with its own formation. This conclusion could also be inferred from previous reported results [15–17]. More recently, Bubani et al. [19] have shown that after introducing a homogeneous distribution of γ nano-precipitates in Cu–Zn–Al single crystals of electronic concentration 1.48, plastic deformation of the 6R phase can be shifted to a higher stress level. This allowed the authors to obtain the stress induced 6R martensitic structure without plastic deformation [19]. Thus, these systems are highly flexible as it is possible to use the β_1 –18R (low hysteresis) and 18R–6R (high hysteresis) individually, or the combination of both, i.e., β_1 –18R–6R (high strain), depending on prospective application requirements (actuator, damping devices, etc.).

Among the Cu-based shape-memory alloys, Cu–Al–Be alloys exhibit a distinct behavior concerning stress induced martensitic transitions. Several papers have shown that, after tensile stressing a β_1 single crystal, an 18R single crystal is formed [21–23]. More recently, it has been shown, however, that the β_1 –18R transition exhibits an increase in mechanical hysteresis and associated deformation with test temperature. In addition, a deviation from linearity in the temperature dependence of critical transformation stresses to transform and retransform is observed [24]. These tendencies allow the behavior of Cu–Al–Be to be considered anomalous with respect to other Cu-based systems. The aforementioned peculiarities could be explained by the presence of a stress induced structural distortion of the 18R martensite in Cu–Al–Be alloys. Its occurrence has been experimentally determined in a wide temperature range. It was found that the distortion takes place in an extended stress range, resembling the stress induced R-phase transformation in NiTi shape-memory alloys [25]. This stress induced distortion of the 18R phase exhibits a positive dependence of the characteristic stresses on temperature (approximately a fifth the value of the β_1 to 18R transformation), no hysteresis and a maximum associated strain close to 1%. A change in the lattice cell parameters on the basal plane, similar to what has been reported for the 2H martensite in Cu–Zn–Al single crystals [12], was suggested to explain the distortion of the 18R structure [24]. Therefore, in the Cu–Al–Be system the 6R structure is stress induced from the distorted 18R phase to which we will refer as 18R'. Additionally, preliminary results have shown that high hysteresis is also present in Cu–Al–Be single crystals and no plastic deformation takes place during transformation.

Considering these attractive properties, the formation of 6R martensite might play an interesting role from the point of view of applications in damping devices. However, a deeper understanding is required to improve the comprehension of the mechanical properties associated to stress induced transitions in this ternary system considering the significant differences reported when compared with Cu–Al–Ni and Cu–Zn–Al alloys. This manuscript focuses on the assessment of the metastable phase transformation

Table 1

Samples used in the present work and type of tests and measurements performed. A and B identify single crystals, the number that follows identifies different samples obtained with each crystal and used in the present manuscript. ER= electrical resistivity measurements (see critical transformation temperatures in the text).

Sample	ER	Temperature range of tensile tests (K)	Phase transition analyzed
A1	x		
B1	x		
A2		303–393	β_1 –18R 18R–18R' 18R'–6R
A3		303–393	β_1 –18R 18R–18R'
A4		203–293	β_1 –18R 18R–18R'
A5		243–353	β_1 –18R 18R–18R' 18R'–6R
B2		373	β_1 –18R 18R–18R' 18R'–6R

diagram of the Cu–Al–Be system. Single crystals are used for this purpose and stress induced transitions under tensile mode are analyzed in detail.

2. Experimental details

Two Cu–Al–Be single crystals with nominal composition Cu–11.4 wt% Al–0.53 wt% Be (Cu–22.63 at% Al, 3.15 at% Be) provided by Nimesis as wires 1.3 mm in diameter and 200 mm in total length were used for the present experiments. Crystals will be referred to as crystal A and crystal B. The orientation of the single crystal axes was determined by the X-Ray Laue method. It lies 7° from [001] towards [011] direction. Crystallographic Miller indexes will be referred to the austenitic β_1 structure unless specifically stated. Specimens with different lengths, 60 mm for mechanical testing and 10–20 mm for electrical resistivity measurements, were obtained from the single crystals by cutting with a low speed saw.

Specimens from crystal A were heat treated in the following way: 900 s (15 min) at 1110 K and quenched into water at 373 K. After 3600 s (60 min) at this temperature, samples were air cooled and then mechanically and electrolytically polished (7 steps, 9 V in a solution 15% of nitric acid in methanol). Crystal B samples were heat treated in this way: 600 s (10 min) at 1173 K, quenched in water at 303 K, kept at 373 K for 4 h and air cooled. Specimens were labeled by a number following the name of the crystal, i.e., specimen A1, etc. Both thermal treatments enable us to obtain the β_1 austenitic structure above room temperature and to reduce the concentration of vacancies [24].

The martensitic transformation temperatures for the heat treated condition were determined by electrical resistivity (ER) measurements by the four leads method. The following characteristic temperatures were obtained: $M_s=296$ K, $M_f=246$ K, $A_s=270$ K and $A_f=302$ K and $M_s=314$ K, $M_f=220$ K, $A_s=268$ K and $A_f=318$ K, for specimens A1 and B1, respectively (Table 1).

Mechanical tests were performed with an Instron 5567 electromechanical testing machine equipped with an Instron 3119-005 temperature chamber which allowed testing in the 203–523 K temperature range. 60-mm-long tensile specimens were cut from the original 200 mm crystals. A free length between grips of 40 mm was used. Deformation was measured with an MTS 632.13F-20 extensometer with a gage length of 10 mm attached to the central portion of the specimen. Tests were performed at a

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