

# Effects of aging on stress-induced martensitic transformation in ductile Cu–Al–Mn-based shape memory alloys

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## Abstract

Effects of aging at 473–573 K on stress-induced martensitic transformation for textured  $\text{Cu}_{71.9}\text{Al}_{16.6}\text{Mn}_{9.3}\text{Ni}_2\text{B}_{0.2}$  and random-textured  $\text{Cu}_{72.1}\text{Al}_{16.9}\text{Mn}_{10.5}\text{Co}_{0.5}$  shape memory alloy (SMA) wires with a large relative grain size  $d/D = 6$  were investigated by cyclic tensile testing at room temperature, where  $d$  and  $D$  indicate mean grain size and wire diameter, respectively. The random-textured  $\text{Cu}_{72.1}\text{Al}_{16.9}\text{Mn}_{10.5}\text{Co}_{0.5}$  wire cannot be uniformly deformed and the ductility is drastically reduced by aging treatment. On the other hand, in the textured  $\text{Cu}_{71.9}\text{Al}_{16.6}\text{Mn}_{9.3}\text{Ni}_2\text{B}_{0.2}$  SMA wire, the critical stress for martensitic transformation  $\sigma_t$  and the tensile strength  $\sigma_f$  are increased by aging without the associated loss of superelasticity (SE). Even in textured wire with a high  $\sigma_t$  of over 750 MPa, an excellent SE strain of about 6% can be obtained due to the formation of a fine bainite phase. Moreover, it was confirmed by in situ observation that stress-induced martensite plates grow, accompanying distortion of the bainite plates.

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**Keywords:** Aging; Bainitic transformation; Martensitic phase transformation; Recrystallization texture; Tension test

## 1. Introduction

Cu-based shape memory alloys (SMAs) in the Cu–Zn and Cu–Al-based systems are commercially attractive for the practical exploitation of the shape memory effect (SME) and superelasticity (SE) because of their low cost as compared with that of commonly used Ti–Ni SMAs. It is known that the recoverable strain of polycrystalline Cu-based SMAs strongly depends on the relative grain size such as  $d/t$  and  $d/D$  and increases by the increment of the relative grain size which results in decrement of the constraint strain among grains, where  $d$ ,  $t$  and  $D$  indicate the grain size, the sheet sample thickness and the wire sample diameter, respectively [1–3]. In polycrystalline Cu–Zn SMAs with huge grains, for example, a recoverable strain of about 10% has been demonstrated [4]. However, the conventional polycrystalline Cu-based SMAs are very brittle,

with intergranular fracture easily occurring, especially in those with coarse grains, and fatigue strength is very low [5,6]. Therefore, many attempts to improve the ductility and the fatigue strength of polycrystalline Cu-based SMAs have been made, mainly by grain refinement [7]. Consequently, in conventional polycrystalline Cu-based SMAs, the attainable SE strain is limited in the region below about 2%, which is insufficient for practical applications in many fields. Furthermore, poor cold-workability of the polycrystalline Cu-based SMAs is a serious drawback for the manufacture of thin sheets, fine wires, tubes and so on.

During the last decade, the present authors developed Cu–Al–Mn-based SMAs with a low Al content showing excellent cold-workability and high fatigue strength [8–10]. The enhancement of ductility in the Cu–Al–Mn-based SMAs is attributable to the low degree of order in the off-stoichiometric Heusler parent phase with an  $L2_1$  structure. Recently, it has been reported that the grain size dependence of the critical stress and the SE strain for the

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stress-induced martensitic transformation (SIMT) in Cu–Al–Mn-based SE wires with a random texture or a  $\langle 110 \rangle$  fiber texture can be predicted using a new model combining the Taylor [11] and Sachs [12] models, in which the grain constraint effect is considered and ignored, respectively [13,14]. Although Cu–Al–Mn-based SMAs with a large relative grain size show a large SE strain of over 7%, the critical stress of the SIMT  $\sigma_t$  ( $<100$  MPa), the ultimate tensile strength  $\sigma_f$  ( $<300$  MPa) of these SMAs and their fatigue strength are low in comparison with those of specimens with a smaller relative grain size [10].

The present authors have reported that the hardness of Cu–Al–Mn–Ni–B alloys drastically increases by aging at around 473–573 K due to bainitic transformation [15]. Therefore, it is expected that the mechanical strength of the Cu–Al–Mn–Ni–B SMAs can also be increased by the bainitic transformation induced by aging. In this study, in order to develop Cu–Al–Mn-based SMAs with both an excellent SE and a high mechanical strength, the effects of aging treatment at around 473–573 K on the SIMT and the other mechanical properties using tensile test for the Cu–Al–Mn–Ni–B SMAs were investigated.

## 2. Experimental procedures

$\text{Cu}_{71.9}\text{Al}_{16.6}\text{Mn}_{9.3}\text{Ni}_2\text{B}_{0.2}$  and  $\text{Cu}_{72.1}\text{Al}_{16.9}\text{Mn}_{10.5}\text{Co}_{0.5}$  alloys were prepared by induction-melting in an argon atmosphere, Ni and B being added for the development of texture [13,16] and for the control of grain size [13], respectively, and Co being added for coarsening of grain size [13]. The obtained ingot was hot-rolled at 1073 K, and then wires with a diameter  $D = 1$  mm were obtained by cold-rolling and drawing with annealing at 873 K. In the Ni-containing alloy, a strong  $\langle 110 \rangle$  recrystallization texture can be formed by the following thermomechanical treatments: (1) cold drawing down to a diameter corresponding to an area reduction ratio of over 30% after annealing in the  $\alpha$  (face-centered cubic, fcc) +  $\beta$  (body-centered cubic, bcc) region at 873 K, and (2) solution-treatment at 1173 K followed by water quenching [13]. It is supposed that the increase of the  $\alpha$  volume fraction in the annealing at 873 K due to the addition of Ni is related to the increase in the intensity of the texture after recrystallization [13,16]. Furthermore, extremely large grains can be obtained by repeating the annealing at 1173 K for 0.12 ks followed by air cooling to room temperature. Although the  $\alpha$  phase precipitated on the grain boundary by air cooling acts as an inhibitor of normal grain growth, secondary recrystallization can be accelerated by the disappearance of the  $\alpha$  phase due to re-solution treatment [17,18]. In the

present study, four cycles of such heat treatment were conducted, and finally, the wires were solution-treated at 1173 K for 0.12 ks to obtain a  $\beta$  single phase. The typical microstructure of the obtained wire is shown in Fig. 1. In both random and textured wires, the wires show a bamboo-like grain structure and the mean grain size  $d$  in the wires that is defined as the mean length of the grains along the drawing direction is about 6  $\mu\text{m}$ , i.e., the  $d/D$  is about 6. The wires were then aged at temperatures ranging from 473 to 573 K in air. The martensitic transformation temperatures were determined by differential scanning calorimetry (DSC) at heating and cooling rates of  $0.17$  K  $\text{s}^{-1}$ . The mechanical and SE properties were investigated by cyclic tensile testing using an Instron machine at a strain rate of  $0.017$  mm  $\text{s}^{-1}$ , where the gauge length was 50 mm. The microstructure change due to the SIMT was observed in situ by scanning electron microscopy (SEM), an acid ferric chloride solution (10 g  $\text{Fe}_3\text{Cl}$ , 25 ml HCl and 100 ml distilled water) being used as an etchant.

## 3. Results and discussion

### 3.1. Random-textured samples

Fig. 2a shows the stress–strain curve obtained by tensile testing at room temperature for superelastic  $\text{Cu}_{72.1}\text{Al}_{16.9}\text{Mn}_{10.5}\text{Co}_{0.5}$  wire with a random texture, the martensitic transformation start temperature ( $M_s$ ) being 224 K. The superelastic wire shows a large SE strain of over 6% and a low critical stress even in the random-textured specimen, as reported in our previous paper [13]. It is important to note that the random-textured wire is not uniformly deformed, as shown by the jagged curve of Fig. 2a. Such inhomogeneous deformation is accelerated by age-hardening. Fig. 2b shows the cyclic stress–strain curve in the  $\text{Cu}_{72.1}\text{Al}_{16.9}\text{Mn}_{10.5}\text{Co}_{0.5}$  wire aged at 523 K for 1.8 ks. It is seen that both the maximum tensile elongation and tensile stress are less than only 2% and 400 MPa, respectively. In the other specimens aged under different conditions, the mechanical properties were basically comparable to this result. It is concluded that strengthening using age-hardening is not suitable for application to random-textured Cu–Al–Mn-based wires with a large mean grain size.

### 3.2. $\langle 110 \rangle$ Fiber textured samples

Fig. 3 shows the cyclic stress–strain curves obtained by tensile testing at room temperature for as-quenched

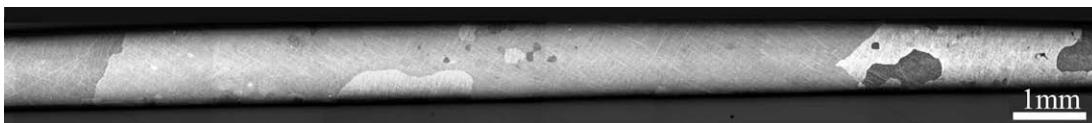


Fig. 1. Optical microstructure of  $\text{Cu}_{71.9}\text{Al}_{16.6}\text{Mn}_{9.3}\text{Ni}_2\text{B}_{0.2}$  wire with a bamboo-like grain structure.

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