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Effects of grain size on plastic deformation in a β CuAlBe shape memory alloy

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ABSTRACT

The effects of grain size on the deformation under compression of a β Cu–11.4Al–0.5Be (wt%) polycrystalline shape memory alloy were analyzed. As the maximum stress increases, major levels of pseudoelastic strain are obtained and the strain retained on unloading increases. The plot retained strain versus applied strain gives a unique curve that is independent of the grain size, with a pure pseudoelastic behavior up to around 3% of applied strain. The largest pseudoelastic strain possible to obtain increases as the grain size increases. Surpassed that strain, plastic deformation occurs and the macroscopic plastic stress follows a Hall–Petch type relation.

In samples with plastic deformation the reverse martensitic transformation occurs in two stages, with a second high temperature calorimetric peak associated to the transformation of plastic deformed martensite.

1. Introduction

The CuAlBe alloys, based on the CuAl system, are receiving high attention, because they exhibit shape memory properties due to a thermoelastic martensitic transformation. Small additions of Be on the Cu–11Al (wt%) system produce an important lowering of the martensitic transformation critical temperature [1], which allows a larger temperature range at which the shape memory effect is operative. In CuAlBe shape memory alloys, the β phase is stable at temperatures higher than 870 K [1], but it can be retained at lower temperatures by rapid cooling [2]. Upon quenching, the metastable β phase becomes ordered and then transforms to 18R martensite [3]. However, the martensitic transformation can also be induced by the application of mechanical stress, producing the pseudoelastic effect. Under appropriate conditions, when the load is removed, the deformation is almost completely recovered and a hysteretic loop is formed, which is associated with energy dissipation. Because of these properties, the use of CuAlBe alloys as passive dampers of seismic energy is highly promising [4,5].

Studies about the pseudoelastic effect of CuAlBe alloys have been reported [6–13]. In a previous work we analyzed the deformation of a CuAlBe polycrystalline alloy with a grain size of 0.5 mm [6]. Compression cycles up to increasing loadings at room temperature were carried out, and a macroscopic plastic limit of around 600 MPa was determined. Such determination was performed through a direct and precise evaluation of the involved deformations, achievable under compression tests. It has been found in CuAlBe alloys that the pseudoelastic behavior is affected by the grain constraint [7], so the martensite-start stress, the pseudoelastic slope and the stress hysteresis decrease as the grain size increases [7,14]. It is well known that in the single phase metallic alloys, the yield stress decreases as the grain size decreases due to the grain boundaries are important obstacles to slip, and the relationship of the yield stress with the grain size is often expressed by the Hall–Petch relation [15,16]. Studies on the relationship between the grain size and the plastic limit in shape memory alloys are scarce, possibly due to the difficulties introduced by the overlapping of pseudoelastic strain and plastic strain. Paradkar et al. [17] has demonstrated the validity of the Hall–Petch relationship for a two phase TiAlNb shape memory alloy, while Khan et al. [18] deformed CuAl in martensitic state and also found it follows that relationship.

In the present work, we study the effects of grain size on the deformation of a β Cu–11.4Al–0.5Be (wt%) polycrystalline alloy. Samples were subjected to compression cycles at room temperature. The influence of the grain size on the strain components and the plastic limit was analyzed.

2. Experimental procedure

A β Cu–11.4Al–0.5Be (wt%) polycrystalline alloy was used in the present work. The chemical composition was determined by

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atomic absorption spectrophotometry. Cylindrical samples around 5.1 mm diameter and 14.3 mm length were prepared for compression tests, with a length/diameter ratio ~2.8. The grain size in the as-received specimens was 0.05 mm. To obtain different grain sizes, samples were kept in a resistance furnace at 1073 K for different times and water quenched at room temperature. For light optical microscopy observations, the samples were electropolished in a satured solution of chromium trioxide in phosphoric acid at 4 V, and immersed for a few seconds in a solution of ferric chloride in order to reveal the microstructural details. Mean grain size (d) was estimated by an area analysis method from optical micrographs [19]. The area of each grain was determined using the software Image Tool 3.0, and the diameter of each grain was estimated as the diameter which would correspond to the measured grain area if they had a circular form. d was obtained as the average value for each sample, considering at least three micrographs. The frequency distributions of the grain size/mean grain size obtained for the different thermal treatment times are essentially similar, with a departure of around ± 1 from the maximal frequency point. Compression tests were performed with a Shimadzu Autograph-DSS-10T universal testing machine at room temperature, and a constant cross-head speed of 1 mm/min. The end faces were covered with a thin Teflon film and lubricated with grease in order to reduce the friction. The stress–strain curves were registered with (i) the paper recorder of the testing machine, (ii) a high-speed digital acquisition system, and the length of the samples in each condition was also measured with a precision digital caliper, $\Delta = 5 \times 10^{-3}$ mm.

From samples subjected to compression, small discs of around 1 mm thick and mass of ~200 mg were cut. Calorimetric measurements were performed with a Rheometric DSC calorimeter at 10 K/min.

3. Results and discussion

Representative stress–strain (σ–ε) curves of samples with different grain sizes are shown in Fig. 1. The first linear part in the loading curve corresponds to the elastic regime of the β phase. The linearity deviation is associated with the beginning of the martensitic transformation, and the stress at this point will be referred hereafter as the martensite-start stress (σ_s). Then, the martensitic transformation progresses, however, other deformation mechanisms could act as will be discussed. A hysteretic loop is formed on removing the load. For cycles at low maximum stresses the strain is almost fully recovered leading to the pseudoelastic (PE) behavior. For a sample with a mean grain size of 0.5 mm, a pure pseudoelastic behavior has been obtained up to around 300 MPa [6]. The grain size of the samples modifies the stress-strain cycles. σ_s decreases as the grain size increases, as we reported in previous works [7,14]. Beyond σ_s, two hardening stages are observed, more marked in the samples with high grain size. This could be understood considering that the largest grains will transform first at lower loads, while the smaller grains do at higher loads [20].

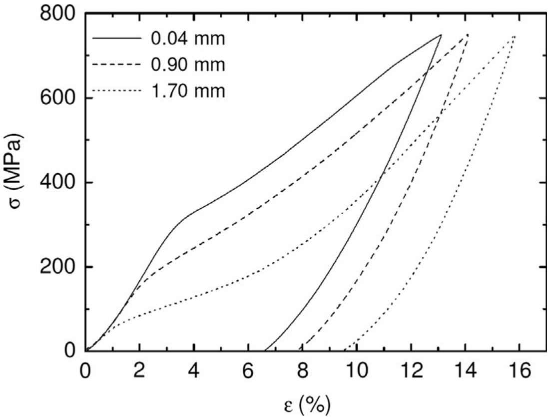

Fig. 1. Stress–strain curves for samples in the β phase with different grain sizes subjected to single cycles of compression.

Micrographs of samples with different grain sizes obtained at room temperature after a single cycle of compression at 750 MPa are shown in Fig. 2. A large amount of martensite plates are observed in the samples, which could correspond to retained and/or plastically deformed martensite. In the samples with small grain size a fine network of martensite plates inside each grain is observed (Fig. 2a), while in those with high grain sizes, large martensite plates forming a grid network can be appreciated (Fig. 2b). It is interesting the observation reported on that point for martensitic CuAl and CuAlZn alloys by Khan et al. [18], who note that the martensite plate thickness (d_{mart}) increases for larger grain sizes, and propose an empirical relationship according to which d_{mart} is proportional to $d^{1/3}$.

To analyze the deformation processes occurring under loading, repeated cycles up to increasing levels of maximum stress (σ_m) in samples with different grain sizes were carried out. With the purpose of distinguish the reversible and irreversible deformation processes, samples were kept for 30 s in a resistance furnace at 1073 K after each cycle, reaching a temperature of around 550 K, and water quenched. For the analysis of the curves, the same methodology explained in reference [6] was used. The maximum strain can be considered as

$$\varepsilon_{max} = \varepsilon_{el} + \varepsilon_{ps} + \varepsilon_{pl} \tag{1}$$

where ε_{el} is the elastic deformation of the β phase, ε_{ps} is the pseudoelastic strain produced by the stress-induced martensite, and ε_{pl} is the strain associated with plastic deformation. The sum of the last two strains will be considered hereafter as the total applied strain (ε_{app}). The contributions to the maximum strain are indicated in Fig. 3a. On removing the loading, the induced martensite could not completely revert to the β phase, leaving retained strain (ε_{ret}). The strain associated to retained martensite is reversible under the flash heating, whereas if some plastic deformation takes place it is not recoverable, so, ε_{pl} will be considered as the measured strain after flash heating, ε_{TT}. Representative measured strains obtained for samples with grain sizes of 0.04 mm and 2.20 mm are shown in Fig. 3b. The stress levels needed to induce the martensite transformation and consequently ε_{app} decrease as the grain size increases. For all the specimens, as σ_m increases, major levels of ε_{ps} are obtained, and ε_{ret} increases. A high recovery is produced by the heat treatment with ε_{TT} values lower than 0.5% up to a certain stress level, above which ε_{TT} increases.

The retained strain levels depend on both the maximum stress and the grain size of the samples (Fig. 3b). However, it was found that the plot ε_{ret} versus ε_{app} for the samples with different grain sizes gives a unique curve that is independent of the grain size, as can be seen in Fig. 4a. This $\varepsilon_{ret} - \varepsilon_{app}$ relationship is an interesting resource for the estimation of the expected retained strain after applying certain total strain in samples with any grain size. For ε_{app} lower than around 3%, ε_{ret} is almost zero and a pure pseudoelastic behavior with closed cycles is obtained. For higher ε_{app}, ε_{ret} continuously increases. The maximum strain associated to the stress-induced martensite that is reversible by unloading and heating (ε_{ps} limit) is shown in Fig. 4b. As the grain size increases, ε_{ps} limit also increases, from ~6% up to around 9% for the highest grain size. This last value is comparable with the deformation of 9.7% product of the formation of a single martensitic variant with a Schmidt factor of 0.482 reported on a CuAlBe single crystal [8].

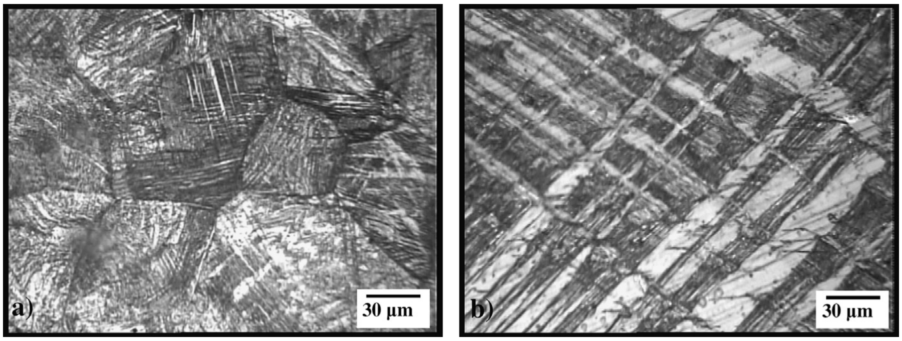

Fig. 2. Optical micrographs of samples with different grain sizes, 0.04 mm (a) and 0.90 mm (b), after a single cycle of compressions at 750 MPa.

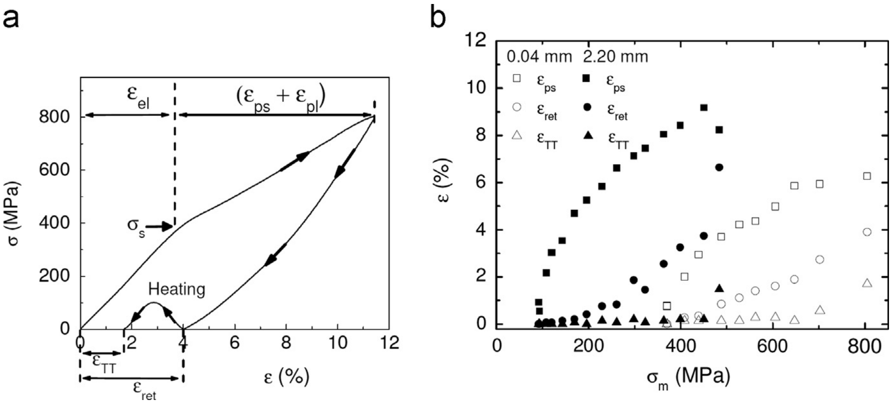

Fig. 3. Samples subjected to compression cycles up to increasing level of σ_m and flash heatings: (a) Stress–strain cycle up to 805 MPa with a posterior heating for a sample with $d=0.04$ mm. (b) Measured strains obtained from samples with different grain sizes.

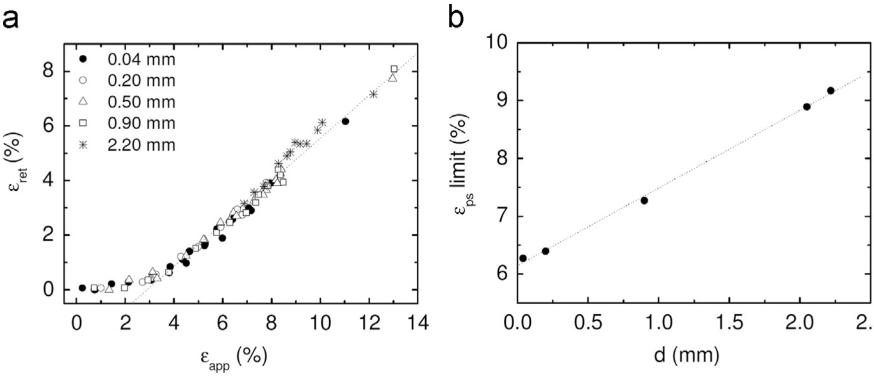

Fig. 4. Analysis of the strain components in sample with different grain sizes: variation of ε_{ret} with ε_{app} (a), and ε_{ps} limit with d (b). The dotted lines are for reference.

The increase of ε_{ret} is attributed to the increase of the martensite retained on unloading [6], which retransform to the β phase with the flash heating. However, for high stress levels ε_{TT} drastically increases, denoting the presence of irreversible plastic deformation. This behavior is shown in Fig. 5a. The stress level which determines the maximum fully recoverable strain is considered as an estimation of the macroscopic plastic limit (σ_{plast}). As can be observed in Fig. 5a, that limiting stresses are dependent on the grain size.

For polycrystalline materials, the dependence of the yield stress on grain size is often expressed by the Hall–Petch relation [15,16]:

$$\sigma_{plast} = \sigma_0 + Kd^{-1/2} \tag{2}$$

where σ_{plast} is the yield stress, σ_0 is the resistance of the material to dislocation motion, K is an empirical factor and represents the dislocation locking stress parameter, and d is the average grain diameter. The obtained values of σ_{plast} as a function of the inverse square root of grain size are shown in Fig. 5b. It is seen that $\sigma_{plast} - d^{-1/2}$ exhibits a very reasonable linear behavior, indicating that the plastic behavior of CuAlBe alloys follows a Hall–Petch type relationship with grain size. As we have analyzed in a previous work [6], the irreversible deformation introduced in the CuAlBe studied alloy at room temperature would correspond to plastic slip in the 18R martensitic phase. The β phase would deform at higher stresses than those here obtained [6,8,21,22]. The Hall–Petch relationship has been previously applied to a martensitic Cu–11.56Al (wt%) alloy [18], with $d=0.8$–1.2 mm, and a value of $\sigma_0 = 192$ MPa has been obtained. For CuAlBe, we obtain $\sigma_0 = 500$ MPa, a value more than twice that obtained for CuAl, denoting an additional strength introduced by the Be addition.

It has been reported for a β TiAlNb [17] and a martensitic NiAlFe [23] shape memory alloys that the plastic limit corresponds with a change in the slope in the σ–ε curve. However, if we compare the determined σ_{plast} for the CuAlBe studied alloy with the curves of Fig. 1, it is no applicable in this case.

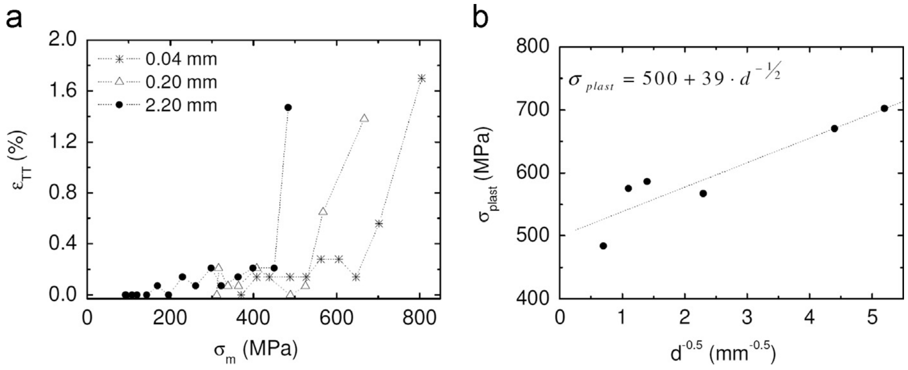

Fig. 5. Variation of ε_{TT} with σ_m (a) and σ_{plast} with $d^{-0.5}$ and (b) for samples with different grain sizes subjected to compression tests at room temperature.

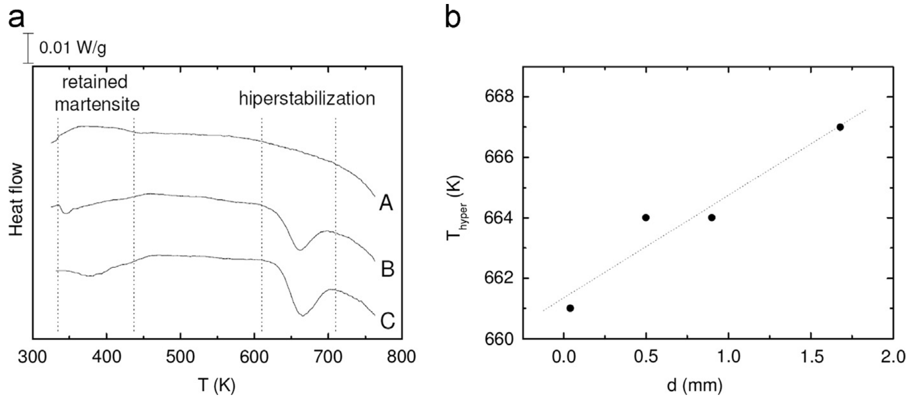

Fig. 6. (a) Heating DSC curves of sample without plastic deformation (A), and with plastic deformation introduced by compression test and different grain sizes: 0.04 mm (B) 0.90 mm (C). (b) Variation of T_{hyper} with the grain size. The dotted line is only for reference.

In order to study the influence of the grain size on the reverse martensitic transformation on samples with plastic deformation, heating DSC curves were carried out on samples with different grain sizes and ε_{pl} around 3% introduced by compression tests. DSC curves of a sample without deformation and a grain size of 0.7 mm as a reference (sample A), and samples with plastic deformation and two representative grain sizes (samples B and C) are presented in Fig. 6a. Sample A corresponds to the β phase, and samples B and C are a mix of β phase, retained martensite and plastic deformation, like that shown in Fig. 2. As is expected, no peak is observed in sample A, while the reverse martensitic transformation occurs in two stages in samples with plastic deformation. A first weak endothermic event is observed in the range 330–430 K, corresponding to the partial transformation of retained martensite [6]. The second and significant endothermic event occurs at around 600–700 K. It is associated to the transformation of the so-called hyperstabilized martensite plates [9,24], consequence of the dislocations introduce under compression. It is important to note about this our observation regarding hiperestablization [6], establishing that it is only appreciable in samples with plastic deformation. The areas under the hyperstabilized martensite transformation peak in samples with different grain sizes present some dispersion. However, we do not found a marked tendency of the area with the grain. Nevertheless, as is shown in Fig. 6b, the temperature corresponding to the peak (T_{hyper}) continuously increases as the grain size increases.

4. Conclusions

The effects of grain size on the deformation behavior under compression of a β Cu–11.4Al–0.5Be (wt%) polycrystalline shape memory alloy have been analyzed.

The grain size of the samples modifies the stress–strain cycles. For higher grain sizes, σ_s decreases and the stress–strain slope presents two hardening stages, where the first lower slope can be associated with the martensitic transformation of the larger grains. As the maximum stress increases, major levels of ε_{ps} are obtained and ε_{ret} increases, so the retained strain levels depend on both the maximum stress and the grain size of the samples. However, it was found that the plot ε_{ret} versus ε_{app} gives a unique curve that is independent of the grain size, with a pure pseudoelastic behavior up to around 3% of ε_{app}. As the grain size increases, the maximum strain produced by the stress-induced martensite, reversible upon unloading and heating, also increases. It was found that the macroscopic plastic limit follows a linear relationship with the inverse square root of grain size, obeying the Hall–Petch relation.

In samples with plastic deformation the reverse martensitic transformation occurs in two stages. A first weak DSC peak in the range of 330–430 K corresponds to the partial transformation of retained martensite. A second and important DSC peak observed at around 600–700 K is associated to the transformation of hyperstabilized martensite plates, consequence of the plastic deformation. It was found that the second peak temperature continuously increases with the grain size.

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