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Thermal treatments and transformation behavior of Cu-Al-Be shape memory alloys

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Abstract

Among the different Cu-based shape memory alloys, the Cu-Al-Be family exhibits a particular technological interest for intermediate and low temperature applications. In this work we studied the martensitic transformation behavior of Cu-Al-Be single crystalline shape memory alloys with three different concentrations. The influence of four specific thermal treatments of quenching and aging on the transformation temperatures has been studied by differential scanning calorimetry and compared with similar studies by mechanical spectroscopy. The analysis of the influence of thermal treatments on the martensitic transformation behavior allows optimize a treatment useful for all the alloys transforming between 200 K and 400 K, avoiding both stabilization and precipitation. In addition, an anomalous jerky behavior has been reported for the alloy with low transformation temperatures, which is discussed in terms of the possible mechanisms.

Keywords: Metals and alloys, shape memory, phase transitions, calorimetry.

1. Introduction

Shape memory alloys (SMA) undergo a reversible thermoelastic martensitic transformation (MT) between a high temperature phase β , called austenite, and a low temperature phase, called martensite [1]. This MT is responsible for the specific thermo-mechanical properties of SMA such as superelasticity, shape memory and high damping [1, 2]. Although Ti-Ni [3] is the most broadly spread SMA for technological applications, Cu-based alloys are being developed as alternative SMA because they offer a broad range of transformation temperatures up to 200°C, a large superelastic window, small thermal hysteresis (even less than 10 K) with a rather large recoverable shear strain up to 10%, as well as high damping coefficient. The main Cu-based SMA are coming form the Cu-Al binary system [4] to which several ternary alloying elements have been added to decrease the extremely high transformation temperatures of the binary alloy and for a better control of the MT and the thermo-mechanical properties. Among the different ternary Cu-based SMA, Cu-Al-Be family exhibits a particular technological interest for intermediate and low temperature applications. Indeed, the addition of Be decreases the transformation temperatures without modifying the concentration of the eutectoid point, which at the same time is lowered by this element. This allows designing alloys for a broad range of transformation temperatures, while maintaining a good thermal stability against overheating.

Cu-Al-Be SMA were initially developed by Higuchi et al. [5, 6], but the most systematic work for developing these alloys was done by Belkahla and Guenin [7-10], who reported the dependence of Ms on Al and Be concentration, Ms(°C)=1245-71%Al-893%Be (wt%), and found that 0.5wt% of Be decreases the eutectoid line by about 50°C. The high temperature β -phase undergoes only one disorder-order phase transition, at about 800 K, from A2 to DO₃ cubic ordered phase according [11, 12], and although there were some controversy because a L2₁ order was also suggested [13, 14], DO₃ order was further evidenced [15], being accepted at present. Then the high temperature β phase transforms by quenching in a β_1 R-18 monoclinic martensite [16]. The main problem of Cu-Al-Be SMA is perhaps the strong tendency to martensite stabilization, which destroy the reversibility of the transformation and as a consequence

all the thermo-mechanical properties of SMA. This is a common problem of Cu-based SMA, and many works were devoted to its study in several alloys and particularly in Cu-Zn-Al, where carefully studies have been performed and recently overviewed [17, 18]. In what concerns Cu-Al-Be, the stabilization after direct quench in martensite was reported [19, 20], being investigated in single crystals and polycrystals [21] and under different conditions as for instance athermal stabilization [22], hyperstabilization [23], and instantaneous stabilization [24]. However, in spite of the number of works on stabilization, there is not yet a clear solution to solve this problem and diverse thermal treatments were applied in the literature, without finding an optimal treatment, which could be systematically used for different alloys exhibiting a broad range of transformation temperatures, as required for technological applications. The aim of the present work is pointing in this direction and several thermal treatments have been tested on three different Cu-Al-Be alloys, and in each case the martensitic transformation and the stabilization phenomena have been analyzed. The results allow us to design a specific thermal treatment, which could successfully work in a broad range of experimental conditions.

2. Experimental methods

For the present study, single crystals of three different Cu-Al-Be alloys have been used. The nominal compositions were 11.4 wt% Al, for all the alloys and 0.46, 0.34 and 0.30 wt% Be, for each one the alloys, and they were chosen to exhibit the transformation temperatures below RT (CAB200), around RT (CAB300) and well above RT (CAB400) respectively. Samples were initially annealed at 1073 K in argon atmosphere during 1800 s and then four different treatments were used.

Treatment IQ: Quenching in iced water at 273 K (IQ).

Treatment IQA: Quenching in iced water (IQ) and then aging in an oil bath at 453 K during 24 hours (IQA).

Treatment BQ: Step-quenching in boiling water at 373 K (BQ).

Treatment BQA: Step-quenching in boiling water (BQ) followed by an up-quench into the oil bath at 453 K followed by an aging at this temperature during 24 hours (BQA). Let us comment the relative interest of the treatment above defined. Treatment IQ is a very fast quenching, which should freeze a high concentration of quenched-in

vacancies, that for the used temperatures and according [25], can be estimated close to 4×10^{-4} . At the same time this quench will partially inhibit the ordering process to DO₃ from the disordered A2 phase, in which the high temperature annealing takes place. Then the goal of the further aging in treatment IQA is to anneal the quenched-in vacancies and allows the meta-stable β phase to reach the equilibrium DO₃ order before transforming into martensite. Treatment BQ is a slow quenching in comparison with IQ, and data from quenching experiments [26] allow estimate that, for a typical sample of 1mm thickness, the cooling rate when quenching in boiling water is about 20 times slower than a quenching in iced water. So it is expected that treatment BQ should freeze a small vacancy concentration and promote ordering during cooling, but boiling water quench is still 5 to 10 times faster than air-cooling, avoiding precipitation problems. However, it is obvious that the sample will not be in equilibrium conditions after treatment BQ and if we cool down to RT, the required time to reach the equilibrium of both vacancies [25] and atomic order [27], would be very long (above one week) and eventually in martensitic state. Consequently the goal of treatment BQA is to solve this problem and allow reaching the equilibrium conditions of the β phase in a reasonable time.

The martensitic transformation after each one of the previous treatments has been studied by differential scanning calorimetry measurements (DSC), and three MT cycles have been carried out by means of a TA DSC Q2000 equipment using a 10 K/min temperature rate. The data used for further analysis are those from the third cycle. In some cases, comparative measurements of internal friction have been performed because of the sensitivity of this technique to the transformation kinetics [28]. In those cases we used a mechanical spectrometer working in sub-resonant torsion mode between 100K and 600K at different frequencies between 10⁻³ and 10 Hz, at an oscillating amplitude of $\varepsilon_m=2x10^{-5}$ and with a heating-cooling rate between 0.5 to 2 K/min [29]. The martensitic transformation has been also observed by optical microscopy in a Leica DM RXA2. All the studied samples show a martensite microstructure formed by self-accommodated groups of variants typical from the β_1^i R-18 monoclinic martensite, as shown in Figure 1.

3. Results and discussion

The DSC curves presented in Figure 2 correspond to the forward and reverse martensitic transformation of the three Cu-Al-Be studied alloys, CAB200, CAB300 and CAB400 after the treatment IQ. Samples with low transformation temperatures CAB200 and CAB300 show an apparently good martensitic transformation, while the transformation in CAB400 alloy has been practically inhibited. This is an expected result because a fast quench directly into martensite, freezing a high concentration of quenched-in vacancies, gives place to an instantaneous stabilization largely reported in the literature [19, 24, 30] The transformation temperatures M_s, M_f, A_s and A_f have been measured at 2% and 98% of the corresponding transformed mass fraction curve obtained by integration of the heating flow during the martensitic (MT) and the reverse (RT) transformations. To follow the influence of the thermal treatments on the transformation and peak temperatures as well as the transformation enthalpies during MT and RT, these DSC data have been reported on Table 1 for the three studied alloys and for the four previously described treatments.

The second treatment IQA, corresponding to an annealing at 453 K, has diverse influence on the three samples. The effect of aging on the CAB200 sample is a slight shifting (about 8 K) of the whole transformation towards low temperature, as is shown in Figure 3. However, this shifting with aging was not observed for the sample CAB300 and cannot be analyzed for sample CAB400, which remains stabilized after quenching. In order to discard a possible artefact on the sample manipulation as responsible for the shift in Figure 3, we performed the same treatments on a macroscopic single crystal of CAB200, with 1.9 mm diameter and 50 mm long, and carried out internal friction measurements. The internal friction spectra during forward and reverse transformations after both treatments IQ and IQA are plotted on Figure 4. The results are very clear and confirm the shifting to low temperatures after the annealing of IQA treatment. A possible explanation could be that the aging promotes the atomic ordering DO₃, that was partially inhibited by the fast quenching, and this ordering process decreases the Gibbs free energy of the metastable β phase, which becomes more stable and consequently shifting the martensitic transformation towards low temperature. However we have not yet understood why the same effect is not observed on sample CAB300 and hence the previous explanation should be regarded with some suspicion. We have to comment another point concerning the shoulder observed in the high-temperature side of the DSC curves during both forward and reverse transformations. This shoulder

means that some amount of β phase transforms into martensite at higher temperature, and we have verified that it is linked to oxidation problems during the high temperature annealing. Indeed, the formation of a beryllium oxide at the surface of the sample produces a small depletion of beryllium beneath the oxide film, given place to some amount of material with poorer beryllium concentration and consequently exhibiting higher transformation temperatures. This effect is noticeable due to the strong sensitivity of the transformation temperatures to the beryllium concentration, being more pronounced for the richer beryllium alloys. This deleterious effect is suppressed when preventing oxidation of Cu-Al-Be alloys during thermal treatments. The DSC curves for the forward and reverse transformation of the three alloys after the slower quenching BQ are presented in Figure 5. The most outstanding difference is the presence of a very good transformation, with an enthalpy of 10 J/g, on the sample CAB400, which for previous treatments IQ and IQA was almost completely stabilized. In what concerns samples CAB200 and CAB300 the transformation is also well developed and when looking at Table 1 it can be realized that their transformation enthalpies of 5.04 J/g (CAB200) and 8.05 J/g (CAB300) are higher than the enthalpies measured after the treatment IQ or IQA, which were about 3 J/g and 6.4 J/g respectively. This means that after the IQ treatment martensite was stabilized not only in sample CAB400 but became also partially stabilized in samples CAB200 and CAB300. Indeed the partial stabilization of sample CAB300 could be expected and can be easily understood because the quenching temperature, 273 K, is below its Ms and consequently the amount of transformed martensite during quenching became immediately stabilized due to the pinning of the interfaces by quenched-in vacancies [17, 18], and a further aging during IQA treatment does not amend such behaviour. What is more difficult to understand is why the sample CAB200 became also so strongly stabilized after the IQA treatment, because the sample remains all time above Ms, this means in β phase, and in this case it should be expected that the quenched-in vacancies in β phase will anneal during aging at 453 K promoting in addition the development of the DO₃ order. It seems that this is not the case and there would be some other mechanism being responsible for the stabilization of this alloy. A possible explanation could be the formation of complex defects from the quenched-in vacancies, which may agglomerate to form voids or prismatic dislocation loops with further negative effects on the martensitic transformation.

The last kind of treatment, BQA, consists in aging at 453 K after quenching in boiling water. The effect of this aging on the CAB200 alloy is the same than the observed after IQA, this means a slight shifting of the whole transformation towards low temperatures, as it is shown in Figure 6a. Once again this effect is not observed in CAB300 neither in CAB400 for which the main part of the transformation remains at the same temperature before and after aging, as indicated in Table 1. We may conclude that the alloy CAB200 exhibits an anomalous behaviour in comparison with the other two alloys. As commented before, at the present state it is difficult give a definitive explanation about the origin of this different behaviour, but looking the curves of Figure 6a it can be remarked that CAB200 is the only alloy exhibiting a clear jerky behaviour, at least in the first half of the martensitic transformation, which has been enlarged in Figure 6b for a better observation. This is not the usual behaviour for the β_1 18R monoclinic martensite, and so we may wonder if for this alloy composition there could be present another kind of martensite, in particular γ'_1 2H orthorhombic martensite, as was already suggested in [31] from stress-induced transformation experiments in a very similar Cu-Al-Be alloy. However, as far as we know, this jerky behaviour has not been reported for the thermal forward transformation in non-stabilized Cu-Al-Be alloys, and it deserves further investigations. Apart from that, the BQA treatment does not seem to have any significant effect on the martensitic transformation in alloys CAB300 and CAB400, but just a slight narrowing of the global transformation cycles, which are plotted in Figure 7 for the three alloys after BQA treatment. Obviously the BQA treatment should promote a complete evolution towards the meta-stable equilibrium conditions of the β phase, which will be free of quenched-in vacancies and with a DO₃ order as good as the stoichiometry of the alloy would allows. This result agrees with the one previously obtained [24] in step-quenched alloy at 440 K and further aged during 1 hour. So the transformation cycles of the three alloys, presented in Figure 7, are rather narrow and very stable, indicating that the BQA treatment is a good one for conditioning the Cu-Al-Be alloys in a broad range of concentrations or, what means the same, in a broad range of transformation temperatures.

4. Conclusions

It may be concluded that the thermal treatment BQA, corresponding to solid solution annealing at 1073 K and then a step-quenching at 373 K followed by an up-quenching to 453 K and aging 24 hours at this temperature, is a good treatment for conditioning Cu-Al-Be shape memory alloys undergoing the martensitic transformation in a very broad range of temperatures, from below 200 K to above 400 K. A particular care must be taken to avoid oxidation during the initial high temperature annealing. The Cu-Al-Be alloy with low transformation temperatures exhibits an anomalous jerky behaviour, which could be attributed to the partial contribution of γ'_1 martensite coexisting with the usual β'_1 martensite, but this point requires further research.

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Figure captions:

Table 1: Data measured for the three Cu-Al-Be alloys CAB200, CAB300 and CAB400, and for the four different performed thermal treatments IQ, IQA, BQ and BQA. In addition to the transformation temperatures, the temperature of the peak is reported for the forward (MT) and the reverse (RT) transformations. The reported enthalpy is the mean value of the enthalpy measured during forward and reverse transformation.

Figure 1: Optical micrograph of a sample of Cu-Al-Be (CAB300) showing the shelfaccommodated group of martensite variants typical of monoclinic β_{i} martensite.

Figure 2: DSC thermograms corresponding to the third cycle of the forward and reverse martensitic transformation for the three studied Cu-Al-Be alloys, after the thermal treatment IQ, iced water quenching from 1073 K.

Figure 3: DSC thermograms for the third transformation cycle during cooling (blue and green) and heating (red and magenta) for the Cu-Al-Be alloy CAB200 just after IQ treatment (blue and red dots) and after aging at 453 K for 24 hours, IQA treatment (green and magenta squares). The annealing produces a clear shifting towards low temperature of the whole transformation.

Figure 4: Internal friction measurements at 1 Hz on the Cu-Al-Be single crystal alloy CAB200, just after quenching IQ (blue and red diamonds), and after aging at 453 K for 24 hours, treatment IQA (violet and green dots). The shifting of the martensitic transformation because of the aging is confirmed.

Figure 5: DSC thermograms corresponding to the third cycle of the forward and reverse martensitic transformation for the three studied Cu-Al-Be alloys, after the thermal treatment BQ, step quenching in boiling water at 373 K from 1073 K. This figure should be compared with Figure 2 corresponding to the IQ treatment. In this case the MT of the CAB400 alloy is not inhibited.

Figure 6: a) DSC thermograms for the third transformation cycle during cooling (blue and green) and heating (red and magenta) for the Cu-Al-Be alloy CAB200 just after BQ treatment (blue and red dots) and after aging at 453 K for 24 hours, BQA treatment (green and magenta squares). b) Enlarged area of the forward transformation of figure (a), to appreciate in more detail the jerky behavior of the martensitic transformation in this alloy.

Figure 7: Transformed mass fraction curves calculated from the integral of the DSC thermograms measured in the three Cu-Al-Be alloys, CAB200, CAB300 and CAB400, after the treatment BQA. The transformation cycles during cooling and heating are stable, evidencing that this treatment is well adapted to the alloys transforming in a broad range of temperature.

Optimized thermal treatments made Cu-Al-Be shape memory alloys ready for technological applications.

A specific thermal treatment has been designed for conditioning different Cu-Al-Be shape memory alloys in a large range of transformation temperatures.

An anomalous jerky behavior on the thermal transformation of Cu-Al-Be shape memory alloys is reported at low temperature.

As (K) Sample and Ms (K) Mf (K) Af (K) Peak Peak Enthalpy MT (K) treatment RT (K) (J/g) 3.56 CAB 200 IQ CAB 200 IQA 3.66 CAB 200 BQ 5.35 CAB 200 BQA 5.41 CAB 300 IQ 6.30 CAB 300 IQA 6.58 CAB 300 BQ 8.05 CAB 300 BQA 8.02 CAB 400 IQ 1.20 CAB 400 IQA 1.31 CAB 400 BQ 10.03 CAB 400 BQA 9.80

Table 1



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