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SOME ASPECTS OF THE TWO WAY SHAPE MEMORY EFFECT INDUCED BY PSEUDOELASTIC CYCLING IN Cu-Zn-Al ALLOYS

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Abstract: The two-way shape memory effect (TWSME) induced by pseudoelastic cycling in Cu-Zn-Al single crystals has been studied. We analyzed the influence of the mechanical cycling characteristics (tensile or compressing) and of the presence of γ-phase precipitates in the material. The two-way memory effect induced by compressing cycling appears after a smaller number of cycles than in tensile pseudoelastic cycling. The presence of γ-phase precipitates also reduces the number of cycles needed to get the TWSME. Samples with TWSME are able to overcome some external stress when going to the martensitic shape until a *threshold stress*, σ_{th}, value is reached. When higher opposing stresses are applied, the degradation of TWSME starts.

1. Introduction

The shape change produced in the direct and reverse thermally induced martensitic transformations without any applied stress is known as the TWSME and must be induced with a thermomechanical treatment [1,2]. Pseudoelastic cycling through the martensitic transformation at a constant temperature is one of the available methods to induce this effect. During this treatment, dislocations are generated, as it has been reported [3]. These dislocations are mostly of mixed character, with Burgers vector and line lying on the plane which transforms to the basal plane of the stress induced martensite variant. Their presence is the most visible change inside the material after pseudoelastic cycling and thus these dislocations are thought to be responsible for the TWSME [4]. This is supported by the fact that their energy is lower in the stress induced variant than in the other ones or in the beta phase [5,6]. Surface defects are also formed but no relation between them and TWSME has been found [5,7].
It has been reported that some differences arise if the stress cycling is performed in tension or compression [7,8]. We mention here only that the dislocation arrangements are not the same. Dislocation bands parallel to the habit and basal plane are mostly found after tensile cycling while a more regular distribution of dislocations is found after compression cycling. This implies that internal stresses

could not be the same after both types of mechanical treatments. This should also have an influence on the TWSME. Some results to evaluate this point will be presented here.

On the other hand, the introduction of γ-precipitates in a beta phase matrix, affects the martensitic transformation. One of the consequences is a shift in the martensitic transformation temperatures, which changes with the size of the precipitates [9-11]. Preliminary experiments to evaluate the effect of precipitates on the TWSME are also presented in this work.

2. Experimental procedure

Single crystals of composition 68.0%Cu- 15.7%Zn - 16.3%Al (%at, e/a=1.48, nominal Ms -10ºC) were grown by the Bridgman method. Metals of 99.999% purity have been used. The crystals were spark-machined in order to give them the appropriate shape for tensile and compressing experiments. The compressing specimens were cylindrical (5 mm diameter and 10 mm length). The tensile samples had thicker ends in order to insert them into the tensile grips. The central part had either a square (3 mm side) or circular (3 mm diameter) section. The samples were annealed for 20' at 850 ºC and then air cooled to room temperature (TT1) or annealed for 20' at 850 ºC, cooled to 520 ºC and quenched into ice-water at 0 ºC (TTB). The latter thermal treatment was performed with a thermocouple welded on the specimen in order to get a good control of quenching temperature. This treatment generates a dense distribution of small γ-phase precipitates (size~10nm) coherent with the matrix. Subsequently, the samples were polished mechanically and electrolytically in a solution of 15% nitric acid in methanol. The tensile or compressing axis orientation was determined by means of X-ray diffraction (Laue method). The axes of several samples used in this study are shown in figure 1. Fatigue testing was carried out in a universal testing deformation machine (Instron model 1123) at constant temperature ~±1ºC, in a temperature chamber. The samples were cycled between a strain corresponding to 0 and 100% of martensitic transformation up to different number of cycles. Some experimental details, like critical stress to induce the transformation, stress mode and number of cycles, are shown in table 1. Tensile and compressing trainings were performed to the samples subjected to TT1, while only tensile cycling was done to samples containing γ-precipitates (TTB).

After the pseudoelastic cycling the samples were again mechanically and electrolytically polished. The two way shape memory effect (TWSME) was checked by cooling the specimen (submerging it into an ethanol bath cooled by liquid nitrogen) and observing by optical microscopy (Olympus BHM) the formation of the stress-induced single variant of martensite. The length change during transformation was measured by dilatometry (Perkin-Elmer TMA7) and, in some cases, by means of optical microscopy, measuring the distance between two marks made on the sample surface. The latter observations were performed first in free transforming samples and after constraining them in the opposite sense of the shape change occurring when going to martensite, i.e. compressing the samples previously submitted to tensile cycling and stretching those which undergone compressing cycling (these samples were previously spark-machined to have the appropriate shape for stretching). The constraining stress was applied by a calibrated spring whose deformation gives the value of the applied stress. Two different constraining devices were designed for tensile and compressing trained specimens.

As mentioned above, the pseudoelastically cycled specimens show surface defects aligned in the habit plane direction either for tensile or compressing cycling. As we have removed these kind of defects by polishing the surface of the samples after training, these surface defects can not be responsible of the features of TWSME that will be presented.

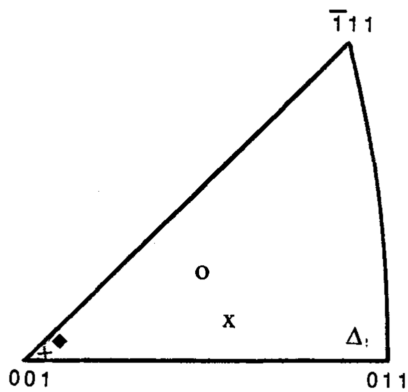

Figure 1.- Orientation of the used samples. Symbols are the same than in table 1.

3. Results and discussion

The experiments performed with samples subjected to TT1/compression, have shown that 300 cycles were enough to induce a partial TWSME (critical stress to induce transformation $\sigma_c \sim 60$ MPa.). The degree of the TWSME (parameter e), defined as the ratio between spontaneous length change during cooling after training and the length change applied during training for the same samples, was also checked. A nearly complete effect was found for 600 cycles. It is observed that the parameter e increases from 0.6 (300 cycles) to 0.9 (600 cycles) and a saturation is obtained after this number of cycles. Some dispersion is observed in the obtained data for the whole set of samples studied. This can be attributed to the different axis orientation of the samples, critical stress applied, friction between the samples and the plates during compression, formation of a second variant, etc.

Samples subjected to TT1/tension, as well as previous results [5] show that a larger number of cycles is needed to induce a similar degree of TWSME, if tensile cycling is performed (order of 1000 cycles).

The results show that compression cycling has a different effect on the TWSME than tensile cycling (a clearly favourable one in the sense that less cycles are needed to induce it). This can be related to the fact that while localized dislocation arrays have been observed after tensile cycling, there is a more regular distribution after compressing cycling. Besides, the presence of retained martensite after compressing cycling has been also observed in the samples used in this work. This does not occur in most of tensile cycling experiments. The retained martensite variants act as preformed nuclei which only have to grow, aiding in this case the TWSME [3,8,12,13].

Table 1.- Experimental conditions for each sample. TT=thermal treatment; c=compressing and t=tensile cycling; N=number of cycles; σ_c=critical stress to transform; σ_{th}= threshold stress; τ_{th}= resolved threshold stress. Samples from the same crystal have the same symbol.

Sample	Symbol	TT	Stress mode	N	σ_c(MPa)	σ_{th}(MPa)	τ_{th}(MPa)
1	x	TT1	c	578	86	3.2	1.2
2	x	TT1	c	2047	88	6.5	2.4
3	o	TT1	c	5000	83	3.9	1.5
4	o	TT1	c	16000	125	9.5	3.7
5	+	TT1	t	8820	47	2.1	1.0
6	♦	TTB	t	507	26	2.3	1.2
7	♦	TTB	t	2200	23	3.3	1.6
8	♦	TTB	t	9642	38	3.9	1.9
9	Δ	TTB	t	22179	119	8.6	2.5

In order to analyse the effect of precipitates, two samples (one containing precipitates -TTB- and the other without them -TT1-) belonging to the same single crystal were trained under similar experimental conditions (500 tensile cycles, $\sigma_c \sim 20$ MPa). The temperature of cycling had to be changed from one sample to the other in order to keep the maximum stress as close as possible, due to the Ms change produced by the precipitates. A favourable effect of the precipitates on the induction of the TWSME exists, since the precipitate-free sample did not show TWSME, while the sample with precipitates presented a complete effect (e=1). Other samples, also submitted to TTB, were used to determine the necessary number of cycles to induce the TWSME. This experiment was performed interrupting the pseudoelastic cycling after a desired number of cycles and thermally transforming the sample under optical microscopy observation. The formation of only the stress induced variant during cooling was considered as the complete induction of the TWSME. Complementary measurements of length change were used to confirm optical observations. A sample with 100 cycles had an incomplete effect, transforming to several variants besides of the induced one during cooling, while another sample with 200 cycles, taken from the same single crystal, showed a complete effect. The pseudoelastic cycling was performed for 2 different stresses (σ_c=23 and 111 MPa), changing the temperature of cycling to adjust the desired stress. The same result was obtained in both cases. From this, it can be stated that the actual distribution of precipitates significantly reduces the number of tensile cycles (about one order of magnitude) to induce the TWSME.

For all kinds of training studied (TT1/tension, TT1/compression and TTB/tension) several samples were used to determine whether mechanical work can be performed by the trained martensite variant during cooling or if the material would choose others martensite variants instead of the trained one, when an opposing force is applied. In all the measured cases, it has been determined that when trained specimens (after tensile or compressing cycling) are thermally transformed to the martensite phase, under an applied opposite stress (compressing or tensile), the pseudoelastically induced variant is still formed until a certain value of the stress is reached, which from now on will be called *threshold stress* σ_{th}. When the opposing stresses are higher than σ_{th} other variants are formed and the degradation of TWSME starts. From the optical micrographs, an important decrease in the length change is observed when the constraining stress is higher than σ_{th}. Further increase of the opposing stress finally leads to the complete disappearance of the TWSME and the variant favoured by this stress is predominantly formed, thus, producing a length change in the external stress direction.

Considering the whole set of studied samples the values of σ_{th} obtained show some dispersion (table 1). Nevertheless, the values corresponding to samples which belong to the same single crystal, and cycled under the same experimental conditions up to different number of cycles increase smoothly going to saturation (σ_{th}=3.9 MPa, for 9642 cycles). The difference between this saturation value and the stress obtained for a sample cycled up to 22179 cycles (σ_{th}=8.6 MPa), decreases if the stresses are resolved in the shear system of the stress induced variant (τ_{th}=1.9 MPa and 2.5 MPa respectively). The threshold resolved stresses (τ_{th}) are also indicated in table 1. However it is not clear if, besides of the orientation of crystal axes, others parameters affect the maximum threshold stress which can be reached.

The capacity to overcome external stresses during the direct transformation can open new possibilities from the point of view of technological applications, specially if more accurate control on the threshold stress could be acquired in the future. On the other hand, the σ_{th} is a parameter which could give a quantitative information on the internal stresses produced by the training method, in this case pseudoelastic cycling, and which are responsible of the TWSME. If the above mentioned parameter e is a measurement of completeness of the effect, σ_{th} gives an idea of how "strongly" the TWSME is induced. In the samples with no precipitates (TT1) these internal stresses are associated with the dislocation arrays formed during cycling [3-6]. In the case of samples containing precipitates (TTB) TEM observations performed to a sample with 507 cycles, with an already induced TWSME, have shown that no dislocation arrays were present. Thus, in this case, the internal stresses arise from other causes. One could be a permanent deformation of the precipitates due to cycling. When increasing the number of cycles (e.g. 9642 cycles), several dislocation arrays are indeed observed, but in a smaller quantity than in precipitate-free specimens. In this case, both factors (deformation of precipitates and dislocation arrays) would contribute to the creation of the internal stresses responsible for the TWSME.

Experiments are being performed to relate the threshold stress to the type of dislocation arrays, density of dislocations, stress mode, etc. In samples containing precipitates, TEM observations are being done with different size of precipitates to check if a permanent deformation of them is produced.

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