

Methods for obtaining two way memory effect and stressed two way memory effect of CuAlNi single crystals^①

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[Abstract] Various training methods for two way memory effect (TWME) and stressed two way memory effect (STWME) were tried on Cu13.4Al4.0Ni (mass fraction, %) single crystals by applying tensile stress along $\langle 001 \rangle$ direction of β phase. The training method of cooling with load can induce a lot of martensite prone to stabilize, thus cause large residual deformation, wide hysteresis and small TWME. Training with constant load can produce STWME larger than 8% with the least residual deformation. By training procedure of martensite reorientation below M_f followed by thermal cycling, the TWME is relatively large with very small residual deformation and with comparatively narrow hysteresis of two-way memory. The obtained two-way memory curve after such training is not a closed loop, and the obtained TWME is not stable. However, these can be improved by thermal cycling. Training with martensite reorientation below M_f and thermal cycling under relatively low constant stress throughout the whole training procedure is the optimum way of obtaining TWME, and more than 1.7% TWME can be obtained. The thermomechanical history of the sample has a pronounced effect on the training result. Thermomechanical cycling has a softening effect on martensite.

[Key words] training; two way memory effect; stressed two way memory effect; CuAlNi single crystal

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1 INTRODUCTION

Two way memory effect (TWME) refers to the reversible spontaneous shape change by heating and cooling between a "hot" austenite shape and a "cold" martensite shape without any macro-stress application^[1]. In other words, this effect is intrinsic which is distinguished from stressed two-way memory effect (STWME, in which an external constant load is applied throughout the whole two-way memory procedure)^[2]. The strains that may be achieved in two-way shape memory are always significantly less than those involved for one-way shape memory in the same alloy, and special processing procedures called training are necessary for two-way shape memory material^[3]. Although several training methods have been reported^[1,4-6], there are many inconsistencies in the compositions, metallurgical and heat treatment histories geometry (thin sheets, wires, cylinders etc.) of samples, experimental conditions as well as the physical mechanisms. It is quite often that the training method proved to be effective for one alloy may be not in effect for others. Until now, there are yet no unified methods and mechanisms that are fully effective and responsible for all the alloys to set up the two way memory effect. This paper summaries the experimental results of uniaxial loading and thermal cycling

procedures made on CuAlNi single crystals and try to provide the basic engineering data for its potential applications.

2 EXPERIMENTAL

The same single crystal sample with a composition of Cu13.4Al4.0Ni (mass fraction, %) as in Refs. [1, 7, 8]. The alloy was heated at 800 °C for 20 min and then water quenched. Phase transformation temperatures of alloy are: $M_s = 96$ °C, $M_p = 84$ °C, $M_f = 60$ °C, $A_s = 73$ °C, $A_p = 96$ °C and $A_f = 102$ °C. The dimension of samples for thermomechanical cycling is $d = 3.0$ mm \times 200 mm with axis direction along $\langle 001 \rangle$ of β phase.

The testing apparatus used is the same as described by Stalmans et al^[1]. The terms used are as follows. SATE: stress assisted transformation effect; SAMS: stress assisted martensite transformation strain; TWMS: two-way martensite transformation strain; RD: residual deformation; ER: elastic recovery; E_{sate} : elastic strain of reverse stress assisted transformation; E_{stab} : elastic strain of reverse stabilized martensite transformation. The gauge length is normally set around 100 mm.

Because the stable and reproducible shape memory performances can be achieved by cycling between

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fully martensitic and fully austenitic condition^[7], especially considering the stabilization effect^[8~10] and the fact that on heating transformation starts at A_s and is completed at A_f , typically some $2 \sim 20$ °C higher depending on loading conditions etc.^[2], the training temperature are set as: $T_L = M_f - 20$ °C and $T_H = A_f + (20 \sim 70$ °C). If during cycling only partial transformations are possible at $M_f < T < A_f$, instabilities may arise, which causes a gradual shift of the shape memory device towards the shape in the fully β or fully martensite condition.

3 RESULTS AND DISCUSSION

3.1 Method 1: Training by cooling under load

All the following experiments are carried out on a same sample in this method. The as-quenched sample is firstly thermal cycled in $50 \sim 136$ °C for two cycles, as shown in Fig. 1. The result demonstrates that the obtained TWME is very small (less than 0.3%), which mainly occurs at $92 \sim 100$ °C. After thermal cycling, the above sample is deformed 0.4% at martensite state (deformation temperature is ($M_f - 20$ °C) under a stress of 87.8 MPa). The deformation can be regarded as martensitic reorientation under external stress. Then the sample is trained with a stress of 48.1 MPa for 6 cycles (load at ($A_f + 20$ °C) \rightarrow cool with load to ($M_f - 20$ °C) \rightarrow unload \rightarrow heat to ($A_f + 20$ °C) \rightarrow cool to ($M_f - 20$ °C)) (as shown in Fig. 1). It can be seen that in the 1st training cycle, the SATE starts at 104 °C and reaches the maximum of 7% at 95 °C, then falls to 5.4% at 86 °C; after the load is removed at 5.4%, the sample has an elastic recovery of 0.6% and has a SAMS of 4.8%. Attention should be paid to the super-large SATE at temperature of $1/2[M_s(\sigma) + M_f(\sigma)]$. Cooling with load can induce a lot of martensite prone to stabilize, i.e. the martensite formed in this way has difficulty to recover during afterwards heating. The retained

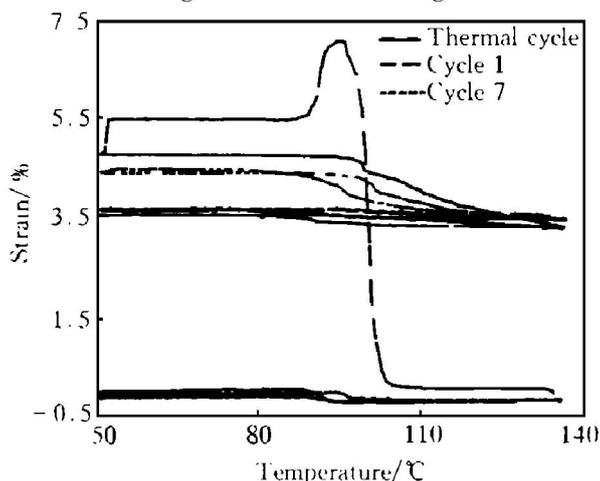


Fig. 1 Shape changes at different training stages (Thermal cycling \rightarrow thermomechanical cycling with stress of 48.1 MPa)

SAMS after the first cycle is quite close to that after the 7th cycle, which implies that the 1st training cycle mainly induces the amount of stabilized martensite to saturate around 3.4%.

Because there is a lot of stabilized martensite, an overheating ($40 \sim 176$ °C) experiment is carried out for 2 cycles on the above sample. The sample recovers almost to its original length, as described in the previous paper^[8].

The above sample is then trained with following procedure for 14 cycles: load with stress of 120.3 MPa at martensite state (50 °C) \rightarrow heat with load to 176 °C \rightarrow cool with load to 50 °C \rightarrow unload, as shown in Fig. 2. It is shown that during the 1st training cycle, when a stress of 120.3 MPa is applied on the sample below M_f , about 3% of strain is produced; then heating with this load, at temperatures of $97 \sim 120$ °C, the strain reaches 6% due to stress assisted transformation, however when temperature is above 120 °C, the strain reduces drastically to 0.5% at 130 °C due to the reverse $M \rightarrow \beta$ transformation, E_{state} of 5.5% is produced. From Fig. 2, it can be seen that heating from below M_f with load causes only very small residual deformation, which is apparently different from that of cooling with load. Heating with load also has effect on retarding of $M \rightarrow \beta$ transformation. When cooling with load, the stress assisted $\beta \rightarrow M$ transformation strain at 100 °C is 8.3%, and the elastic recovery during unloading at martensite state is 1.4%. It is also shown by comparing results of different cycles that with the increase of training number, E_{state} decreases, E_{stab} increases, the stress assisted $\beta \rightarrow M$ transformation strain decreases and ER during unloading at martensite state increases. With thermomechanical cycling going on, the differences between ($M_s - M_f$) and ($A_f - A_s$) gets larger. These results mean that the training method of cooling with load induces the stabilization of martensite.

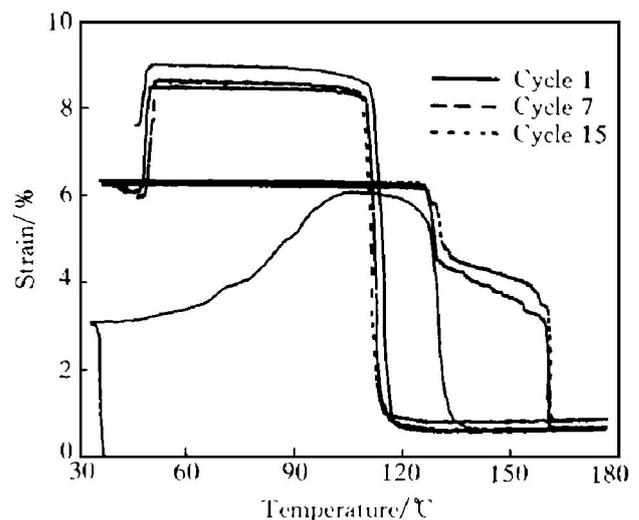


Fig. 2 Training with constant stress (Load at martensite state \rightarrow heat to 176 °C and cool with load \rightarrow unload)

By comparing the results of the 15th cycle with that of thermal cycling without load, the position of E_{stab} during thermal cycling is 10 °C lower than that of thermomechanical cycling (with load), which means that training by cooling under load shifts reverse transformation temperatures to higher region. This is also a stabilization effect of martensite.

Fig. 3 shows the shape change during training (load with stress of 144.4 MPa and unload → heat to 156 °C, reload and cool with load → heat with load, unload and cool). By comparing Fig. 2 with Fig. 3, it is seen that although both are obtained with constant loads, there is difference of loading methods. 1) the load is applied at temperature above A_f ; 2) the load is applied below M_f ; 3) the load is kept constant throughout the whole training procedure; 4) at constant load condition, the load is freed and applied again at each cycle. Results show that under the condition of thermomechanical cycling with respect to loading at temperature above A_f and then the load being kept constant throughout the whole procedure, the STWME is the largest (over 8%) with the least residual deformation and there is no stabilization of martensite, and the relevant transformation temperatures are shifted towards higher temperature region.

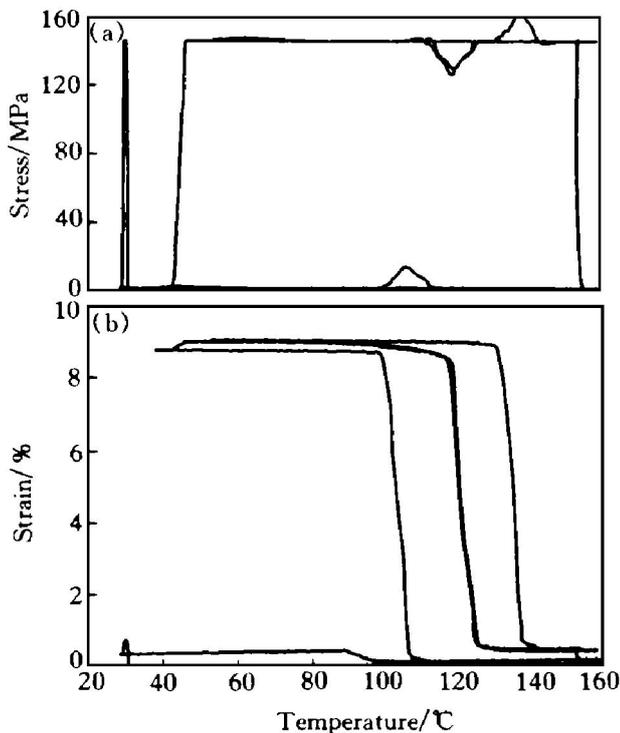


Fig. 3 Training results
(Load and unload below M_f → heat to 156 °C → load → cool with load → heat with load → cool with load again → unload → heat → cool)
(a) —Stress —temperature curve;
(b) —Strain —temperature curve

In order to avoid the stabilization of martensite, the above sample is loaded at martensite state ($< M_f$) with relatively high stress ($\sigma = 144.4$ MPa), and unload, then thermal cycled at 40~176 °C. The sample

is trained for 100 cycles, as shown in Fig. 4. The TWME is relatively large with very small residual deformation. Fig. 5 shows the evolution of SATS, TWME and RD with the cycling number. It can be seen that within the first 30 training cycles, both the SATS and TWME increase with training number, and the RD is negligible. The reverse transformation temperatures is only shifted 3.2 °C after 100 cycles. According to Perkins review on the training methods on polycrystals, typically the TWME will be perhaps 1/5~1/4 of the training strain, for example, if the strain induced during training is 6%, the TWME is likely in 1.2%~1.5%. These studying results of CuAlNi single crystals are consistent well with this point.

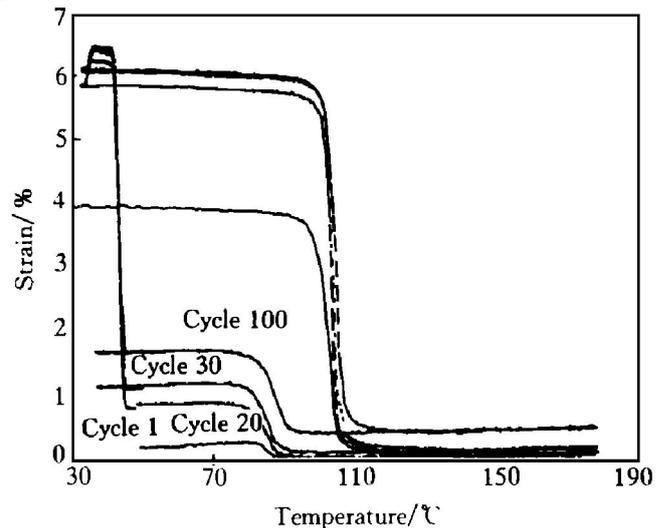


Fig. 4 Training results of martensite reorientation
(Load and unload below M_f → thermal cycling)

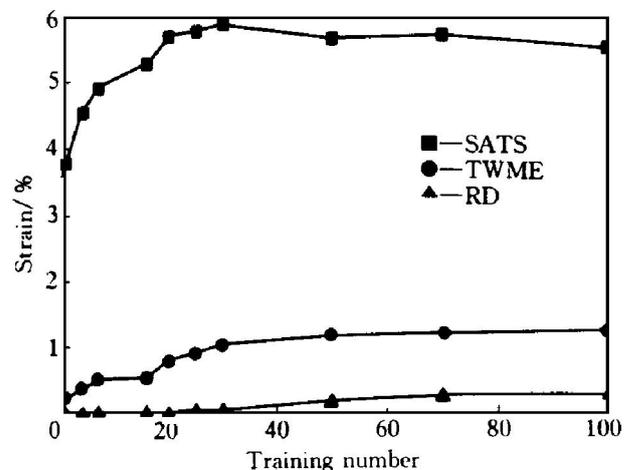


Fig. 5 Evolution of strains vs training number
(Training with martensite reorientation method)

The above sample is thermal cycled in 40~176 °C for one time, and the results are also shown in Fig. 6. In this thermal cycling, TWME after the 100th cycle is not very stable from the point of both recovery temperature and the value of TWME. The reaction temperature at thermal cycling is 8 °C lower than that at the 100th training cycle. This suggests

that the martensite reorientation structure formed below M_f with load is more stable than that during cooling at free load, which can be reflected by modified Clausius-Clapeyron formula^[1]. During cooling from temperature above A_s , the forward transformation temperature is always the same, which reveals that stress distribution structure built in the previous training does not have obvious change on the forward transformation temperature.

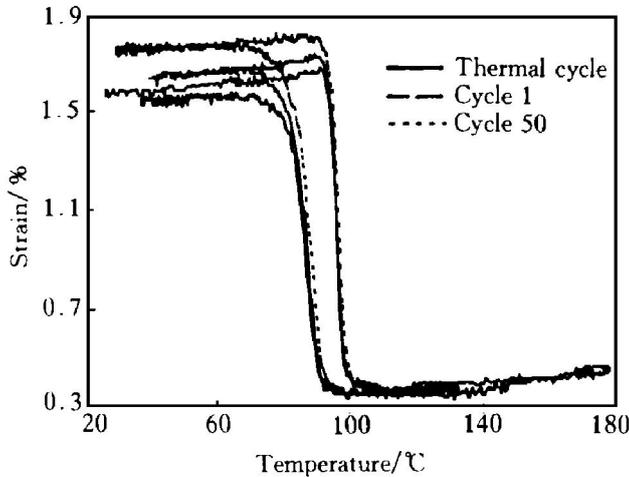


Fig. 6 Thermal cycling after training 100 cycles

It is important to notice that although during heating the strain changes 1.38% in ($A_s \sim A_f$), during cooling the strains changes only 1.19% in ($M_s \sim M_f$), which means that after training the obtained TWME is not stable. The two-way memory curve is not a closed loop. This instability of TWME after training may be caused by overheating which reduces the level of internal stress which governs, under accompany of temperature changing, the formation of thermodynamic anisotropy of martensite.

In order to investigate the stability of obtained TWME, following experiments are carried out. First, the above sample is thermally cycled in (30~130 °C) for 50 cycles, the results are shown in Fig. 7. It is revealed that during thermal cycling, the shape recovery values for the forward transformation increase and the TWME is relatively stable (more than 1.3% of fully two-way memory) with a full close memory loop. This experiment shows that thermal cycling after training has a positive influence on the TWME. Then the above sample is kept at 125 °C for 4h and cooled to 30 °C, thermally cycled again, it can be seen in Fig. 7 that such isothermal treatment has a small creep-like effect, which reduces the obtained TWME a little bit.

3.2 Method 2: Training by martensite reorientation and thermal cycling

The specimen for this experiment is originally subjected to neither training nor thermal cycling from quenching state. At each training cycle the specimen is firstly deformed under a stress of 144.4 MPa at

temperature around 50 °C, then thermally cycled from 50 °C → 126 °C → 50 °C, the results are shown in Fig. 8. TWME in the 30th cycle is only about 0.6% although the two way memory is progressing with training, which forms a significant contrast with the training results for the thermomechanical cycled specimen in Fig. 5. Therefore, the thermomechanical history of the specimen may have a pronounced effect on the training results. The change of the concentrations of quenched-in vacancies, other defects as well as the stress fields during thermomechanical training or thermal cycling may have a great influence on the training behavior. After thermal treatment, the yield stress at martensite state is decreased, and the corresponding strain at the same stress level is larger than that of non-thermal cycling, as shown in Fig. 9. It can be seen that the strain after each martensite reorientation is increased with the training number. Thus it seems to be a conclusive point that the thermo-

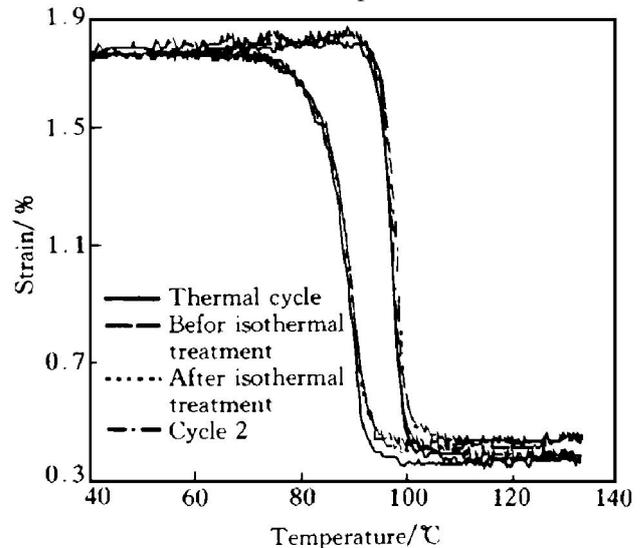


Fig. 7 Isothermal results at 125 °C to specimen thermal cycled 50 times in Fig. 6

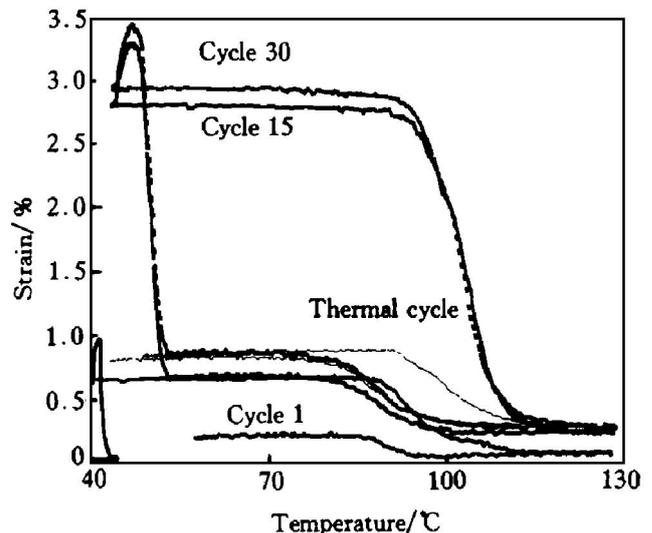


Fig. 8 Training results
(Load with stress of 144.4 MPa and unload below M_f → heat → cool)

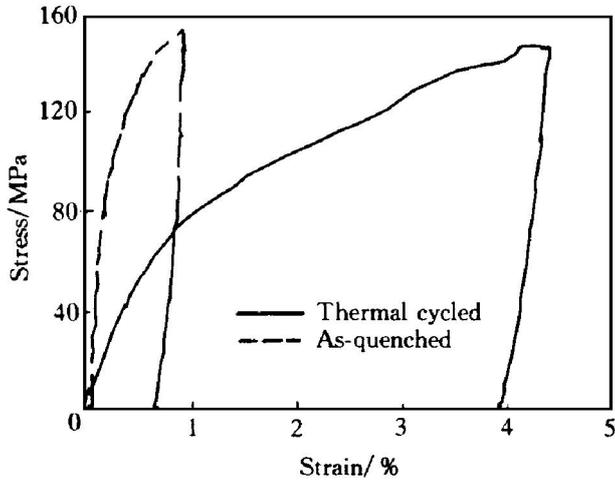


Fig. 9 Stress—strain curves at ($M_f - 20\text{ }^\circ\text{C}$) for specimen with different thermal histories (cycle 1)

mechanical cycling has a softening effect on martensite.

An additional experiment was carried out on the above-trained sample. At each training cycle, the specimen was first deformed below M_f with a stress of 144.4 MPa and then thermally cycled under constant stress of 12.0 MPa throughout the whole training procedure. After training for 30 cycles (as shown in Fig. 10), TWME considerably increases to 1.75%. Training by deformation below M_f with large stress and then thermal cycling under relatively low constant stress throughout the whole training procedure is a good way of obtaining large TWME.

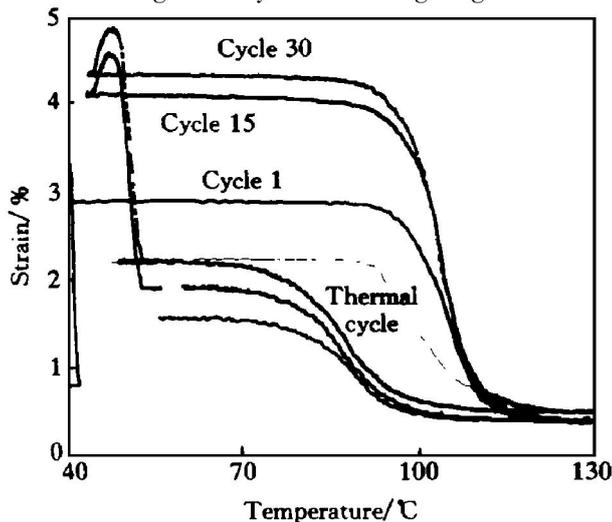


Fig. 10 Training results
(Load and unload below M_f heat and cool with constant load of 12.0 MPa)

After obtaining TWME at 1.75%, specimen is thermally cycled under a stress of 24.0 MPa (without unloading procedure), as shown in Fig. 11, the STWME is 2.2% in the 31st training cycle. However, after thermal cycling without load, the corresponding TWME is only half of the previous value although the training stress is doubled.

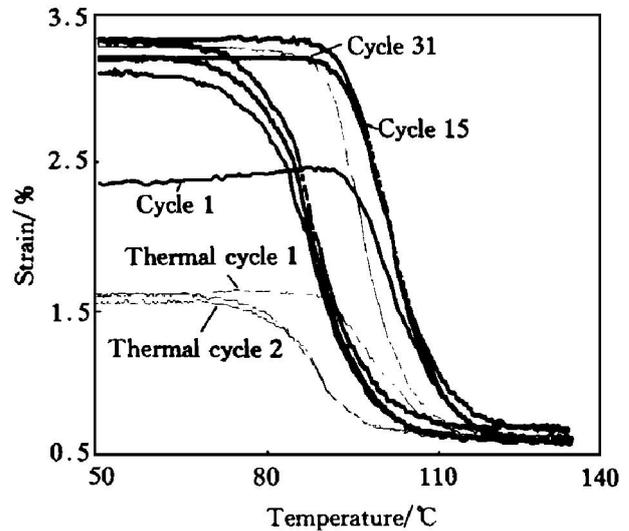


Fig. 11 Training results under constant load of 24.0 MPa
(Load below M_f heat \rightarrow cool)

3.3 Method 3: Training by cooling with load and thermal cycling

This training method is the same as that for CuZnAl polycrystals^[11]. The training stresses used are 17.8 MPa, 48.1 MPa and 144.4 MPa respectively, and the results are shown in Fig. 12.

3.3.1 SATE

When training with relatively low stress, there is a competition between the stress assisted preferential formation of martensite and the self-accommodation of martensite. The later is considered as a resisting effect of training. At higher training stress level, there is a saturation of SATE. This upper limit probably due to texture limitations and/or the necessity of strain accommodation on the whole. From the training results, it can be seen that with increasing training stress level, the amount of stress assisted $\beta \rightarrow M$ transformation strain (SATS) increases greatly until it reaches 9% (this value is the maximum value of superelasticity originated from β_1 for studied CuAlNi single crystal). Under relatively high training stress (greater than 48.1 MPa), this kind of strain also decreases during the stabilization of stress assisted martensite formed under cooling with high stress, which results in the reduction of the amount of “active austenite” for further training.

3.3.2 TWME and RD

It is shown that by this training method, only very small TWME with large hysteresis of transformation temperature can be obtained for this alloy, although more than 1.2% of TWME can be obtained in CuZnAl polycrystals^[11]. The reason may be the lack of “active austenite” because of the stabilization of martensite and thus incurs the difficult formation of thermodynamic anisotropy structures. In Fig. 12, large amount of RD exists after several cycles due to the presence of stabilized martensite. The martensite

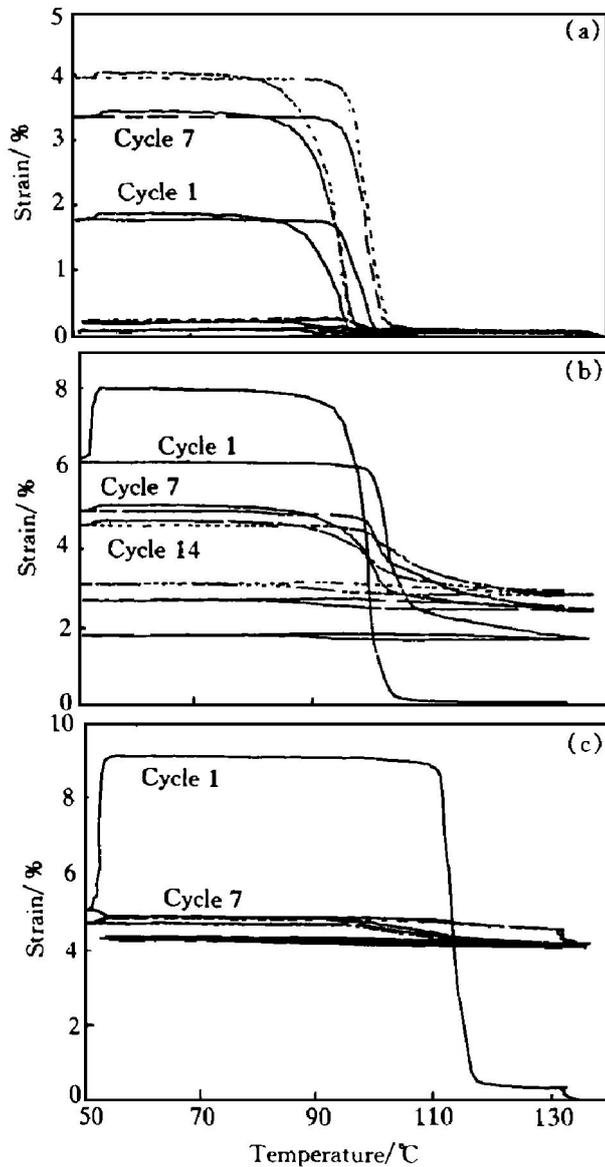


Fig. 12 Training with different loads
(Load at 136 °C → cool to 50 °C → unload → heat → cool)
(a) -17.8 MPa; (b) -48.1 MPa; (c) -144.4 MPa

is formed under the stress at certain temperature because the driving force for the forward transformation is increased a lot by applying uniaxially external stress on the single crystal. However, once such martensite is formed, the reverse transformation of stabilized martensite can be obtained by either raising heating temperature (overheating) or under the same favorable stress assisted condition.

3.3.3 ER during unloading at martensite state

By comparing the results of 1st training cycle at different stress levels in Fig. 13, it is clearly shown that ER at martensite state increases with training stress level (about 4% at training stress of 144.4 MPa). This kind of “superelasticity” at martensite state shows the great effect on stress assisted $\beta' \rightarrow M$ transformation. Once the training stress is removed, the driving force for $\beta' \rightarrow M$ transformation is decreased drastically, which induces the backward motion of β'/M variant interface. However, during the afterwards training cycle, due to the fact that the

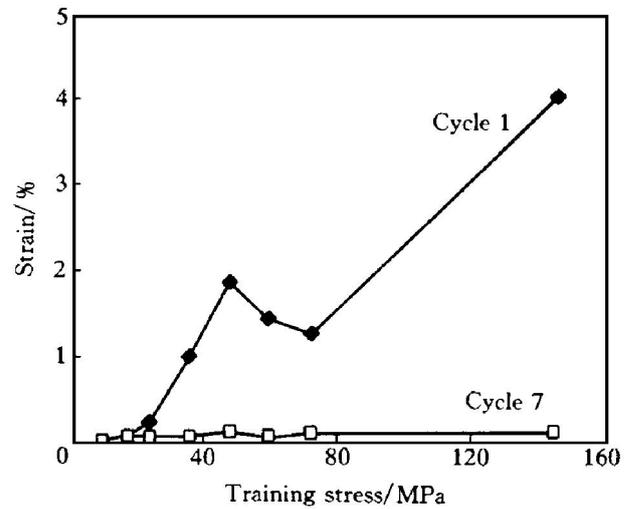


Fig. 13 Elastic recovery vs training stress during unloading below M_f

amount of “active austenite” is considerably decrease, the elastic recovery during unloading at martensite state decreases.

3.3.4 Shift of transformation temperatures

Experiments show that the transformation temperatures are shifted towards higher temperature region. As an estimation, the tangent line method^[1] from the strain-temperature curve is used to evaluate the evolution of transformation temperatures. It should be aware that in the DSC measurement for the sample before training, the peak position of $\beta' \rightarrow M$ transformation (M_p) is 84 °C, the peak position of $M \rightarrow \beta'$ transformation (A_p) is 97 °C. It is shown in the results of this method that the forward transformation temperature is higher in the training cycle than that in the following thermal cycle due to the difference in applied stress, which is also true for CuZnAl polycrystals. For example, at training stress of 144.4 MPa, during cooling with load, $M_s = 117$ °C and $M_f = 110$ °C.

The stress applied during training has a significant influence on the shift of transformation temperature. With training stress level increasing, the shifts of transformation temperature increases. These can be explained quantitatively by using modified Clausius-Clapeyron equation.

3.4 Method 4: Training by combination of cooling with load and overheating

The training is programmed as: load at ($A_f + 20$ °C) (136 °C) → cool with load to below ($M_f - 20$ °C) → unload → heat to ($A_f + 70$ °C) → cool, repeat 14 times. The training stresses used are 48.1 MPa, 72.2 MPa and 144.4 MPa respectively. Fig. 14 shows the results. The only difference between Method 4 and Method 3 is that an overheating cycle replaces thermal cycling following each thermomechanical cycling. The purpose is to retransform the stabilized martensite

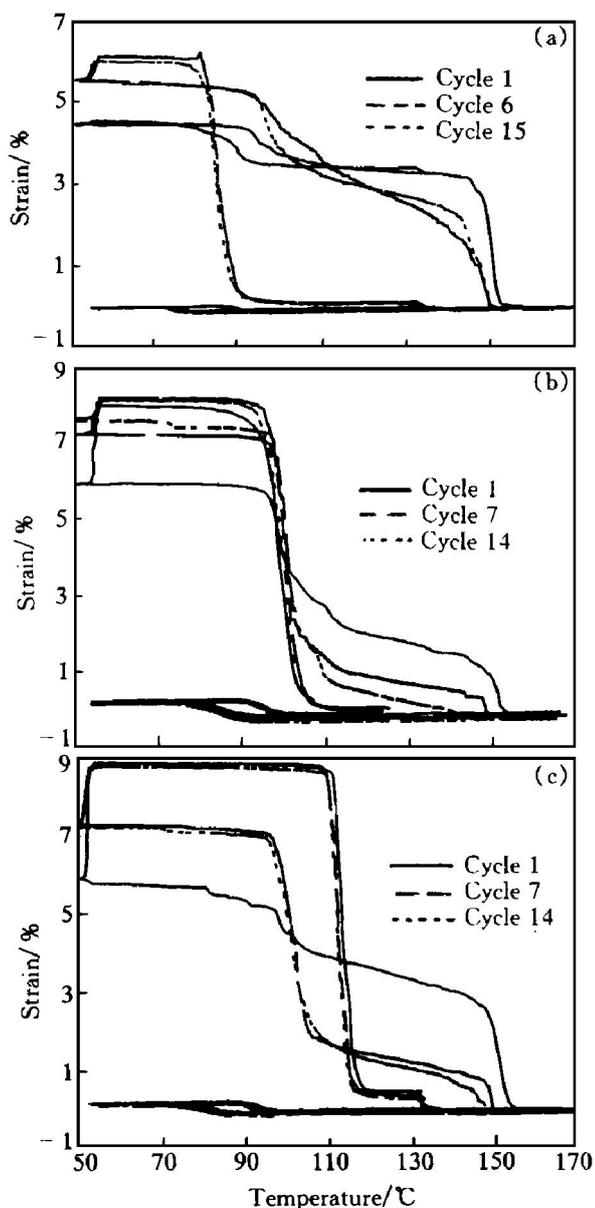


Fig. 14 Training with different load
(Load at 136 °C → cool → unload → overheat to 176 °C → cool)
(a) -48.1 MPa; (b) -72.2 MPa; (c) -144.4 MPa

site back to austenite to provide “active austenite” for further training. It is shown from the training results that the shape recovery during overheating consists of three parts:

$$E_{total} = E_{sate} + E_{thermal} + E_{stab} \quad (1)$$

E_{sate} associates with the reverse stress assisted transformation. The slope of the recovery curve at this stage is large. $E_{thermal}$ associates with the thermally activated reverse transformation of partially stabilized martensite. The slope of recovery curve during this stage is relatively small. E_{stab} associates with the reverse transformation of stabilized martensite, which takes place around 150 °C when training stress is greater than 48.1 MPa. According to the shape recovery curves during overheating as well as DSC measurements^[8], once E_{stab} reverse transformation occurs, it will proceed drastically within a very narrow

temperature range and the slope of recovery is quite large. These are the characteristics of E_{stab} . The proportions of the three recovery parts change with the training stress level and the training number. By comparing the results of different training stresses, it can be seen that the forward stress assisted transformation temperatures (M'_s and M'_f) increase with training stress. The small RD in such training and overheating procedure indicates that during each overheating cycle, the stabilized martensite formed during the previous training cycle can be completely recovered by overheating. E_{stab} occurs at about 20 °C higher than the maximum training temperature^[1]. The fact that for training with relatively high stress and overheating for the reverse transformation, E_{sate} increases while E_{stab} decreases with training number suggests that the amount of stabilized martensite decreased with training going on. Consequently it can be deduced that after a sample is such trained for a certain number of cycles, there is less possibility for stabilization of martensite. And this sample can be used for further training for two way memory effect with less residual deformation and large TWME because of the large value of SATE at high training stress level, as shown in section 3.1 and Figs. 4~7.

It is clearly shown that after such training and overheating, there is no residual deformation, but the obtained TWME is also very small although the hysteresis of two way memory loop is very small (less than 17 °C). According to the fact that based on the experimental results of CuZnAl polycrystals, if the SATE is very large, it is expected that the TWME should be correspondingly large if TWME is linearly related to the SATE^[1,5]. However the results in this case are not like this, the reason may be in that the overheating could disturb the stress built in the previous training. If the upper temperature limit is set at just above the occurrence of E_{stab} , the result could be much better.

3.5 Method 5: Training by cooling with load and heating with load

Specimens are heated to above ($A_f + 20$ °C) (136 °C) → load → cool with load to below ($M_f - 20$ °C) → unload and load again → heat to ($A_f + 20$ °C) → unload as one cycle, then repeat. The results are shown in Fig. 15. The training stresses used are 12.0 MPa and 24.0 MPa respectively. Training with constant load can produce large STWME, which is considerably higher than the value of TWME. However, there is large RD due to the stabilization of martensite, which is produced on cooling with load. By comparing this with Fig. 4 and Fig. 1(b) in Ref. [8], it can be noted that under the load, the distribution of stress within the sample is favorable for the reverse stress assisted $\beta \rightarrow M$ transformation.

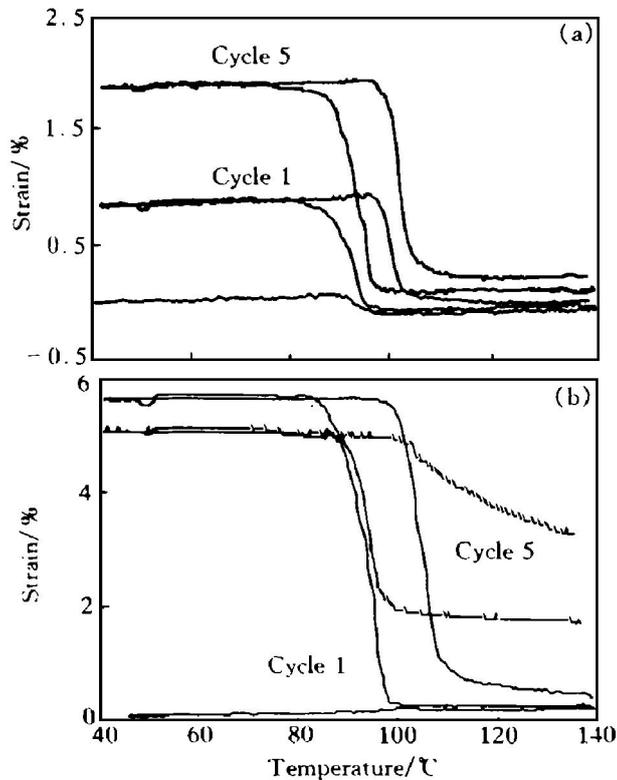


Fig. 15 Training with different stresses
(Load at 136 °C → cool to 50 °C → unload and
load again → heat to 136 °C)
(a) -12.0 MPa; (b) -24.0 MPa

4 CONCLUSIONS

1) Training by cooling with load induces a lot of martensite prone to stabilize. Training by heating from temperature below M_f with load causes only very small residual deformation. Training by preferential formation of martensite during cooling is not an efficient way for obtaining TWME in CuAlNi single crystal.

2) The shape recovery during overheating consists of three parts. The first part E_{state} results from the reverse stress assisted transformation; the second part $E_{thermal}$ from the thermally activated reverse transformation of partially stabilized martensite; the third part E_{stab} from the reverse transformation of stabilized martensite. The amount of stabilized martensite produced in the training and overheating procedure decreases with training cycle. With training number increasing, E_{state} decreases and E_{stab} increases.

3) Training with constant load produces large stressed two way memory effect. When cooling with load from temperature above A_f and then the load be-

ing kept constant throughout the whole thermomechanical cycling procedure, the stressed two way memory effect is the largest with the least residual deformation.

4) Training by martensite reorientation followed by thermal cycling, the TWME is relatively large with small residual deformation and comparatively narrow hysteresis of two way memory.

5) The thermomechanical history of the sample has a pronounced effect on the training result. Thermomechanical cycling has a softening effect on martensite. The previous thermomechanical cycling may be beneficial to the set-up TWME.

6) Training with martensite reorientation below M_f and thermal cycling under relatively low constant stress throughout the whole training procedure is the optimum way to obtain TWME. More than 1.7% of TWME can be obtained.

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