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Stress-Induced Transformation and Temperature-Induced Transformation in Cu-Zn-Al Single Crystals

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Abstract
The stress-induced martensitic transformation has been studied at different temperatures T>Ms. The application of a stress σ on a shape memory alloy leads to the apparition of oriented variants of martensite in the direction of the stress. It causes the superelastic effect. Beyond a critical stress σ_c, variants grow until parent phase diseappears. But martensitic transformation can be also induced by temperature.
During a superelastic test, the sample has been overheat, then cooled at a constant deformation. It could be noticed that stress moves with temperature and that a low hysteresis appeared.

1. INTRODUCTION

A number of investigation have been carried out about the martensite transformation because of interest of shape memory phenomena and its properties as shape memory effect, two way shape memory, rubber-like behaviour and superelasticity. The shape memory alloys presents an ability to undergo a thermoelastic martensitic transformation[1,2]. The thermoelastic martensitic transformation can be induced by a temperature variation. However, it is also common for a similar form of martensite to be induced by an applied stress and to shrink when the load is removed. It is implicit that the transformation is reversible but includes a hysteresis of about 3 MPa. The superelasticity phenomenon observed above Ms temperature is due to the formation of stress-induced martensite on a load cycling. In order to study the superelasticy and the connexion existing between stress-induced martensite and temperature-induced martensite, a tensile machine was required.
The purpose of the present paper is to report the results of the mechanical studies and to verify the stress behaviour versus temperature.

2. EXPERIMENTAL

The samples are CuZnAl single crystals, containing 15,5% wt Zn and 8% wt Al. Polycrystals are obtained by melting in an induction furnace with nitrogen protection and then single crystals are obtained by a Bridgman method. Specimen for tensile tests were cut, the gauge lengh portion being 15x1x3.5mm^3. The Ms temperature is 14°C. The heat treatment consisted of a homogenization 10 minutes at 850°C, a quench in water and an ageing in boiled water 30 minutes in order to cancel vacancies and in order to obtain a beta phase with a DO_3 order[3,4,5].
A tensile machine has been designed in order to obtain data on shape memory behavior [6]. It is driven by a computer. Its characteristics are:

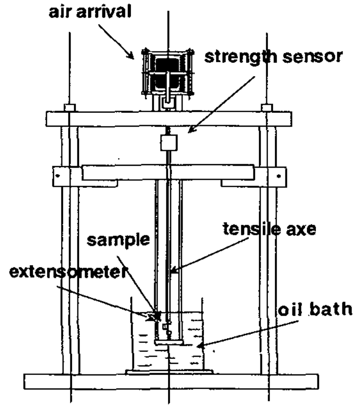


figure 1: tensile machine

- a pneumatic controlled movement
- an uniaxial (vertical) motion
- an absence of external friction

An electrovalve controls the air arrival between two inox chambers. The sensors are:

- a strenght gauge, with working limits of 0-500N
- a classical linear velocity displacement transducer (LVDT)
- an extensometer, associated with the sample.

This tensile machine (figure 1) can work in a strain controlled regime (with an infinite stiffness between 0 and 500 N) or in a stress controlled regime (with no stiffness). The sample is immersed in a silicone oil bath in order to obtain a constant and homogeneous temperature.
Under a strain controlled regime, a constant strain rate (2%/mn) is imposed and the variation of the stress is measured as a function of strain. The computer records stress, strain and temperature.

3. RESULTS AND DISCUSSION

The CuZnAl specimen exhibits a superelastic effect. In figure 2 a strain-stress curve is plotted, at a temperature of T=21°C. Upon initial loading, sample shows an elastic behaviour (from A to B). With further loading, before a critical stress σ_c, when martensite variants start their development, sample undergoes a martensitic transformation (from B to C). The stress-strain relationship is very different from that of conventional polycrystalline materials. Single crystals exhibit a plateau, which presence can be explained by the growth of a single variant.

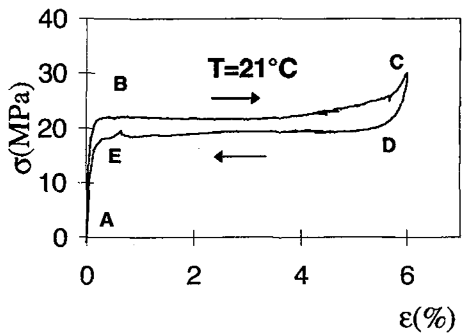


figure 2: strain-stress curve at T= 21°C

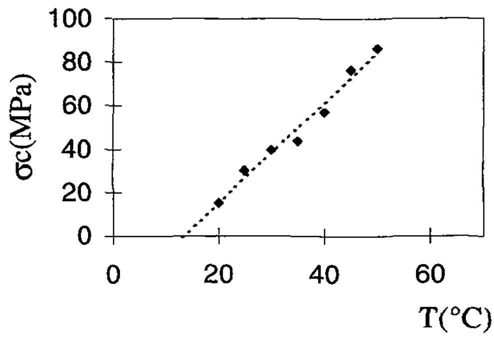


figure 3: critical stress behaviour versus temperature

Upon unloading, the stress rapidly drops at first and then becomes nearly horizontal. A hysteresis loop appears (from C to D), which is characteristic of the martensite transformation. The sample undergoes a reverse transformation (from D to E). Then, stress linearly decreases with strain, in an austenitic elastic return (from E to A). Stress which is required to induce the transformation, noted σ_c, increases in respect with the temperature, according to the Clausius-Clapeyron law (equation n°1).

$$\frac{d\sigma_c}{dT} = \frac{\Delta S}{\varepsilon_u}$$ **équation n°1**

with σ_c the critical stress, ε_u the strain associated to shape change of the transformation, ΔS the entropy change per volume unit[7,8].
Figure 3 shows a straight line behaviour and a slope of about 2,3 MPa/°C. The critical stress had been defined when the curve deviated from the elastic slope. It is possible to know the Ms temperature by extrapoling the line when stress upon loading becomes zero.

3.1 Temperature Variation experiments (TV curves)

3.1.1 *Experimental procedure*

A sample undergoes a stress-induced transformation at a constant temperature T_1 (from A to B). The stress is released till a chosen deformation ε_0 inferior to 1% (from B to C). Then the deformation ε_0 is remained constant and the specimen is overheated till a temperature T_3 then cooling till T_1.(from C to D). The small graph on the right shows the variation of the stress versus temperature during a temperature cycle.
The results are as follows:
- from T_1 to a temperature $T_2<T_3$, an increase of the stress is observed according to a straight line behaviour.
- from T_2 to T_3, the stress remains constant and a plateau appears.

When temperature decreases, a few hysteresis appears.

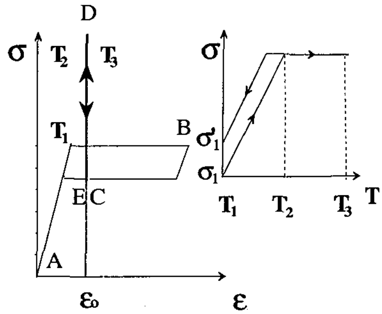


figure 4: after a superelastic cycle, a strain is held constant and the sample is overheated

3.1.2 Results

An exemple of superelastic effect, performed at T=21°C and at a strain rate of 2%/mn, can be noticed in figure 5a: the deformation ε_0 is remained constant and the specimen is overheated till a temperature of about 55°C.

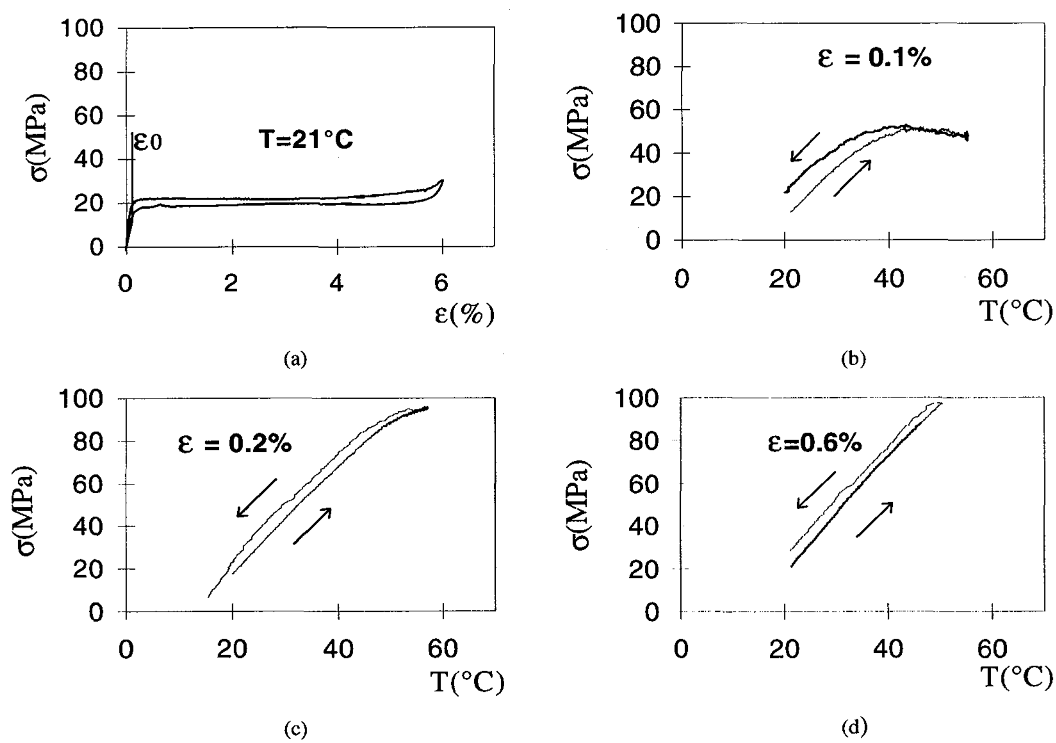


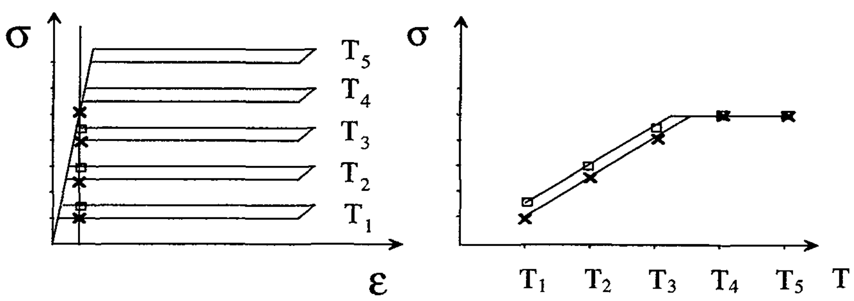
figure 5: (a) after a superelastic cycle,the deformation is held constant and the sample is overheated; (b) increase of the stress versus temperature at 0.1%; (c) at 0.2% (d) at 0.6%.

The figures 5b, 5c, 5d experimentally show the evolution of the stress versus temperature for respectively ε_0=0.1%, 0.2%, 0.6%.When temperature increases, the sample cannot recover its high temperature shape. The reverse transformation takes place but, under a strain controlled regime, in order to keep a constant deformation, a more important stress must be generated. So, an increase of the stress is observed. Similarly, for the same reasons, cooling the sample brings about a decrease of the stress. But a small temperature hysteresis can be observed (about 2°C). The slope is about 2.5 MPa/°C, close to the slope measured for the critical stress.

3.2 Constant Temperature experiments (CT curves)

3.2.1 Experimental procedure

In figure 6a, strain-stress curves are plotted at different temperatures. A constant deformation was held and corresponding stress is noted for each temperature as it is drawn in figure 6b. Stress upon loading and upon unloading is reported as a function of test temperature. The plot of loading and unloading stress versus temperature results in straight lines which present a few temperature hysteresis. However, a plateau appears before a critical stress σ_0.

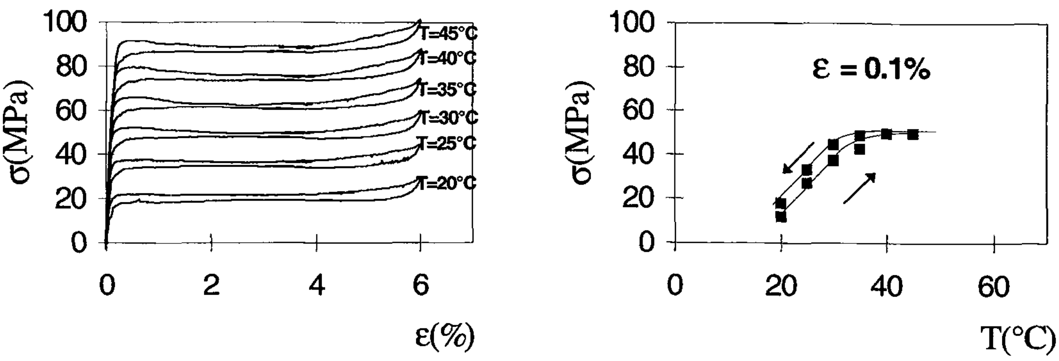


(a) (b)

figure 6: theorical curves: (a) superelastic cycle evolves with T. A strain is held constant and corresponding stresses on loading and on unloading are collected for various temperatures; (b) On unloading stress increases as a function of the temperature and on loading stress decreases. A hysteresis appears.

3.2.2 Results

A constant deformation ε_0=0.1% has been chosen. In figures 7a and 7b, the curves obtained by experiments are plotted. A hysteresis of about 2°C is found.



(a) (b)

figure 7: experimental results: (a) Superelastic cycle evolution with T; (b) Stress increases versus temperature.

3.3 Discussion

At T above Af, the specimen is in an austenitic phase. When a strain is applied, a single variant of martensite, oriented by an uniaxial stress, appears in the single crystal. The stress-induced transformation progresses by nucleation of new plates which cross the whole specimen. The increase of the strain forces the growth of the variant plates and leads the specimen in a total martensitic phase [9,10].

At T_1, when a constant strain ε_0 is applied, the sample which is submitted to a stress σ_1 contains a given amount m_1 of martensite plates and a given amount of austenite a_1. The sample is deformed and its length can be decomposed into two lengths L_{m1} and L_{a1} such as:

$$L_{m1} + L_{a1} = L_0$$

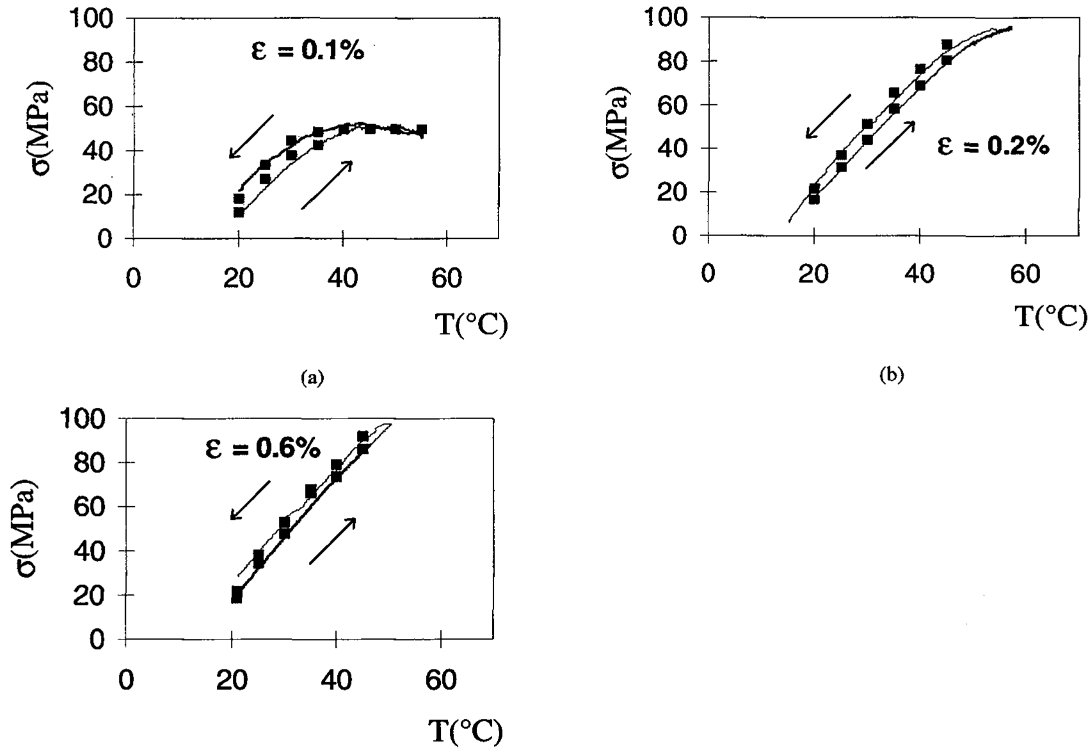
with L_{m1} the length component due to the martensite and L_{a1} the length component due to the austenite at T_1. L_{m1} is function of the amount of martensite m_1.

When the sample is overheated till a temperature T_2, superior to T_1, at ε_0 constant, the total length remains constant but stress increases as a function of the temperature as it can be noted in our experiments. At T_2, the sample undergoes a stress σ_2. The amount of martensite is now m_2.

If total length remained constant, the martensite component and the austenite component have changed:

$$L_{m2} + L_{a2} = L_0$$

with L_{m2} the length component due to the martensite and L_{a2} the length component due to the austenite at T_2.



(c)

Figure 8: superposition of TV and CT curves: (a) at 0.1%; (b) at 0.2%; (c) at 0.6%.

When the stress increases, the length of the specimen which corresponds to the austenitic phase L_a increases, because of the elasticity of the austenitic phase. As the total length L_0 is constant, the length which is due to the martensite phase L_{m1} decreases. So L_{a2} is higher than L_{a1} and L_{m2} is lower than L_{m1}.

Accordingly, the amount of martensite decreased and m_2 is lower than m_1. When the martensite has disappeared, the stress becomes constant.

In figure 8, the TV curves and the CT curves are superposed. As it can be seen, the two experiments made with the specimen lead to a comparable result. The effect of increasing or decreasing the applied load is respectively equivalent to cooling or heating the sample.
Figure 9 shows two stress-strain curves, performed at two temperatures T_2 and T_1, with $T_2>T_1$. However, reverse transformation plateau for the T_2 temperature corresponds with direct transformation for the T_1 temperature. At a constant deformation ε_0, loading stress, σ_1, at $T=T_1$, and unloading stress σ_2, at $T=T_2$, are the same. So, for two different temperatures, we can find the same stress. What can explain the temperature hysteresis of about 2°C.

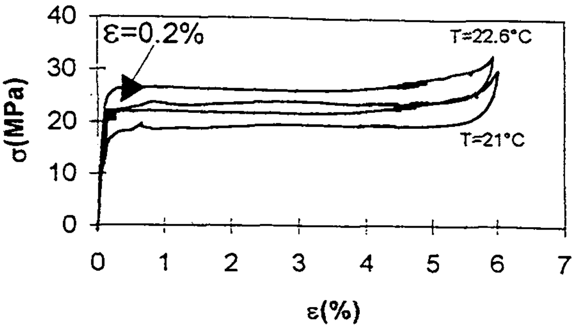


figure 9: stress-strain curves at T_1 and at T_2.

4. CONCLUSION

Superelastic effect and temperature-induced transformation have been investigated in a CuZnAl single crystal in vue to explain the similarity of both transformation. It has been shown that the stress-induced martensite can be recovered and can be reformed with a temperature variation at a constant deformation. This transformation presents a few hysteresis of about 2°C. A similar result can be obtained by reporting the data collected on the superelastic curves measurements at constant temperatures.

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