

**STUDY OF THE Cu-Zn-Al MARTENSITE AGING BY INTERNAL FRICTION MEASUREMENTS**

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**Résumé.**— Des mesures de résistivité ont montré que la transformation martensitique est partiellement ou complètement stabilisée après trempe suivie d'un vieillissement en phase martensitique. L'hypothèse qui a été proposée est basée sur le piégeage des interfaces de martensite par des défauts ponctuels. Des mesures de frottement interne et de résistivité ont été réalisées pour vérifier cette hypothèse.

**Abstract.**— It has been shown by resistivity measurements that the martensitic transformation is partially or completely stabilised after fast quenching and aging in martensitic phase. The hypothesis which has been proposed is based on the pinning of martensitic interfaces by point defects. Some internal friction and resistivity measurements made during heating at different temperature rates have been realized to verify this hypothesis.

Introduction.

The martensitic transformation of Cu-Zn-Al  $\beta$ -phase alloys is very sensitive to the thermal treatments, like quenching and aging in the  $\beta$ -phase or in the martensitic phase (1,2,3). When a sample is quenched at temperature  $T_M$  below the  $M_f$  temperature (in the martensitic phase) and then aged in this phase, a progressive increase of the reverse transformation temperatures and decrease of the reverse transformation magnitude occurs. This phenomenon is called stabilization of the martensite.

To have no stabilization of the martensitic phase, the sample should be step quenched to a temperature  $t_B$  above  $A_f$ . This treatment decreases the vacancy concentration and changes the order of the  $\beta$ -phase ( $B_2 \rightarrow DO_3$ ). After this treatment, the transformation temperatures and the magnitude of the transformation do not change with aging in martensitic phase. This treatment has been called "Standard".

Some authors (4) have shown that the damping capacity of stabilized martensite is very low compared to the damping of standard martensite. In this work, the stabilization of the martensite has been studied during heating at different temperature rates by internal friction measurements. Resistivity measurements have also been done after each experiment to estimate the transformation temperatures and the ratio of stabilized martensite.

Experimental procedure.

The samples have been prepared from rolled plates of one millimeter thickness. The composition is:

Cu 70.75 / Zn 25.25 / Al 4 wt%.

The same sample of size  $50 \times 4 \times 1 \text{ mm}^3$  has been used for internal friction and resistivity measurements. Two thermal treatments were performed:

-To obtain standard martensite, the sample was annealed at 1123 K during 900 s and then step quenched at  $T_B = 373 \text{ K}$  during 3.6 ks. After this treatment, the transformation temperatures were:

$$M_S = 350 \text{ K} \quad M_F = 328 \text{ K} \quad A_S = 338 \text{ K} \quad A_F = 360 \text{ K}$$

-To study the stabilization of martensite, the sample was annealed at 1123 K during 600 s, quenched directly at room temperature in water, dried, and quickly immersed in liquid nitrogen. Then, the sample was set in the pendulum at low temperature, this one being always kept below 170 K.

The damping ( $\lg \phi$ ) was measured with a forced oscillation torsional pendulum between 170 and 420 K. Measurements have been done at two temperature rates,  $5 \times 10^{-3}$  and  $5 \times 10^{-2} \text{ K/s}$ , and frequency was in the range  $5 \times 10^{-2}$  to 2 Hz.

During the measurements, the maximum strain was kept constant at  $\epsilon_m = 4 \times 10^{-4}$ .

### Experimental results.

-A/ Figure 1 shows the internal friction and modulus variation as function of temperature for a "standard" sample ( $dT/dt = 5 \times 10^{-3} \text{ K/s}$ ,  $f = 0.05 \text{ Hz}$ ). On this figure the variations of resistivity have also been reported to estimate the characteristic temperatures of the transformation.

In the temperature range of the transformation, the maximum internal friction associated with a minimum of modulus is observed (5). In the martensite phase, the damping capacity is very high, with a small maximum on heating at 300 K and a minimum just before the inverse transformation at 330 K. This effect is also reported by J. Van Humbeeck (4). In the high temperature phase, internal friction is very low.

-B/ Figure 2 concerns the same experiment ( $dT/dt = 5 \times 10^{-3} \text{ K/s}$ ,  $f = 0.05 \text{ Hz}$ ), but with a directly quenched sample. After this treatment, resistivity curves do not exhibit any slope change indicating that no reverse transformation occurs, therefore a complete stabilization of the martensite is induced by the low heating rate in this temperature range. At low temperatures the damping of the standard and direct quenched sample is of the same order of magnitude. During heating, the damping shows a maximum  $\delta_M$  at  $T_M = 250 \text{ K}$  and decreases rapidly to a very low value above 285 K. During these variations, the modulus only increases, especially when the damping decreases.

On further cooling, and for the next thermal cycles between 190 K and 360 K, the internal friction remains very low, and the modulus is stable. It is therefore confirmed that the internal friction of stabilized martensite is very low, in opposition to the one of the standard martensite.

-C/ The curves of the figure 3 are obtained when the sample is heated at a higher rate ( $5 \times 10^{-2} \text{ K/s}$ ) after direct quench. For this rate, some differences exist with respect to lower heating rate:

-The internal friction maximum  $\delta_M$  is higher.

-The temperature  $T_M$  of this maximum is higher ( $T_M = 255 \text{ K}$ ).

-The temperature at which internal friction is minimum is also higher ( $T_m = 300 \text{ K}$ ).

-A small maximum of internal friction appears at 320 K, with a modulus minimum. From the resistivity curves, it can be seen that these effects are due to incomplete reverse martensitic transformation (compare with the resistivity curve of figure 1). The temperatures  $A_S$  and  $A_F$  of this transformation are lower than for the standard sample. This is due to the fact that the martensite

inherits the order of the  $\beta$ -phase. For direct quenched sample, this order is B2, which induces a lower transformation temperature than when the  $\beta$ -phase is ordered DO<sub>3</sub> (standard sample).

- Internal friction on heating is always higher than  $2 \times 10^{-2}$  even at temperature higher than the martensitic transformation. To explain the difference of the damping at  $T > 340$  K between the heating and the cooling ( $\tan \delta \approx 2 \times 10^{-3}$ ), the heating has been stopped at 370 K (figure 4). This induces a dip in the internal friction curve, which proves that the damping up to 340 K is temperature rate dependent (on this figure, the internal friction maximum  $\delta_M$  is lower, due to the frequency change). This damping is certainly due to the reverse transformation of small martensite plates which is not perceptible with resistivity measurement, but which has been observed with acoustic emission measurement (1)(figure 5).

### Discussion.

Recent works (6) have shown that the stabilization of Cu-Zn-Al martensite is a thermally activated process. Two phenomena, vacancy pinning and reordering in martensitic phase are considered as responsible for this stabilization (7). In a previous work (8) we have also shown that internal friction of martensite in the range of  $\epsilon_m = 10^{-4}$  strain amplitude is due to the hysteretic movement of the interfaces between the plates variants. Our experimental results seems to indicate that the martensite stabilization is due to the pinning of the interfaces between the variants by the vacancies. This pinning induce the martensite stabilization and also the disappearance of the high damping capacity of the martensite phase.

When the sample is directly quenched, a maximum amount of vacancies is inherited in the martensite phase. At low temperatures, directly after the quench, a random distribution of frozen in vacancies is obtained. These vacancies do not disturb the interface-motion: the damping of direct quenched martensite is of the same order of magnitude than for the "standard" martensite, which contains much less vacancies.

When the temperature is gently increased ( $5 \times 10^{-3}$  K/s) the damping first increases (from 220 K) and then decreases. In the same time, the modulus increases. These effects can be due to the pinning of the interfaces by the vacancies. In a first time, the vacancies move to the interfaces and begin to disturb their motion: the damping increases. When the number of vacancies which move to the interfaces becomes sufficiently high, the interfaces are progressively pinned and the damping decreases to reach a low value. In the same time the modulus strongly increases. This pinning of the interfaces by the vacancies is then permanent between 170 K and 380 K: no reverse transformation occurs due to the fact that interfaces cannot move. On cooling the damping of the martensite remains low, even at low temperature. The martensite is "stabilized". At low temperatures, the modulus of the stabilized martensite is higher than the modulus of the direct quenched martensite.

When the temperature rate after the direct quench is higher ( $5 \times 10^{-2}$  K/s), some new effects appears:

- The time for the vacancies to move to the interfaces is smaller, which induces a shift of the maximum of the damping  $T_M$  to the higher temperatures.
- As all the interfaces are not pinned at 310 K an incomplete reverse transformation occurs. This is demonstrated by resistivity measurements, the internal friction peak and the modulus dip.
- An other small part of stabilized martensite is continuously transformed at higher temperature ( $t > 340$  K), inducing a temperature rate damping and acoustic emission.

### Conclusion.

In this work the stabilization of direct quenched martensite has been studied during heating at two different temperature rates.

The internal friction and modulus results agree with a progressive pinning of the interfaces between the variants by the quenched-in vacancies. When the temperature rate is sufficiently high, the martensite stabilisation is incomplete, and a partial reverse martensite transformation occurs.

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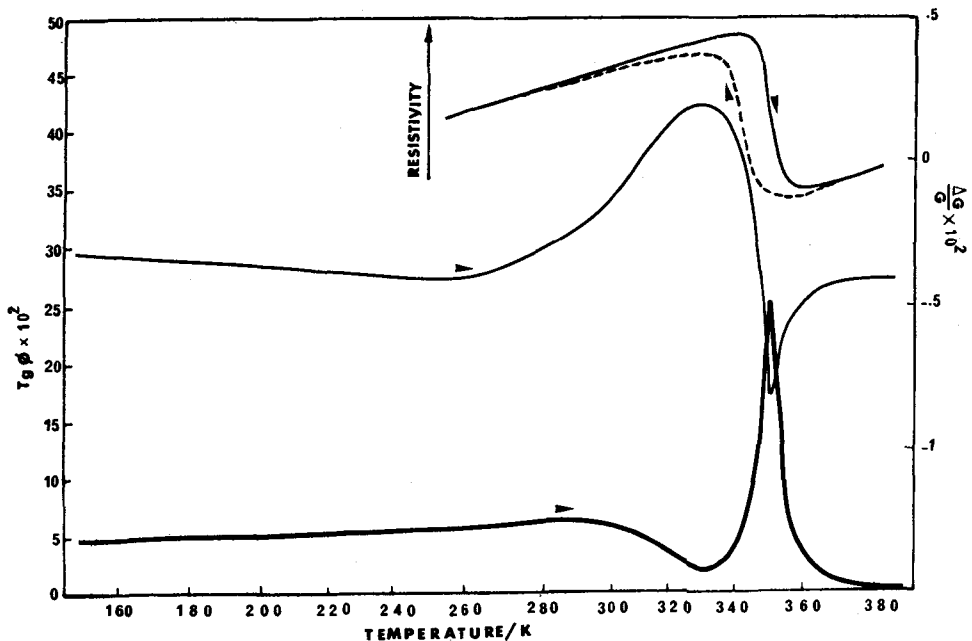


Figure 1.- Internal friction, modulus, and resistivity versus temperature.

- Standard sample.  $\epsilon_m = 4 \times 10^{-4}$ ;  $\dot{T} = 5 \times 10^{-3} \text{ K/s}$ ;  $f = 0.05 \text{ Hz}$ .
- All the resistivity curve are in the same scale.

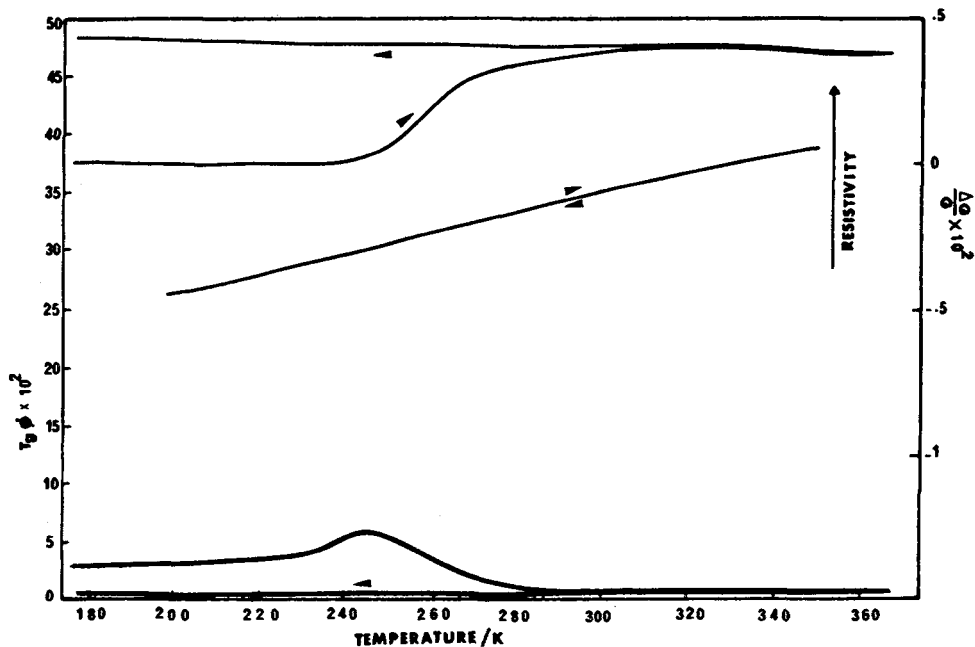


Figure 2.- Internal friction, modulus and resistivity versus temperature.

- Resistivity as been measured after the damping measurement.
- Direct quenched sample.  $\epsilon_m = 4 \times 10^{-4}$ ;  $\dot{T} = 5 \times 10^{-3}$  K/s;  $f = 0.05$  Hz

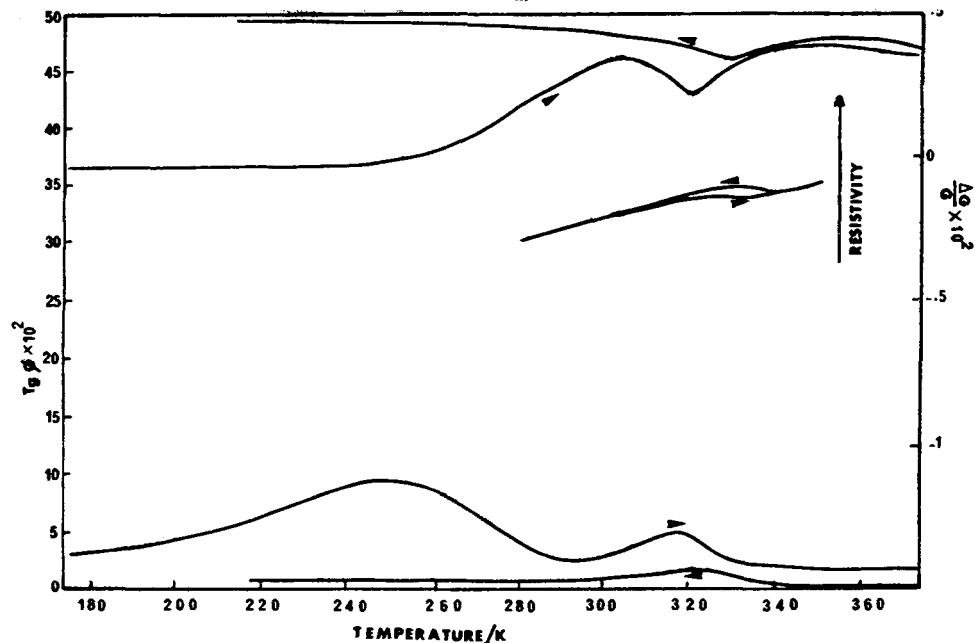


Figure 3.- Internal friction, modulus and resistivity versus temperature.

- Resistivity as been measured after the damping measurement.
- Direct quenched sample.  $\epsilon_m = 4 \times 10^{-4}$ ;  $\dot{T} = 5 \times 10^{-2}$  K/s;  $f = 0.05$  Hz.

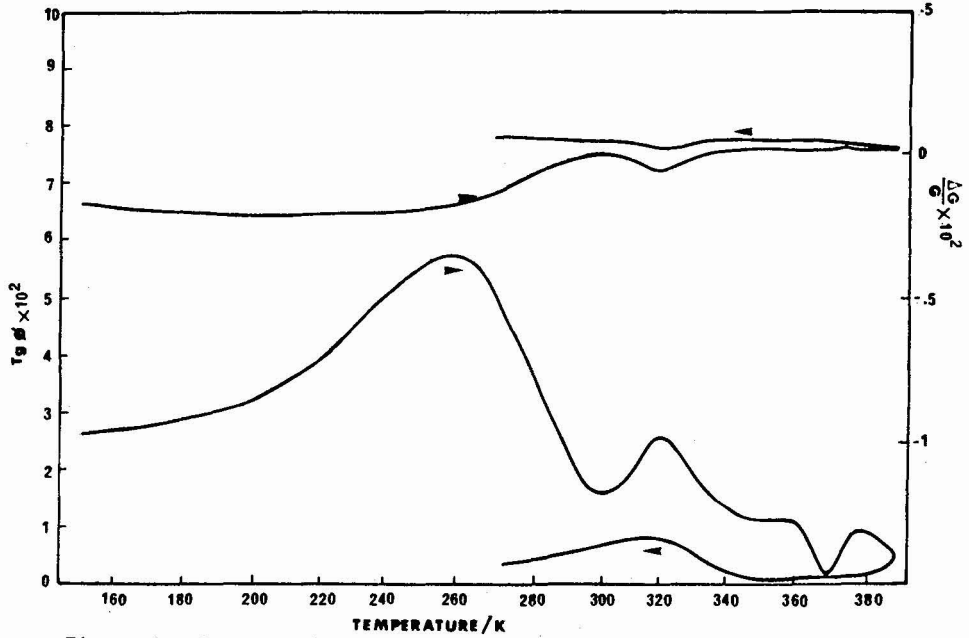


Figure 4.- Internal friction and modulus versus temperature.

-Direct quenched sample.  $\epsilon_m = 4 \times 10^{-4}$ ;  $\dot{T} = 5 \times 10^{-2}$  K/s;  $f = 0.1$  Hz.  
 - The heating has been stopped at 370 K.

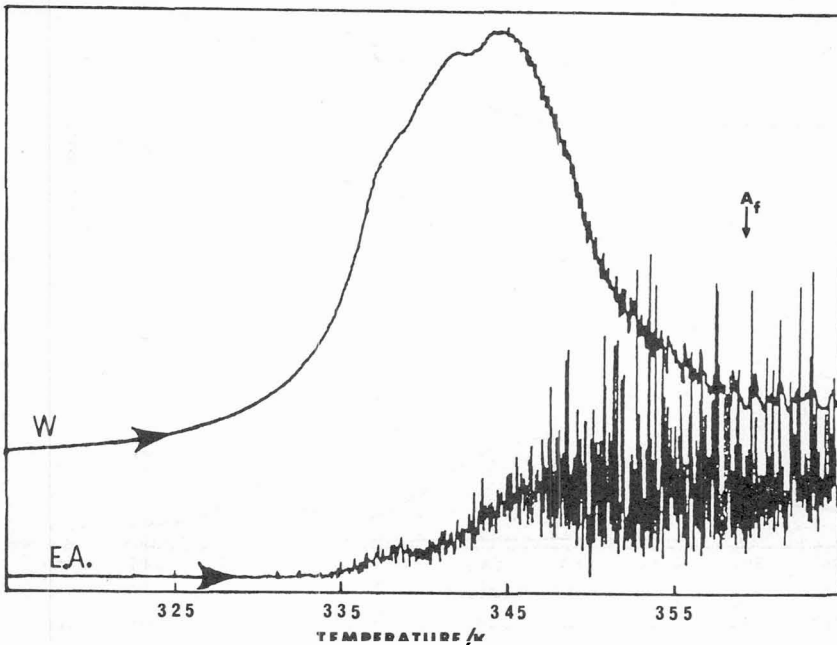


Figure 5.- Acoustic and Calorimetric curves for a partial stabilized sample (1).