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THE INFLUENCE OF MARTENSITE STABILISATION ON CHANGES IN DISLOCATION DENSITY IN Cu-Zn-Al ALLOY

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Résumé - On a examiné l’alliage Cu-Zn-Al avec l’effet de mémoire de forme, utilisant la méthode du frottement interne. En étudiant les dépendances d’amplitude du frottement interne on a constaté que pendant la stabilisation de la martensite la température ambiante, la densité et la mobilité des dislocations augmentent la présence des lacunes trempées.

Abstract - From analysis of the amplitude dependence of internal friction for Cu-Zn-Al alloy exhibiting the shape memory effect, the changes taking place in the structure of the martensite during ageing at room temperature were determined. It was ascertained that during stabilisation of the martensite, both the density and mobility of dislocations increase due to the presence in the martensite of a substantial concentration of quenched-in vacancies.

I - INTRODUCTION

The practical application of Cu-Zn-Al alloys exhibiting the shape memory effect associated with reversible martensitic transformation is governed by the condition of ensuring complete shape recovery and stability of transformation temperatures. Considerable attention has recently been devoted to the stability of properties and structure, and also to the understanding of the mechanism responsible for the process of stabilisation of these alloys, employing such research methods as: electron microscopy, X-ray diffraction, microcalorimetric measurements, measurements of mechanical properties and also of internal friction (IF) [1 - 10].

From investigations already carried out it was ascertained that the process of martensite stabilisation is a complex thermally activated process, markedly dependent on the concentration of quenched-in vacancies formed during quenching from a high temperature, leading to an increase in the temperatures of transformation, relative to those for nonstabilised alloys. The object of the investigations reported here was to study the influence of the process of martensite stabilisation in a Cu-(14.5%wt.)Zn-(8.5%wt.)Al alloy on variations in density and mobility of dislocations arising during this process taking place at room temperature, by making use of measurements of IF-amplitude dependence.

II - TEST MATERIALS AND METHODOLOGY

Tests were performed on samples of Cu-Zn-Al obtained by melting copper, zinc and aluminium of technical purity in an induction furnace. The ingot was homogenised by annealing at 1220K for 12 hours and then hot-rolled (370K) to a thickness of 0.8 mm. Samples of dimensions 35x10x0.8 mm were cut from the sheet obtained and used for IF measurements. To achieve a martensitic structure, samples were heated...
at 1020K for 15 minutes, next quenched to 573K at which temperature they were held for 15 seconds and then quenched in ice-water. Measurements of IF amplitude dependence were conducted at room temperature using a DMA (Dynamic Mechanical Analyser) acoustic frequencies relaxator, delivered by the DUPONT company [6] in a range of vibration amplitudes from $3 \cdot 10^{-5}$ to $6 \cdot 10^{-4}$, both immediately following heat treatment and also after various ageing times at room temperature. Parallel with measurements of curves of $Q^{-1} = f(\varepsilon)$, resonance frequency of sample vibrations was also measured. Based on these measured values it was possible to determine variations in Young's modulus, which is proportional to the square of the frequency.

III - MEASUREMENT RESULTS AND DISCUSSION

Fig. 1 shows the curves of internal friction and $f^2$ as a function of measurement time, measured at room temperature for a sample immediately after heat treatment.

![Fig. 1 Internal friction and square of resonance frequency of the sample as a function of time of ageing at room temperature ($T = 293K$); $\varepsilon = 3.2 \cdot 10^{-5}$](image)

On the $Q^{-1} = f(t)$ curve may be seen a distinct internal friction peak ($t = 2$ min) and a corresponding reduction in value of Young's modulus after which the modulus value increases with increase in ageing time. This observed IF peak is known as the "peaking effect of the first kind" (PEI) and occurs due to the interaction between vacancies and dislocations at the boundaries of the martensite plates [7,10]. With the aim of studying the changes taking place during stabilisation of the martensite, curves of IF-amplitude dependence were determined after various ageing times. As an example, on Fig. 2 are shown the curves $Q^{-1} = f(\varepsilon)$ and $f^2(\varepsilon)$ obtained directly after heat treatment (curve a) and also after 10 min (b), 30 min (c) and 60 min ageing (d).
On the curves $Q^{-1} = f(\varepsilon)$ may be observed initially a drop in IF, after which in the range of vibration amplitudes from $5 \cdot 10^{-5}$ to $1 \cdot 10^{-4}$ a small IF maximum is found whose height rapidly decreased during 20 minutes and next rose slightly (Fig. 3).
For $\varepsilon > 1 \cdot 10^{-4}$ a zone of IF weakly dependent on amplitude was observed, which increased with increasing ageing time, after which IF showed a rise. An IF maximum occurring on the IF amplitude curves has also been observed for, among others, pure aluminium samples containing a slight quantity of copper or magnesium, subjected to cold plastic deformation [11]. In the initial phase of the martensite stabilisation process the vacancies migrate to the dislocations, causing them to be pinned. The concentration of dislocation pinning points is not very large so that they can easily be unpinned which leads to a slight rise in the height of the IF peak as the ageing time is prolonged. For short ageing times the value of Young's modulus shows a slight increase, which is a corroboration of the postulated mechanism of formation of this peak in the IF-amplitude dependences. With increase in length of ageing time a weak increase in modulus value may be observed.

The IF-amplitude dependence being analysed was the Granato-Lücke model. Fig. 4 shows the curves of $\ln(Q^{-1}\varepsilon)$ as a function of $1/\varepsilon$ obtained from the $Q^{-1} = f(\varepsilon)$ curves given on Fig. 2 for a range of vibration amplitudes from $2 \cdot 10^{-4}$ to $6 \cdot 10^{-4}$. It may be seen that the points lie along straight lines, which is a confirmation that the high damping in the martensite phase is related to dislocations.

Then knowing the parameters of the Granato-Lücke curves and the strain amplitude $\varepsilon_c$, corresponding to the stress value needed to tear the dislocation segments away from the strong pinning points ($l_N$), using the relation $\varepsilon_c = b/l_N$ [12] (where b is the Burgers vector), the change in dislocation density as a function of time was estimated. On Fig. 5 (curve a) are shown the changes in dislocation density $\Lambda_t/\Lambda_o$ ($\Lambda_o$ is the dislocation density observed after 5 minutes ageing at room temperature, counting from the moment of finishing heat treatment) taking place during the process of martensite stabilisation. It may be seen that during 40 minutes ageing time the dislocation density does not change, after which it rises, achieving after 60 minutes a level 1.5 times that observed after 5 minutes. Increase in dislocation density in the martensitic phase is also observed after cyclic martensitic transformations [13 - 16].

Using the measured values of vibration frequency $f$ and knowing the variations of Young's modulus $\Delta E/E_o$ and also dislocation density and value $\varepsilon_c$, the dislocation mobility was estimated, using the following relation [17]:

$$\dot{\varepsilon} = 4f\left(\frac{\Delta E}{E_0}\right)\frac{\varepsilon_c}{\Lambda b}$$
Variations in dislocation mobility $\delta_i/\delta_o$ taking place during martensite stabilisation at room temperature are shown on Fig. 5 (curve b).

After 40 minutes ageing mobility rises to 4 times its initial value, and next begins to decrease reaching after 60 minutes a value 3.5 times greater than that found after 5 minutes. The observed changes in both density and mobility of dislocations suggest that the vacancies frozen due to step-quenching from a high temperature, during the ageing process form clusters leading to the formation of dislocation loops and stacking faults. Stabilisation of the martensite is governed both by pinning of the martensite boundaries and dislocations by the migrating vacancies and also by the formation of stacking faults.

IV - CONCLUSIONS

- Analysis of IF-amplitude dependences showed that the martensite stabilisation process in Cu-Zn-Al alloy exhibiting the shape memory effect, is associated with freezing of the saturated vacancies in the martensite.
- During the martensite stabilisation process the quenched-in vacancies form clusters which are, presumably, the cause of the formation of dislocation loops and stacking faults, which are responsible for the variations in density and mobility of dislocations.
- After 60 minutes ageing at room temperature a 1.5 times growth in density and a 3.5 times growth in mobility of dislocations is observed, relative to the values found immediately after heat treatment.

REFERENCES