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Nucleation of Martensite in CuZnAl Alloys as Investigated by Young's Modulus and Acoustic Emission Measurements

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Abstract. Young's modulus (E) has been measured during cooling/heating cycles carried out between a fixed upper temperature T_2 ($T_2=340$ K) and a gradually decreasing lower temperature T_1 ($T_1=320, 310, 300, 290, 285, 281$ and 279.5 K). The $E(T)$ curves show closed thermal hysteretic loops, which are needle-like for $T_1 > M_s$ while they assume a downward bent shape for $T_1 < M_s$. The occurrence of thermal hysteresis in the cooling/heating $E(T)$ curves is interpreted as evidence of the formation, far above M_s , of static martensite embryos, which are stabilized by preexisting lattice defects. The formation, on cooling, and the delayed disappearance, on heating, of these static embryos is believed to be responsible for the needle shaped $E(T)$ loops. Acoustic emission is found to occur only during the heating stages of partial thermal cycles carried out above M_s , suggesting that only the disappearance of a stabilized embryo is associated with an abrupt release of the stored energy.

1. INTRODUCTION

Many different aspects of martensitic transformation (MT) have attracted attention during the past decades, among these are pre-transitional [1-6] and nucleation/growth phenomena [7-12]. By virtue of the considerable amount of experimental [7-8] and theoretical [9-12] work carried out so far, it is now generally accepted that MT is initiated by heterogeneous nucleation, that is, nucleation triggered by localized strain fields associated with pre-existing lattice defects. It is also well established by now that precursor effects, such as softening of the C' shear elastic constant, usually accompany weak first order MT. Although pre-martensitic effects have not been revealed by NMR experiments [13], nonetheless, CuZnAl alloys exhibit substantial softening of the C' shear elastic constant, as well as strong anharmonic behavior [14,15]. In this same series of alloys synchrotron x-ray topography has been able to reveal heterogeneous nucleation followed by unlimited growth of martensite at sub-grain boundaries and only in the close vicinity of M_s [8]. No evidence of the formation of static embryos above M_s seems to have been provided so far.

The main aim of the present work has been that of studying pre-martensitic phenomena in CuZnAl alloys, far above M_s , by accurate Young's modulus (E), acoustic emission (AE) and differential scanning calorimetry (DSC) measurements. In the present paper results regarding Young's modulus and AE are presented and discussed.

2. EXPERIMENTAL

The atomic composition of the alloy used in the present work was 67.83 Cu, 19.03 Zn and 13.4 Al. After melting, the material was homogenized at 1073 K for about 4×10^4 s, then machined to the form of rectangular bars of dimensions $2 \times 5 \times 50$ mm³. Prior to any measurement, the samples were treated at 1050 K for 1200 s, quenched into boiling water in the β phase and then stored at room temperature for several months. This sequence of thermal treatments guarantees that vacancies are at equilibrium. The Young's modulus was derived from the resonant frequency, which at room temperature was about 2720 Hz. The experiments were performed in vacuo at cooling/heating rates of about 10^{-1} K/s. Measurements of AE were carried out on a sample that had been cut from the same bar as that used for the elastic modulus observations, prior to any experiment.

3. RESULTS

From Young's modulus, DSC and AE data taken during complete thermal cycles of the material in the same conditions the mean characteristic temperatures for the direct and reverse transition are the following : $M_s=280$ K, $M_f=271$ K, $A_s=282$ K, $A_f=288$ K

Figures 1a-1d show data taken during thermal cycles carried out in sequence between a fixed upper temperature T_1 ($T_1=350$ K) and a gradually decreasing lower temperature T_2 ($T_2=320, 310, 300, 290, 285, 281$ and 279.5 K). As can be seen in fig.1a the cooling and heating stages of the cycle perfectly overlap one another indicating full absence of any nucleation process of the martensitic phase above 320 K. The temperature dependence of the Young's modulus only slightly departs from linearity, the temperature coefficient dE/dT becoming gradually less negative with decreasing T . With cycles extending further down in temperature, cooling and heating curves do not match anymore and tend to display increasingly wider thermal hysteric loops. For $T_2 \geq 290$ K these loops are needle like, while they assume a curved shape for lower values of T_2 .

An interesting aspect of all the Young's modulus cooling/heating curves is the fact that they tend to become closed loops provided the sample is heated up to sufficiently high temperatures (typically 340 K) and that the cooling branches are retraced during successive cycles. This means that whichever is the source of the hysteresis this source disappears on heating above 340 K.

Figure 1: Young's modulus as measured during thermal cycles between a fixed higher temperature $T_1=350$ K and a gradually decreasing lower temperature $T_2=320$ K (a), 310 K (b), 300, 290 K (c) and 285, 281, 279.5 K (d)

Acoustic emission events recorded during a complete heating/cooling cycle are reported in fig. 2, where is also shown the noise level as deduced from a dummy sample. As can be seen in this figure, the number of counts is about two times higher for the reverse than the direct transition, in agreement with other experiments [16]. Fig 3 shows AE data taken during partial cycles carried out between 290 and 330 K and 300 and 330 K that is, at temperatures higher than M_s . As can be seen, during cooling the emission is at noise level, while during heating it is about 20 times higher. This observation, which has also been observed in three additional partial cooling/heating cycles, is in agreement with results by [16], who also measured

Figure 2: AE counting rate as measured in a sample and in a dummy sample during a thermal cycles between 255 and 330 K

Figure 3: AE cumulative counts as measured in a sample and a dummy sample during thermal cycles at $T > M_s$ (281 K)

AE at temperatures higher than M_s during the heating portion of thermal cycles carried out over narrow temperature ranges. Optical microscopy did not reveal the occurrence of any martensitic platelet during these cycles.

4. DISCUSSION

The bulk of the experimental evidence presented in this paper clearly shows that some sort of martensite nucleation starts to occur far above M_s . It is unlikely that the hysteretic effects observed in the Young's modulus are associated with the formation of fully developed martensite platelets at high temperature as a consequence of chemical inhomogeneity of the material. As a matter of fact, the temperature ranges of both the direct and reverse transitions are narrow and well defined. Early formation of martensite platelets at the external surface of the sample can be ruled out as a source of AE above M_s , since optical microscopy did not succeed in detecting them [16]. Also the absence of AE on cooling and of anomalies in the heat flow (on cooling and on heating) [17], strongly support the idea that martensite platelets are not formed far above M_s . This conclusion is also in accordance with synchrotron x-ray topography observations, which provide evidence of martensite platelets just at temperatures close to M_s .

On the basis of these considerations the observed elastic hysteresis can more reasonably be attributed to the formation of embryos of subcritical size stabilized by the strain field of strong lattice defects [18]. The situation is illustrated by fig.4, taken from [18], where the work for the formation of a martensite embryo interacting with a strong defect is schematically plotted as a function of the embryo size (r), for two different levels Δg_1 and Δg_2 ($\Delta g_1 < \Delta g_2$) of the driving force of the transition, that is, for two different temperatures T_1 and T_2 ($T_1 > T_2$). At temperature T_1 an embryo of size r_c smaller than the critical size r^* is stabilized by the insurmountable energy barrier ΔW^* . This embryo will start growing

only at temperatures $T \leq T_1$ where ΔW^* becomes surmountable by thermal activation. For $T \leq T_2$ the interaction with the lattice defect leads to barrierless nucleation, immediately followed by unlimited growth, which will

Figure 4: Schematic plots of the energy (ΔW) vs. size r of a martensitic nucleus formed near a weak and strong defect for different levels of the driving force ΔG (from 18)

give rise to an autocatalytic nucleation process. The actual features of embryo stabilization depend on the detailed structure of the nucleating defect and are expected not to be identifiable on the basis of the present results.

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