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Internal Friction and Elastic Properties of Nanocrystalline Fe-Si-Nb-Cu Alloy

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Abstract. The anomalous elastic modulus reduction in the nanocrystalline $\text{Fe}_{73.4}\text{Cu}_1\text{Nb}_{3.1}\text{Si}_{13.4}\text{B}_{0.1}$ alloy was found. Sound wave velocity and internal friction as a function of annealing temperature were measured in a magnetic field by the vibrating reed technique. In the as-prepared state a large modulus defect was observed which disappeared after annealing above a characteristic temperature (much less than the crystallization temperature) defined as a temperature of the structural rearrangement. The "in-situ" measurements revealed the irreversible internal friction peak and an accompanying elastic modulus increase at this temperature. In the same temperature region the irreversible changes of the ΔE -effect were observed.

1. INTRODUCTION

In the last years much attention was attracted to the investigations of the unique physical properties of the materials with nanocrystalline structure. The elastic and dissipative properties of nanocrystalline materials exhibit a behaviour which differs strongly from that of coarse grain ones [1 to 3] (the anomalous moduli reduction and irreversible internal friction peak at the temperature of the primary recrystallization). These results were obtained for the specimens prepared by gas-condensation process followed by consolidation at high pressure (grain size was from 5 to 60 nm) and by severe plastic deformation (grain size from 100 to 200 nm).

In this paper we investigated the same phenomena in the specimens with nanocrystalline structure but prepared by crystallization from amorphous state. We used amorphous ferromagnetic alloy Fe-B-Si containing additions of Cu and Nb. Heat treatment of this amorphous alloy above the crystallization temperature produces a homogenous ultrafine grain structure with a typical grain size 5 to 20 nm and random texture.

2. EXPERIMENTAL

The effect of magnetic field and temperature on elastic and dissipative properties were studied. All experiments were carried out on the nanocrystalline specimens of the $(20 \times 2 \times 0.025)$ mm³ Fe-Si-Cu-Nb-B ferromagnetic alloy. This material was prepared from a melt - spun amorphous ribbon by annealing at a temperature higher than the crystallization temperature in a tin bath at 580°C for a short time (from 10 to 30 s), followed by water quenching [4]. For comparison, ribbons were used which were annealed in the furnace in an argon atmosphere for 1 hr at 560°C, followed by cooling in the furnace. The microstructure produced by the procedure described consisted of ultrafine grains of about 10 nm and residual amorphous phase ($\approx 25\%$), revealed by the X-ray diffraction technique. The elastic and dissipative properties of the specimens were measured by the vibrating reed method [5] at the frequencies from 100 to 300 kHz within the temperature range from room temperature up to about 500°C. The static magnetic field was created by Helmholtz coils and was applied parallel to the long axis of the specimen. In order to obtain the "pure" elastic contribution to the total magnitude of an effective elastic modulus the measurements were conducted in the saturating magnetic field.

Fig.1: The relative dependences of Young's modulus E on magnetic field for amorphous (1), rapidly crystallized (2) and furnace annealed (3) specimens. E_s is the magnitude of Young's modulus in saturating magnetic field.

3. RESULTS

The dependences field of relative change of Young's modulus E on magnetic field for amorphous, rapidly crystallized and furnace annealed nanocrystalline specimens at room temperature are shown in Fig.1. The magnitude of Young's modulus E_s in saturating magnetic field was greater for the furnace annealed specimens (on $\approx 7\%$ in comparison with rapidly crystallized and $\approx 30\%$ in comparison with amorphous specimens). It is seen that the magnitude $\Delta E/E = (E_s - E)/E$ (so-called ΔE -effect) in zero magnetic field is approximately the same for all specimens, but the saturating field for the nanocrystalline specimens is much lower than for amorphous ones.

The results of the temperature measurements for rapidly crystallized specimens are given in Figs.2 and 3. Fig.2 illustrates the temperature dependence of the internal friction δ and Young's modulus E during the primary and repeated heating "in situ" with a rate of ≈ 2 deg/min. One can see the irreversible internal friction peak at $\approx 200^\circ\text{C}$ and an accompanying Young's modulus change. It is very important to note that the temperature dependences of the internal friction and the Young's modulus are very similar to those observed in nanocrystalline Pd [1] and in submicrocrystalline Cu [3].

It will be noted that such irreversible variations in Young's modulus and attenuation were observed only for as-prepared specimens obtained by rapid crystallization. For as-prepared specimens obtained by the furnace annealing there were no irreversible changes during and after heatings up to 470°C . Within the accuracy of measurements the room temperature value of Young's modulus in such specimens was the same as that for rapidly crystallized specimens after annealing at temperatures above 300°C .

The temperature dependence of ΔE -effect upon heating and cooling is given in Fig.3. The irreversible variation of ΔE -effect for the as-prepared rapidly crystallized specimen is observed in the temperature range up to 300°C , but the room temperature value of the ΔE -effect does not change significantly.

DISCUSSION

According to the structural investigations [4,6,7,8] the Fe-Cu-Nb-Si-B nanocrystalline alloy obtained by the procedure described in the Experimental is a composite which consists of three metastable

Fig.2: The temperature dependences of logarithmic decrement δ and Young's modulus E during primary and repeated heating.

Fig.3: The temperature dependences of ΔE -effect for the as-quenched rapidly crystallized specimen at the heating and cooling.

phases (nanocrystalline matrix, residual amorphous phases and inclusions of Cu clusters). The latter occupies a very small volume. Therefore we can ignore its contribution to the total elastic modulus. Since Young's modulus of the amorphous state is lower than in the crystalline one, Young's modulus of such composite must be higher than that of the initial amorphous one. Although the nanocrystalline and amorphous phases considered are metastable, only long-term annealings at temperatures above the crystallization temperature leads to grain growth and to a decrease of the residual amorphous phase volume. From this fact one can conclude that below about 500°C the structure of the three phases is fixed, that is the relation between the volumes of these phases is unchanged. If we use the elastic consideration it means that annealing below 500°C should not influence Young's modulus. However, the experimental fact that the annealing increases significantly (about 7%) Young's modulus of the as-prepared nanocrystalline specimens makes us think that the problem is more complicated than one can assume on first sight. As we have pointed out above, the curves in Fig.2 are very similar to those obtained for nanocrystalline Pd [1] and submicrocrystalline Cu [3]. Therefore, by analogy with the explanations given in [2,3,9] we can assume that the phenomenon observed could be caused by the anelastic relaxations of structural defects in the internal stress field. The annealing is likely to lead to the structural rearrangement and decrease of internal stresses that results in a lowering of the anelastic relaxation contribution to Young's modulus.

One of the possible explanations of such structural rearrangement can be made on the basis of the theoretical work [10], where it was shown that the high level of internal stresses in as-prepared ultrafine grain materials is due to the very high density of the different kinds of grain boundary dislocations. If it is so, the elastic modulus reduction observed in our experiments may be explained by the anelastic effects due to the movement of glissile grain boundary dislocations under applied oscillating stresses. In terms of such a suggestion the observed increase of Young's modulus at the temperatures near 200 C (characteristic temperature) is caused by the decrease of the density of glissile dislocations. The structural rearrangement of such kind should lead to the irreversible internal friction similar to that discussed in our previous paper [9].

The assumption on the structural rearrangement is confirmed indirectly also by ΔE -effect studies since the irreversible changes of the ΔE -effect occur in the same temperature region (Fig.3). The lowering of the level of internal stresses σ_i caused by the structural rearrangement is manifested in the decrease of the magnitude of the saturating magnetic field for ΔE -effect (Fig.1).

However such rearrangement (as one can see from Fig.1) does not change the value of ΔE -effect at room temperature. One can explain this fact from the following considerations. It is well known that ΔE -effect is directly proportional to the magnetostriction value and inversely proportional to the magnitude of internal stresses. Since σ_i decreases after heat treatment while the value of the ΔE -effect does not change it means that the value of magnetostriction for this materials is proportional to the value of internal stresses.

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