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Crystallization of Metallic Glasses After Severe Plastic Deformation

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Abstract

The crystallization kinetics of Co based amorphous alloys after severe plastic deformation was studied by differential scanning calorimetry, X-ray diffraction and scanning probe microscopy. Severe plastic deformation was achieved by barocryodeformation (all-round compression). The results are compared with biaxial stretching by ion irradiation. It is shown that the crystallization rate increases after biaxial stretching and decreases after all-round compression.

Keywords: crystallization, metal glass, severe plastic deformation

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1. Introduction

Severe plastic deformation (SPD) changes the characteristics of phase transitions in metallic glasses. This was shown by the example of the crystallization of metallic glasses $CoFe_{3.2}Si_{2.5}Mn_{3.1}B_{15.7}$ after ion irradiation [1]. The crystallization heat of glass is increased by 30% compared with the crystallization heat of initial unirradiated glass as was shown by the DSC measurements after low temperature irradiation by 30 keV Ar^+ ions. Ion irradiation leads to nanostructuring of the metallic glass surface. This indicates that the severe plastic deformation takes place during the ion irradiation.

According to [2] ion irradiation leads to the biaxial stretching of material. The induced strain reaches $(10^{-3}–10^{-2})E$ (E - Young's modulus). Such strain exceeds the yield strength of the metal at a depth of tens of microns. Nanostructure of plastic deformation is formed at depth much deeper than the projective ion range.

In this paper the crystallization process of metallic glass $CoFe_{4.9}Si_{14.9}B_{10}$ after severe plastic deformation by all-round compression were studied. All-round compression was achieved by barocryodeformation method - quasi-hydroextrusion at cryogenic temperature.

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2. Experimental results

The amorphous alloys $CoFe_{4.9}Si_{14.9}B_{10}$ obtained by melt spinning technique had the form of a band with a width of 13 mm and a thickness of 30 μm. All initial samples were in an X-ray amorphous state.

Samples were subjected to SPD method of low-temperature quasi-hydroextrusion [3, 4] with all-round compression of ~ 2.5 GPa at a temperature 77 K. The structure and composition of the initial and subjected to severe plastic deformation samples was studied using a differential scanning calorimeter 204 F1 Phoenix, X-ray diffractometer DRON-8, a scanning probe microscope Certus.

Figure 1 shows results of DSC with heating rate of 10 K/min in the temperature range from 100 to 600°C. Three well-defined exothermic peaks can be observed on the DSC curves of the samples. The first peak at T = 378–385°C corresponds to a certain structural relaxation of the amorphous metallic alloy. The second and the third crystallization peaks are observed at temperatures of 512°C and 554°C.

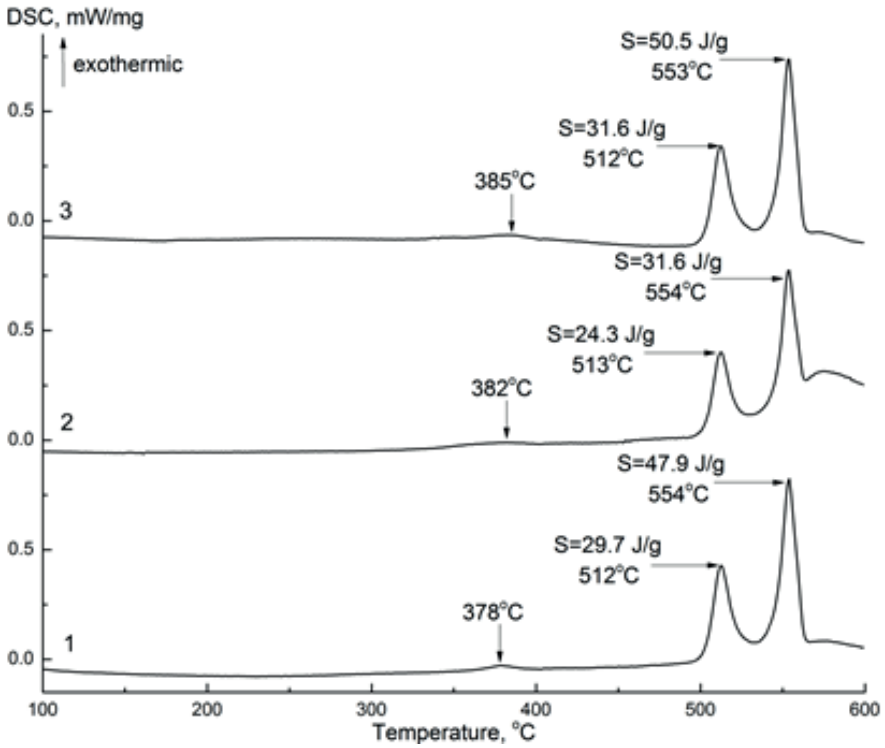

Figure 1: DSC of the amorphous alloy $CoFe_{4.9}Si_{14.9}B_{10}$, in initial state (1), after SPD (2, 3). S – peak area.

Exothermic crystallization peaks of samples 1 and 3 are almost identical. Peak areas of the sample 2 at temperatures of 512°C and 554°C is less by 15% and 35% respectively.

XRD patterns of samples after short-time annealing at 600°C are shown on Figure 2. Crystalline phases peaks appear against the background of an amorphous halo that indicates partial crystallization of the samples.

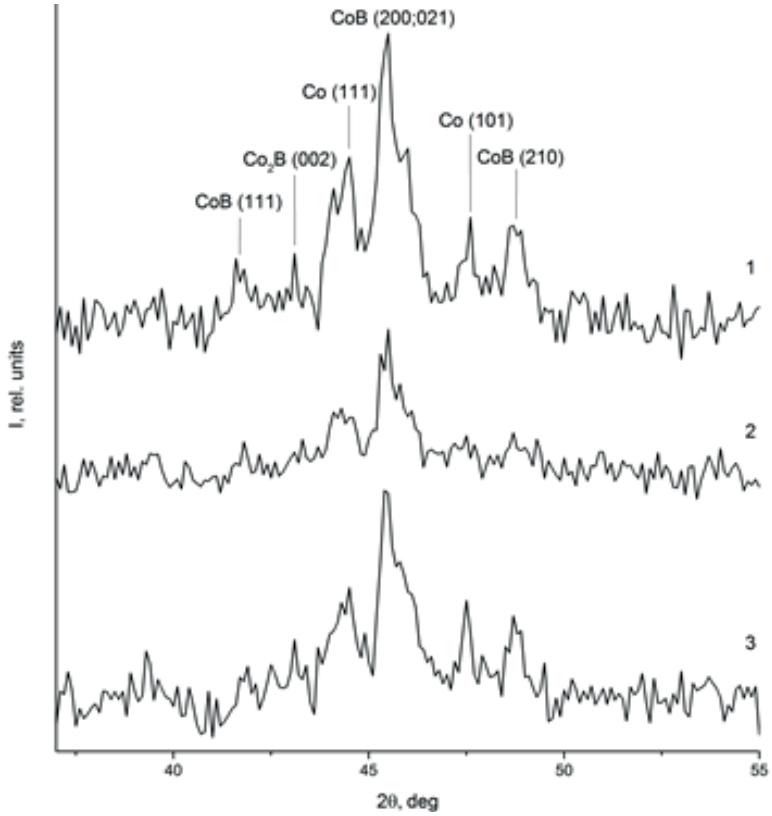

Figure 2: XRD patterns of the amorphous alloy $CoFe_{4.9}Si_{14.9}B_{10}$ after short-time annealing at 600°C, in initial state (1), after SPD (2, 3).

Peaks on XRD patterns correspond to crystalline phases of a solid solution of cobalt and cobalt borides CoB, Co_2B. The XRD pattern of sample 2 shows crystalline phase peaks with less intensity.

Micro- and nanostructures of samples 2 and 3 differ from one another. After barocryodeformation samples were subjected to room temperature. As a result cracks formed through the volume of sample 3 with size up to 0.1 mm. And complex network of deformation bands formed on the surface of sample 2.

Figure 3 shows the nanostructure samples 2 and 3. There are shear bands in the form of steps 400 nm in height in sample 2. Nanocrystalline inclusions are ordered along the shear bands. A large number of cracks and inclusions of a size of about 100 nm can be seen on the surface of the sample 3.

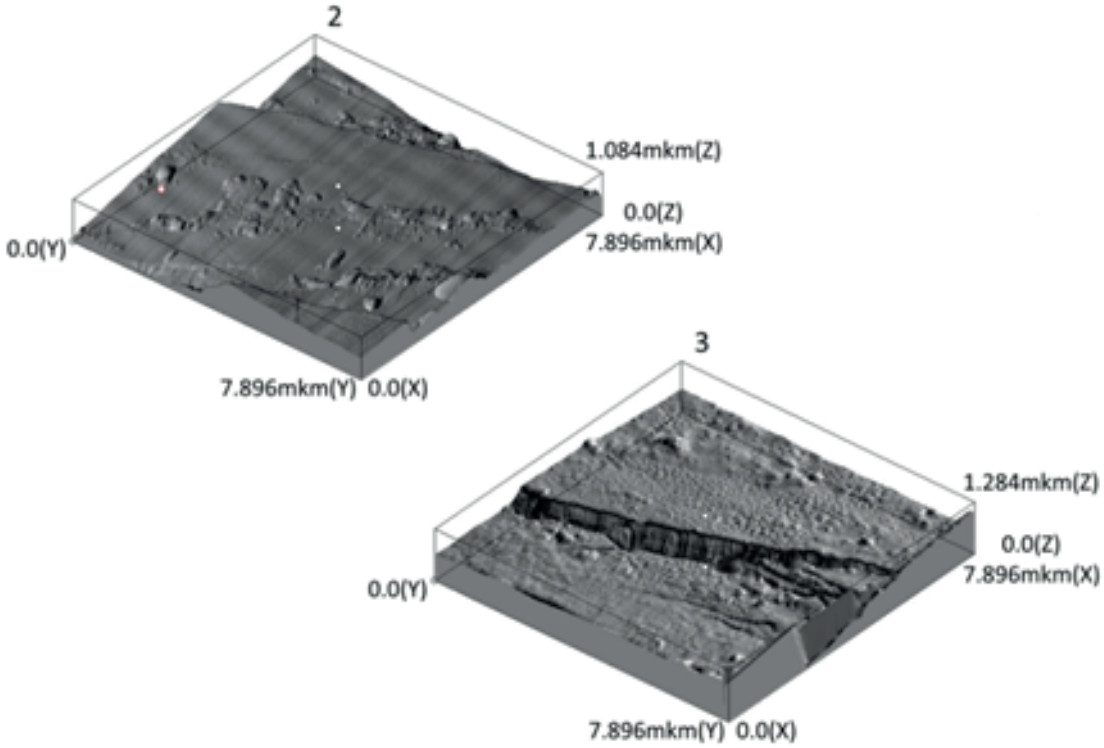

Figure 3: SPM images of nanostructure of samples 2 and 3 (after SPD).

3. Discussion

The crystallization rate of metallic glass is defined as the probability of the transition:

$$W \sim \exp\left(-\frac{H}{kT}\right) \tag{1}$$

where H is the transition enthalpy.

$$H = \Delta U + P\Delta V \tag{2}$$

where ΔU is difference of internal energy of the amorphous and crystalline phases. The elastic strain energy is added to (2) in the presence of internal strain P. ΔV is the excess of atom volume in the amorphous phase, which is up to 10% in metal glasses [5]. Crystallization kinetics of amorphous alloy is affected by internal strain. Defrosting after severe all-round compression induces positive residual internal strain, enthalpy (2) increases, the crystallization rate (1) decreases. Ion irradiation (stretching deformation) induces negative residual internal strain and the crystallization rate increases [1].

The residual strain in sample 2 leads to a reduction in the crystallization rate up to 30%. This causes the reduction of peaks intensity on DSC curves (Figure 1) and on X-ray spectrum (Figure 2). Crystallization kinetics of sample 3 has not changed. In this sample strain relaxation by crack formation has occurred during defrosting after deformation

(Figure 3). Assuming that the crystallization rate ratio of the original glass and the glass 2 is $W_1/W_2 \sim 1.3$ and $\Delta V \sim 6.4 \cdot 10^{-3}$ nm^3 (10% of atomic volume) and using expression (1) gives an estimated value of the residual internal strain in the deformed metallic glass ~ 460 MPa.

4. Conclusion

Severe plastic deformation induces residual internal strain in the metallic glasses. The crystallization rate increases if the internal strain is negative. The crystallization rate decreases if the internal strain is positive.

It has been shown by differential scanning calorimetry, X-ray diffraction analysis and scanning probe microscopy that severe plastic deformation achieved by barocryodeformation of the metallic glass $CoFe_{4.9}Si_{14.9}B_{10}$ induces positive residual internal strain. The estimated value of residual strain is 460 MPa. The residual strain decreases crystallization rate by 30%.

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