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The Use of Atomic-Force Microscopy for Studying the Crystallization Process of Amorphous Alloys

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Abstract

The crystallization process of amorphous alloys is accompanied by the volume changes as a result of structural phase transitions. This leads to changes in the surface topography, which was studied by atomic force microscopy (AFM). The changes of the surface topography, structure and phase composition during multistage crystallization process of the metallic glasses with composition Ni71,5Cr6,8Fe2,7B11,9Si7,1 and Ni63,4Cr7,4Fe4,3Mn0,8B15,6Si8,5 (AWS BNi2) has been investigated. The obtained results on changing of the surface topography in crystallization process are in good agreement with the data of X-ray diffraction analysis (XRD). The nature of redistribution of some alloy components in the crystallization process has been suggested.

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Keywords: atomic force microscopy; AFM; metallic glass; amorphous alloy; crystallization; crystalline structure; nickel; boron; silicon; boride; BNi2; surface topology; phase composition

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1. Introduction

X-ray diffraction (XRD), scanning and transmission electron microscopy (SEM and TEM) are commonly used to study the crystallization process of amorphous alloys. Atomic force microscopy (AFM) can provide more information about the processes. High sensitivity of AFM to changes in surface topography (not less than 0.1 nm) allows to detect such changes of surface during the initial stage of crystallization, which cannot be detected using SEM. However, systematic studies of the surface topography changes during the crystallization of multicomponent amorphous alloys have not been conducted yet.

Particularly important is consideration of changes in the surface topology under the structural and phase transformations occurring in the sample volume. Such research is essential to ensure the absence of oxides on the surface of the sample. Thus, amorphous nickel-based alloys were chosen for research, containing silicon and boron in addition to the nickel, in amounts close to the eutectic composition (BNi2 according to the American Welding Society classification). It is known that crystallization of such amorphous alloys proceeds in three stages [1-3].

The first stage of crystallization (450–500°C) is associated with the formation of solid solution crystals based on nickel (α-Ni), the second stage (500–550°C) – with the formation of a metastable τ-phase with $M_{23}B_6$-type structure, in the final stage (550–650°C) the formation of Ni_3B and Ni_3Si as a result of the decomposition of τ-phase was reported [1, 2].

The purpose of this work is to explore the possibility of application of atomic force microscopy for the analysis of the processes occurring during crystallization of amorphous alloys of complex composition, as well as clarification of the mechanisms of microstructure transformations in the multistage crystallization of amorphous nickel-based alloys.

2. Materials and Methods

The amorphous alloys with two similar to BNi2 compositions $Ni_{71,5}Cr_{6,8}Fe_{2,7}B_{11,9}Si_{7,1}$ (STEMET® 1301A) and $Ni_{63,4}Cr_{7,4}Fe_{4,3}Mn_{0,8}B_{15,6}Si_{8,5}$ (STEMET® 1301) (hereinafter referred to as 1301A and 1301) were obtained in the form of tapes with a thickness of 40–50 μm by the rapid solidification techniques [4]. The annealing of ribbon samples were carried out in a vacuum furnace with heating rate 20 deg/min. The modes of annealing of samples and their phase composition are given in table 1.

Table 1. The modes of annealing and phase compositions of the amorphous alloys.

Sample	Alloy	Annealing	Phase composition	Note: The crystal structures of phases [2, 3]
A	1301A	500°C, 1min	α-Ni +A (amorphous phase)	α-Ni: FCC structure (a = 0.3530 – 0,3535 nm)
B	1301	550°C, 1min	α-Ni + τ + A (amorphous phase)	τ-phase: cubic structure (a = 1,0496 nm);
C	1301	550°C, 15min	α-Ni + τ + Ni_3B (small amount)	Ni_3B: orthorhombic cubic structure;
D	1301	600°C, 60 min	α-Ni + τ + Ni_3B + Ni_3Si	β_1-Ni_3Si: ordered FCC structure Cu_3Au (a = 0,3513 nm)
E	1301A	650°C, 60 min	α-Ni + Ni_3B + Ni_3Si	

The first stage of crystallization was studied on the alloy 1301A because in the alloy 1301 first and second stage occurs almost simultaneously. The second and third stages were studied on the alloy 1301 with a higher content of boron, which produced more of boride and silicide phases. The study of the surface topology was performed on the scanning probe multi-microscope SMM 2000 (Proton-MIET, Russia) in the contact mode of atomic force microscopy. X-ray diffraction phase analysis was carried out on D8 DISCOVER (Bruker) at CuK_α-radiation.

The studies were conducted on the free surface of the tape, as opposed to the contact one it had fewer surface defects and, furthermore, according to the paper [5], the contact surface of the rapidly quenched alloys from the liquid phase of the Fe-Ni-Si-B system is depleted of boron and silicon. Previously, we have shown that the microstructures formed during the crystallization on both surfaces of the tape hardly differ [2], however formation of crystalline phases on the contact surface begins a little earlier than at the free one [6].