In-Situ TEM Observation of $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ Amorphous Alloy

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Crystallization process of amorphous $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy is observed at 733K by in-situ TEM. Results show that crystalline process of this alloy presents three-step type, i.e. incubation, phase separation and crystallization. Before precipitation of bcc-Fe, [200] and [220] diffraction rings of cubic-carbon appear first. With the increase of annealing time, these diffraction rings of cubic-carbon disappear.

The bcc-Fe grains of $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ and $\text{Fe}_{79.5}\text{P}_{12}\text{C}_6\text{Cu}_{0.5}\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy, annealed at 673K, present different morphology. Copper is necessary in the formation of FePC based nanocrystalline alloy.

Key words: in-situ TEM, amorphous alloy, crystallization, phase separation, morphology

1. Introduction

Nanocrystalline $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ ¹⁾, $\text{Fe-Zr-B-1\%Cu}$ ²⁾, and $\text{Fe}_{78}\text{P}_{16}\text{C}_2\text{Cu}_{0.5}\text{Ge}_3\text{Si}_{0.5}$ ³⁾ have drawn much attention for their excellent magnetic properties in last few years. Much work is done on the crystallization of Finemet and FeZrB(Cu) alloys by TEM⁴⁾⁻⁶⁾. Results show that copper is an important element in the formation of homogeneous nano-size bcc-Fe in both Finemet and FeZrB(Cu) alloys. The role of copper has not been clearly illustrated on FePC based nanocrystalline alloy.

From our previous work, $\text{Fe}_{79.5}\text{P}_{12}\text{C}_6\text{Cu}_{0.5}\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy, annealed at the temperature range of 613K to 673K, has good soft magnetic properties. The alloy has a nanocrystalline structure with grain size of bcc-Fe about 22nm. For the sake of contrast, copper-free $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy will be studied too.

In-situ transmission electron microscopy (TEM) is a useful method for observing crystallization process of amorphous alloys, and provides much information about nucleation and growth of grains. In order to understand the crystallization process of copper-free FePC based amorphous alloy, the in-situ TEM observation is done on $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy first in this paper. Then the morphology and distribution of bcc-Fe gains in $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ and $\text{Fe}_{79.5}\text{P}_{12}\text{C}_6\text{Cu}_{0.5}\text{Mo}_{0.5}\text{Si}_{1.5}$ alloys, annealed at 673K, are compared.

2. Experimental methods

Amorphous $\text{Fe}_{79.5}\text{P}_{12}\text{C}_6\text{Cu}_{0.5}\text{Mo}_{0.5}\text{Si}_{1.5}$ and $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloys are prepared by single roller method. Then zero-field annealing at N_2 atmosphere is performed for both alloys. The microstructure is observed with H-800 transmission electron microscope. JEM-2000CZ transmission electron microscope is used for in-situ TEM observation.

Thin foils for TEM observation are prepared by ion thinning. An electron microscopy specimen holder heats the foil during observation.

3. Results and Discussion

3.1 Crystallization of $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ amorphous alloy

Figure 1 shows the in-situ transmission images and selected area diffraction patterns (SAD) of $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy at different stages of annealing at 733K. The as-quenched sample presents typical character of amorphous alloy (see Fig.1 (a), (b)). Then we heat the sample to 733K rapidly and keep the temperature at 733K for different time. No change in the bright field image takes place when the sample is annealed for 1200 seconds (Fig.1(c)). But two diffraction rings different from that of the amorphous alloy are observed from SAD pattern (Fig.1 (d)). The two rings are titled as [200], [220] diffraction rings of cubic-carbon. When the annealing time is up to 2700s, a contrast in the amorphous matrix emerges (Fig.1 (e)). The diffraction rings of bcc-Fe are observed, while the diffraction rings of cubic-carbon disappear (Fig.1 (f)). When the annealing time is any longer, an obvious structure change occurs.

Fig.1 In-situ transmission electron microphotographs (TEM) and selected area diffraction patterns (SAD) of $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ amorphous alloy ($T=733\text{K}$): bright field images at (a) $t=0\text{s}$, (c) $t=1200\text{s}$, (e) $t=2700\text{s}$ and SAD patterns at (b) $t=0\text{s}$, (d) $t=1200\text{s}$, (f) $t=2700\text{s}$

First, small grains are precipitated in the dark region. Then the grains grow rapidly. Since all of these take places in tens seconds, no photography is taken. In spite of this, three different steps of crystallization of $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy i.e. incubation, phase separation and crystallization could be confirmed by in-situ TEM observation.

3.2 Morphology of bcc-Fe

Fig.2 (a), (b) show transmission electron images of

Fig. 2 Transmission electron microphotographs of $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy annealed at 673K : bright field images (a), (b), and selected area diffraction patterns of bcc-Fe(c) and Fe_5C_2 (d) compounds.

$\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy after isochronal annealing at 673K for 0.5h. It consists of spherical Fe_5C_2 compound and bcc-Fe grains, which are identified by SAD patterns (Fig.2(c), (d)). The bcc-Fe grains in $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy annealed at 673K assemble into an irregular shaped cluster (Fig.2 (b)).

Fig. 3 Transmission electron microphotograph (a) and selected area diffraction (b) of $\text{Fe}_{79.5}\text{P}_{12}\text{C}_6\text{Cu}_{0.5}\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy annealed at 673K.

By comparing the bcc-Fe grains in $\text{Fe}_{79.5}\text{P}_{12}\text{C}_6\text{Cu}_{0.5}\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy under the same condition (Fig.3), it can be drawn that copper increases the number of bcc-Fe grains and makes them distribute homogeneously. Copper influences the morphology and distribution of bcc-Fe grains.

4. Conclusion

Crystallization of amorphous $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy is composed of three different steps, i.e. incubation, phase separation and crystallization. Before the precipitation of bcc-Fe, cubic-carbon appears first. With increasing annealing time, cubic-carbon disappears.

The bcc-Fe grains of $\text{Fe}_{80}\text{P}_{12}\text{C}_6\text{Mo}_{0.5}\text{Si}_{1.5}$ alloy annealed at 673K present irregular shaped cluster. By addition of 0.5at% copper, the number of bcc-Fe grains increases and they are homogeneously distributed. Copper is an important element in crystallization of FePCMoSi based nanocrystalline alloy.

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