acta physica slovaca vol. 46 No. 2, 155 – 158 April 1996

STRUCTURE CHANGES AND CRYSTALLIZATION OF THIN FILMS OF Fe-Cu-Nb-U-Si-B TYPE ALLOYS[1]

I. Gościańska[†2], H. Ratajczak[‡], P. Sovák[¶], P. Matta[¶], M. Konč[¶]

[†] *Institute of Physics, A. Mickiewicz University, ul. Matejki 48/49, 60-769 Poznań, Poland*

[‡] *Institute of Molecular Physics, Polish Academy of Sciences, Smoluchowskiego 17/19, 60-179 Poznań, Poland.*

[¶] *Department of Experimental Physics, P. J. Šafarik University, Park Angelinum 9, 040-154 Košice, Slovakia.*

Received 31 July 1995, accepted 8 February 1996

Thin films of $Fe_{73.5}U_4Si_{13.5}B_9$ and $Fe_{72.5}U_5Si_{13.5}B_9$ alloys were prepared by flash evaporation technique. Changes in the structure upon annealing were studied using electron diffraction and transmission electron microscopic observations. The course of the electrical resistivity dependence on continuous heating at a rate of 10 K/min was analysed. No particular structural properties which could indicate a transition with appearance of any fine-grained phase were observed.

1. Introduction

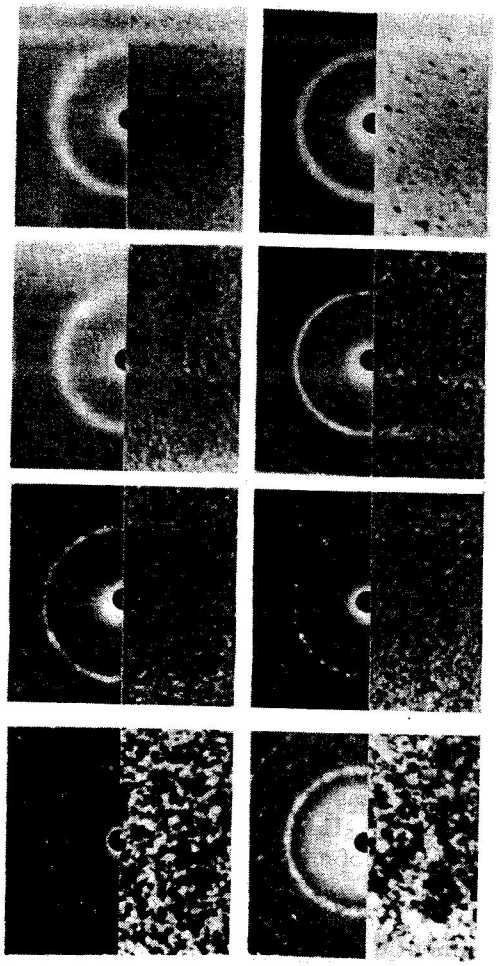
According to the results of [1], a replacement of high- temperature melting metals like Nb, Ta, or Mo by uranium in FINEMET type alloys should allow us to obtain nanocrystalline structures. So we have already studied thin films of $Fe_{73.5}Cu_1Nb_{3-x}U_xSi_{13.5}B_9$ alloys [2] and now we report results for the alloys of $Fe_{73.5}U_4Si_{13.5}B_9$ and $Fe_{72.5}U_5Si_{13.5}B_9$. As the structure determines the magnetic properties of alloys, the present study concerns observations of changes in their structure in the course of annealing.

2. Experimental

Thin films of the alloys studied were obtained, as in the previous works [2,3], by flash evaporation, onto glass substrates and rock-salt plates. The latter films were placed onto microscope meshes. The structure of the films was investigated by electron diffraction and by observation of the micrographs of transmission electron microscope (TEM). The results of microscopic observation have been supplemented with the study of temperature dependences of electrical resistivity measured on heating at a rate of about 10 K/min.

[1] Presented at 9th Czech and Slovak conference on magnetism, Košice, Slovakia, August 28-30 1995

[2] E-mail address: igosc@plpuam11.amu.edu.pl



a b

Fig.1 Representative sequences of diffractograms and TEM microphotographs of the structure of as-deposited and annealed for 10 min at 603 K, 773 and 823 K films: a) of the alloy $Fe_{73.5}U_4Si_{13.5}B_9$ and b) $Fe_{72.5}U_5Si_{13.5}B_9$

3. Results and discussion

According to the previous paper [2], the structure of thin film alloys of the Fe-Cu-(Nb-U)-Si-B type, in which Nb was partially or totally replaced by uranium, did not reveal characteristic features dependent on the Nb and U concentration. The as-deposited films were amorphous and they crystallized in the course of annealing, similarly as those of other alloys produced by the rapid quenching. Some of the films considered, containing both, uranium and niobium appeared fine-grained. However, identification

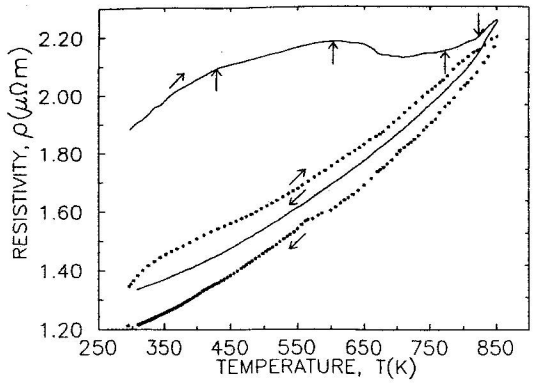


Fig.2 Exemplary temperature dependence of electrical resistivity of a thin film of the alloy $Fe_{73.5}Cu_1Nb_1U_2Si_{13.5}B_9$. The continuous line represents the initial heating up to 850 K and down to room temperature, and the dotted one - the second course of the temperature. The vertical arrows on the initial curve denotes temperatures 430, 603, 773 and 823 K.

of the solid solution α - FeSi phase as nanocrystalline is rather uncertain.

The thin films studied did not contain any Nb and Cu, but these components were replaced by U in concentrations -U_4 and - U_5. Fig. 1 shows electron diffractograms and microphotograms of the structures of: a)$Fe_{73.5}U_4Si_{13.5}B_9$ and b) $Fe_{72.5}U_5Si_{13.5}B_9$ alloy films. The former sample did not show any crystallization after annealing at T_{a1} = 603 K, while the latter one crystallized forming rather large grains. At T_{a2} = 773 K this sample recrystallized to smaller size but better ordered grains. After annealing at T_{a3} = 823 K, the alloys of both samples were already completely crystallized.

Fig. 2 presents the temperature dependence of electrical resistivity of a thin film of the alloy $Fe_{73.5}Cu_1Nb_1U_2Si_{13.5}B_9$. Electron diffraction study suggested, that nanocrystalline phase could be generated in an alloy of this composition. The ρ(T) initial curve reveals a distinct change already below 450 K. A most significant transition takes place at about 600 K, where the resistivity decreases with increasing temperature and after reaching a minimum at about 750 K it increases again. Such a course of the ρ(T) dependence indicates the stress relaxation and the removal of the excess free volume in the amorphous phase, in low temperature range. In the higher temperatures, the first stage of crystallization and recrystallization occurs in the most of the film volume. The nanostructure generation should begin above 800 K, but the shape of the curve in Fig.2 does not indicate any particular behaviour. Moreover, annealing of the sample above 800 K leads to its complete crystallization, which is the normal structural transition of rapidly quenched alloys at high temperatures.

4. Conclusions

In thin films of FINEMET type alloys, niobium and copper were replaced by uranium. The films of the composition of $Fe_{73.5}U_4Si_{13.5}B_9$ when as-deposited were amorphous and

crystallized upon annealing. Moreover, the films studied did not show any particular structural properties and they were found to behave similarly as other alloys produced by rapid quenching, which could indicate a transition with appearance of the fine grained phase.

References

[1.] P. Sovák, P. Petrovič, P. Kollár, M. Zatroch, M. Konč: *J. Magn. Magn. Mat.* **140-144** (1995) 427

[2.] I. Gościańska, H. Ratajczak, P. Sovák, P. Matta, M. Konč: *EMMA 95*, Vienna, T03-B061

[3.] H. Ratajczak, I. Gościańska, P. Sovák, M. Konč, P. Matta: *Phys. stat. sol. (a)* **136**, (1993) 119