Hyperfine Interact
DOI 10.1007/s10751-011-0495-1

Surface crystallization of Co-containing NANOPERM-type alloys

Marcel Bruno Miglierini · Tomas Hatala · Jiri Frydrych · Klara Šafářová

© Springer Science+Business Media B.V. 2012

Abstract Formation of crystalline phases is detected at different depths of the surface regions in $(Fe_{1-x}Co_x)_{76}Mo_8Cu_1B_{15}$ ($x = 0, 0.25$) alloy by CEMS and CXMS techniques. Magnetite is predominantly present on the wheel sides of the ribbons while the alloy is amorphous. During annealing, formation of bcc-Fe and bcc-Fe,Co starts earlier on the air sides of the $x = 0$ and $x = 0.25$, respectively.

Keywords Nanocrystalline alloy · Surface crystallization · CEMS · CXMS

1 Introduction

Magnetic properties of nanocrystalline alloys play an important role when employed in practical applications [1]. The improvement of magnetic parameters is often looked for by substitution of Fe with Co [2]. Consequently, both structure and magnetic arrangement of the alloy are affected. In this paper, we concentrate on the investigation of surface features of Co-substituted NANOPERM-type alloys.

2 Experimental details

Ribbons of $(Fe_{1-x}Co_x)_{76}Mo_8Cu_1B_{15}$ ($x = 0, 0.25$) metallic glass were prepared by rapid quenching (width 2 mm, thickness 23 μm) using iron enriched in ^{57}Fe to 50%.

M. B. Miglierini (✉) · T. Hatala
Institute of Nuclear and Physical Engineering, Slovak University of Technology,
Ilkovičova 3, 812 19 Bratislava, Slovakia
e-mail: marcel.miglierini@stuba.sk

M. B. Miglierini · J. Frydrych · K. Šafářová
Regional Centre of Advanced Technologies and Materials, Departments of Experimental Physics and Physical Chemistry, Faculty of Science, Palacky University, 17. listopadu 12,
771 46 Olomouc, Czech Republic

Published online: 21 February 2012

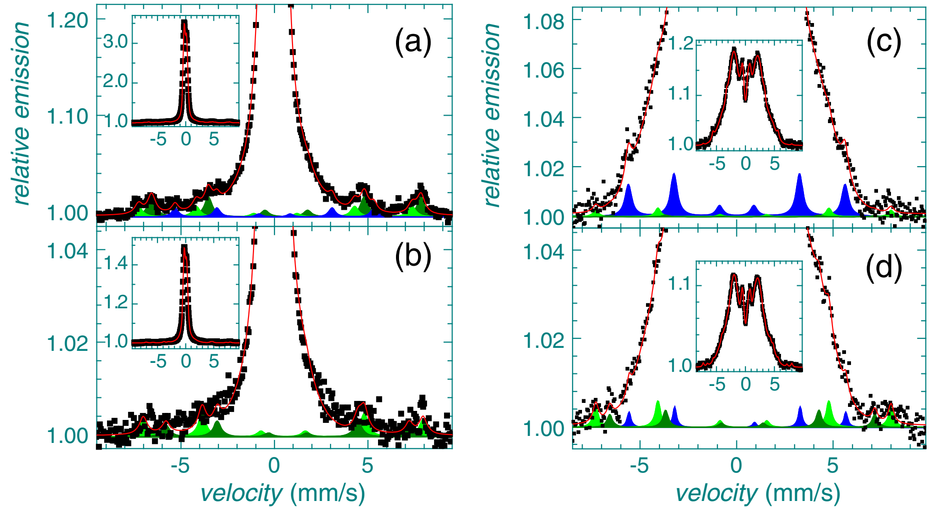

Fig. 1 Air sides of $(Fe_{1-x}Co_x)_{76}Mo_8Cu_1B_{15}$ alloy for $x = 0$ (**a**, **b**) and $x = 0.25$ (**c**, **d**) annealed at 370°C and recorded by CEMS (**a**, **c**) and CXMS (**b**, **d**) techniques. Crystalline components are given in colour: magnetite–*green*, bcc-Fe/Fe, Co–*blue*. Insets show spectra in full range

Both wheel and air surfaces were investigated by conversion electron Mössbauer spectrometry (CEMS) and by conversion X-ray Mössbauer spectrometry (CXMS) that provide information from the depths of about 200 nm and 1 μm, respectively. Evaluation of Mössbauer spectra was performed by the Confit software [3].

The acquisition of atomic force microscopy (AFM) surface topographic images and magnetic force microscopy (MFM) images was carried out by scanning probe microscope NTEGRA Aura in a map topography mode and in magnetic properties mode, respectively.

Samples were studied in the as-quenched (amorphous) state and after heat treatment in a vacuum furnace for 30 min which has eventually ensured formation of nanocrystalline grains with different amounts.

3 Results and discussion

Examples of CEMS and CXMS spectra recorded from air sides of the $(Fe_{1-x}Co_x)_{76}Mo_8Cu_1B_{15}$, $x = 0, 0.25$ ribbons annealed at 370°C are shown in Fig. 1. Broad spectral lines are assigned to the amorphous part of the alloy. They were reconstructed by distributions of quadrupole splitting and hyperfine magnetic fields that correspond to non-magnetic and magnetic regions, respectively. Presence of crystalline phases is indicated by magnetically split spectral components. According to the hyperfine field values they correspond to (disordered and/or substituted) magnetite, bcc-Fe ($x = 0$) and bcc-Fe,Co ($x = 0.25$) phases. The latter was fitted with up to four sextets of Lorentzian lines.

Relative areas of the crystalline phases derived from all spectra are plotted against temperature of annealing in Fig. 2. In $x = 0$, the formation of bcc-Fe starts earlier in

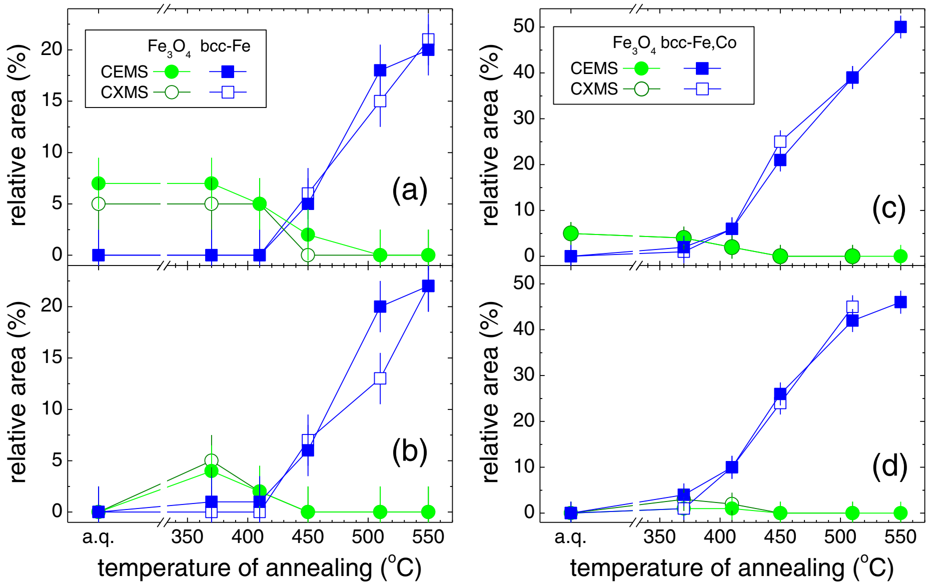

Fig. 2 Relative areas of crystalline phases (magnetite–*green*, bcc-Fe/Fe, Co–*blue*) derived from CEMS (*full symbols*) and CXMS (*opened symbols*) spectra and plotted against temperature of annealing (a.q.–as-quenched) for wheel (**a**, **c**) and air (**b**, **d**) sides of $(Fe_{1-x}Co_x)_{76}Mo_8Cu_1B_{15}$ alloy for $x = 0$ (**a**, **b**) and $x = 0.25$ (**c**, **d**)

the uppermost surface films (CEMS–Fig. 2b) on the air side which has faced the surrounding atmosphere (air) during the production of the ribbons. No bcc-Fe is detected on the wheel side (Fig. 2a) that was in direct contact with the rotating wheel and thus was exposed to better quenching conditions (more effective dissipation of heat). On the other hand, the amount of magnetite is higher at the wheel side but only in the samples that are still amorphous, i.e. below the temperature of annealing of 410°C. With the onset of crystallization traces of magnetite vanish. We can speculate that magnetite, as a corrosion product, might be formed owing to residual humidity that is trapped inside the surface pockets. These are fingerprints of the wheel's surface roughness into the quenching melt. That is why the wheel side of the ribbon is matt while the air side is shiny.

Addition of Co ($x = 0.25$) significantly alternates magnetic microstructure of this NANOPERM-type Mo-containing alloy which is demonstrated by notable magnetic splitting of the spectra in Fig. 1c and d. Surface crystallization is also different. Presence of bcc-FeCo nanocrystals is revealed already after moderate annealing at 370°C and evolves more rapidly on the air side. As in the previous case of $x = 0$, more abundant magnetite is detected on the wheel side but extends to deeper surface regions because CEMS and CXMS results are almost equal.

A tendency towards magnetic order in $x = 0.25$ is demonstrated by appearance of magnetic domains. They can be visualised by the help of MFM in Fig. 3b. A part of a domain is seen in which a tendency of an internal structure to develop occurs. Position of magnetic moments is different inside nanocrystalline grains the positions of which are documented by AFM in Fig. 3a.

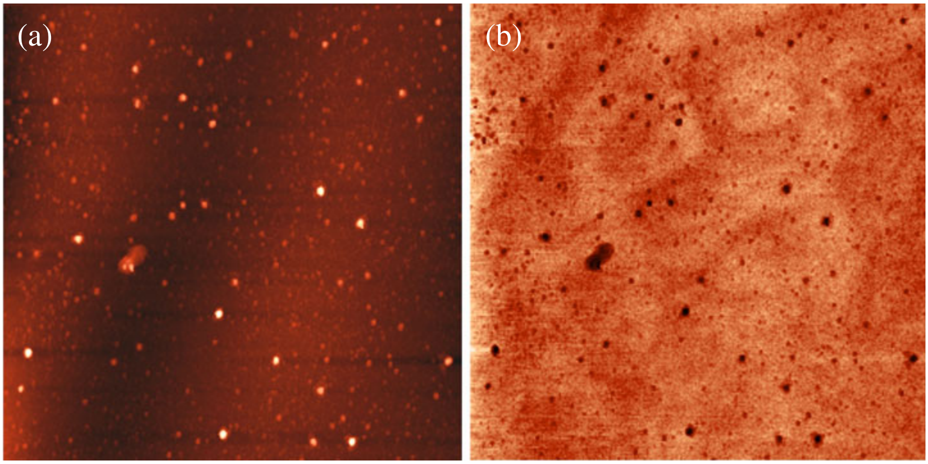

Fig. 3 AFM (**a**) and MFM (**b**) images taken from the air side of $(Fe_{0.75}Co_{0.25})_{76}Mo_8Cu_1B_{15}$ alloy annealed at 550°C. The displayed area is of 50 × 50 μm^2

4 Conclusion

After annealing at low enough temperatures when the alloys are still amorphous, presence of magnetite was revealed on both surfaces of the ribbons. Its content is higher at the immediate surface (CEMS) than in deeper regions (CXMS). With the onset of crystallization, only bcc-Fe and/or bcc-Fe,Co phases were identified.

Acknowledgements This work has been supported by research projects VEGA 1/0286/12, SK-PL-0013–09 and by the Operational Program Research and Development for Innovations–European Regional Development Fund (project CZ.1.05/2.1.00/03.0058 of the Ministry of Education, Youth and Sports of the Czech Republic).

References

1. McHenry, M.E., Laughlin, D.E.: Acta Mater. **48**, 223 (2000)
2. Willard, M.A., Laughlin, D.E., McHenry, M.E., et al.: J. Appl. Phys. **84**, 6773 (1998)
3. Žák, T., Jirásková, Y.: Surf. Interface Anal. **38**, 710 (2006)