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Structure and phase analysis of electrochemically synthesized Fe–W

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Abstract

Structural and magnetic study of electrochemically synthesized $Fe_{91.2}W_{8.8}$ amorphous powder was realized. Mössbauer spectrum of the as prepared material with its paramagnetic and magnetic part includes γ-Fe plus components of Fe–W solid solution overlapped by amorphous magnetic and paramagnetic phases, stable almost down to 20 K. After thermomagnetic curve measurement, the increase in the intensity of the magnetic part was observed. Phases resulting from thermal decomposition agree with those in the equilibrium Fe–W phase diagram.
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Recent intense development of modern powder metallurgy has provoked sudden interest in amorphous powders, particularly metallic ones, as composition range of multiatomic system can be extended in this way. E.g., electrochemically synthesized Fe–P amorphous powder alloys are active hydrogen absorbers in the temperature range from 100°C to 300°C and that they are transformed into crystalline state above this temperature range. To synthesize an amorphous alloy of increased structural stability, with no intention to stabilize the alloy additionally by crystallization over a wide temperature range, tungsten was used as amorphizer instead. However, the Fe–W amorphous system prepared by mechanical alloying has been studied. When studying the structure and phase composition of electrochemically synthesized $Fe_{91.2}W_{8.8}$ amorphous powder by means of Mössbauer spectroscopy (at room temperature and 20 K) and by magnetic methods, interesting structural and magnetic properties of the treated material were revealed and compared with some of those of mechanically alloyed ones.

Mössbauer spectra of the electrochemically synthesized powder material were taken in the standard transmission geometry using a Co^{57}(Rh) source. Calibration was done against α-iron foil data. Computer processing of spectra yielded intensities of individual components, their hyperfine inductions, isomer shifts, and quadrupole splittings. The amount of iron containing phases is supposed to be equal to intensities of the corresponding spectral components. The phase analysis of Fe–W materials published in [1–3] was applied comparing the above-mentioned characteristics of individual components. The thermomagnetic (TM) curve was measured on compacted material of cylindrical shape with a diameter of 2 mm and thickness of about 1.5 mm in the field of 3.98 kA/m with heating and cooling rates of 4 K/min. During the measurement, temperature of 800°C was reached and kept for 30 min between the heating and cooling process ramps.

In Mössbauer spectra, the prevailing paramagnetic part is formed by single line component (arising from γ-Fe particles) and by doublets. The doublets together

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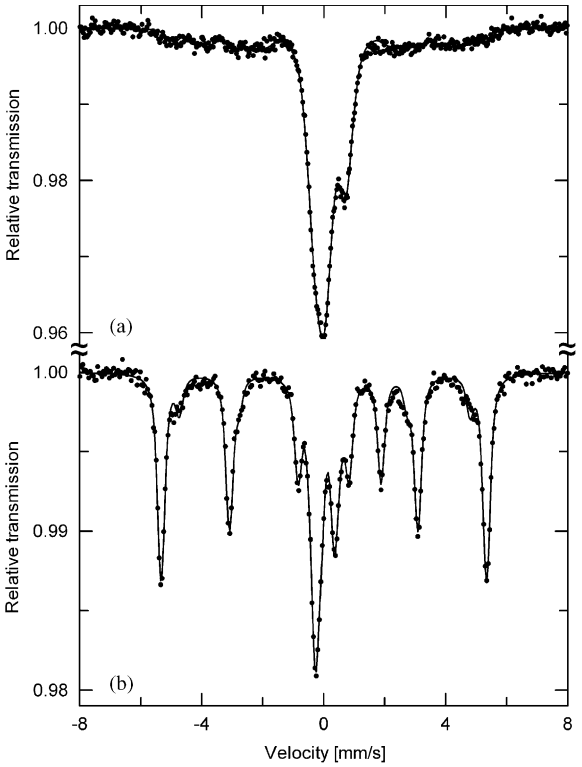

Fig. 1. Room temperature Mössbauer spectra of $Fe_{91.2}W_{8.8}$ amorphous powder in the as prepared state (a) and after thermomagnetic curve measurement—as vacuum annealing 800°C/30 min (b).

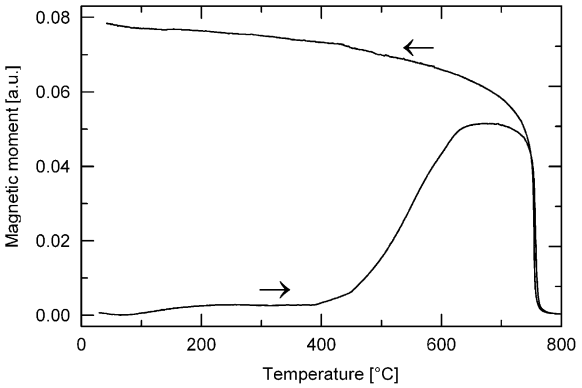

Fig. 2. Thermomagnetic curve of the as prepared $Fe_{91.2}W_{8.8}$ amorphous powder measured at the field of 3.98 kA/m with heating and cooling rate of 4 K/min and 30 min dwell time at 800°C.

with the small-splitting part of six-line components represent an amorphous phase. However, the isomer shifts of doublets are different from those found in literature for the nanocrystalline or ball milled Fe–W [4,5]. As all paramagnetic components and their intensities remain almost stable up to 20 K, they do not represent superparamagnetic particles and the γ-Fe phase does not transform to an antiferromagnetic state. The sextets with large magnetic splitting cannot be simply ascribed to crystalline solid solution of W in α-Fe only. They are probably overlapped by components of magnetically ordered amorphous phase or components representing crystallites of small coherent volumes.

After the heat treatment during the measurement of the thermomagnetic curve, the increase in the intensity of the magnetic part at the expense of the paramagnetic one was observed. The distribution of magnetic components is close to solid solution of W in α-Fe. The content of W can be estimated to approximately 3 at%. The components of the paramagnetic part were ascribed to W(Fe) solid solution, λ-Fe_2W, and Fe^{2+} phases. The Fe^{2+} phase probably arises from $FeO \cdot WO_3$ oxide, the traces of which were also observed in the spectra of ball milled Fe–W [5]. The Mössbauer phase analysis of the final product shows that the decomposition takes place during the annealing and that the detected phases agree with those in the equilibrium Fe–W phase diagram [6] (Fig. 1).

The thermomagnetic curve reflects some structural changes, especially above 500°C. Sharp increase in magnetic moment at about 600°C can be ascribed to crystallization of the amorphous phase and its decomposition into several tungsten rich phases and major iron rich α-phase enlarging the total magnetic moment of the sample. The Curie temperature derived from the branches for increasing and decreasing temperatures is approximately 755°C, which indicates low amount of W in the solid solution of α-Fe(W). The main curve features are almost identical with those of $Fe_{50}W_{50}$ in Ref. [5] (Fig. 2).

From this hysteresis loop measurements the decrease in coercivity from 11.8 to 4.1 kA/m and the increase in total magnetic moment was observed. These changes can be explained by phase decomposition (crystallization of α-Fe and λ-Fe_2W phases) and grain coarsening. The increase in iron atoms magnetic moment corresponds nicely with changes in content of magnetic phases as determined from Mössbauer phase analysis. The magnetic properties of the material studied are close to those of the magnetically soft ones.

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