14th Czech and Slovak Conference on Magnetism, Košice, Slovakia, July 6–9, 2010

Properties of Ni_3Fe Nanoparticles Prepared by Chemical Method

K. Zábranský*, O. Schneeweiss, N. Pizůrová and B. David
Institute of Physics of Materials, Academy of Sciences of the Czech Republic
Žižkova 22, CZ-616 62 Brno, Czech Republic

Ni_3Fe nanoparticles prepared by calcination of the iron–nickel oxalate were investigated by X-ray diffraction, Mössbauer spectroscopy and magnetic measurements. The high temperature X-ray diffraction shows two stages of transformation of oxalate: (i) amorphization and (ii) formation of nanocrystalline Ni_3Fe particles. The development of coercivity, magnetization and mean coherence length ($\approx$ particle size) is discussed.

PACS numbers: 61.05.cp, 61.46.−w, 75.20.En, 75.50.Bb

1. Introduction

Ni_3Fe alloy is well-known for their good soft magnetic properties. The bulk Ni_3Fe phase crystallizes in fcc structure and its magnetic properties depend on atomic ordering [1]. Several methods for preparation of this material in nanocrystalline form e.g. ball milling, chemical synthesis, etc., have been recently developed [2–5]. In this paper we present the preparation of the nanocrystalline Ni_3Fe using solid state decomposition.

2. Experimental details

The Ni_3Fe nanocrystalline powder was prepared by the calcination of $Ni_{0.75}Fe_{0.25}C_2O_4$. The phase analysis of the samples was carried out by X-ray diffraction (XRD) using Co K_α radiation at room temperature (RT) and at high temperature (HT) in the furnace with H_2 atmosphere where the precursor was deposited on heating platinum strip. The XRD patterns were taken in the temperature range $100 \div 600\,^\circ$C with the step $\Delta T = 20\,^\circ$C. The mean coherence length (MCL) ($\approx$ particle size) was calculated using the Scherrer formula from the XRD patterns. The magnetic measurements were performed by vibration sample magnetometer (VSM) EG&G model No. 4500 Princeton Applied Research Corporation. Hysteresis loops were measured at RT. The magnetic field was increased from the 0 to 796 kA/m with the step of 3.2 kA/m. Thermomagnetic curve (TMC) was measured in the temperature range $25 \rightarrow 800/30\,\text{min} \rightarrow 25\,^\circ$C under H_2 atmosphere with the temperature sweep $4\,^\circ$C/min in the magnetic field $H = 4$ kA/m. Transmission electron microscopy (TEM) and energy dispersive X-ray analysis (EDAX) were used for the determination of the chemical composition.

3. Results and discussions

The RT XRD patterns of the precursor sample show Fe–Ni oxalate compound with traces of impurities remaining from precursor preparation. Two stages of transformation of the precursor to nanocrystalline powder were observed by HT XRD measurement. The first stage begins at $160\,^\circ$C where an amorphization occurs. It is represented by broad peaks in the XRD patterns. The second stage starts at $260\,^\circ$C and it is characterized by appearing of crystalline fcc NiFe phase. The intensity of the diffraction peaks and MCL increases with the increasing annealing temperature. The temperature dependence of MCL is depicted in Fig. 1 where two stages of MCL growth of the nanocrystalline NiFe phase can be recognized.

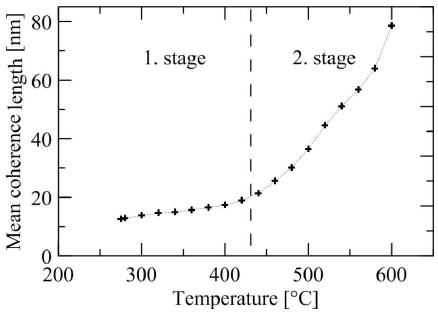

Fig. 1. The mean coherence length MCL in dependence on temperature.

The slight increase up to $\approx 420\,^\circ$C is followed by a more pronounced increase at higher temperature. Similarly, a rapid slope can be observed on TMC at $\approx 370\,^\circ$C (Fig. 2) with the maximum at the same temperature ($\approx 420\,^\circ$C) where the pronounced increase in MCL begins. The decrease in magnetic moment above this temperature is connected with the MCL growth and increase in magnetocrystalline anisotropy of crystalline Ni_3Fe phase. The Curie temperatures (T_C) determined from the TMC are $600\,^\circ$C for the increasing temperature and $610\,^\circ$C for the cooling. The difference is probably associated with structural changes at high temperature up to $800\,^\circ$C. The reported T_C of the bulk Ni_3Fe phase is $600\,^\circ$C for a disordered state and $690\,^\circ$C [1]. Therefore the shift of the T_C on TMC between increasing and decreasing temperature can be related to a derivation from the stoichiometric composition towards a higher iron concentration and with changes in atomic ordering [6, 7]. This was con-

* corresponding author; e-mail: zabransky@ipm.cz

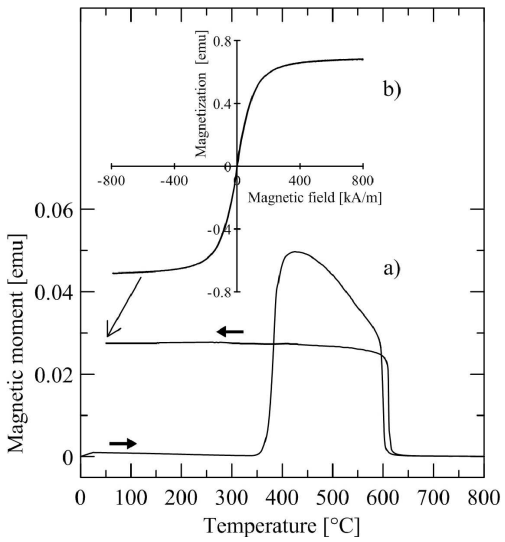

Fig. 2. (a) Temperature dependence of the magnetic moment of the precursor by the annealing in H_2 atmosphere. The arrowheads indicate the increase and decrease of the temperature. (b) The hysteresis loop of the sample after TMC measurement.

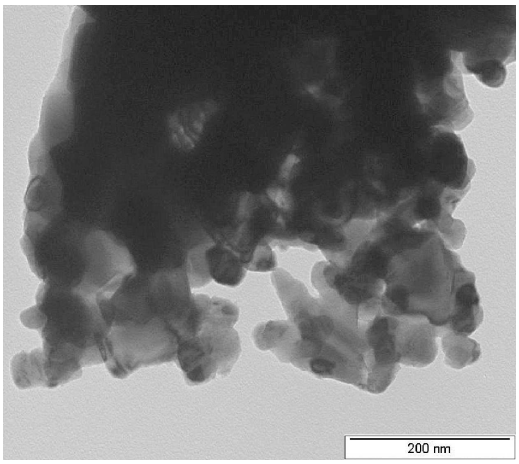

Fig. 3. TEM image of the sample after the annealing at 560 °C in H_2 in the furnace.

TABLE

The parameters of the hysteresis loops taken after the different heat treatments of the precursor in H_2 (coercivity, H_c, saturation magnetization σ_s, remanent magnetization σ_r).

Heat treatment: after	H_c [kA/m]	σ_s [T]	σ_r [T]
TMC measurement up to 800 °C	1.01	1.32	0.173
HT XRD up to 600 °C	4.87	1.30	0.135
furnace annealing up to 560 °C	9.98	1.25	0.141

firmed by EDAX measurement taken in TEM where the mean chemical composition of the nanocrystalline particles 62.6 ± 0.4 at.% Ni and 37.4 ± 0.4 at.% Fe was determined.

The TEM image in Fig. 3 shows the nanoparticles after annealing at 560 °C in H_2.

The results from hysteresis loops measurement are summarized in Table. The σ_s value taken on the sample after the annealing at 560 °C in H_2 corresponds to chemical composition $Fe_{40}Ni_{40}$ reported in [8]. This is in a good agreement with the values determined by TEM. Higher values of σ_s and lower H_c were found on the samples annealed at higher temperatures. It can be ascribed to an increase in particle size [9, 10] and atomic ordering, respectively.

4. Conclusions

Ni_3Fe nanoparticles were prepared by the calcination of nickel–iron oxalate precursor. The data derived from XRD and TMC measurements indicate two stages of the particle growth. The slight increase in MCL up to $\approx$ 420 °C is followed by a more pronounced increase at higher temperatures. The maximum magnetic moment was observed at $\approx$ 420 °C. The higher annealing temperature the higher saturation magnetization σ_s and lower coercivity H_c were found which can be explained as the result of an increase in particle size and atomic ordering.

Acknowledgments

This work was supported by the Ministry of Education, Youth and Sports of the Czech Republic (project No. 1M6198959201) and by the Grant Agency of the Czech Republic (project No. 106/08/1440).

References

[1] L.J. Swartzendruber, V.P. Itkin, C.B. Alcock, in: *Phase Diagram of Binary Iron Alloys*, Ed. H. Okamoto, ASM International, Materials Park, OH 1993, p. 256.

[2] I. Chicinas, V. Pop, O. Isnard, *J. Magn. Magn. Mater.* **242-245**, 885 (2002).

[3] A. Datta, M. Pal, D. Chakravorty, D. Das, S.N. Chintalapudi, *J. Magn. Magn. Mater.* **205**, 301 (1999).

[4] I. Chicinas, V. Pop, O. Isnard, J.M. LeBreton, J. Juraszek, *J. Alloys Comp.* **352**, 34 (2003).

[5] P. Kollar, D. Oleksakova, J. Fuzer, J. Kovac, S. Roth, K. Polanski, *J. Magn. Magn. Mater.* **310**, 2609 (2007).

[6] R.J. Wakelir, E.L. Yates, *Proc. Phys. Soc.* **66**, 221 (1953).

[7] G. Inden, *Z. Metallkd.* **66**, 577 (1975).

[8] *Magnetic Properties of Metals. D-Elements, Alloys and Compounds*, Ed. H.P.J. Wijn, Springer-Verlag, Berlin 1991, p. 33.

[9] G. Herzer, *Scr. Metal. Mater.* **33**, 1741 (1995).

[10] E. Artz, *Acta Mater.* **46**, 5611 (1998).