



Originally published as:

Olekšáková, D., Füzer, J., Kollár, P., Bednarčík, J., Lathe, C. (2014): Isobaric Thermal Expansion and Isothermal Compression of Powdered NiFe Based Alloys Studied by In-Situ EDXRD. - *Acta Physica Polonica A*, 126, 1, p. 128-129

DOI: <http://doi.org/10.12693/APhysPolA.126.128>

Isobaric Thermal Expansion and Isothermal Compression of Powdered NiFe Based Alloys Studied by In-Situ EDXRD

D. OLEKŠÁKOVÁ^{a,*}, J. FÜZER^b, P. KOLLÁR^b, J. BEDNARČÍK^c, C. LATHE^d

^aDpt. of Appl. Mathematics and Informatics, Fac. of Mechanical Eng., TUKE, Letná 9, 042 00 Košice, Slovakia

^bInstitute of Physics, Faculty of Sciences, P. J. Šafárik University, Park Angelinum 9, 041 54 Košice, Slovakia

^cHASYLAB am Deutschen Elektronen Synchrotron, DESY, Notkestrasse 85, D-22607 Hamburg, Germany

^dGeo Forschungs Zentrum Potsdam at DESY/HASYLAB, Notkestrasse 85, D-22607 Hamburg, Germany

The aim of the present work was to study the isothermal compression and isobaric thermal expansion behaviour of ball-milled NiFe (81 wt.% of Ni) and NiFeMo (79 wt.% of Ni, 16 wt.% of Fe) alloy and follow its phase evolution when exposed to high pressure and temperature. In-situ pressure–temperature energy dispersive X-ray (EDXRD) diffraction experiments were performed at the MAX80 instrument (beamline F2.1). The compressibility of NiFe alloy at 400 °C was evaluated for pressure values of up to 3.5 GPa. The EDXRD spectra revealed the presence of cubic FeNi₃ phase as determined from the shift of (111), (200) and (220) reflection lines in corresponding EDXRD spectra.

DOI: [10.12693/APhysPolA.126.128](https://doi.org/10.12693/APhysPolA.126.128)

PACS: 75.50.Bb, 61.05.cp

1. Introduction

In the recent years, nickel-iron alloys have generated a lot of the interest in the both fundamental and applied science. Their remarkable magnetic properties have made them the subject of study over the last about half of a century. The highest permeability and near to zero magnetostriction characterize the alloys with the nominal composition Ni₈₁Fe₁₉, called permalloy. Supermalloy Ni₇₉Fe₁₆Mo₅ has an improved loss characteristic over permalloy materials, which target at a higher operating frequency [1].

After heat treatment permalloy has the initial permeability much larger than the one of the pure iron and is produced usually in the form of thin sheets. Therefore it is logical to attempt to prepare such material in more “bulk” form, for example in the form of a cylinder or a ring, which would be more convenient for some industrial applications. One of the methods, suitable for this preparation is for example the mechanical milling or mechanical alloying.

Mechanical milling is a useful powder processing technique that can produce a variety of equilibrium and non-equilibrium alloy phases [2, 3]. Some researches concerning Ni–Fe, Ni–Fe–Mo powders system produced by mechanical milling were reported in [4–6].

2. Experimental

We have prepared two types of powder samples [4]. The sample NiFe was prepared by mechanical milling of microcrystalline ribbon NiFe (81 wt.% of Ni) obtained by melt-spinning, which is suitable for milling. The sample NiFeMo was prepared by the milling of the swarfs of NiFeMo (79 wt.% of Ni, 16 wt.% of Fe), which were

prepared from an ingot by turning. We used swarfs, because it was not possible to prepare ribbons with this chemical composition. The milling of both samples was performed in a protective argon atmosphere in a high-energy planetary ball mill (RETSCH PM4000) with ball-to-powder-ratio of 6:1 and with a speed of 180 rpm [4, 5]. The Ni₇₉Fe₁₆Mo₅ sample was milled in a vial cooled in liquid nitrogen. The temperature stability of the high-energy ball-milled Ni₈₁Fe₁₉ and Ni₇₉Fe₁₆Mo₅ specimens was investigated by in situ angular-dispersive X-ray diffraction using high-resolution diffractometer at the B2 beamline in HASYLAB/DESY. Heating experiments were performed in situ, using an image plate detector and a STOE furnace. In-situ pressure–temperature energy dispersive X-ray diffraction experiments were performed at the MAX80 instrument (beamline F2.1). The compressibility of Ni₈₁Fe₁₉ alloy at 400 °C was evaluated for pressure values of up to 3.5 GPa. The thermal expansion of Ni₇₉Fe₁₆Mo₅ alloy under pressure of 3.9 GPa was evaluated within temperature range 30–800 °C.

3. Results

The aim of this work was to study the isothermal compression and isobaric thermal expansion of ball-milled Ni₈₁Fe₁₉ and Ni₇₉Fe₁₆Mo₅ alloys and follow their phase evolution when exposed to high pressure and temperature.

The diffraction patterns collected upon heating of the powder, prepared by the milling of the ribbon Ni₈₁Fe₁₉ for 15 hours at constant heating rate, are shown in Fig. 1. These investigations confirmed that Fe–Ni₃ phase as well as fcc Fe–Ni solid solution occur in powdered NiFe samples and this single-phase system is precondition for the creation of Ni–Fe based soft magnetic materials. These results correspond with previous DSC results (Netzsch DSC 404) [4] and with results of X-ray diffraction, obtained with Fe-filtered Co–K_α radiation (Philips PW 1050) with diffracted-beam graphite monochromator [6].

*corresponding author; e-mail: denisa.oleksakova@tuke.sk

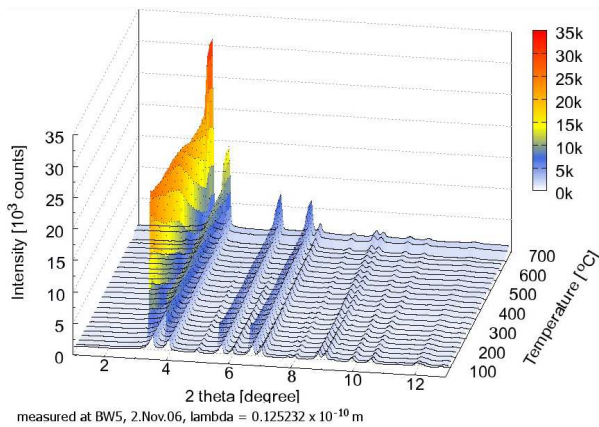


Fig. 1. XRD patterns of the in-situ annealing of sample NiFe, milled for 15 hours at BW5.

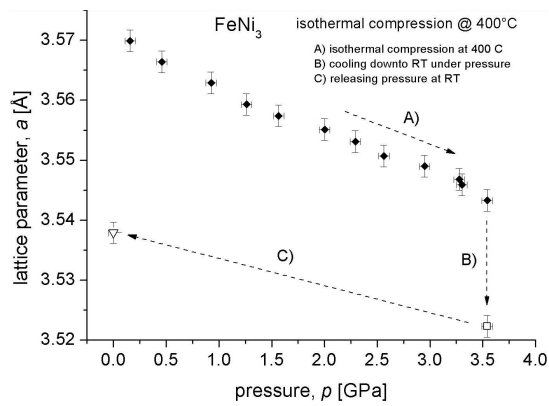


Fig. 2. The evolution of the lattice parameter of cubic FeNi₃ phase of the sample Ni₈₁Fe₁₉ during A) isothermal compression at 400 °C, B) cooling down to room temperature under pressure of 3.5 GPa and C) after releasing pressure at room temperature.

The compressibility of Ni₈₁Fe₁₉ alloy at 400 °C was evaluated for pressure values of up to 3.5 GPa. The EDXRD spectra revealed the presence of cubic FeNi₃ phase. Figure 2 illustrates the experimental compression behaviour of major constituent phase FeNi₃, as determined from the shift of (111), (200) and (220) reflection lines in corresponding EDXRD spectra [7]. The thermal expansion of Ni₇₉Fe₁₆Mo₅ alloy under pressure of 3.9 GPa was evaluated within temperature range 30–800 °C. The EDXRD spectra revealed the presence of cubic FeNi₃ phase. Figure 3 shows the temperature dependence of the lattice parameter a of major constituent phase FeNi₃ as determined from the shift of (111), (200) and (220) reflection lines in corresponding EDXRD spectra. After reaching 500 °C, the thermal expansion of cubic lattice deviates from linear behaviour and tends to increase at a higher rate [7].

4. Conclusions

We found out that the decrease of lattice parameter of cubic FeNi₃ after isothermal compression of ball-milled

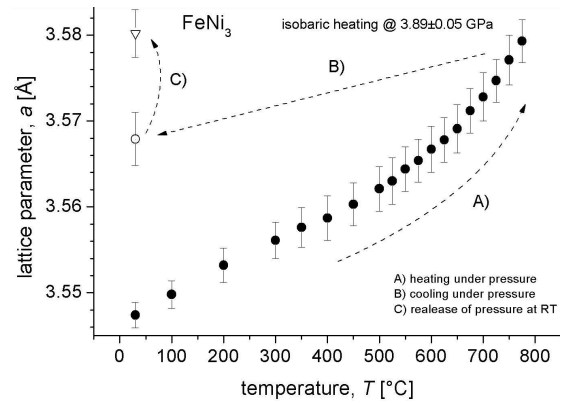


Fig. 3. The evolution of the lattice parameter of cubic FeNi₃ phase of the sample Ni₇₉Fe₁₆Mo₅ during A) isobaric heating at 3.9 GPa, B) cooling down to room temperature under pressure of 3.9 GPa and C) after releasing pressure at room temperature.

Ni₈₁Fe₁₉, and subsequent release of compression and temperature, is about 0.03 Å. The increase of lattice parameter of cubic FeNi₃ after isobaric thermal expansion of ball-milled Ni₇₉Fe₁₆Mo₅ alloy is also about 0.03 Å. High compression and heating, which simulate the conditions at milling of NiFe alloys, influence negligibly the crystal structure. We assume that compaction of a milled NiFe-based powder results in material with a similar structure as the one of the origin alloy.

Acknowledgments

This work was supported by the Slovak Research and Development Agency through project APVV-0222-10 MAGCOMP, by the projects, nanoCEXmat I, ITMS 26220120019 and nanoCEXmat II, ITMS: 26220120035, supported by the Operational Program “Research and Development” financed through European Regional Development Fund and by the Scientific Grant Agency of the Ministry of Education of Slovak Republic and the Slovak Academy of Sciences, projects Nos. VEGA 1/0861/12 and No. 1/0862/12.

References

- [1] R.C. O’Handley, *Modern Magnetic Materials*, Wiley, New York, 2000.
- [2] R. Besmel, M. Ghaffari, H. Shokrollahi, B. Chitsazan, L. Karimi, *J. Magn. Magn. Mater.* **323**, 2727 (2011).
- [3] B.V. Neamtu, O. Isnard, I. Chichinas, V. Pop, *J. Alloys Compd.* **509**, 3632 (2011).
- [4] D. Olekšáková, P. Kollár, J. Füzér, M. Kusý, S. Roth, K. Polanski, *J. Magn. Magn. Mater.* **316**, e838 (2007).
- [5] D. Olekšáková, J. Füzér, P. Kollár, T. Švec, J. Kováč, J. Briančin, K. Polański, *Czechoslovak Journal of Physics* **54**, D93 (2004).
- [6] D. Olekšáková, S. Roth, P. Kollár, J. Füzér, *J. Magn. Magn. Mater.* **304**, e730 (2006).
- [7] D. Balzar, H. Ledbetter, *Journal of Appl. Cryst.* **26**, 97 (1993).