Magnetic and structural data used to monitor the alloying process of mechanically alloyed Fe$_{80}$Ni$_{20}$

Michael W.R. Volk$^{a,*}$, Michael R. Wack$^b$, Bernd Maier$^b$

$^a$ Institute for Rock Magnetism, Department of Earth Sciences, University of Minnesota, 116 Church Street SE, Minneapolis, MN 55455, United States
$^b$ Department of Earth and Environmental Sciences, Ludwig Maximilians Universität, Theresienstr. 41, 80333 Munich, Germany

**Abstract**

In the last decades, much attention was given to mechanical alloying as it proved to be a cheap and easy way to produce (even metastable) nanostructured alloys. Especially Fe-Ni alloys have been studied intensely due to their technological and scientific importance. The MA process, however, is not fully understood. Furthermore, remanence properties of Fe$_{80}$Ni$_{20}$ are not well known. In our article “Monitoring the alloying process of mechanically synthesized Fe$_{80}$Ni$_{20}$ through changes in magnetic properties (DOI: j.jallcom.2017.10.090, Volk et al., 2018) [1]” we investigated structural and magnetic properties of the intermediate and final alloys. Elemental Fe (99.5%) and Ni (99.7%) powders were filled in a 80 ml zirconia vials together with 3 mm zirconia milling balls and milled at 400 PRM with a planetary ball mill (Fritsch Pulverisette Premium 7). By subsampling the product at 14 different times during the process, the data presented here shows how crystalline structure (X-ray diffraction) and magnetic properties, induced as well as remanent, of the metastable Fe$_{80}$Ni$_{20}$ change during the mechanical synthesis.

© 2018 The Authors. Published by Elsevier Inc. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).
### Specifications Table

| Subject area               | Material sciences, Earth sciences, Planetary Sciences |
|----------------------------|-------------------------------------------------------|
| More specific :subject area| Mechanical alloying, Mechanical synthesis, Intermetallic alloys, Alloying, Meteorite studies, Magnetic properties of Fe—Ni alloys |
| Type of data               | - Unprocessed magnetic data (hysteresis, direct current demagnetization and isothermal remanent magnetization acquisition) in Princeton Vibrating Sample Magnetometer (VSM) file format |
|                           | - X-ray diffraction xy data |
| How data was acquired      | XRD: STOE Stadi P diffractometer in Debye-Scherrer geometry using Mo-\(\kappa\) |
|                           | (\(\lambda = 0.0709\) nm) |
|                           | Magnetic Measurements: Princeton Measurements Corporation model 3900 vibrating sample magnetometer |
| Data format                | Raw-data and figures |
| Experimental factors       | Milling parameters: |
|                           | - 400 RPM |
|                           | - Fe = 7.92 g, Ni = 2.08 g |
|                           | - 100 g, 3 mm Zirconia milling balls |
|                           | - 80 ml zirconia milling vial |
|                           | All sample preparation steps (e.g. filling of gel capsules) were done in a glovebox with Argon atmosphere to minimize oxidation. \(O_2\) levels were monitored constantly (< 2%) |
|                           | Subsamples were taken after 1, 2, 4, 6, 10, 16, 30, 60, 120, 240, 480, 960, 1440 and 2160 min of milling |
|                           | XRD: Powder was measured in glass capillaries with 100 \(\mu\)m diameter and 10 \(\mu\)m. |
|                           | Magnetic measurements: approx. 50 mg of sample was measured and filled into a gel-capsule. The remaining space was filled with high purity quartz-wool |
| Experimental features      | XRD: |
|                           | - samples are placed in the capillary |
|                           | - measurement is done from 10–70° 2 Theta in 0.15° steps with a measurement time of 360 s per step |
|                           | Hysteresis: |
|                           | - Magnetization is measured while magnetic field is ramped from 0 T - > +1.5 T - > -1.5 T - > +1.5 T |
|                           | - Magnetization is measured every 1mT with an integration time of 100 ms, using sweeping mode in the VSM (continuous field sweep compared to discrete steps) |
|                           | IRM acquisition: |
|                           | - The sample is AC demagnetized in the VSM prior to the IRM ac. measurement |
|                           | - Starting from a demagnetized state increasingly strong magnetic fields are applied to the sample. After each field step, the remanent magnetization is measured in the residual field of the VSM |
|                           | - The field steps are chosen by the VSM software (logarithmic) |
|                           | - Measurement time per point = 1 s, maximum field = 500 mT |
**Value of the data**

- Detailed structural analysis as well as changes in magnetic properties during mechanical synthesis is essential for understanding of formation of metastable bcc Fe—Ni alloys (i.e., of Fe₈₀Ni₂₀).
- Evolution of magnetic properties need to be well characterized so they can be used as a proxy for the homogeneity and alloying status of Fe₈₀Ni₂₀ and possibly other alloys.
- Remanence properties of most Fe—Ni alloys unknown. But knowledge of the magnetic remanence properties of Fe₈₀Ni₂₀ is important to our understanding of the magnetic signature of meteorites and early planetesimals recovered from meteorites.

**1. Data**

Mechanical alloying proved to be a simple way to synthesize intermetallic alloys. The data set shows the influence of increasing milling times on magnetic properties determined from magnetic hysteresis, direct current demagnetization (DCD) and isothermal remanent magnetization (IRM) acquisition [1]. Furthermore, the X-ray diffraction patterns of the FeNi₂₀ alloy are included, which show the progression of alloying, strain, and crystallite size.

**2. Experimental design, materials, and methods**

We used a Fritsch Pulverisette Premium (P7) planetary ball mill to synthesize Fe₈₀Ni₂₀. Iron (99.5%, 7.92 g) and Ni (99.7%, 2.08 g) were filled into an 80-ml zirconia vial together with 100 g 3 mm sized milling balls. The vials were prepared in an Ar-filled glove box (O₂ < 2%).

X-ray diffraction was measured with a STOE Stadi P diffractometer in Debye-Scherrer geometry using Mo-kα₁ (λ = 0.0709 nm) from 10° to 70° 2Θ in 0.15° steps (360 s).

**Figs. 1–4** show the unprocessed Hysteresis (HYS, left column), DCD (middle column) and IRM acquisition curves (right column). For each milling time three specimens were measured. Each specimen was prepared by filling ca. 50 mg of the powder together with quartz wool into a gel-capsule. Subsampling and sample preparation were done inside a glovebox, filled with Ar. The O₂
Fig. 1. Unprocessed hysteresis loops (first column), direct current demagnetization curves (second column) and acquisition of an isothermal remanent magnetization (right column) for milling times 0–4 min. Data shows the three specimens (S1–S3).
Fig. 2. Unprocessed hysteresis loops (first column), direct current demagnetization curves (second column) and acquisition of an isothermal remanent magnetization (right column) for milling times 6–30 min.
concentration was monitored consistently and never exceeded 2%. A Princeton Measurements Corporation model 3900 vibrating sample magnetometer (VSM) was used for the measurements. For each specimen, the same measurement parameters were used. HYS, IRM and DCD were done in sequence.

Fig. 3. Unprocessed hysteresis loops (first column), direct current demagnetization curves (second column) and acquisition of an isothermal remanent magnetization (right column) for milling times 60–480 min.
Acknowledgments

This work was supported by Deutsche Forschungsgemeinschaft project WA3402/1-1 under the auspices of SPP1488, Planetary Magnetism. We thank Prof. Dr. Stuart Gilder for the helpful comments and discussions. Furthermore, we would like to thank Martin Leberer and Johannes Heinbuch for their help in the laboratory.

Transparency document. Supplementary material

Transparency document associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2018.06.036.

Reference

[1] M.W.R. Volk, M.R. Wack, B.J. Maier, Monitoring the alloying process of mechanically synthesized Fe80Ni20 through changes in magnetic properties, J. Alloy. Compd. 732 (Suppl. C) (2018) S336–S342. http://dx.doi.org/10.1016/j.jallcom.2017.10.090.