Magnetic properties of Fe-Ni nanoparticles prepared by co-precipitation method

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Abstract. Many methods have been extended for the preparation of nanoparticles. One of the most important methods is the chemical wet process, e.g. the co-precipitation method that has been used for the preparation of Fe-Ni nanoparticles by the authors. XRD patterns show that the nanoparticles are amorphous before calcination and crystallized after calcination. SEM images show that the grain size of the Fe-Ni particles is in 50-300 nanometre range and that the texture of the nanoparticles after calcination was smoother than before calcination. Hysteresis loops show that the Fe-Ni nanoparticles are superparamagnetic before calcination, because the carbonate phase still exists in the sample, and that they are ferromagnetic materials after calcination. For 40Fe-60Ni nanoparticles after calcination, $H_C = 0.12$ and $B_S = 4800$ Oe, being very different in respect to the bulk 40Fe-60Ni alloy.

1. Introduction
There are many methods for the preparation of nanoparticles, these include: 1) Gas process; Plasma Arch (PA), Physical Vapour Deposition (PVD), Chemical Vapour Deposition (CVD), Inert Gas Condensation (IGC), Combustion Flame Spraying (CFS)) and Cryogenic Melting (CM), 2) Liquid process; Sol-Gel (SG), Chemical Wet (CW), Electrical Condensation (EC). 3) Solid-State process; Mechanical Alloying (MA) and Chemical-Mechanical (Chem-Mech) methods.

One of the most important methods of the Chemical Wet process is the co-precipitation method that has been used by different research groups such as Mirzaei et al [1,2]. In this research the authors have used this method for the preparation of Fe-Ni nanoparticles in the catalytic research centre of the University of Sistan and Baluchestan, IRAN and they have investigated the magnetic properties of the sample for this report.

2 Chemical reactions
Fe-Ni nanoparticles have been prepared by the co-precipitation method. Solutions of iron nitrate and nickel nitrate were prepared with distilled water solvent [2]. Then these solutions were mixed together in a container.

2.1 Chemical reaction before calcination
By adding sodium carbonate, sediments of iron carbonate and nickel carbonate were formed at the bottom of the container.

The chemical reaction (1) shows the reaction between iron nitrate and sodium carbonate.

$$2Fe(NO_3)_3 \cdot 9H_2O + 3Na_2CO_3 \rightarrow Fe_2(CO_3)_3 + 6Na^+ NO_3^-$$ (1)

The chemical reaction (2) shows the reaction between nickle nitrate and sodium carbonate.

$$Ni(NO_3)_2 \cdot 6H_2O + Na_2CO_3 \rightarrow NiCO_3 + 2Na^+ NO_3^-$$ (2)
2.2 Chemical reaction after calcination
Then the sediments are filtered and strained and calcined at 115 °C, reactions (3) and (4) show the use of annealing for the removal of impure ingredients of the sediment.

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\begin{align*}
NiCO_3 & \xrightarrow{\text{Annealing}} NiO + CO_2 & (3) \\
Fe_2(CO_3)_3 & \xrightarrow{\text{Annealing}} \begin{cases} 
Fe_2O_3 + CO_2 \\
FeO + CO_2 \\
Fe_3O_4 + CO_2 
\end{cases} & (4)
\end{align*}
\]

3. Experiments
A number of techniques were used to study these samples, including:

I) X-ray diffraction (XRD) technique was used to determine the ingredients of the sample. XRD measurements were performed using a Bruker AXS Company, D8 Advance diffractometer. Scans were taken with a 2θ step size of 0.02 and a counting time of 1.0 s using Cu Ka radiation source generated at 40 kV and 30 mA. Specimens for XRD were prepared by compaction into a glass-backed aluminum sample holder. Data were collected over a 2θ range from 4° to 70° and phases were identified by matching experimental patterns to entries in the Diffrac\text{plus} Version 6.0 indexing software.

II) The morphology of the sample and their precursors were observed by means of a CamScan model of the Oxford S range of Scanning Electron Microscopes (SEM). The SEM technique was used to determination of grain size of the sample.

III) The Vibrating Sample Magnetometer (VSM) technique with an applied field \(-6000 \text{ Oe} \leq H \leq 6000 \text{ Oe}\) used to determination of magnetic properties of the Fe – Ni nanoparticles. The Fe - Ni nano structure has been also studied by X W. Wei et al [3,4] with different methods.

4. Results and discussion
XRD patterns showed that the nanoparticles are amorphous before calcination and crystallized after calcination. The XRD spectra have not been shown here.

Figures (1a) and (1b) show SEM images. The range of grain size of the Fe-Ni particles is in 50-300 nanometre range and the texture of the nanoparticles after calcination is smoother than before calcination.

**Figure 1a** SEM image of 40Fe60Ni nanoparticles before calcination.

**Figure 1b** SEM image of 40Fe60Ni nanoparticles after 6 hrs calcination.
Figure (2) shows hysteresis loop of 40Fe – 60Ni nanoparticles. It exhibits that the sample is superparamagnetic before calcination, because the XRD spectra showed carbonate in the sample.

Figure (3) shows hysteresis loop of the same sample after calcination, so that they are ferromagnetic particles. For 40Fe-60Ni after calcination, $H_c = 0.12$ and $B_s = 4800$ Oe, being very different in respect to the bulk state of Fe –Ni alloy. Figure (4) shows hysteresis loop of bulk iron.

Figure 2.
Hysteresis loop of 40Fe60Ni nanoparticles before calcination.

Figure 3.
Hysteresis loop of 40Fe60Ni nanoparticles after calcination.
Table (1) shows numerical results of VSM of 40Fe-60Ni nanoparticles and bulk iron in this work and the same quantities for bulk 40Fe – 60Ni alloy from [5]. It can be considered that the saturation magnetic field (B_s) of the sample on the nanoscale is much lower than the bulk.

|                  | Percentage (%) of Fe and Ni | Hc(Oe) | Bs(Oe) |
|------------------|----------------------------|--------|--------|
| **Bulk [5]**     | 40Fe-60Ni                  | 0.4    | ~15000 |
| **Bulk (this work)** | Fe                        | 0.08   | 4980   |
| **Nano particles (this work)** | 40Fe-60Ni | 0.12   | 4800   |

5. Conclusions

SEM images showed that the grain sizes of 40Fe-60Ni particles were in 50 - 300 nanometre range and that the topography of the nanoparticles after calcination was smoother than before calcination.

Magnetic measurements demonstrated that the 40Fe-60Ni nanoparticles were superparamagnetic before calcination, and that they were ferromagnetic materials after calcination with values of H_c = 0.12 and B_s = 4800 Oe, showing that the properties of the nanostructures were very different in respect to the 40Fe-60Ni bulk alloy.

Acknowledgments

The authors acknowledge the University of Sistan and Baluchestan, Iran for support of this work. A.M. DAVARPANAH would like to thanks Dr M. Almasi (Head of Faculty of Science, Kashan University, Iran) for his scientific co-operations.

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