### The Optical Properties of Nano Crystalline Fe$_{50}$Ni$_{50}$ Alloys Prepared by Mechanical Alloying Method

Iman Farahbakhsh

**Department of Engineering, Quuchan Branch, Islamic Azad University, Quuchan, Iran**

#### ABSTRACT

Mechanical alloying (MA) is a solid-state powder processing technique involving repeated cold welding and fracturing of powder particles in a high-energy ball mill. The present study is focused on optical properties and the effect of the milling time on the size of Fe$_{50}$Ni$_{50}$ alloy particles by X-ray diffraction (XRD), Field Emission scanning electron microscopy (FESEM), Transmission Electron Microscopy (TEM), vibration sample magnetometer (VSM) and Fourier transform infrared (FTIR) spectroscopy. Fe and Ni elemental powders have been ball milled in a planetary mill (Pulverisette 5, Fritsch) for milling times: 5, 30, 70 h and for BPR=30:1. The obtained FTIR spectra were analyzed using Kramers–Kronig (K-K) method. Optical studies showed that determined grain size based on hysteresis curve for 70h of milling time is in the same order of radiation wavelength.

© 2014 AENSI Publisher All rights reserved.

To Cite This Article: Iman Farahbakhsh, The Optical Properties of Nano Crystalline Fe$_{50}$Ni$_{50}$ Alloys Prepared by Mechanical Alloying Method. *Adv. Environ. Biol.*, 8(22), 691-697, 2014

#### INTRODUCTION

Mechanical alloying is a useful powder processing technique that can produce a variety of equilibrium and non-equilibrium alloy phases [1, 2]. The advantage of this process technology is that the powder can be produced in large quantities and the processing parameters can be easily controlled, thus it is a suitable method for commercial applications. It can also be used to produce amorphous and nanocrystalline materials in commercially relevant amounts and is also amenable to the production of a variety of alloy composition [3]. Magnetic alloys are the key to the future with high power electronics industry. The recent development of transformers, chokes, inductors, filters and the emergence of completely new technologies call for entirely new types of magnetic alloys prepared by mechanical alloying (MA) methods [4]. The main fields of application of high permeability Ni-Fe alloys are fault –current circuit breakers, LF and HF transformers, chokes, magnetic shielding, switching and storing cores, high sensitivity relays, for temperature compensation in permanent magnet systems and optical fiber magnetic sensor [5, 6].

The aim of the present work is first to produce the binary Fe$_{50}$Ni$_{50}$ system by high-energy ball alloying technique and evaluation of optical properties.

#### MATERIALS AND METHODS

Mechanical alloying as mixture of pure Fe (99.5%) powder and Ni (99.98%) powder was carried out in a commercial Fritsch Pulverisette 5 planetary ball mill. To prevent oxidation phenomena, the mixed powder was sealed in a cylindrical vial with capacity of 125 ml with stainless steel balls (d=10 mm) under an argon atmosphere in a glove box. The weight ratio of balls to powder was 30:1 for the 5, 30 and 70 hours milling time and vial rotation speed was 300 rpm. To prevent agglomeration of powder particles, we've used toluene as process control agent (PCA). The affection of medium as PCA, which doesn't participate in any chemical reaction in MA process, has not been specially reported [7].

To avoid excessive heating during milling, each 1 h of milling was followed by a stay during 10 min under the argon atmosphere at room temperature. After different milling times, the process was interrupted and small amounts of milled powders were taken out for analysis.

**Corresponding Author:** Iman Farahbakhsh, Department of Engineering, Quuchan Branch, Islamic Azad University, Quuchan, Iran. E-mail: ifarahbakhsh@gmail.com
X-ray diffraction patterns were recorded using D8 advanced model, product of 2002 in Bruker company (with CuKα radiation; λα=1.54Å). X-ray patterns were analyzed by X’pert Highscore plus program. Scherer method is used for calculating the crystallite size of samples. Scherer formula:

$$D = \frac{0.9 \lambda}{\beta \cos \theta}$$

Where D is crystallite size, λ is wavelength of used ray, β is peak wide in half of the most intensity and θ is Bragg angle.

Field emission scanning electron microscopy (FESEM) has been used for microstructure and crystallites size observations. FESEM device used has Hitachi label, S4160 model, product of Japan in 1996. In order to correctness of crystallite size examined using Transmission Electron Microscopy (TEM). The magnetic measurements were performed, using a vibration sample magnetometer (VSM) at room temperature. The optical measurements were performed, using a FTIR-8400 S product of Shimadzu Company for the absorption region 400 - 4000 cm⁻¹.

RESULTS AND DISCUSSIONS

X-ray diffraction studies (XRD):

(Fig.1b) shows the evolution of X-ray diffraction CuKα patterns as a function of milling time for the Fe50Ni50 powders. XRD (marked 0 h milled) shows reflections corresponding to body-centered-cubic (BCC) Fe (Im-3m, lattice constant a=2.8530Å) and face-centered-cubic (FCC) Ni (Fm-3m, a=3.5140Å) metals (Fig.1a). The process continues up to 5 hours of milling time, FeNi nanocrystalline alloy with Fm-3m space groups is formed. One clearly observes that the diffraction Bragg peaks broaden, with increasing milling time. This suggests a continuous decrease of the crystallite size because based on mechanisms discussed in [8] and a final value (for 70 h) of about 13nm. This value is smaller than the 45 nm [9] and 16.5 nm [10] for the iron–nickel ultra-fine particles obtained by a gas-condensation method, with the same composition. Reduction of crystallite size resulted in the increase of magnetization and improved the magnetic properties because the crystallite size approaches that of a magnetic domain, offering thus the possibility to eliminate the influence of the domain walls [11,12].

![Fig. 1: X-ray diffraction patterns of (a) pure Fe and pure Ni (b) Fe₅₀Ni₅₀ powdered alloys synthesized in high-energy mill for various milling times.](image-url)
Microstructure:

**FESEM analysis:**

The morphologies of Fe–Ni powders after various milling time are shown in Fig. 2. We can see in this figures that with increasing milling time, grain size became smaller, although size distribution is not uniform.

![Fig. 2](image1.png)

**Fig. 2:** FE SEM Micrograph of (Fe, Ni) powders prepared by mechanical alloying after different milling times (a) 5 h; (b) 30 h; (c) 70 h

**TEM analysis:**

The TEM image of the nano-FeNi alloy particles after 70 h of milling time are shown in Fig 3. With increasing milling time for 70 h, grain size is decreased and we found that the TEM results as well as agree with the results obtained of X-ray diffraction and FE SEM Micrograph.

![Fig. 3](image2.png)

**Fig. 3:** TEM Micrograph of (Fe, Ni) powders prepared by mechanical alloying after 70 h of milling time.

**Calculating magnetic grain size based on hysteresis curve:**

Fig 4. shows hysteresis curve (magnetization vs. applied field) for synthesized FeNi nanoparticles after 70 hours of milling with BPR=30:1. To calculate maximum size of magnetic grain, we can assume particles as
spheres with radius (r) and use (eq. 5) [13]. In this equation, K is Boltzmann constant (1.38×10^{-16} \text{erg/K}), T is temperature (300 K), dM/dH is gradient near zero field (=0.02 in fig. 4.), \rho is nanoparticles density for FeNi (=8.02 gr/(cm^3)), M_s is magnetic (=120 emu/g). Based on these values, particle size is about (3×10^4) nm.

\[
d_{\text{max}} = \left[ \frac{18KT (dM/dH)_{H=0}}{\pi \rho M^2 s} \right]^{1/3}
\]

(2)

**Fig. 4:** Magnetization vs the field applied to milled after 70 h of milling time.

**Fig. 5:** Transmittance of the synthesized FeNi nanopowders for various milling times versus wave number.

**Optical properties:**

**FTIR Analysis:**

The room temperature transmittance of the synthesized FeNi nano powders for various milling times was investigated by Fourier transform infrared (FTIR) spectroscopy in the range of 400–4000 cm^{-1} and the results are presented in Fig. 5.

Comparing this with spectrum of pure FeNi [14], we can see that there is no peak in wave number 2400 cm^{-1} in pure FeNi, because there is no bond between Fe and Ni atoms in this condition and no optical mode excitation is expected. Existing peaks in other wave numbers for spectrum of pure FeNi is because of acoustic modes and also vibration and rotation modes of grains. Based on Fig. 5 we can see that for wave number of 2400 cm^{-1} and for 70 hours of milling, wavelength will be about 27 μm, which is in good agreement with calculated grain size base on hysteresis curve. As is obvious from this figure, and assuming one valley or one
peak for each wave number, it is evident that increasing milling time will cause shifting these valleys and peaks to left (lower wavelength), so it is expected that increasing milling time cause smaller grain size. This is also in agreement with X-ray and Electron Microscopy results.

**Theory of K–K method:**

The K–K method was used to evaluate the optical constants of the FeNi nanopowders prepared by the Mechanical alloying method using FTIR transmittance spectra data. Absorption is characterized by a decrease in transmitted light intensity through the sample. The quantity used to discuss the absorption, as a function of the wavenumber, is the transmittance (T), which is the ratio of the intensity of the light transmitted through the sample (I) to the incident light intensity (I_0). Transmittance is usually given by the more common percent transmittance (T%) by T% = 100T. The absorption (A) is defined as log10 (1/T), according to Lambert’s principle [15].

\[
A(\omega) = 2 - \log_{10}(T(\omega))
\]

\[
R(\omega) = 100 - A(\omega) - T(\omega)
\]

Where R(ω) is the reflectance at a particular wavenumber. The refractive index N is the most widely physical quantity used in optical design. Generally, it is a complex quantity given as:

\[
N(\omega) = n(\omega) + ik(\omega)
\]

Where n(ω) and k(ω) are the real and imaginary parts of the complex refractive index, respectively obtained from the following equations:

\[
n(\omega) = \frac{1 - R(\omega)}{1 + R(\omega) - 2 \cos \varphi(\omega) \sqrt{R(\omega)}}
\]

\[
k(\omega) = \frac{2 \sin \varphi(\omega) \sqrt{R(\omega)}}{1 + R(\omega) - 2 \cos \varphi(\omega) \sqrt{R(\omega)}}
\]

Where R(ω) is the reflectance and is the phase change between the incident and reflected signals for a particular wavenumber ω. This phase change can be derived from K–K dispersion relation [16]. Also, can be obtained from Fourier transform of K–K dispersion relation [17].

\[
\varphi(\omega) = \frac{\omega}{\pi} \int_0^\infty \frac{\ln R(\omega') - \ln R(\omega)}{\omega'^2 - \omega^2} \, d\omega'
\]

The Fourier transform of K–K was chosen to avoid the singularity that may happen in the integral. The dielectric function is the square of the refractive index. Therefore, the real and imaginary parts of the complex dielectric function can be written as:

\[
\varepsilon(\omega) = [N(\omega)]^2 = [n(\omega) + ik(\omega)]^2
\]

\[
\varepsilon'(\omega) = n^2(\omega) - k^2(\omega)
\]

\[
\varepsilon''(\omega) = 2n(\omega)k(\omega)
\]

Fig. 6 shows imaginative part of refractive index based on wave number for various milling time. This part of refractive index is related to waste. Comparing Figs 5 and 6 shows that each valley in transmittance spectrum is corresponding with respective absorptive peak. Waste spectrum may be as a result of single energy electrons excitation in solid or collectively excitation of valence electrons, namely plasmon. Namely plasmons are fluctuations in charge density. Incident electrons will lose integer multiplication of plasmon energy to cause excitation. plasmons will be seen as sequential peaks in waste energy spectrum. Roots of real part for dielectric function are requirement for plasmons vibration. We can understand this by comparing figures 6 and 7.

**Fig. 6:** The imaginary part of the refractive index of synthesized FeNi nanopowders for various milling times versus wave number.
**Fig. 7**: The real part of the dielectric function of synthesized FeNi nanopowders for various milling times versus wave number.

Fig. 8. shows imaginative part of the dielectric function based on wave number for various milling time. Imaginative part of dielectric function can be used to examine and describe real transitions between occupied and unoccupied states. Existing peaks in imaginative part diagram of dielectric function is according to possible electronic transitions. Fig. 9 shows reflection spectrum based on wave number for various milling time.

**Fig. 8**: The imaginary part of the dielectric function of synthesized FeNi nanopowders for various milling times versus wave number.

Comparing Figs 5 and 9 shows that each valley in transmittance spectrum is corresponding with respective absorptive peak. In reflection spectrum for areas with higher energies (higher wave numbers), which electrons have major roles in the reflection process, increasing milling time caused to widen and then faded peaks. So more electrons had excited in valence band and transferred to conductivity band. We can say that increasing milling time resulted in more bilateral permeation of Fe and Ni in each other's networks, then crystallite symmetry increased and so energy gap decreased.

**Fig. 9**: Reflection spectrum of synthesized FeNi nanopowders based on wave number for various milling time.
Conclusion:
In this paper we studied optical properties of nanostructured Fe$_{50}$Ni$_{50}$ alloy. Optical studies showed that determined grain size based on hysteresis curve is in the same order of radiation wavelength. Increasing milling time caused to decreasing grain size, and these results is supported by optical studies.

REFERENCES