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Magnetic Properties Evaluation of Fe-Al Nanocrystalline Alloys Fabricated by Mechanical Alloying Technique as a Function of Milling Time

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ABSTRAK

Sifat-sifat magnetik dari paduan kristal nano $Fe_{50}Al_{50}$ yang disiapkan dengan teknik paduan mekanik (dengan menggunakan bubuk Fe dan Al sebagai prekursor) telah diteliti dengan detil sebagai fungsi dari waktu penggilingan dengan variasi waktu dari 1 sampai dengan 24 jam. Analisa strutur diasarkan pada X-ray diffraction *(XRD) dan* Extended X-ray Absorption Fine Structure Spectroscopy *(EXAFS) menunjukkan bahwa proses paduan terjadi setelah 12 jam penggilingan. Tentang sifat magnetik, data yang diperoleh dari* Superconducting Quantum Interference Device *(SQUID) menunjukkan bahwa* magnetic saturation *(Ms) and* the coercivity *(Hc) sangat tergantung waktu penggilingan. Dari hasil yang diperoleh, ditemukan bahwa dengan mengatur waktu penggilingan, merupakan faktor kunci dalam memperoleh transformasi struktur dan harga-harga besaran magnit yang tepat.*

Kata Kunci: *Paduan kristal nano $Fe_{50}Al_{50}$, struktur lokal, sifat-sifat magnetik, waktu penggilingan, EXAFS, SQUID*

ABSTRACT

Magnetic properties of nanocrystalline $Fe_{50}Al_{50}$ alloys prepared by mechanical alloying technique (using commercial Fe and Al powders as precursors) were studied in detail as a functions of the milling time ranging from 1 to 24 hrs. The structural analyses based on X-ray diffraction (XRD) and extended X-ray absorption fine structure spectroscopy (EXAFS) revealed that the alloying process took place after 12 hrs of milling time. Concerning the magnetic behavior, the data that were obtained from a superconducting quantum interference device (SQUID) showed that both the magnetic saturation (*Ms*) and the coercivity (*Hc*) depend strongly on the milling time. From the results that we obtain, we found out that by adjusting the milling time, is a key factor in obtaining an appropriate structural transformation and appropriate magnetization values.

Keywords: *$Fe_{50}Al_{50}$ nanocrystalline alloys, Local Structure and Magnetic Properties, Miling time, EXAFS, SQUID*

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INTRODUCTION

In recent years, nanocrystalline materials have been the subject of scientific interest because of their attractive properties often superior to those of conventional polycrystalline materials and also amorphous alloys of the same composition. Their properties are quite different from those of the corresponding crystalline materials. Nanocrystalline materials are novel materials that are not only scientifically interesting but also holding great potential for a number of technological applications.[1-3]

Nanocrystalline and amorphous magnetic materials have been studied for many applications in industrial products, including transformers, motors, and a wide variety of magnetic components in sensors, power electronics, electrical energy control/management systems, telecommunication equipment and pulse power devices.[4-5]

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The wide range of applications arises from the versatile nature of these materials which can provide fast magnetization reversal with minimal magnetic losses. One aspects of the versatility is that materials can be prepared without stoichiometric restrictions characteristic of crystalline materials and by post-fabrication heat treatment. In certain cases, alloys can be designed for specific applications. There are, however, constraints that limit the fabrication of these materials. In developing a magnetic material, two extreme cases are of interest from an application point of view: one is a material with as high permeability as possible and the other is a material with the saturation induction (*Bs*) as high as possible which is an important area of research and development.[6-7]

The nanocrystalline supersaturated solid solutions and amorphous phase in the powders are obtained during mechanical alloying.[8-9] Nanocrystalline materials obtained by high-energy ball milling are of great interest since it is known that those materials may exhibit different electrical, magnetic, optical, and other physical properties in the nano-regime due to finite size effects. [10]

Mechanical alloying (MA) is non-equilibrium solid-state alloying technology for powders and can be used to synthesize novel alloys impossibly obtained by conventional technology. It is well known that mechanical alloying of elemental metal mixtures can generate equilibrium and non-equilibrium structures, including supersaturated solid solution, nanocrystalline, metastable compounds and amorphous solids.[1,11]

During the last years the mechanical alloying technique has been found to be very effective in producing powders with interesting properties. By this means it is possible to synthesize alloys or composite materials with highly dispersed components far away from thermal equilibrium state, e amorphous or nanocrystalline materials.[1-2] Last two decades, the various mechanical routes used in producing soft magnetic powders (ferrites and alloys) were reported.[10-12] Mechanical alloying is able to produce nanostructure materials with unique chemical, structural, electrical and magnetic properties, due to type of disorder created by the high density of defects and the small crystal size.[13]

The formation of metastable phases and disorder in the crystal lattice through MA gives interesting mechanical and magnetic properties. This is particularly evident in the case of the Fe-Al inter metallic system because the complicated phase diagrams and dependence of magnetic properties on disorder and microstructure make them interesting to study through the MA process. Furthermore, the Fe-Al systems are technologically important because of their superior mechanical properties and resistance to corrosion. Moreover, their being amenable to easy substitution by other metal atoms. For these reasons, Fe-Al alloy systems with various compositions have been investigated in recent years.[14,15] More particularly, the Fe-Al inter metallic compounds offer many attractive properties, such as high specific strength, good strength at intermediate temperatures and excellent corrosion resistance at elevated temperatures under oxidizing, carburizing and sulfidizing atmosphere.[16,17]

The degree of order in Fe-Al inter metallic alloys has an important influence on their magnetic properties. Moreover, the deformation of ordered alloys causes a dramatic increase in magnetization.[18] In Fe-Al alloys with the bcc structure, the magnetic moment of Fe atoms depends on the local structure. As a rule, Fe atoms with less than four nearest Fe neighbors possess no localized magnetic moments, and Fe atoms become magnetic only when they have four or more nearest neighbors.[19]

The magnetic properties of an assembly of small grains depend on the counter play between the local magnetic anisotropy energy and the ferromagnetic exchange energy.[20] In this work, we studied $Fe_{50}Al_{50}$ alloys by various the milling time. Their structural and magnetic properties were then studied by means of X-ray diffraction (XRD), extended X-ray absorption fine structure spectroscopy (EXAFS), and superconducting quantum interference device (SQUID).

EXPRIMENTAL DETAILS

$Fe_{50}Al_{50}$ nanocrystalline alloys were prepared by mechanical alloying using a SPEX 8000 mixer with stainless-steel balls and a stainless-steel

vial. The starting mixture of $Fe_{50}Al_{50}$ was formed using commercial powders of Fe (53 μm, 99.9%) and Al (53-106 μm, 99.9%). For the purpose of milling, the weight ratio of the ball to powder was 5:1. $Fe_{50}Al_{50}$ alloys were mixed and ground ed for different times of 1, 2, 4, 6, 12, and 24 hrs. The process was performed in an Ar gas bient to avoid oxidation. After the preparation, magnetic measurements were carried out using a superconducting quantum interference device (SQUID). The sizes of the particles and their morphologies were analized preliminarily by using scanning electron microscopy (SEM). XRD data were obtained using an X-ray diffractometer with the Cu- Kα radiation. The data were analyzed using Materials Data Inc. DI) software. Also, based on these data, the crystallite size and the strain of the samples were estimate terms of the Williamson Hall method. The Extended X-ray absorption fine structure (EXAFS) data were obatined from the EXAFS surements, which was operated at energy of 2.5 GeV and a maximum current of 200 mA. EXAFS spectra obtained at the Fe K-edge (7111.181 eV) in the transmission mode at room temperature. After that, the EXAFS data were analyzed by Ifeffit software, an interactive program for XAFS analysis.

RESULTS AND DISCUSSION

Figure 1a, 1b and 1c show a typical SEM images revealing the variations in particle shape and size of the $Fe_{50}Al_{50}$ powders after 2, 12, and 24 hrs of milling time. Our SEM study revealed that the particles present in the $Fe_{50}Al_{50}$ samples had quite similar shapes, where very small particles were located on the surfaces of big particles. The particle size varied as we changed the milling time. As can be seen from Figure 1a, 1b and 1c, the average particle sizes estimated from the SEM images decreased with increasing milling time. Such results are in good agreement with the data estimated by using the Williamson-Hall plot.

XRD patterns obtained from the $Fe_{50}Al_{50}$ nanocrystalline alloys are shown in Figure 2. From Figure 2 it can be seen that the peaks after 12 hrs of milling are broader and shifted to smaller angles, which are due to the deformation of the structure and variation in the crystallite size. The deformation is due to the replacement Fe atoms by Al atoms, which signals the formation of an alloy.

Based on XRD data, the crystallite size and the lattice ain can be evaluated from the intercept and slope of the Williamson-Hall plot: [21]

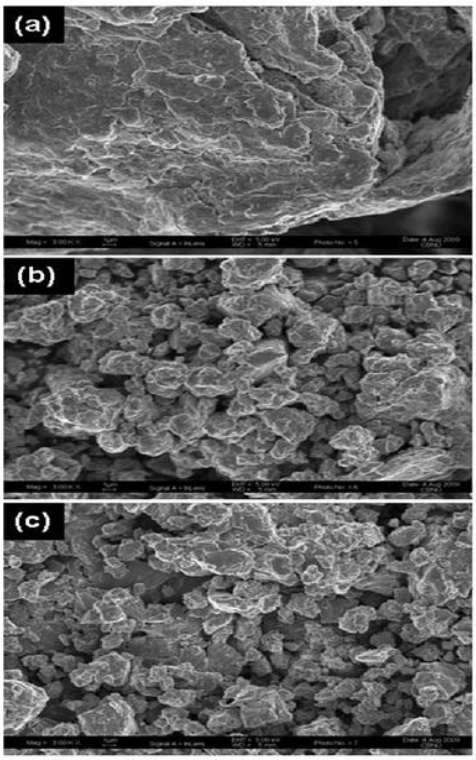

Figure 1. Typical SEM images for $Fe_{50}Al_{50}$ nanocrystalline alloys with milling times of (a) 2 hrs, (b) 12 hrs, and (c) 24 hrs.

$$B \cos \theta = (K\lambda/D) + 2\epsilon \sin \theta, \quad (1)$$

where B is the full width at half maximum (FWHM) in radian D is the average crystallite e, ϵ is the strain, K is the shape factor, λ is the X-ray wavelength and θ is the Bragg angle.

Figure 3 shows the crystallite sizes and the strain of the samples as a function of the milling time. The crystallite sizes and the strain of the sample after a 1hr of milling were found to be about 220 nm and 5.32×10^{-3}, respectively. After a 24-hr of milling, the crystallite size decreased

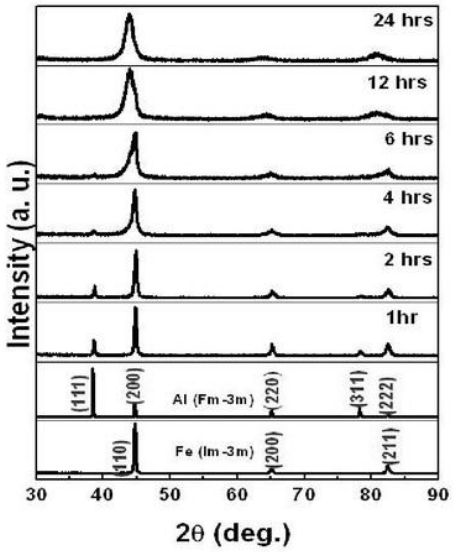

Figure 2. XRD patterns of $Fe_{50}Al_{50}$ with different milling time

to ~13.5 nm while the strain increased to 19.03×10^{-3}. This trend could be explained by the formation of defects during the milling process. The collision energy was introduced and thus promoted a distortion of lattice parameters of the powder. However, at 24 hrs of milling, it is shown that the internal strain decreases. Since the crystallite size almost reached its saturation value at around 12 hrs of milling, the impact produced by MA was to anneal the high amount of stress within Ni-Al-C particles, thus decreasing the lattice strain.[22]

The inset of Figure 3 presents the variation of the lattice parameter, calculated from Scherrer's formula (here, the 110 peak of the $Fe_{50}Al_{50}$ was fitted to a Gaussian function); it increases slightly from 2.01 to 2.06 Å.[23]

The EXAFS can give direct information about the variation of local structure.[24] Figure 4 show the normalized EXAFS spectra of $Fe_{50}Al_{50}$ at Fe-K near edge. The processed samples were similar to each other but above the edge the spectra gradually changed. This suggests that the electronic configuration for the Fe central atoms was unchanged but the surrounding atoms around the atom central Fe was changed during the MA processing.

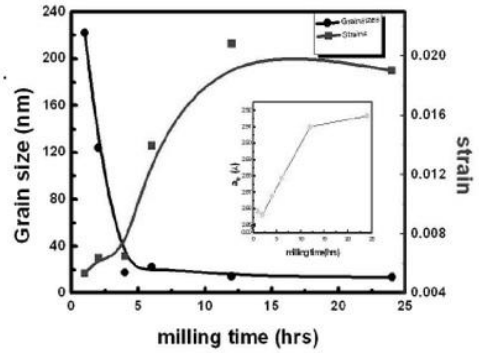

Figure 3. Grain size and strain for $Fe_{50}Al_{50}$ nanocrystalline alloys as functions of the milling time. The inset shows the variation of the lattice parameter based on (110) peak with respect to the milling time.

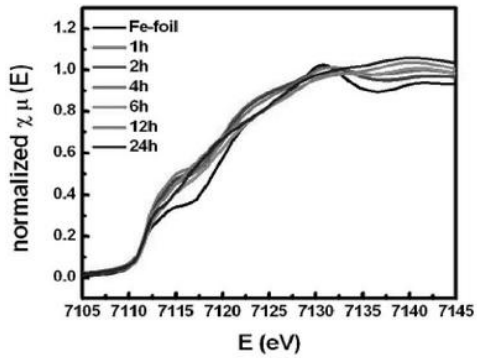

Figure 4. Normalized XAFS spectra of $Fe_{50}Al_{50}$ at Fe-K edge

Figure 5 shows the k-weighted EXAFS spectra of $Fe_{50}Al_{50}$ powders processed for 1, 2, 4, 6, 12, and 24hrs. The decreasing in the amplitude before the 12 hrs milling indicates that the fracture and the cold welding of both Fe and Al were dominant and that a minor change in the local structure had occurred. However, a significant change in the phase took place for the sample with a milling time of 12 hrs. This indicates that the alloying process was dominant, and new phase was formed for this sample. The amount of the new phase increased as the milling time was increased. The radial atomic density in real space can be seen in the Fourier Transformation Spectra that is shown in Figure 6.[24]

The dotted lines shown in Figure 6 indicate the first, second, third, and fourth shells of pure Fe, which are used as a guide lines to compare with alloyed samples. The magnitude of the Fourier Transformation Spectrum decreased when the milling time was increased. This suggests that the number of Fe-Fe bonds are decreased due to the inter-diffusion of Fe and Al atoms. In addition, the first and the fourth shells were shifted, corresponding to the increased of Fe-Al bonding. This indicates that the short- range and the long-range orders increased with increasing milling time.

Clearly seen that, the 12 and 24 hrs milled samples exhibit different positions of the phase in the first and the fourth shells compared to the 1, 2, 4, and 6 hrs milled samples. This can be explained by a change in the Fe-Fe order due to alloy formation of Fe-Al. Such results are in good agreement with the XRD data. These changes in the local structural order caused the variation in the magnetic properties of the samples. Here, EXAFS was used to examine the local structure of the samples. Variations in amplitude and the shift of an EXAFS peak give information on the structural changes occurring in the MA process on an atomic scale. The reduction of the amplitude in the EXAFS spectrum can be caused by spatial and chemical disorders. Most spatial disorder causes variations in the phases in EXAFS spectra. Such a situation influences the magnetic properties of the samples strongly, as will be presented below.

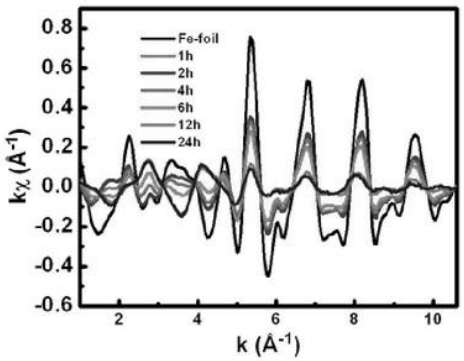

Figure 5. The k-weighted EXAFS spectra of $Fe_{50}Al_{50}$ with indicated processing times

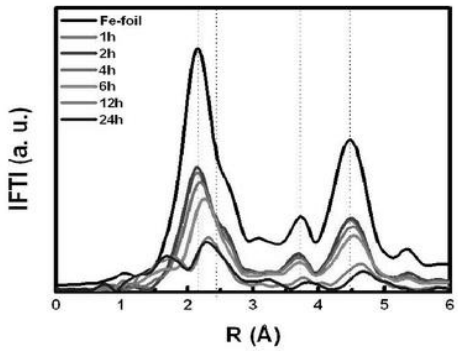

Figure 6. The Fourier Transformation of the EXAFS Spectra of the $Fe_{50}Al_{50}$ alloys measured at the Fe K-edge for various milling times

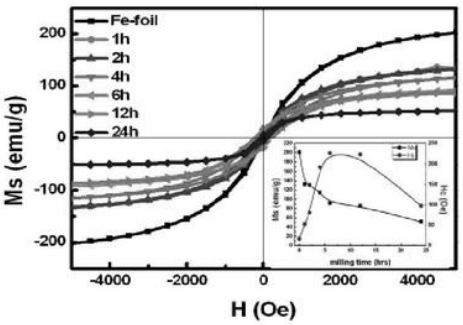

Figure 7. The hysteresis loop of $Fe_{50}Al_{50}$ alloys as functions of the milling time and the Ms and Hc (inset)

Figure 7 shows the magnetization and the coercivity as functions of the milling time. One can see that magnetization rapidly decreases in the initial stage of the milling (lower milling time) because of the changes in the structure of mixed powder from Fe grains to $Fe_{50}Al_{50}$ grains occur.[25,26] Begin at 2 hrs milling time and after, the magnetization decreased slowly. The variation of the magnetization could be caused by the dilution of the magnetic lattice of Fe by Al with increasing milling time. In Figure 7, one can also see that the coercivity (*Hc*) increases with increasing the milling time, reaching a maximum value at about 225 Oe after a 6 hrs milling time, and it decreases to about 100 Oe after a 24 hrs milling time. The *Hc* increase for a relatively short milling time can be attributed to a reduction

in the crystallite size. Meanwhile, the decreases in *Hc* for a longer milling time indicate an increased formation of the Fe-Al alloy.

Further milling tended to make all samples highly disordered, then, they lost part of their high magnetic anisotropy, which makes the *Hc* dereased.[24] Furthermore, one can also see from Figures 3 and 7 that magnetic saturation decreased as the lattice parameter increased.

CONCLUSION

The formations of $Fe_{50}Al_{50}$ metastable alloys were explicitly shown in the XRD patterns with shifted and broadened peaks. EXAFS spectra showed variations in the amplitude and the phase for the samples with milling times of 12 and 24 hrs. The significant change in the phase confirmed that new Al atoms were introduced around the central Fe atoms during the MA process which were alloying occurred. The magnetization saturation was decreased due to magnetic dilution caused by the Al. Meanwhile, the coercivity decreased due to growing single-domain size and reduced particle size.

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