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Local Structure and Magnetic Properties of $Fe_{50}Cr_{40}Si_{10}$ Nanocrystalline Alloys

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Abstract: The structural and the magnetic properties of nanocrystalline $Fe_{50}Cr_{40}Si_{10}$ alloys were prepared via mechanical alloying by using Fe, Cr, and Si elements with 1- to 24 hrs milling times. Structural analysis based on X-ray diffraction and Extended X-ray absorption fine structure spectroscopy. Concerning the magnetic behavior, the data obtained from a vibrating sample magnetometer at 300 K shows that both the magnetic saturation and the coercivity are dependent strongly on the milling time and the crystallite size. By adjusting the milling time, both appropriate structural transformation and magnetization values are obtained.

1. Introduction

In recently 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]. 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 B_s as high as possible [6] is an important area of research and development [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].



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Mechanical alloying (MA) is a non-equilibrium solid-state alloying technology for powders and can be used to synthesize novel alloys impossible to obtain 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]. MA has been shown to be capable of synthesizing a variety of equilibrium and non-equilibrium alloy phases starting from blended elemental or pre-alloyed powders. The non-equilibrium phases synthesized include supersaturated solid solutions, metastable crystalline and quasicrystalline phases, nanostructures, and amorphous alloys [1]. 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 like amorphous or nanocrystalline materials [1-2].

Besides the incipient crystallisation of amorphous solids, mechanical alloying is nowadays one of the widely used preparation nanocrystalline structures. Mechanical alloying techniques involve the synthesis of materials by high-energy ball milling, in which elemental blends (or pre-alloyed powders, oxides, nitrides, etc) are milled to achieve alloys or composite materials. High-energy ball-milling offers indeed complementary degrees of freedom in the choice of possible for synthesizing new materials and appears further as an attractive method of synthesis in view of its potential for large scale production. These techniques allow producing non-equilibrium structures/microstructures including amorphous alloys, extended solid solutions, metastable crystalline phases, nanocrystalline materials and quasi crystals. 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].

Nanocrystalline magnetic materials are intensively investigated because of their remarkable properties such as coercivity and saturation magnetization, which significantly differ from those of microcrystalline materials and are sensitive to the structure and microstructure. It is known that the interplay between the crystallite size and the magnetic properties i.e. saturation magnetization, coercivity of FeCr and FeCrSi alloys with nanometer crystallite size is not yet understood fully. This research constitutes a contribution to the comprehension of the relation between the physical properties (local structural and magnetic) of nanostructure some alloys elaborated by high energy ball milling method.

Although important progress has been made in the study of nanocrystalline magnetic materials prepared by mechanical alloying methods, there are numbers of open questions, whose solution could help to advance both their theory and practical applications. Among the challenges facing experts are those concerned with the correlation between structural and magnetic properties of materials at nanocrystallite size scales.

The main objective of this study is the investigation of the relationship between the structure and the magnetic properties of nanocrystalline alloys which were made by mechanical alloying. To understand the mechanism of solid state reaction by mechanical alloying on the atomic scale the particle size of these alloys will be examined by the structural evolution by using X-ray diffraction (XRD) and Extended X-ray Absorption Fine Structure (EXAFS), and the magnetic properties are measured by using a vibrating sample magnetometer (VSM).

2. Experimental method

$Fe_{50}Cr_{40}Si_{10}$ nanocrystalline alloys were prepared by mechanical alloying using SPEX 8000 mixer with stainless steel balls and vial. The starting material were a mixture of pure Fe(53 μ m, 99 %), Cr(75 μ m, 99 %) and Si(105 μ m, 99 %) powders which were used commercial powders as the precursors. The weight ratio of balls-to-powder mixture was 5:1. $Fe_{50}Cr_{40}Si_{10}$ alloys were mixed and ground for different times 1-, 3-, 6-, 12-, and 24 hrs. The process was performed in Ar ambient to prevent oxidation during the alloying process. Magnetic measurements were carried out on VSM in magnetic field of 10 kOe. Structure data were obtained by using the X-ray diffractometer (XRD) Cu-K α radiation. The data were analyzed using Match. Based on these data, crystallite size of the samples were estimated in terms of the Williamson-Hall plot. EXAFS data were collected with energy of 2.5 GeV, and a maximum current of 200 mA. EXAFS spectra were obtained at Fe K-edge (7112 eV) in the transmission mode at room temperature. The sample chamber was filled with pure nitrogen gas. Then the EXAFS data were analyzed using IFEFFIT software, an interactive program for XAFS analysis.

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3. Results and discussions

Figure 1 shows the X-ray diffraction of the $Fe_{50}Cr_{40}Si_{10}$ powder. The XRD pattern of un-milled powder indicates the characteristic reflection of individual element constituent of the Fe, Cr, and Si elements. From 1- up to 24 h, Si patterns were disappeared, it resolved in Fe and Cr structure, only the bcc phase peaks are visible (A2 solid solution α -Cr (Fe, Si)).

Based on the line in Figure 1, that Fe and Si atoms dissolved into the Cr lattices, so that the intensity of the bcc phase diffraction peaks decreases with increasing of milling time.

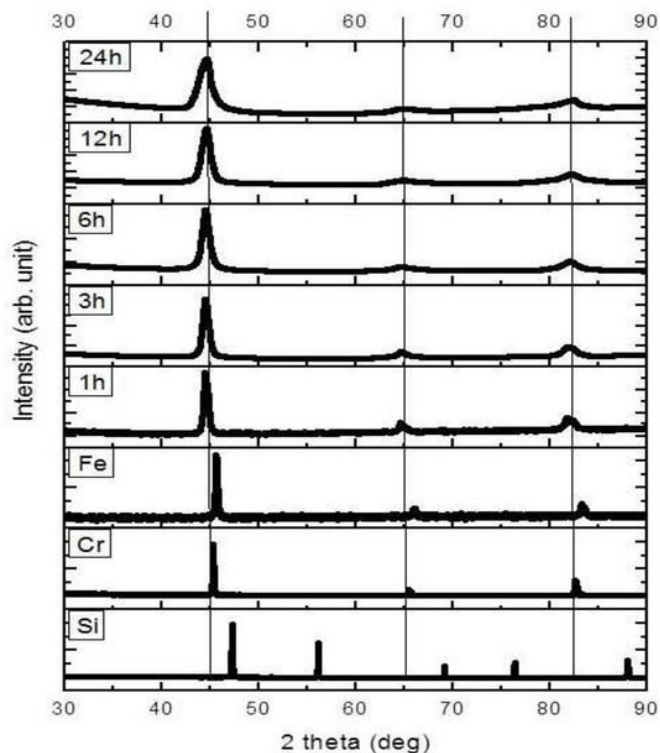


Figure. 1: The XRD of $Fe_{50}Cr_{40}Si_{10}$ of 1- to 24 h, respectively.

In Figure 2, the deformation is due to the replacement Cr atoms by Fe and Si atoms, which signals the formation of an alloy. Based the data, the crystallite size and the lattice strain can be evaluated from the intercept and slope the Williamson-Hall plot, $B \cos \theta = \frac{K\lambda}{D} + 2\epsilon \sin \theta$, where B is the PWHM in radians, D is the average crystallite size, ϵ is the strain, K is the shape factor, λ is the X-ray wavelength and θ is the Bragg angle. In general, both of crystallite size and lattice parameter are decreased.

In inset of Figure 2, the lattice parameter decreases for increasing milling time, except for 1 h milled which is the sample has not stabled yet. The peak after 3 h milled are broader, which is due to the deformation of the structure and variation in the crystallite size.

Figure 3 of the EXAFS give direct information about the variation of local structure. Its use to examine local structure around the Fe ions in the $Fe_{50}Cr_{40}Si_{10}$ alloys. Figure 3 shows the normalized near edge spectra for the processed sample were similar to each other but above the edge the spectra gradually changed. This suggests that electronic configuration for the Fe central atom was unchanged during the MA processing.

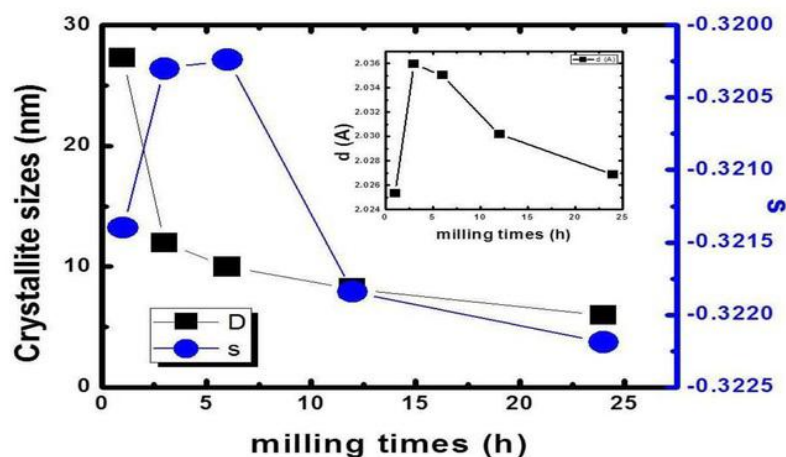


Figure 2. The crystallite size of $\text{Fe}_{50}\text{Cr}_{40}\text{Si}_{10}$ of milled powder for 1- to 24 h. Inset is the lattice parameter respect to milling times.

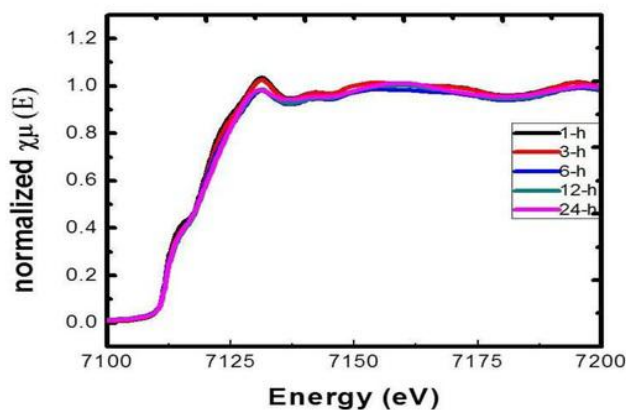


Figure 3: The EXAFS spectra of $\text{Fe}_{50}\text{Cr}_{40}\text{Si}_{10}$ of milled powder for 1- to 24 h

Figure 4 shows the EXAFS spectrum of $\text{Fe}_{50}\text{Cr}_{40}\text{Si}_{10}$ alloys for 1- to 24 h. The reduction of amplitudes is related to the disorder of local structure, and the variation of the phase is related to the change of chemical order. In Figure shows the significant change in the amplitude and the phase. Its mean there is a huge changed in local structure. The systemic variations of the amplitude and phase in the EXAFS spectra confirmed that alloying at atomic scale occurred during MA process.

The decrease of the amplitude before 12 h indicates that the fracture was dominant. After 12 h milling, the phase was shifted significantly. This indicates that the Fe and Si atoms were diffused into the Cr structure and new materials were produced during this process.

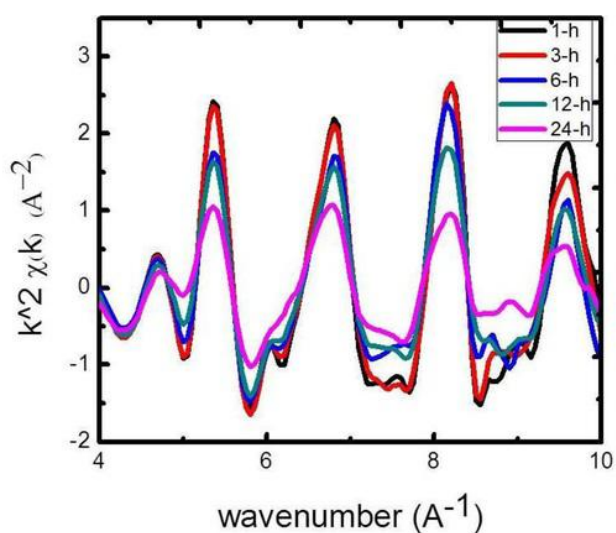


Figure 4. The k-weighted EXAFS spectra of milled powder for 1- to 24 h

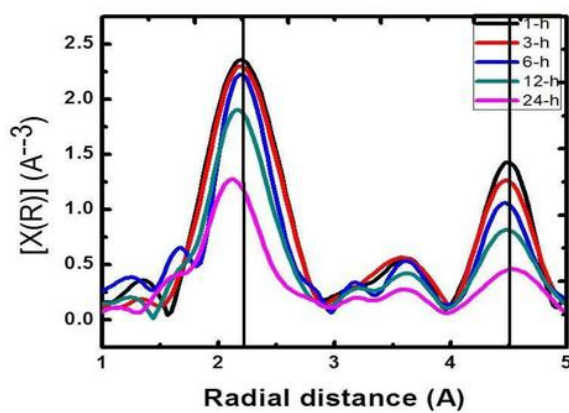


Figure 5: The Fourier transformation of EXAFS spectra of powder for 1- to 24 h

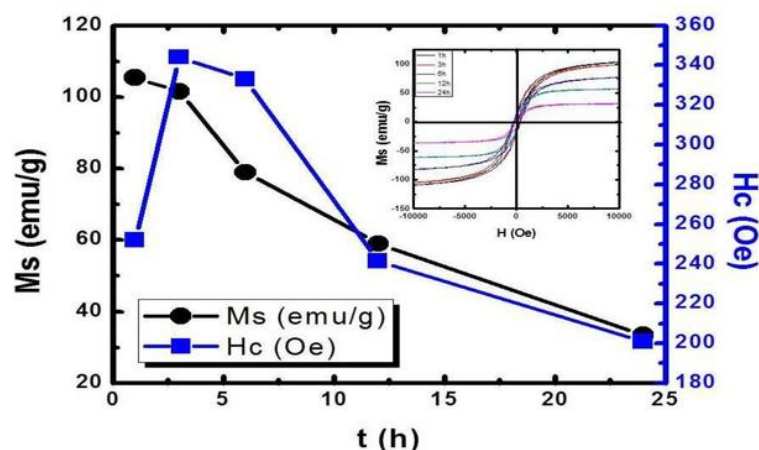


Figure 6. Variation of magnetization and Coercivity of $Fe_{50}Cr_{40}Si_{10}$ alloys with various milling times. The inset shows the variation of the hysteresis loop with magnetic saturation respected to the milling times.

Concerning the magnetic behaviors, both magnetization saturation (M_s) and coercivity (H_c) depended strongly on milling times, t_m . The M_s is decreased because of particle sizes is decreased and the transition from Fe, Cr, and Si grains to $Fe_{50}Cr_{40}Si_{10}$ grain. The H_c in beginning time is increased quickly because of fractures in process unstable yet. After 3 h milling times H_c is decreased continuously. The coercivity (H_c) is decreased continuously until 24 h milled as effect of parameter d is decreased. The variation of M_s and H_c can be seen in hysteresis loop in inset of the Figure 6.

Conclusion

The relatively new phase of $Fe_{50}Cr_{40}Si_{10}$ alloy is explicitly shown in the EXAFS spectra by the variation of amplitude and phase after 12 h milling times. The significant change of the structural phase revealed that new atom neighbors, the atom central Fe substituted by Cr and Si atoms were increased during the MA process. The magnetic saturation (M_s) is decreased in long milling times as effect of crystallite size is decreased. The coercivity (H_c) is decreased continuously until 24 h milled as effect of parameter d is decreased.

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