Magnetic properties and Structural characterization of nanocrystalline Fe-20%A (Ni, Co and Si) alloys powders synthesized by mechanical alloying process

Abderrahmane Younes, Abderahim Abada, Amirouche Bouamer and Rachid Amraoui

Abstract− This study focuses on the preparation and characterization of nanocrystalline Fe-A (Ni, Co, and Si) alloy powders using mechanical alloying technique with Retsch PM 400 high energy planetary ball mill. The evolution of the phases and magnetic properties of the powders are investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray (EDX), and vibrating sample magnetometer (VSM) as a function of grinding time. The XRD results indicate that after 20 hours of milling, the FeNi, FeCo, and FeSi phases are completely formed. The lattice deformation of FeSi alloy is 0.7%, and the grain size decreases to 17 nm, 13.5 nm, and 10 nm for FeNi, FeCo, and FeSi, respectively. SEM observations of the powder morphologies at different stages of alloy formation are also conducted. Moreover, elemental maps of Fe, Ni, Co, and Si obtained by EDS experiments confirme the XRD results on the evolution of the alloy formation. Finally, the VSM results showed that the magnetic properties of FeNi, FeCo, and FeSi alloy nanoparticles are influenced by the composition, size, and morphology of the particles. Overall, this study provides valuable insights into the preparation and characterization of nanocrystalline Fe-A (Ni, Co, and Si) alloy powders, which may have potential applications in various fields.

Keywords− Nanostructured FeNi, FeCo and FeSi, Structural properties, Magnetic behavior

NOMENCLATURE

VSM	Vibrating Sample Magnetometer.
XRD	X-ray Diffraction.
SEM	Scanning Electronic Microscopy.
EDS	Energy Dispersive Spectroscopy.
Ms	Saturation Magnetization.
Hc	Coercivity.

I. INTRODUCTION

The science and technology of nanocrystalline materials has a significant advance in the recent years. Mechanical alloying is a technique for the synthesis of such materials, which are of interest for several application fields. The study of these materials is a significant feature that provides an indication of the technological development. Fe-A (Ni, Co and Si) alloys have a range of magnetic properties that make them useful for various applications, including high magnetic permeability, high magnetization saturation, and low coercivity field [1-3]. These materials are applied in different areas, for example, magnetic application, biomedical application, and Turbomachinery components [4-6]. Magnetic evaluation, which refers to the use of various testing techniques to investigate magnetic properties of materials, is an essential tool in different industrial applications owing to its many advantages [7-11]. Several studies have tackled with these techniques to investigate diverse properties compared to conventional techniques. Among the many magnetic testing processes, the analysis of Vibrating Sample Magnetometers (VSM) is a highly sensitive method that can detect variations in chemical composition, lattice strain, and microstructural properties.

The main scientific purpose of this work is to investigate the preparation and characterization of nanocrystalline Fe-20%A (Ni, Co, and Si) alloy powders using mechanical alloying technique. The study aimed to understand the evolution of the phases and magnetic properties of the powders as a function of grinding time. X-ray diffraction, scanning electron microscopy, energy dispersive X-ray, and vibrating sample magnetometer were used to investigate the structural and magnetic properties of the powders. The study is important because nanocrystalline Fe-20%A (Ni, Co, and Si) alloy powders have potential applications in various fields such as magnetic storage devices, catalysts, and sensors. The findings of this study can provide valuable insights into the preparation and characterization of these powders, which can lead to the development of improved materials with desirable properties for specific applications.

II. EXPERIMENTAL PROCEDURE

The synthesis of nanocrystalline Fe-20%A (Ni, Co and Si) was performed in a PM 400 planetary mill with tungsten carbide (WC) pots. The elemental powders were purchased from Sigma Aldrich (USA). The first one is pure iron with a particle size of 80 mm and purity of 99.5%; the second one is pure Ni, Co and Si with a particle size about of 71 µm, 66 µm and 61 µm and 99.98%, 99% and 98% of purity, respectively. Mechanical grinding was done for 20 h; the ball-to-powder mass ratio was 1:10.

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The elaboration of the samples was performed with a speed of 300 r/min for 15 min of work, followed by a 15 min of pause in order to avoid an excessive rise in temperature in the interior of the pots.

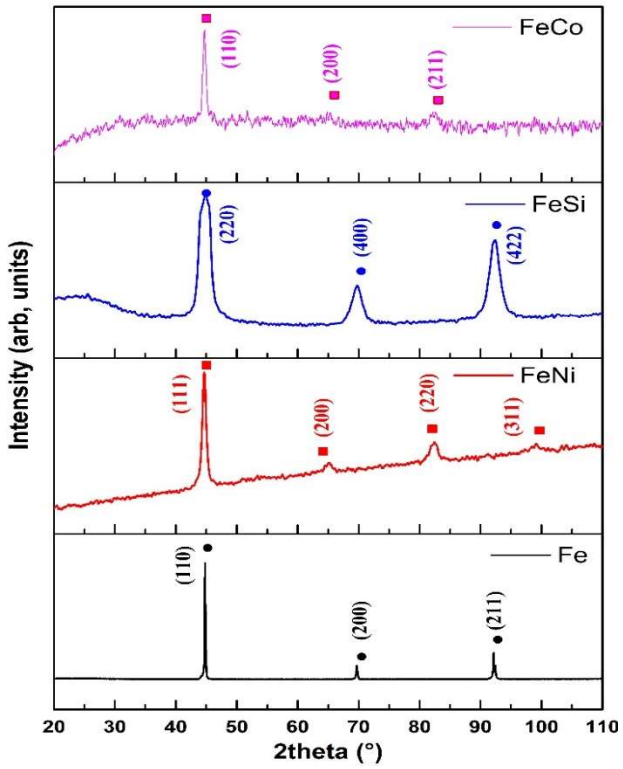

Figure 1. XRD patterns of nanocrystalline milled Fe-20%A alloy for 20 h

The microstructure of the obtained samples was examined using a scanning electron microscope (SEM) - Gemini SEM 300, operated at 30 kV acceleration voltage and equipped with an X-ray dispersive energy spectrometer (EDS) for elemental analysis. The kinetics of the formation during mechanical milling was analyzed using an X-ray diffractometer (XPERT PRO) with Co Ka radiation (wavelength ƛ=1.7889 Å) in the range of 2θ from 20° to 120°. The magnetic behavior of the samples was analyzed using a vibrat**b)**;-sample magnetometer (model EV9) at room temperature with a sweep rate of 10 Oe/s.

III. RESULTS AND DISCUSSION

III.1. Structural analysis

The kinetics of the formation of nanocrystalline during mechanical grinding was monitored using the X-ray diffraction (XRD) technique. The XRD patterns of the milled powders were evaluated to investigate the variation in structural properties that occurs during mechanical grinding. Figure 1 shows the XRD patterns of nanocrystalline milled Fe-A alloy for 20 h of grinding. For the preliminary powder of Fe, the XRD pattern indicates the existence of a body-centered cubic (bcc) iron. After 20h of milling, appearance of FeNi, FeCo and FeSi solid solution with different intensity and Bragg angle [12-15]. The crystallite size (D) was calculated using the Scherrer equation, which relates the broadening (ß) of a diffraction peak to the size of the crystallites. The Scherrer equation is expressed as follows [16-17]:

$$D = K\lambda/\beta\cos\theta \qquad (1)$$

where D is the crystallite size, λ is the wavelength of the X-ray radiation used for diffraction (in this case, λ=1.54059 Å), θ is the diffraction angle (in radians), β is the full width at half maximum (FWHM) of the diffraction peak, and K is a constant that depends on the shape of the crystallites and is typically taken to be around 0.9. Similarly, the lattice strain (ε) was determined using the same diffraction lines and can be calculated from the following equation:

$$\varepsilon = (\beta\cos\theta)/\lambda \qquad (2)$$

where β is the FWHM of the diffraction peak, θ is the diffraction angle (in radians), and λ is the X-ray wavelength.

Table 1 shows the Crystallite size and Lattice strain of nanocrystalline of Fe-20%A during 20 h of milling. The lattice deformation reached 0.7 % in FeSi alloy, while the grain size decreases to 17 nm, 13.5 nm and 10 nm for FeNi, FeCo and FeSi, respectively.

Table 1. Crystallite size and lattice strain of Fe-20%A

Alloy	Crystallite size D (nm)	Lattice strain ε (%)
FeNi	17	0.13
FeCo	13.5	0.36
FeSi	10	0.7

III.2. Morphologies characterization

The morphology of nanocrystalline Fe-A (Ni, Co and Si) during 20h of mechanical grinding is shown in Figure 2. Studies by Davis et al. [18] and Rodriguez et al. [19] have shown that the lamellar structure observed in some particles of nanocrystalline Fe-Ni alloy, as seen in Figure 2(a), is formed by a superposition of Ni and Fe layers. This structure is typical of materials prepared by mechanical milling from ductile (Fe) or brittle (Ni) elements. In a study by Yousefi et al. [8], it was observed that the unmilled FeCo alloy powders consisted of spherical iron particles and skinny, plate-shaped cobalt particles. After mechanical milling, the particles were flattened and changed in shape, likely due to the compression force generated during severe plastic deformation. The resulting particles were smaller than those in the starting mixture, but differences in particle ductility and some larger particles resulting from agglomeration and/or clustering of smaller particles could still be observed.

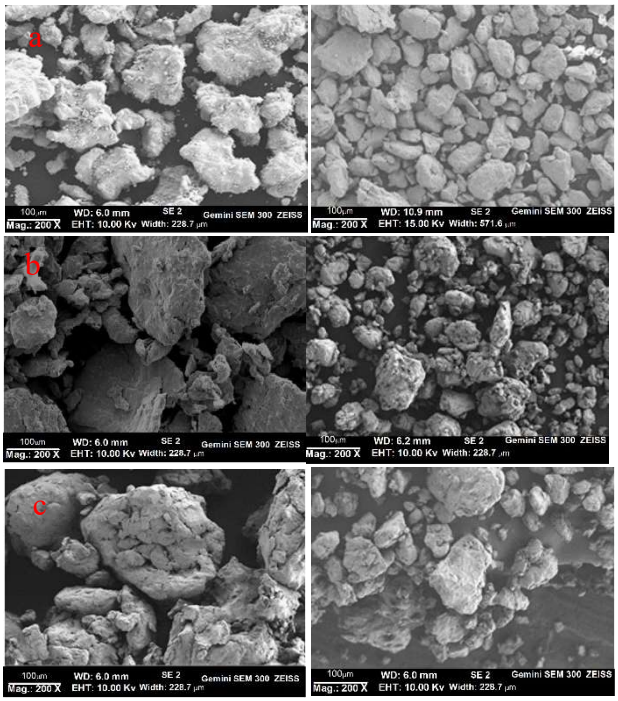

Figure 2. SEM micrographs of nanocrystalline Fe-20%A milled until 20h (a) FeNi, (b) FeCo, and (c) FeSi

The morphology of the FeSi alloy after 20 hours of milling is shown in Figure 2(c). The particles exhibite irregular shapes and varying sizes, which can be attributed to the severe plastic deformation process. The silicon particles were welded and fractured with iron particles to form a nanostructured FeSi alloy. The average particle size of the Fe-Si binary alloy is found to be smaller than that of pure iron and pure silicon. Similar morphologies and grain refinements have been reported in previous studies by Clark et al. [20] and Herting et al. [21].

The energy-dispersive X-ray spectroscopy (EDS) spectrum of the Fe-20%A mixture milled after 20 h is shown in Figure 3. It shows the presence of all elements constituting the alloys. The formation of the Fe-A alloy crystalline structure is given in Figure 3.

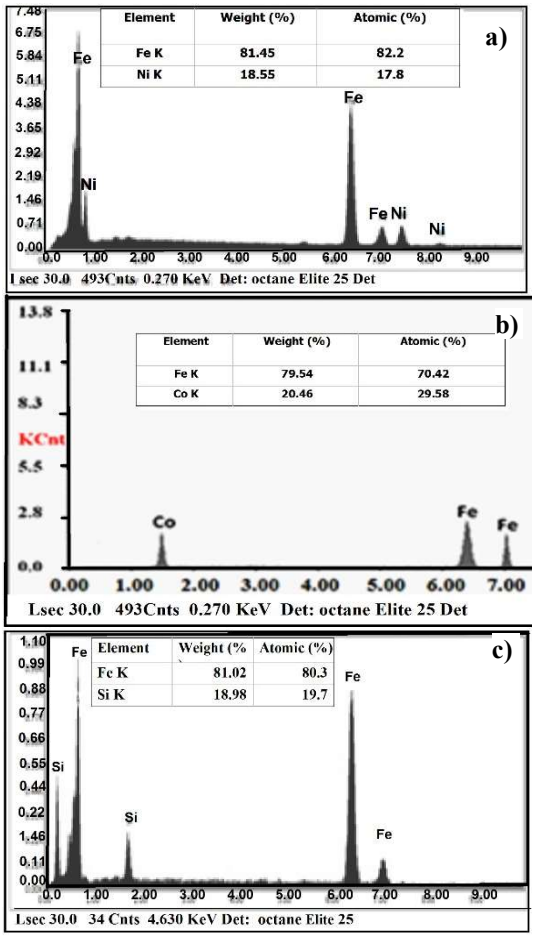

Figure 3. EDS analysis of nanocrystalline Fe-20%A milled until 20h (a) FeNi, (b) FeCo, and (c) FeSi

III.3. Magnetic measurement

Figure 4 shows the hysteresis loops (M–H) for the nanocrystalline Fe-A alloys as a function of the mechanical grinding.

Figure 4 and Table 2 illustrate the magnetic properties of mixtures of FeCo, FeNi, and FeSi alloys. These mixtures exhibit a similar magnetic behavior, with a lower coercivity and higher saturation magnetization. Notably, FeCo shows a significant increase in saturation magnetization, indicating the formation of a solid solution of Fe and Co compared to FeNi and FeSi. Additionally, the maximum coercivity is reached in the FeCo alloy, which is attributed to a decrease in crystallite size and the influence of Co in close proximity to Fe.

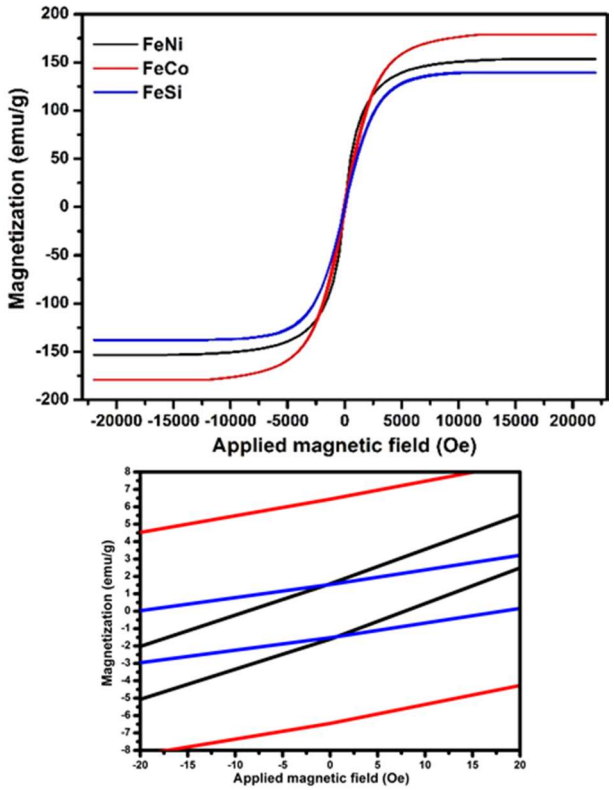

Figure 4. Hysteresis loops of nanocrystalline Fe-20%A (A: Ni, Co and Si) milled until 20h

Table 1. Magnetic parameters of Fe-20%A nanostructured alloys

	FeCo	FeSi	FeNi
Hc (Oe)	96.08	29.38	16.01
Ms (emu/g)	178.24	139.19	153.40
Mr (emu/g)	6.42	1.53	1.59

IV. CONCLUSION

The effective production of a nanocrystalline Fe-20%A (Co, Ni, and Si) alloy through mechanical grinding was confirmed. The magnetic properties of the nanocrystalline Fe-20%A alloy were found to be influenced by various structural and chemical factors, including the crystallite size and the formation of FeCo, FeNi, and FeSi solid solutions. Additionally, the saturation magnetization was found to be highly dependent on the variation in the chemical composition and the introduction of defects during the mechanical alloying process. These findings underscore the importance of carefully controlling the structural and chemical factors during the production of nanocrystalline Fe-20%A alloys to achieve optimal magnetic properties. Future research efforts can focus on optimizing these factors to further enhance the magnetic properties of these alloys for a range of applications.

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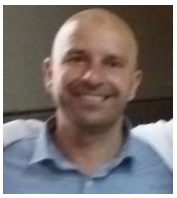

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