

## Silicothermic Production and Characterization of FeSiNi/SiO<sub>2</sub> Magneticnanocomposite Via Mechanical Alloying

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### ABSTRACT

In recent years, a lot of research has been done in the field of producing soft magnetic nano composites by means of mechanical alloying. These materials indicate more electrical resistance and permeability in comparison with soft magnetic composites (SMCs). In this research, reduction of silicothermal mixed powder (Nickel-oxide, Silicon and Iron) was carried out by mechanical alloying to form soft nano composites of FeSiNi/SiO<sub>2</sub>. Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) were used to detect physical characterization and finding the effect of milling time on microstructural properties. It was found that initiation of forming the homogenous alloy occurred in 4 hours of milling, and increasing the time of milling to 40 hours caused decreasing the grain size to 13 nm and formation of a homogenous alloy. Magnetic properties of the milled powder were studied by alternating gradient force magnetometer (AGFM) and it was observed that increasing the milling time will cause improvement in soft magnetic properties (decreasing magnetic coercivity and increasing magnetic saturation).

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### 1. Introduction

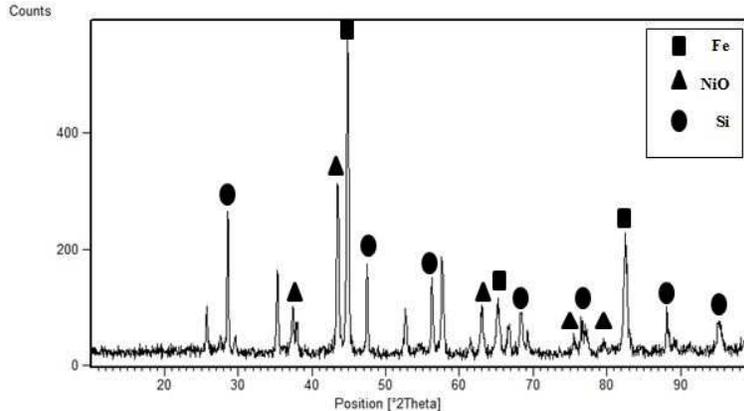
Mechanical alloying is a new method of producing materials with nano structure. During this process powder materials undergo an intensive plastic deformation and develop cold welding and fracture. Consecutive repetition of this process leads to produce nano-structured productions. Generally, gaining nano size particles causes different physical and mechanical properties [1]. Another advantage of mechanical alloying is production of different alloy phases such as solid solution, amorphous, crystal, and semi crystal phases, and possibility of acceleration of chemical reactions due to mechanical stimulation in the

mixed powder. One of chemical reactions that occurs during this process is reduction of metallic oxides in the presence of metallic or nonmetallic reduction agent [2]. Metals are used in order to reduce reaction, and if the reduction of metallic oxide happens by carbon, the reduction is called carbon thermic reduction [3]. Metal thermic of a reaction is highly pyrogenic and regarding to the reduction agent, metal thermic reactions are divided into alumina and silicon thermic reactions. In recent years, soft-magnetic nano composites have had a lot of applications in different industries such as electrical micro-motors, low power motors for automation, robotic and shuttle due to

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**Fig. 1.** X-ray diffraction pattern of the FeSiNi/SiO<sub>2</sub> nano composite powders for the initial milling time

suitable magnetic properties and high durability. These composites are made of pure iron-powder and high density and high magnetic-saturation. Low electric resistance of iron causes too much eddy current loss. Hence, for increasing electrical resistance and decreasing eddy current loss, alloys such as iron-nickel, iron-silicon, and iron-nickel-aluminum are used instead of pure iron.

Nano-crystal materials with iron-base has shown soft magnetic behavior and at the same time indicate properties of amorphous iron-base alloys such as high magnetic and advantages of amorphous cobalt-base alloys such as low-magnetic coercivity [4]. Also, using these nano crystal powder alloys of iron-base in soft-magnetic composites cause increasing in electrical resistance and magnetic diffusivity in high frequencies.

The aim of this research is to produce nano composites of FeSiNi/SiO<sub>2</sub> in the first step and then physical and magnetic characterization.

## 2. Experimental

In this research, the used materials were iron powder, nickel-oxide, Silicon with 10 Micron grain size and purity of 98%. These mixed powders had 85 wt% iron powder, 12.5 wt% Silicon powder and 5 wt% nickel-oxide powder milled by a planetary mill (Model FP4) with weight proportion of ball to powder (20 to 1) with 5 steel balls of 20 mm in a box made of Chromium steel and with speed of 600 rpm in different times at room temperature and under argon atmosphere with 99.99 % purity. Milling

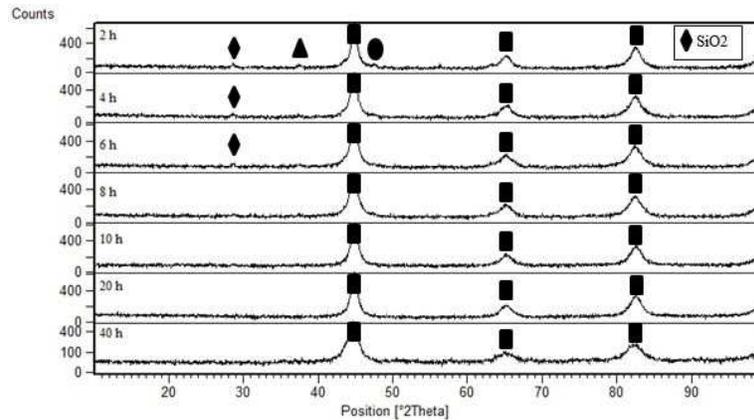
was carried out for 0, 2, 4, 6, 8, 10, 20, and 40 hours. Then the milling powders were characterized by X-Ray diffractometer (Philips, X-Pert) with Cu K<sub>α</sub> radiation and 1.5406 Å wavelength. Grain size and internal strain were calculated by Williamson-Hall method and lattice parameter was calculated by Bragg's law. Magnetic properties of the milled powder at 6, 10 and 40 hours were measured by alternating gradient force magnetometer (AGFM) at room temperature with field of 9000 Oersted (Oe), and microstructure changes of the powders was probed by scanning electron microscopy (SEM), model VEGA TESCAN at 2, 10, and 20 hours.

## 3. Results and Discussion

Fig. 1 and 2 show XRD patterns of the alloyed powder sample milled for 0 to 40 hours. It was observed that after 4 hours of grinding, peaks of nickel-oxide and Si diminished and a peak of SiO<sub>2</sub> appeared due to Silicothermal reduction of nickel-oxide and dissolution of nickel and Si atoms in iron crystal structure.

By increasing the time of milling to 8 hours, removal of SiO<sub>2</sub> peak was observed because of intensive grinding and SiO<sub>2</sub> became amorphous. Continuing grinding to 40 hours caused formation of homogenous solid-solution which can be due to formation of inner-strain or decreasing of the grain-size during mechanical milling.

Table 1 indicates changes in lattice-parameter and grain-size vs. increase in the milling time. Comparing lattice-parameter with increasing



**Fig. 2.** X-ray diffraction patterns of the FeSiNi/SiO<sub>2</sub> nano composite powders for different milling times

**Table 1.** Changes of lattice parameter and grain size according to the milling time

Time (h)	Lattice parameter (nm)	Grain size (nm)
0	0.2867	-
2	0.2862	38
4	0.2858	33
6	0.2863	29
8	0.2864	17
10	0.2863	15
20	0.2858	14
40	0.2856	13

**Table 2.** Changes of coercivity ( $H_C$ ) and saturation magnetization ( $M_S$ ) according to the milling time

Time (h)	$H_C$ (Oe)	$M_S$ (emu/g)
6	118	132
10	84	155
40	58	166

grinding-time to 4 hours it was observed that lattice-parameter decreased from 0.2867 to 0.2858 because of diffusion of Si-atoms into iron crystal structure. Since Si-atom size is significantly smaller than iron-atom, so this decline in lattice-parameter is acceptable [5].

By increasing the time of milling to 8 hours, increasing of lattice-parameter was observed to be  $6 \times 10^{-4}$  nm. This change in lattice-parameter can be due to small difference between nickel and iron atom-size. Increasing the time of grinding to 40 hours causes more diffusivity of Si and nickel atoms into iron structure and leads to decreasing in lattice-parameter; however, this lattice-parameter change is too small because of similar atom-size of nickel and iron, but this small change in the lattice-parameter can be the cause of diffusivity of Si atoms into iron structure.

Table 1 shows that by increasing the time of milling, grain-size decreases because of hardness of intensive plastic deformation during mechanical alloying [6].

Table 2 and Fig. 3, 4, and 5 show that by increasing the milling time magnetic-saturation increased and magnetic-coercivity decreased. Increasing in magnetic-saturation is because of finer grain-size, so that each grain acts as a magnetic-field and in the presence of nickel and formation of solid-solution non-isotropic magnetic decreases and magnetic-saturation increases. Magnetic-coercivity is not an inherent property of materials and depends on conditions; this property has a maximum value in the beginning of milling, because grain-size exceeds width of field partition and grain-boundary acts as obstacle for moving of partitions; when width of field partition

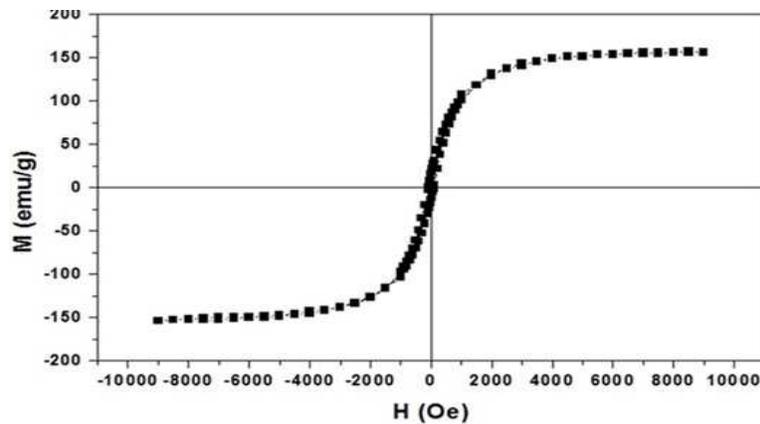


Fig. 3. Residual loop of the FeSiNi/SiO<sub>2</sub> nano composite powders after 6 hours of milling

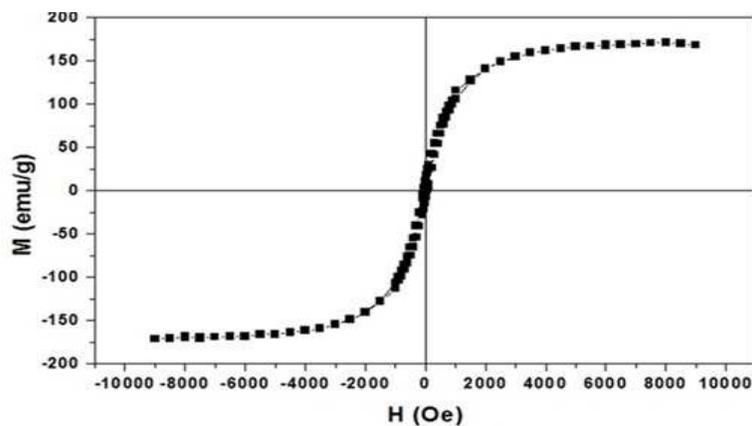


Fig. 4. Residual loop of the FeSiNi/SiO<sub>2</sub> nano composite powders after 10 hours of milling

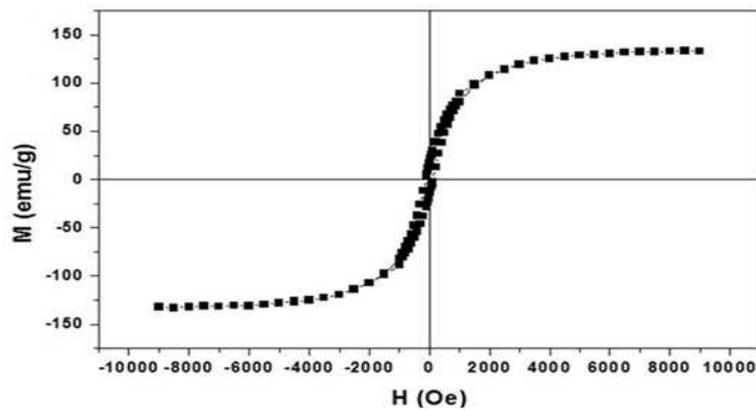
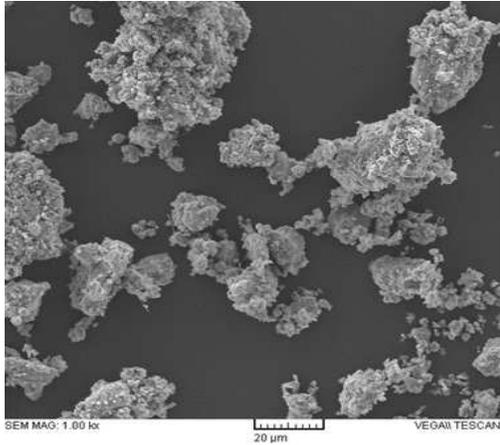


Fig. 5. Residual loop of the FeSiNi/SiO<sub>2</sub> nano composite powders after 40 hours of milling

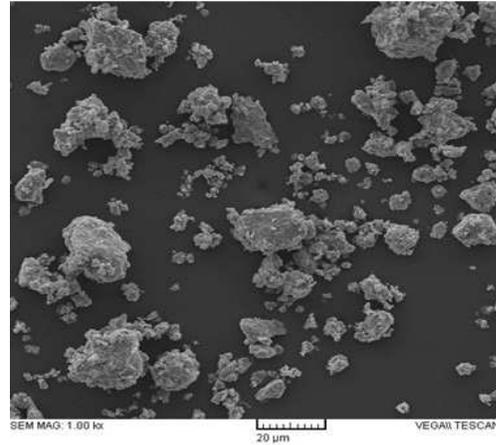
exceeds grain-size magnetic-coercivity decreased [7].

Figs 6 to 8 show that particles in the beginning of milling are agglomerated with the layer structure. Generally, pressure of the milling-

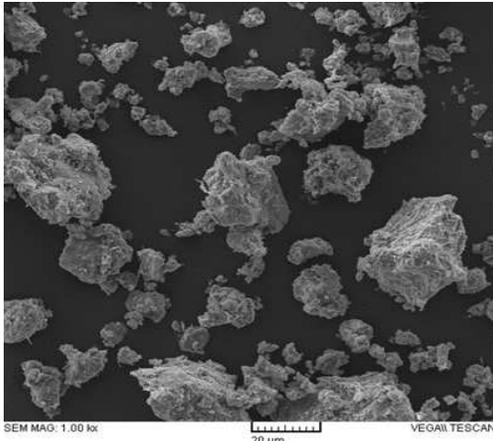
balls causes formation of micro-structure layers. In this condition, cold-welding mechanism is dominant and it doesn't consist of work-hardening during alloying and there is a tendency to welding and formation of layer



**Fig. 6.** SEM image of the sample that was milled for 2 hours



**Fig. 7.** SEM image of the sample that was milled for 10 hours



**Fig. 8.** SEM image of the sample that was milled for 20 hours

structure. By increasing the milling time to 20 hours, because of work-hardening and high brittleness the agglomerated particles start to fracture. Under this situation, it can be concluded that there is a balance between cold-welding mechanism and fracture [8]. Therefore, smaller particles with more homogenous distribution and more spherical shapes will be observed.

#### 4. Conclusion

1. In this research, soft magnetic nano composite powder of FeSiNi/SiO<sub>2</sub> was produced by means of mechanical alloying and Silicothermal.
2. By increasing the grinding time, the grain size and lattice parameter decreased, so that

after 40 hours of milling, particles with grain size of 13 nm were produced.

3. SEM images showed that increasing the time of milling causes morphologic changes in the grinded powder from layer to spherical shape with more homogenous distribution and smaller size of particles due to formation of work-hardening which is the result of mechanical alloying.

4. Increasing the time of milling causes decrease in magnetic coercivity, increase in magnetic-saturation and achievement of soft magnetic conditions.

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