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# Magnetic, Structural and Morphological Properties of Mechanically Alloyed FeSi and $FeSiO_2$ Nanocomposites

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**Abstract**: Nanostructured FeSi and $FeSiO_2$ nanocomposites were synthesized via mechanical alloying to investigate the effects of milling time (0–30 h) on their structural, morphological, and magnetic properties. Comprehensive characterization was performed using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), and vibrating sample magnetometry (VSM). XRD analysis revealed a transition to a disordered FeSi solid solution with a body-centered cubic structure after prolonged milling, alongside a crystallite size reduction and increasing lattice strain. For FeSi, the lattice parameter increased from 0.2861 nm (unmilled) to 0.452 nm after 30 h, with an average crystallite size of 22 nm. In contrast, the $FeSiO_2$ nanocomposite exhibited crystallite sizes ranging from 79–28 nm, with $Fe/SiO_2$ showing a lattice parameter decrease from 0.286 nm to 0.283 nm. Morphological evolution was evident through SEM. Magnetic properties improved with extended milling, with FeSi attaining its highest coercivity, saturation magnetization, and remanent magnetization at 30 h. Similarly, $FeSiO_2$ exhibited notable coercivity and remanence values, with $Fe/SiO_2$ achieving the highest saturation magnetization of 177.08 emu/g. These results emphasize the potential of FeSi and $FeSiO_2$ nanocomposites for high-frequency magnetic applications.

**Keywords:** Nanostructured FeSi and $FeSiO_2$ alloys, Magnetic behavior, Structural properties, SEM observations

## Introduction

Nanostructured materials and nanocomposites have garnered significant attention due to their exceptional magnetic, thermal, optical, dielectric, and catalytic properties. These properties, often superior to their bulk counterparts, are strongly influenced by factors such as particle size, shape, inter-particle distance, and distribution homogeneity. Reducing particle size to the single-domain limit enhances magnetic characteristics such as superparamagnetism, higher coercivity, and optimized magnetic coupling, making nanostructured materials highly desirable for advanced technological applications (Abbassi et al., 2022; Baig & Kammakakam, 2021; Paras et al., 2022; Kim et al. 2013; Yao et al. 2010). Among various synthesis techniques, mechanical alloying (MA) stands out as an effective approach for producing nanostructured materials and nanocomposites. MA offers advantages such as uniform element dispersion at the nanoscale, production of metastable phases, and improved properties, including magnetic and mechanical characteristics. The process involves repeated welding, fracturing, and rewelding of powder particles in a high-energy ball mill. Key parameters like milling


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time, ball-to-powder ratio, and milling speed critically influence the structural and magnetic properties of the final material (Ouadah & Younes, 2023; Younes et al., 2024; Younes et al., 2023). In this context, FeSi alloys and $FeSiO_2$ nanocomposites have emerged as promising candidates for magnetic applications, including sensors, transformers, and high-frequency devices, due to their high saturation magnetization, low coercivity, and superior soft magnetic behavior. Nanostructuring FeSi further enhances these properties, while incorporating Fe nanoparticles into a $SiO_2$ matrix introduces additional benefits such as increased electrical resistivity and reduced eddy current losses (Leveneur et al., 2011; Krings et al., 2016).

This study focuses on the comparative synthesis and characterization of FeSi and $FeSiO_2$ nanocomposites using MA, with milling times ranging from 0 to 30 h. Advanced characterization techniques, including scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), and vibrating sample magnetometry (VSM), are employed to investigate the evolution of structural, morphological, and magnetic properties. By elucidating the relationships between milling parameters, crystallite structure, and magnetic behavior, this research aims to optimize the properties of FeSi and $FeSiO_2$ nanocomposites, contributing to the development of efficient materials for magnetic and electronic applications.

## Method

The nanostructured FeSi and $FeSiO_2$ powders were synthesized using a high-energy planetary ball mill (PM 400) equipped with tungsten carbide pots and balls. High-purity Fe and Si powders (99.5%) with an average particle size of approximately 70 µm were used as starting materials, procured from Sigma-Aldrich (USA). Each sample consisted of a 20 g powder mixture placed in a 250 ml tungsten carbide jar along with tungsten carbide balls (20 mm diameter). The milling process was conducted under an inert atmosphere to prevent oxidation. A ball-to-powder weight ratio of 20:1 was maintained, with the milling speed set to 400 rpm. To avoid excessive heating during the process, a sequence of 15 minutes of milling followed by a 15-minute resting period was applied. The duration of the milling process was varied systematically (0 and 30 hours) to investigate the influence of milling time on the structural and magnetic properties of the powders.

The structural and morphological evolution during milling was analyzed using a Zeiss Gemini SEM equipped with an energy-dispersive X-ray spectroscopy (EDS) system for elemental analysis. Phase identification, crystallite size, and lattice parameters were determined using a Panalytical X'Pert Pro X-ray diffractometer with Cu Kα radiation ($\lambda$ = 1.5406 Å) over a 2θ range of 10°–90°. The magnetic properties of the samples, including coercivity, saturation magnetization, and remanence, were characterized using a MicroSense EV9 vibrating sample magnetometer (VSM) with a maximum applied field of 15 kOe. This integrated approach enabled a comprehensive evaluation of the effects of milling time on the structural, morphological, and magnetic properties of the FeSi and $FeSiO_2$ nanocomposites.

## Results and Discussion

### Structural Study

Figure 1 and Table 1 provide a detailed representation of the structural evolution of FeSi and $FeSiO_2$ nanocomposites during mechanical alloying, as determined through X-ray diffraction (XRD) analysis. For FeSi, the milling process induced a significant transformation, with the initial Fe and Si phases disappearing and forming a disordered body-centered cubic (bcc) Fe(Si) solid solution (Rodriguez et al., 2007). This was accompanied by a progressive reduction in crystallite size, from 33 nm in the unmilled state to 22 nm after 30 hours of milling.

Additionally, the lattice strain increased markedly from 0.07% to 0.23%, reflecting the high mechanical deformation and energy imparted during milling, which led to increased lattice distortions. For $FeSiO_2$ nanocomposites, the XRD patterns showed a similar trend of structural refinement, with the crystallite size decreasing significantly from 79 nm to 28 nm over the same milling duration. The reduction in lattice parameters, from 0.286 nm to 0.283 nm, is indicative of the amorphization of the $SiO_2$ matrix and the diffusion of Fe into the matrix. The disappearance of distinct $SiO_2$ peaks and the broadening of Fe peaks suggest the formation of an Fe-$SiO_2$ composite with enhanced homogeneity and reduced crystallinity (Suryanarayana et al., 2022, and Alonso-Sanudo et al.,2000).

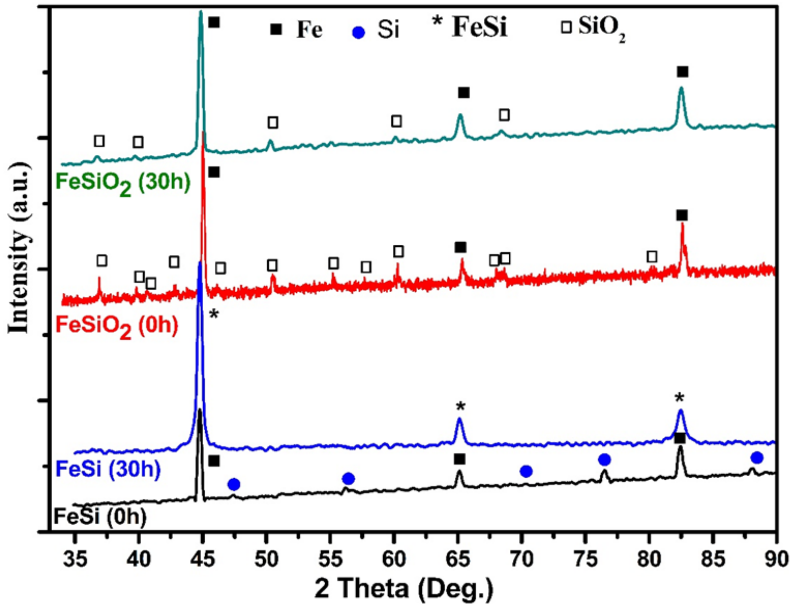


Figure 1. Structural evolution of FeSi and $FeSiO_2$ nanocomposites during mechanical alloying

These results clearly demonstrate that prolonged milling not only refines the crystallite size but also increases lattice strain and structural disorder, key indicators of effective mechanical alloying. The interplay between mechanical energy, grain refinement, and lattice distortion highlights the efficacy of this process in producing nanocomposites with tailored structural properties for advanced applications.

Table 1. Structural parameters of nanostructured Fe/SiO2mixture milled with defferent times.

| Milling time (h) | Crystallite size (nm) | Lattice strain (%) | Lattice parameter of Fe (nm) |
|---|---|---|---|
| FeSiO2 (0h) | 79 | 0,01 | 0.286 |
| FeSi (0h) | 33 | 0.07 | 0.452 |
| FeSiO2 (30h) | 28 | 0,02 | 0.283 |
| FeSi (30h) | 22 | 0.23 | 0.285 |

**Morphology Analysis**

Figure 2 provides a detailed Scanning Electron Microscopy (SEM) analysis, showcasing the morphological evolution of FeSi and $FeSiO_2$ nanocomposites during mechanical alloying. In the case of FeSi, the initial coarse particles (~70 µm) underwent substantial size reduction, reaching ~10 µm after 30 hours of milling. This transformation is attributed to the cyclic processes of cold welding, fracturing, and rewelding inherent to high-energy ball milling. Early stages of milling produced irregularly shaped, agglomerated particles, which progressively became finer and more homogeneous with extended milling time. The reduction in particle size indicates higher energy input and enhanced mechanical deformation, which together facilitate the formation of a nanostructured material (Gokçe & Ovecoglu, 2023; Banerjee et al., 2020).

For $FeSiO_2$, a similar trend was observed, where milling influenced the morphology by enhancing particle dispersion, reducing agglomeration, and contributing to the homogenization of the nanocomposite. The mechanical forces applied during milling not only fragmented the particles but also facilitated the uniform mixing of Fe and $SiO_2$ phases, as well as the integration of dopants (Gaona et al., 2021, and Dad et al., 2017). By 30 hours of linmilg, $FeSiO_2$ exhibited a well-dispersed, fine-grained morphology, indicative of efficient alloying and composite formation. Energy-dispersive X-ray spectroscopy (EDS) analyses corroborated these observations by confirming the uniform elemental distribution in both nanocomposites.

The consistent alloy composition and absence of impurities or contamination highlight the effectiveness of the milling process in achieving homogeneity. Additionally, EDS mapping revealed that the diffusion of elements, particularly silicon into the iron matrix for FeSi and dopants into the $SiO_2$ matrix for $FeSiO_2$, was complete by 30 hours, ensuring the successful formation of the desired nanostructured materials. These results underline the critical role of mechanical alloying in optimizing particle size, morphology, and compositional uniformity for advanced material applications.

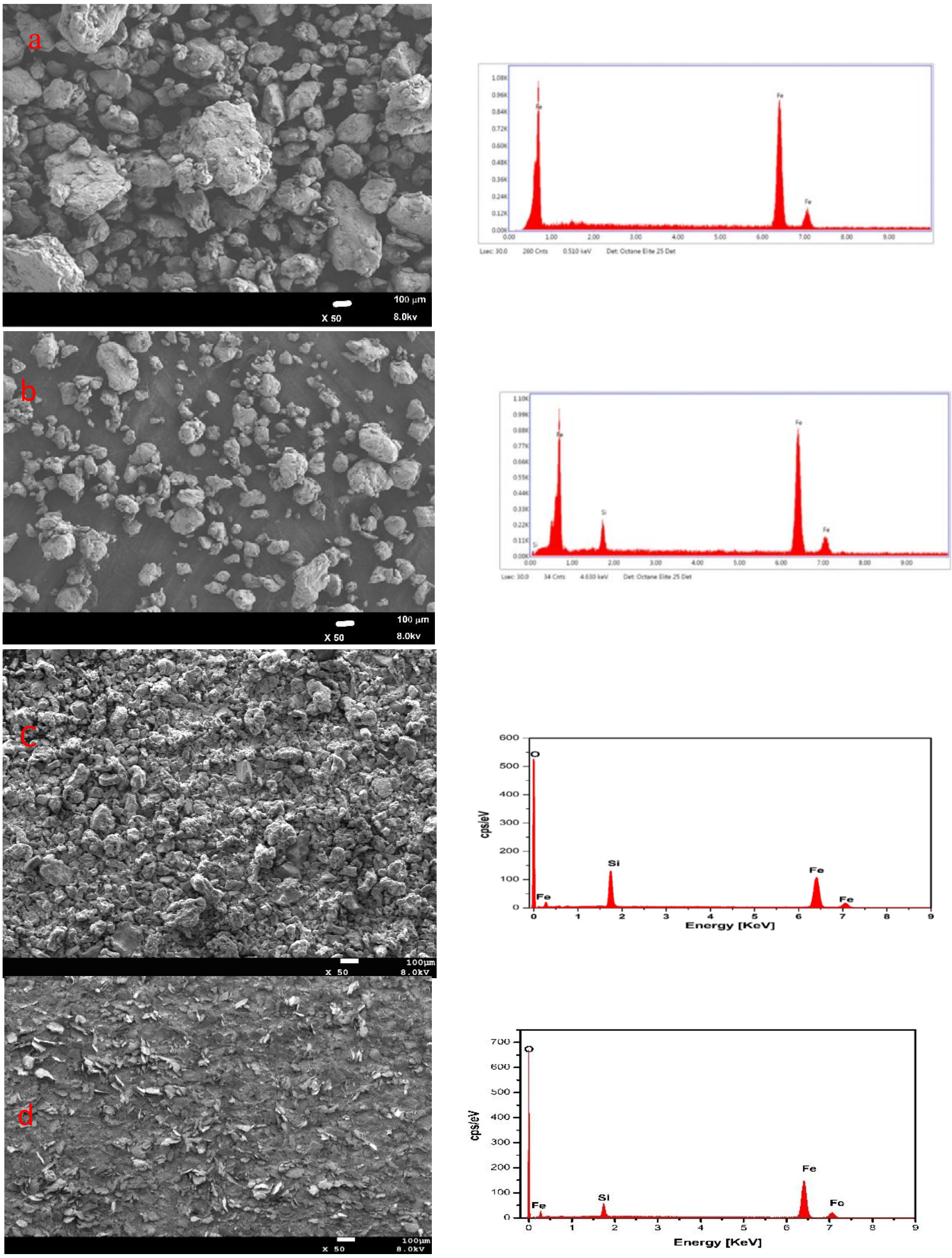


Figure 2. Morphological evolution of FeSi and $FeSiO_2$ nanocomposites during mechanical alloying (a)FeSi(0h), (b) FeSi(30h), (c) $FeSiO_2$(0h) and $FeSiO_2$(30h)

**Magnetic Investigation**

Figure 3 and Table 2 detail the magnetic measurements of FeSi and $FeSiO_2$ nanocomposites, highlighting significant enhancements in their magnetic properties with increased milling time. For FeSi, the mechanical alloying process resulted in a remarkable improvement in magnetic performance. At 30 hours of milling, coercivity (Hc) increased to 68.70 Oe, saturation magnetization (Ms) reached 159.89 emu/g, and remanent magnetization (Mr) rose to 8.71 emu/g.

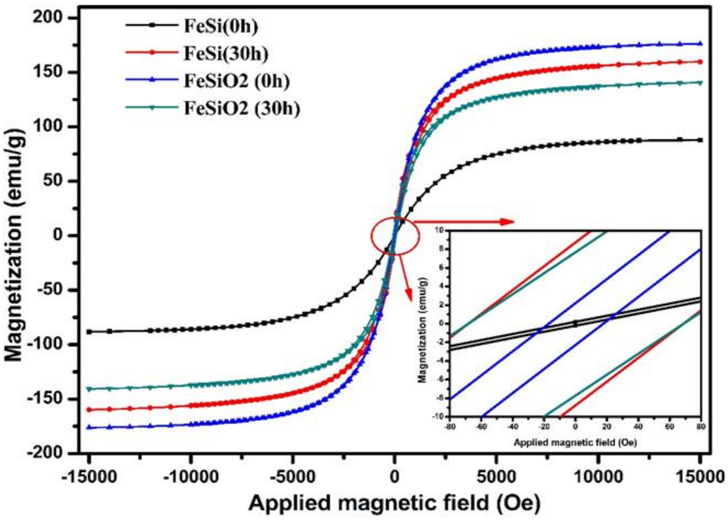


Figure 3. Magnetic properties of FeSi and $FeSiO_2$ nanocomposites improved with milling time.

These improvements are primarily attributed to the reduction in crystallite size, which increased the effective surface area of the magnetic domains, and the rise in lattice strain, which altered the magnetic anisotropy and domain wall motion. The fine crystallite structure and increased lattice defects induced by milling likely contributed to higher magnetic energy storage and domain wall pinning, enhancing the material's overall magnetic performance (Guo et al., 2020, and Overman et al., 2018). For $FeSiO_2$, extended milling resulted in distinct magnetic behaviors, reflecting the influence of structural refinement and phase evolution. After 30 hours, the saturation magnetization (Ms) peaked at 140.9 emu/g, while coercivity (Hc) and remanence (Mr) were measured at 69 Oe and 7.66 emu/g, respectively. The dopants played a pivotal role in enhancing magnetic coupling within the composite, as they improved the dispersion of Fe particles within the $SiO_2$ matrix and reduced magnetic interactions between neighboring particles. The presence of non-magnetic $SiO_2$ as a spacer reduced eddy current losses and further contributed to the soft magnetic behavior of the composite, making it suitable for high-frequency applications (Liu et al., 2019, and (Onderko et al., 2022).

Table 2. Magnetic parameters of nanostructured Fe/SiO2 mixture milled with defferent times.

| Milling time (h) | Hc (Oe) | Mr (emu/g) | Ms (emu/g) | Mr/Ms |
|---|---|---|---|---|
| FeSi (0h) | 7.72 | 0.19 | 87.53 | 0.002 |
| FeSi (30h) | 68.70 | 8.71 | 159.89 | 0.054 |
| FeSiO2 (0h) | 17.96 | 2.21 | 177.08 | 0.012 |
| FeSiO2 (30h) | 69 | 7.66 | 140.9 | 0.054 |

The results clearly demonstrate that mechanical alloying and dopant incorporation are powerful tools for tailoring the magnetic properties of nanocomposites. The interplay between reduced crystallite size, increased lattice strain, and the introduction of dopants significantly influenced the coercivity, remanence, and saturation magnetization of these materials. These findings highlight the potential of FeSi and $FeSiO_2$ nanocomposites for advanced applications, such as magnetic sensors, transformers, and other devices requiring precise control of magnetic properties.

## Conclusion

This comparative study underscores the transformative effects of mechanical alloying in tailoring the structural, morphological, and magnetic properties of FeSi and $FeSiO_2$ nanocomposites. For FeSi, prolonged milling facilitated the formation of a disordered body-centered cubic (bcc) Fe(Si) solid solution, significantly enhancing its magnetic behavior, including higher coercivity, saturation magnetization, and remanence. This improvement is attributed to the reduction in crystallite size, increased lattice strain, and the uniform dispersion of silicon within the iron matrix. In the case of $FeSiO_2$, mechanical alloying promoted superior magnetic performance, characterized by higher saturation magnetization, improved particle dispersion, and diminished agglomeration. The amorphization of the $SiO_2$ matrix and the interaction between Fe and $SiO_2$ further contributed to enhanced structural and magnetic properties. These dopants played a pivotal role in promoting magnetic coupling, refining the particle size, and ensuring homogeneity throughout the nanocomposite. The findings of this study emphasize the critical influence of milling parameters, including time and environmental conditions, as well as material composition on the final properties of nanocomposites. By optimizing these factors, FeSi and $FeSiO_2$ nanocomposites demonstrate significant potential for advanced magnetic applications, including high-frequency devices, magnetic sensors, and transformers. This research provides valuable insights for developing high-performance magnetic nanocomposites and paves the way for future innovations in the field of materials science.

## Recommendations

Future research should optimize milling beyond 30 h to further refine grain structure and magnetic response. Exploring dopants or oxide phases may enhance coercivity and thermal stability. Advanced tools like TEM or Mössbauer spectroscopy could provide deeper microstructural insights. Post-milling annealing effects on magnetic properties should be systematically studied. Application-specific testing and scalability assessments will aid industrial adoption of FeSi and $FeSiO_2$ nanocomposites.
Scientific Ethics Declaration

## Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

## Conflict of Interest

* The authors declare that they have no conflicts of interest

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