The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM)
ISSN: 2602-3199

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2025

Volume 38, Pages 210-216

IConTES 2025: International Conference on Technology, Engineering and Science

Magnetic and Structural Behavior of Nanocrystalline Iron-Silicon and Iron-Tin Produced via Mechanical Milling

Abderahim Abada
University of Saad Dahleb Blida

Abderrahmane Younes
Research Center in Industrial Technologies (CRTI)

Rachid Amraoui
Research Center in Industrial Technologies (CRTI)

Djilali Allou
Research Center in Industrial Technologies (CRTI)

Abstract: This study presents a comparative investigation of the structural, morphological, and magnetic properties of nanostructured FeSi and FeSn alloys synthesized via the ball milling process. The alloys were analyzed at various milling times from 0 to 20 hours using X-ray diffraction (XRD), scanning electron microscopy (SEM), and vibrating sample magnetometer (VSM). XRD analysis confirmed the formation of FeSi and FeSn phases after 20 hours of milling, with characteristic peaks indicating structural evolution. SEM micrographs revealed significant morphological changes and particle refinement with increased milling time. Magnetic measurements highlighted contrasting behaviors between the two alloys. For FeSn, increased milling time enhanced coercivity, remanence magnetization, and squareness, accompanied by a reduction in saturation magnetization. Conversely, FeSi exhibited superparamagnetic behavior with notable saturation magnetization and significant coercivity (Hc). These findings emphasize the distinct magnetic and structural properties of FeSi and FeSn nanocrystalline alloys and their potential for tailored applications in advanced magnetic systems.

Keywords: Nanocrystalline FeSi and FeSn, Structural and morphological properties, Magnetic behavior

Introduction

Nanostructured materials, especially those based on iron alloys, have garnered significant attention due to their unique properties that emerge at the nanoscale, such as enhanced magnetic, mechanical, and electronic characteristics. FeSi and FeSn alloys are particularly important for their potential applications in electronics, energy storage, and sensors due to their tunable magnetic properties (Suryanarayana et al., 2022, Wang et al., 2014, Younes et al., 2024, Abada et al., 2020, and Abada et al., 2024). Mechanical milling, a solid-state processing method, has been widely used to fabricate nanocrystalline materials because it induces changes in the alloy's phase, particle size, and crystallinity. The milling time and conditions significantly affect the microstructure and, consequently, the magnetic behavior of these materials. In this study, we compare the structural, morphological, and magnetic properties of FeSi and FeSn alloys synthesized through the ball milling process, exploring the effects of different milling times (Ouadah et al., 2023, Jaldurgam et al., 2021, Yelsukov et al., 2000, and Chen et al., 2007). The aim of this research is to investigate the magnetic and structural properties of nanocrystalline FeSi and FeSn alloys synthesized through the mechanical milling process. Specifically, the study seeks to examine how varying milling times (0–20 hours) affect the structural evolution, including changes in grain size, phase formation, and crystallinity, as well as the morphological characteristics such as particle refinement. Additionally, the research aims to analyze the magnetic behavior of these alloys, focusing on key properties such as saturation magnetization, coercivity, and remanent magnetization, using vibrating sample magnetometer (VSM). By correlating the structural and magnetic characteristics, this research aims to understand the impact of mechanical milling on the alloys' performance and their potential applications in advanced magnetic systems, such as sensors and energy devices.

Method

FeSi and FeSn alloys were synthesized using high-purity iron (Fe), silicon (Si), and tin (Sn) powders, which were mixed in the desired stoichiometric proportions. The powders were subjected to a high-energy ball milling process using a planetary ball mill. Milling times of 0, and 20 hours were chosen to study the evolution of the material properties with increasing milling duration. During milling, the powders were subjected to a speed of 300 rpm, and a ball-to-powder weight ratio of 10:1 was maintained. The synthesized powders were characterized using; X-ray Diffraction (XRD): To determine the crystalline phases and assess the structural evolution of the alloys as a function of milling time. XRD patterns were collected using a Cu Kα radiation source (λ = 1.5406 Å) in the 2θ range from 20° to 80°. Scanning Electron Microscopy (SEM): To investigate the morphological changes and particle size reduction during milling. SEM imaging was performed using a field-emission scanning electron microscope. Vibrating Sample Magnetometer (VSM): To evaluate the magnetic properties of the alloys, including saturation magnetization, coercivity, and remanence. Measurements were carried out at room temperature and 300 K.

Results and Discussion

SEM Examination

Figures 1a and 1b display SEM micrographs of the FeSi and FeSn alloys before and after 20 hours of mechanical milling, providing detailed insight into the morphological evolution of the particles. Before Milling (0 hours): The initial powders of both FeSi and FeSn alloys exhibit a relatively coarse and irregular structure, characterized by large, angular particles that are typical of the raw materials used. These particles have relatively broad size distributions, with the majority of them being in the micron range. The large particle sizes and irregular shapes suggest that the powders are not yet refined and are in their original form, with limited surface area available for interactions. The SEM images show that the particles are not homogeneously distributed, and there is an absence of finer particles, which is indicative of the need for milling to induce structural changes in the alloys. After 20 Hours Milling: After 20 hours of ball milling, significant refinement in particle size is observed in both FeSi and FeSn alloys. The particles are reduced to a finer, more uniform distribution, with the majority of the particles now being in the nanometer range (Jensen et al., 2017; Younes et al. 2019; Yin et al 2017).

The increased surface area of the nanostructured particles after milling is expected to significantly enhance the magnetic properties of these alloys. This is because the high surface area allows for more efficient interactions between the individual particles and the external magnetic field. The smaller particle size and increased surface energy also enhance the material's potential for exhibiting superparamagnetism and may improve the performance of the alloys in applications such as magnetic sensors, data storage, and electromagnetic shielding. The fine, uniform distribution of the nanoparticles, along with the enhanced surface area, plays a crucial role in determining the alloy’s magnetic anisotropy and overall magnetic performance.

The SEM images show a more homogeneous particle size distribution, with the particles appearing more spherical or faceted, a result of the high-energy collisions within the milling process. This refinement of particle size is a direct consequence of the mechanical milling, which induces repeated fracturing and welding of the powder particles, progressively reducing their size. The particle surfaces also appear smoother, further suggesting that the milling process has facilitated the refinement of the particles into nanoscale dimensions. Furthermore, the SEM images reveal an increased degree of agglomeration of the particles after milling. Agglomeration is a common phenomenon in ball milling, where fine nanoparticles tend to cluster together due to the high surface energy created during the milling process. The agglomerates formed are typically the result of van der Waals forces and other attractive forces between the particles. This clustering can increase the particle size slightly, but it also contributes to the creation of complex microstructures.

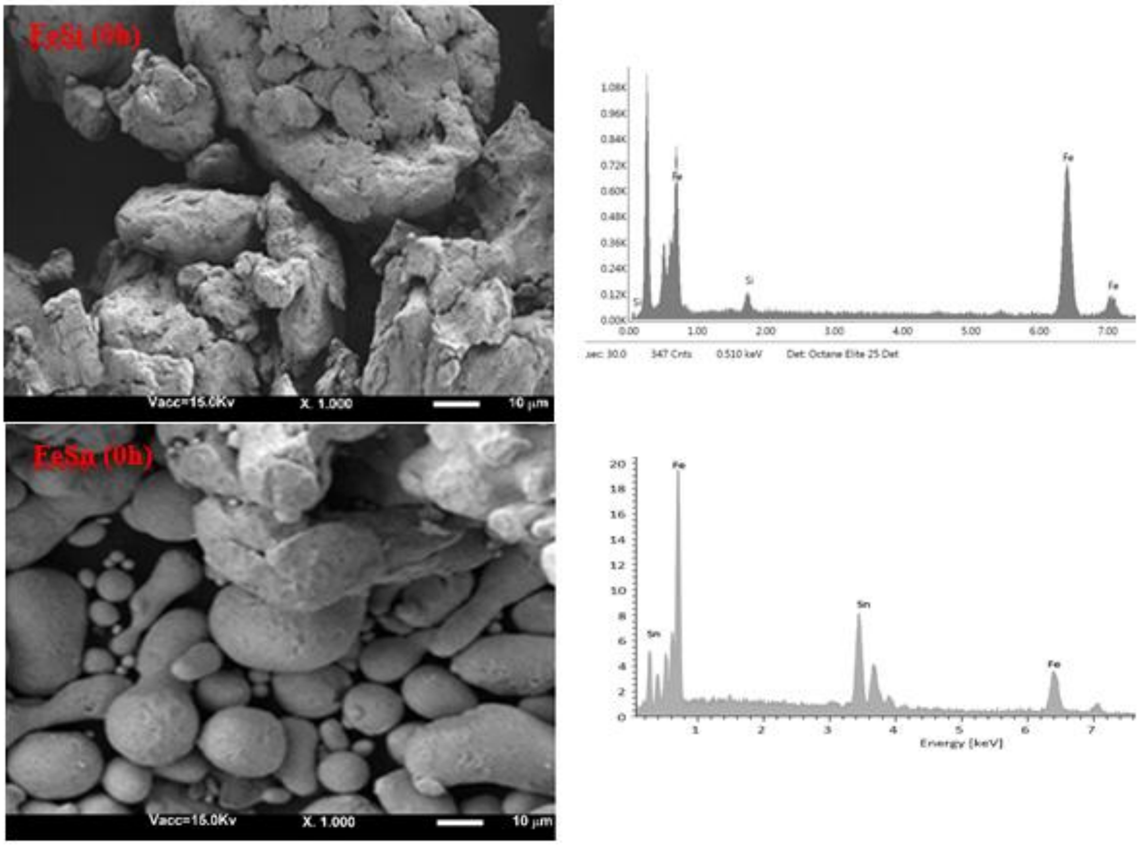

Figure 1a. SEM micrographs of nanocrystalline FeSi and FeSn alloys before milling

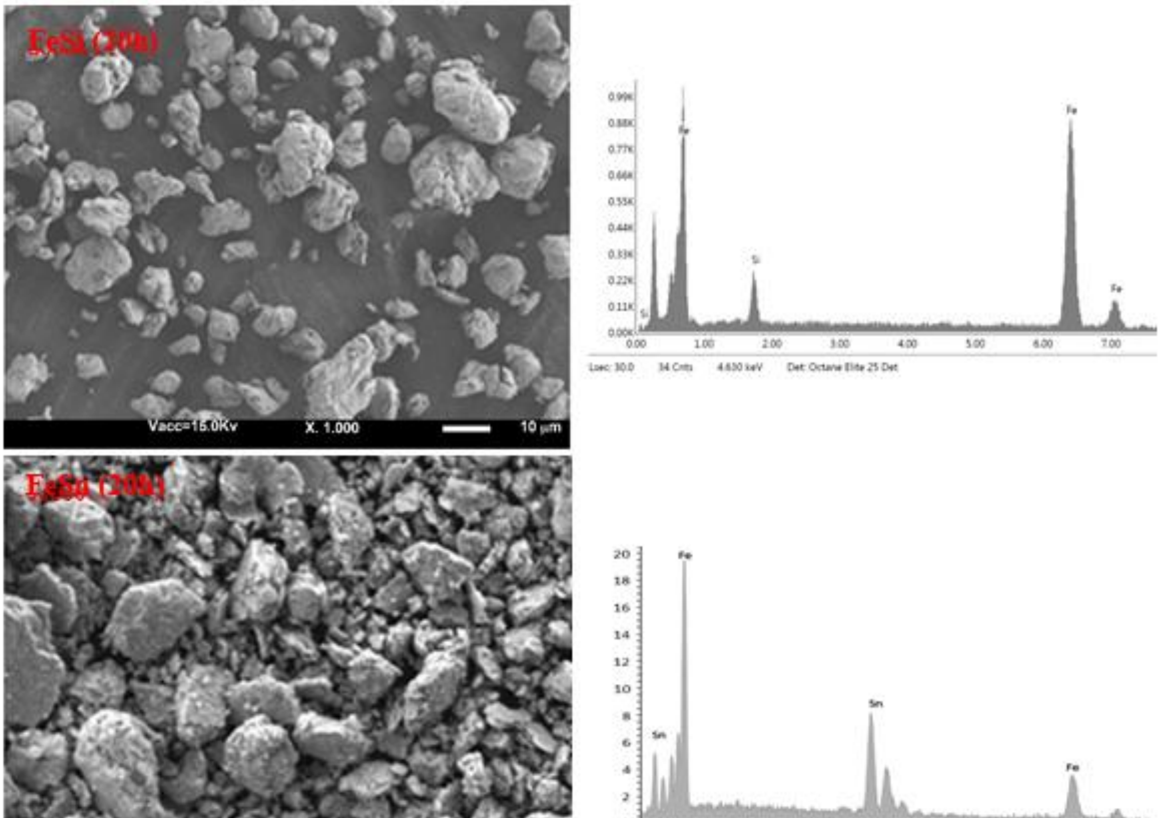

Figure 1b. SEM micrographs of Nanocrystalline FeSi and FeSn alloys milled for 20h

XRD Analysis

The XRD patterns of the FeSi and FeSn alloys at various milling times are displayed in Figure 2, showcasing the structural evolution of both alloys as a function of milling duration. After 20 hours of milling, the XRD data reveal the formation of well-defined FeSi and FeSn phases, with characteristic diffraction peaks at 2θ = 30.7°, 35.1°, and 50.4° for FeSi, and 2θ = 32.8°, 36.2°, and 45.5° for FeSn. These peaks correspond to the (110), (200), and (211) crystallographic planes for FeSi, and the (110), (101), and (211) planes for FeSn, indicating the successful formation of the FeSi and FeSn phases after extensive milling. The sharpness of the peaks reflects the crystallinity of the materials at this stage. As the milling time increases, however, the broadening of the diffraction peaks becomes more pronounced, which is a hallmark of reduced crystallite size typical of nanocrystalline materials. This broadening occurs because smaller grains produce more grain boundaries, which hinder the coherent diffraction of X-rays, resulting in a wider peak. Additionally, the intensity of the peaks gradually decreases with increasing milling time, suggesting a decrease in the crystallite size and the emergence of more amorphous regions within the alloys. This observation is indicative of the refinement of the crystal structure into smaller nano-sized grains as the milling process continues. The gradual reduction in peak intensity and the continuous broadening of diffraction peaks with increasing milling time further confirm that the mechanical milling process is progressively breaking down the larger crystalline structures into finer, nanoscale crystallites, leading to the formation of a nanocrystalline phase in both FeSi and FeSn alloys. The overall structural evolution observed in the XRD data underscores the impact of milling time on the phase transformation and grain refinement of these materials, emphasizing the nanocrystalline nature of the synthesized alloys after extended milling (Wang et al., 2011, Wang et al., 2019, and Ozduran et al., 2021).

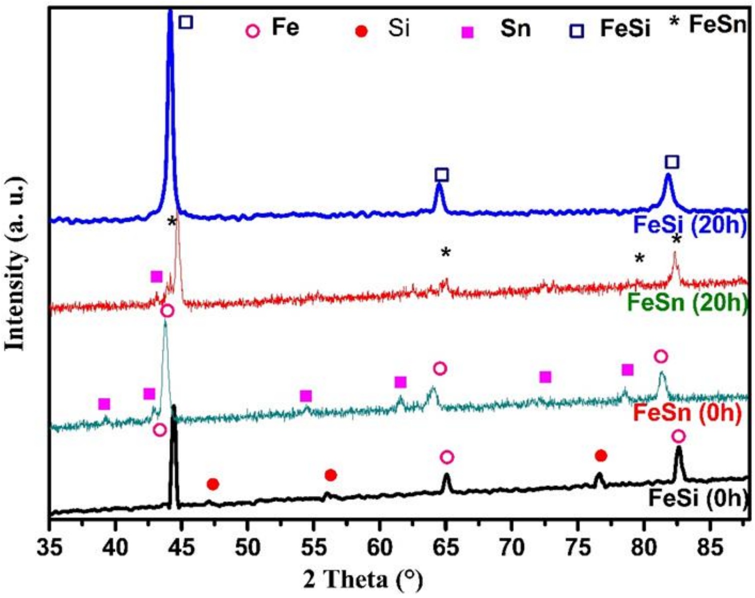

Figure 2. XRD patterns of Nanocrystalline FeSi and FeSn alloys milled for different times

Magnetic Investigation

Figure 3 presents the hysteresis loops of the FeSi and FeSn alloys at different milling times, providing key insights into the evolution of their magnetic properties as the milling duration increases. FeSn Alloys: As the milling time progresses, a clear trend emerges in the magnetic behavior of the FeSn alloys. Notably, there is a significant increase in both coercivity (the field required to reduce the magnetization to zero) and remanent magnetization (the residual magnetization remaining when the external magnetic field is removed). This suggests a transition toward more hard magnetic behavior, which is typically associated with materials that retain their magnetization after the external magnetic field is removed. The increase in coercivity and remanent magnetization indicates that the FeSn alloy is becoming magnetically more resistant to external fields as the milling time increases. However, a noticeable decrease in saturation magnetization is observed, which implies that the material is losing its ability to reach its full magnetization under a strong external field. This decrease in saturation magnetization is likely due to the increased magnetic anisotropy and domain wall pinning caused by the nanostructuring effect. As the milling time increases, the alloy's crystal structure is refined, and smaller crystallites with more surface defects are created. These defects act as pinning sites for the magnetic domain walls, hindering their movement and, therefore, contributing to higher coercivity. This behavior is typical of hard magnetic materials, where the increased number of domain wall pinning sites prevents the alignment of domains in the presence of a magnetic field.

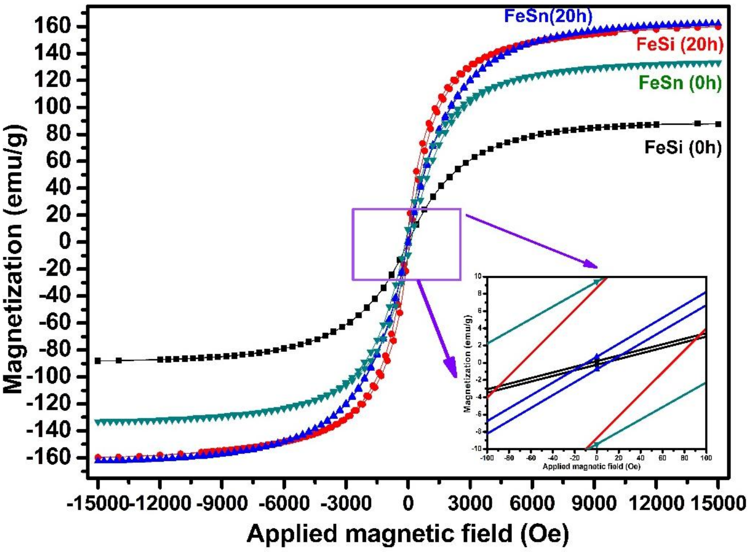

Figure 3. Hysteresis curve (a) and zoom part (b) of Nanocrystalline FeSi and FeSn alloys milled for different times

FeSi Alloys: In contrast to FeSn, the FeSi alloys exhibit superparamagnetic behavior even after 20 hours of milling. This is evident from the hysteresis loops, which show no distinct coercivity, and the lack of a well-defined remanence. Superparamagnetism is a phenomenon typically observed in nanomaterials where, at room temperature, the thermal energy is sufficient to overcome the anisotropy energy of the individual nanoparticles, causing them to rapidly flip their magnetization direction in the absence of an external magnetic field. This results in the absence of a stable magnetization and, consequently, no coercivity or remanent magnetization. Despite this, FeSi alloys exhibit high saturation magnetization, indicating that the material has a strong magnetic response when subjected to an external magnetic field. This suggests that the particles are still able to align with the field, but their small size (due to the ball milling process) and the associated high surface-to-volume ratio lead to superparamagnetic behavior at room temperature. This behavior is often observed in nanoparticles with diameters smaller than the critical size for ferromagnetic or ferrimagnetic ordering, where the particles behave like single magnetic domains (Kakihana et al., 2019, Piamba et al., 2020, Yu et al., 2015, Sales et al., 2014, and Rodmacq et al., 1980) . In summary, the FeSn alloys demonstrate increasing hard magnetic characteristics with higher coercivity and remanent magnetization but reduced saturation magnetization as the milling time increases, reflecting the development of more anisotropic and domain-pinned structures. On the other hand, FeSi alloys exhibit superparamagnetic behavior, with no observable coercivity but a high saturation magnetization, attributed to the small particle sizes and their tendency to behave like single magnetic domains at room temperature. These contrasting behaviors highlight the significant influence of milling time and particle size on the magnetic properties of these nanocrystalline alloys.

Conclusion

This study investigates the structural and magnetic properties of FeSi and FeSn alloys synthesized by mechanical milling, confirming the formation of nanocrystalline structures through XRD and SEM analysis, with particle refinement occurring as milling time increases. Magnetic measurements revealed distinct behaviors: FeSn alloys exhibited enhanced coercivity and reduced saturation magnetization, indicating the development of hard magnetic characteristics, while FeSi alloys displayed superparamagnetic behavior with high saturation magnetization. The SEM images showed a transition from coarse, irregular particles to fine, uniform nanoparticles after 20 hours of milling, which improved the alloys' surface area and magnetic properties. These findings suggest that the magnetic properties of FeSi and FeSn alloys can be tailored by adjusting the milling time, making them suitable for advanced magnetic devices such as sensors, memory devices, and high-performance magnets. The contrasting magnetic behaviors of the two alloys underscore the influence of milling time and particle size in determining the alloys' performance, offering potential for customized applications in next-generation magnetic technologies.

Recommendations

Future research should extend the milling duration beyond 20 hours to further refine the microstructure and enhance magnetic uniformity in both FeSi and FeSn systems. Investigating the influence of controlled annealing or the introduction of minor dopants could improve coercivity, thermal stability, and magnetic anisotropy. Employing advanced characterization tools such as transmission electron microscopy (TEM), energy-dispersive X-ray spectroscopy (EDS), and Mössbauer spectroscopy would provide deeper insight into phase formation and local atomic environments. Additionally, exploring the correlation between milling parameters and magnetization dynamics could help optimize performance for targeted applications. Finally, assessing long-term stability and integrating these nanostructured alloys into prototype magnetic devices would facilitate their transition toward practical and industrial uses.

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

Funding

* This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

Acknowledgements or Notes

* This article was presented as a poster presentation at the International Conference on Technology, Engineering and Science (www.icontes.net) held in Antalya/Türkiye on November 12-15, 2025.

References

Abada, A., Bergheul, S., & Younes, A. (2020). Mechanical and structural behaviour of TiAlV nanocrystalline elaborated by mechanical milling technique. *Micro & Nano Letters, 15*(14), 1023–1027.

Abada, A., Younes, A., & Manseri, A. (2024). Magnetic and structural properties of nanostructured FeSn, FeSnTi, FeSnV and FeSnTiV alloys elaborated via ball milling process. *Journal of the Korean Physical Society, 84*(1), 33–43.

Chen, D., Chen, J., Yan, H., & Chen, Z. (2007). Synthesis of binary and ternary intermetallic powders via a novel reaction ball milling technique. *Materials Science and Engineering: A, 444*(1–2), 1–5.

Jaldurgam, F. F., Ahmad, Z., & Touati, F. (2021). Synthesis and performance of large-scale cost-effective environment-friendly nanostructured thermoelectric materials. *Nanomaterials, 11*(5), 1091.

Jensen, W. A., Liu, N., Rosker, E., Donovan, B. F., Foley, B., Hopkins, P. E., & Floro, J. A. (2017). Eutectoid transformations in Fe-Si alloys for thermoelectric applications. *Journal of Alloys and Compounds, 721*, 705–711.

Kakihana, M., Nishimura, K., Aoki, D., Nakamura, A., Nakashima, M., Amako, Y., Takeuchi, T., Harima, H., Hedo, M., Nakama, T., & Ōnuki, Y. (2019). Electronic states of antiferromagnet FeSn and Pauli paramagnet CoSn. *Journal of the Physical Society of Japan, 88*(1), 014705.

Ouadah, M. H., & Younes, A. (2023). Effects of silicon concentration on the magnetic and structural properties of nanostructured Fe-Si alloy synthesized by ball mill process. *The International Journal of Advanced Manufacturing Technology, 127*(7), 3655–3663.

Ozduran, M., Altay, M. O., Iyigor, A., Çanlı, M., & Arıkan, N. (2021). Structural, electronic, elastic, magnetic, phonon and thermodynamic properties of inverse-Heusler Ti_2FeX (X = Si, Ge, and Sn): Insights from DFT-based computer simulation. *Materials Today Communications, 26*, 102036.

Piamba, J. F., Ortega, C., Hernández-Bravo, R., Carmona, J. G., Tabares, J. A., Alcázar, G. P., & Alvarado-Orozco, J. M. (2020). Theoretical and experimental study of FeSi on magnetic and phase properties. *Applied Physics A, 126*(7), 520.

Rodmacq, B., Piecuch, M., Janot, C., Marchal, G., & Mangin, P. (1980). Structure and magnetic properties of amorphous FexSn1−x alloys. *Physical Review B, 21*(5), 1911–1918.

Sales, B. C., Saparov, B., McGuire, M. A., Singh, D. J., & Parker, D. S. (2014). Ferromagnetism of Fe_3Sn and alloys. *Scientific Reports, 4*, 7024.

Suryanarayana, C., Al-Joubori, A. A., & Wang, Z. (2022). Nanostructured materials and nanocomposites by mechanical alloying: An overview. *Metals and Materials International, 28*(1), 41–53.

Wang, C., Meyer, J., Teichert, N., Auge, A., Rausch, E., Balke, B., Hütten, A., & Felser, C. (2014). Heusler nanoparticles for spintronics and ferromagnetic shape memory alloys. *Journal of Vacuum Science & Technology B, 32*(2), 020802.

Wang, W. L., Li, Z. Q., & Wei, B. (2011). Macrosegregation pattern and microstructure feature of ternary Fe-Sn-Si immiscible alloy solidified under free fall condition. *Acta Materialia, 59*(14), 5482–5493.

Wang, X., Chen, X., Su, X., Yin, F., & Li, Z. (2019). The zinc-rich corner of the Fe-Si-Sn-Zn quaternary system at 450 °C. *Metals, 9*(8), 908.

Yelsukov, E. P., Konygin, G. N., Voronina, E. V., Korolyov, A. V., Ulyanov, A. I., Godovikov, S. K., & Zagainov, A. V. (2000). Magnetic behaviour of high Si (Sn) concentration nanocrystalline Fe-Si and Fe-Sn alloys obtained by mechanical grinding. *Journal of Magnetism and Magnetic Materials, 214*(3), 258–268.

Yin, M., Nash, P., Kaduk, J. A., & Schuster, J. C. (2017). Experimental investigation of the Fe-Sn-Ti ternary isothermal section at 873 K. *Journal of Alloys and Compounds, 693*, 76–86.

Younes, A., Amraoui, R., Bouamer, A., Guessoum, M., Boutaghou, Z., Smaili, F., & Mendoud, A. (2024). Effect of milling time on the structural and magnetic properties of nanostructured Fe90Si10 alloys. *Journal of Electronic Materials, 53*(10), 6098–6109.

Younes, A., Khorchef, M., Bouamer, A., & Amar, H. (2019). Magnetic and structural behavior of Fe-CoO nanocomposites mechanically milled. *IOP Conference Series: Materials Science and Engineering, 557*(1), 012064.

Yu, H., Wen, Y., & Bi, X. (2015). Magnetic and mechanical properties of the gradient FeSi alloys fabricated by magnetron sputtering. *Journal of Alloys and Compounds, 634*, 83–86.

Author(s) Information

Abderahim Abada
Laboratory of Aeronautic Science, Aeronautic Institute,
University of Saad Dahleb Blida 1, Blida, Algeria
Contact e-mail: *abada_75@yahoo.fr*

Abderrahmane Younes
Research Centre in Industrial Technologies (CRTI)
P.O Box 64, Cheraga 16014 Algiers, Algeria

Rachid Amraoui
Research Centre in Industrial Technologies (CRTI)
P.O Box 64, Cheraga 16014 Algiers, Algeria

Djilali Allou
Research Centre in Industrial Technologies (CRTI)
P.O Box 64, Cheraga 16014 Algiers, Algeria

To cite this article:

Abada, A., Younes, A., Amraoui, R., & Allou, D. (2025). Magnetic and structural behavior of nanocrystalline iron-silicon and iron-tin produced via mechanical milling. *The Eurasia Proceedings of Science, Technology, Engineering and Mathematics (EPSTEM), 38,* 200-216.