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Effect of Crystallite Size on Magnetic Properties of Nanostructured FeAlTiBZr Alloy

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Abstract: This study investigates the influence of crystallite size (D) on two key magnetic parameters, saturation magnetization (MS) and coercivity (HC), in nanostructured FeAlTiBZr alloy. The fabrication of this nanostructured material involved a mechanical grinding process. Structural, morphological, and magnetic properties of the nanostructured FeAlTiBZr alloy at various production stages were thoroughly examined using advanced characterization techniques, including Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), X-ray Diffraction (XRD), and Vibrating Sample Magnetometer (VSM). The crystallite size decreased from 38 to 26 nm, while the lattice strain increased from 0.17933% to 0.25194%. This shift in lattice parameters, from 0.2867 to 0.2876, can be attributed to the milling time effect. Additionally, the morphology of particles underwent changes, with particle size increasing over the milling period. However, the magnetic properties exhibited a contrasting trend: coercivity, saturation magnetization, remanence magnetization, and squareness decreased from 55.78 Oe, 77.35 emu/g, 3.08 emu/g, and 0.039 to 42.83 Oe, 66.39 emu/g, 2.04 emu/g, and 0.030, respectively.

Keywords: Nanostructured FeAlTiBZr alloy, Magnetic properties, Structural and morphology

Introduction

In recent years, the development of nanostructured materials has garnered significant attention due to their unique properties and potential applications in various fields, including electronics, energy storage, and magnetic devices. Among these materials, nanostructured FeAlTiBZr alloy holds particular promise for its intriguing magnetic properties and structural characteristics (Xu et al., 2019; Ji et al., 2011; Zhang et al., 2016,; Kuchibhatla et al., 2007; Gao et al., 2013). This study delves into the effects of crystallite size (D) on two fundamental magnetic parameters, saturation magnetization (Ms) and coercivity (Hc), in nanostructured FeAlTiBZr alloy. Fabricated through a mechanical grinding process, this alloy offers a compelling avenue for exploring the intricate interplay between structural, morphological, and magnetic properties at the nanoscale. Utilizing advanced characterization techniques such as Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), X-ray Diffraction (XRD), and Vibrating Sample Magnetometer (VSM), this

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research meticulously investigates the evolution of the nanostructured FeAlTiBZr alloy throughout different production stages. Key focus is placed on elucidating changes in crystallite size, lattice strain, morphology, and magnetic behavior as influenced by milling time.

The findings presented herein shed light on the complex relationship between crystallite size and magnetic properties in nanostructured materials, offering valuable insights into the design and optimization of magnetic devices. Moreover, this study underscores the significance of structural and morphological characterization in tailoring the properties of nanostructured alloys for diverse applications.

Method

The synthesis process employed a PM 400 ball mill with two WC jars, operated at 280 rpm. Nanostructured FeAlTiBZr was synthesized by blending elemental powders of Fe, Al, Ti, B, and Zr at a mass ratio of 1:12. The raw materials exhibited mean particle sizes of 62 µm, 55 µm, 51 µm, 43 µm, and 58 µm for Fe, Al, Ti, B, and Zr, respectively, with purities of 99.7%, 99.1%, 99.3%, 98.8%, and 98.9%, respectively.

Morphological changes were examined using SEM microscopy equipped with EDS spectroscopy. X-ray diffraction analysis, performed with an XPERT PRO instrument using Co Kα radiation (wavelength λ = 1.7902 Å) in the 2θ range from 0° to 120°, facilitated the identification of different phases during milling and determination of structural parameters. The magnetic properties, influenced by milling time, were evaluated utilizing a Micro Sense EV9 vibrating sample magnetometer.

Results and Discussion

Structural Analysis

In Figure 1, the progress of XRD patterns for nanostructured FeAlTiBZr alloys milled for various periods is depicted. Each sample exhibits distinct diffraction peaks, accompanied by observable broadening and intensity fluctuations, indicative of particle size reduction and strain accumulation throughout the milling process. Upon detailed analysis of alloys milled for different durations, notable transformations emerge. The diffraction peaks exhibit increased broadness and reduced relative intensity, hinting at phenomena extending beyond conventional grain refinement and lattice strain.

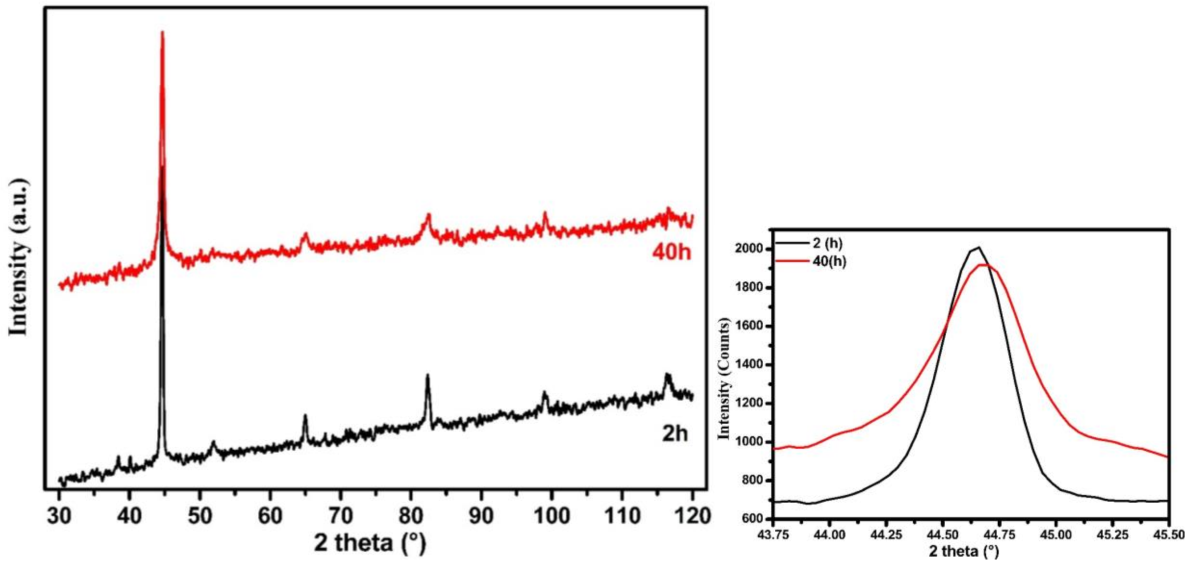

Figure 1. XRD patterns of nanostructured FeAlTiBZr alloy milled with different times

Table 1 presents the dynamic changes in structural parameters, including crystallite size, lattice strain, and lattice parameters, observed in nanostructured FeAlTiBZr throughout the milling process. The data indicates a significant reduction in crystallite size, from an initial value of 38 nm to 26 nm, concomitant with an increase in lattice strain, rising from 0.17933% to 0.25194%. These alterations are primarily attributed to the milling time effect, where prolonged mechanical milling leads to enhanced dislocation densities and lattice defects, thereby contributing to strain accumulation and crystallite refinement. Furthermore, a discernible shift in lattice parameters is evident, transitioning from 0.2867 to 0.2876, indicative of lattice distortion induced by mechanical deformation during milling. This phenomenon underscores the intricate interplay between processing parameters and resultant structural modifications in nanostructured alloys (Metidji et al., 2023; Metidji et al., 2020; Metidji et al., 2022)

Table 1. Structural parameters of nanostructured FeAlTiBZr alloy milled with different times

Milling time (h)	Crystallite size (nm)	FWHM (°)	lattice strain (%)	lattice parameters (nm)
2h	38	0.25	0.17933	0.2867
40h	26	0.269	0.25194	0.2876

Morphology

Figure 2 depicts the scanning electron microscope (SEM) analysis of nanostructured FeAlTiBZr alloy subjected to varying grinding durations. Initially, at 2 hours of grinding, the Fe, Al, Ti, B, and Zr particles exhibit a coarse or agglomerated state, resulting in irregularly shaped particles and clusters. However, with prolonged grinding durations extending up to 40 hours, the mechanical forces exerted by the grinding tool, including the grinding balls, induce continuous deformation and fragmentation of the particles.

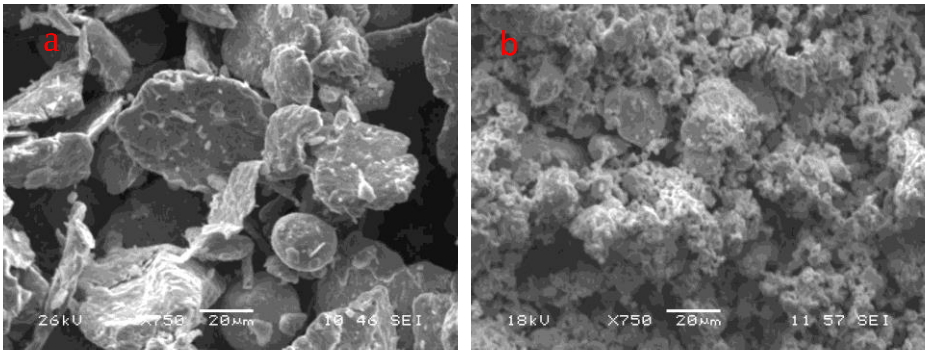

Figure 2. SEM examination of nanostructured FeAlTiBZr alloy milled with different times

This mechanical action leads to a progressive reduction in particle size and alters their morphology. Specifically, the nanostructured alloy particles undergo a transformation towards smaller and more regular shapes compared to their initial state. This phenomenon arises from the cumulative effect of particle fracture and refinement mechanisms such as cold welding and grain boundary rearrangement induced by prolonged mechanical milling. The resulting nanostructured morphology holds implications for the material's properties and performance in various applications (Peng et al., 2019; Fan et al., 2020; Pereira et al., 2007; Shen et al., 2023).

Magnetic Investigation

The hysteresis analysis of the nanostructured FeAlTiBZr alloy was conducted by a Vibrating Sample Magnetometer (VSM) apparatus, employing an applied magnetic field ranging from -17 kOe to 17 kOe, as illustrated in Figure 3. It was observed that all samples exhibit ferromagnetic behavior, characterized by narrow hysteresis loops, indicative of the formation of soft magnetic materials. Notably, the nanostructured FeAlTiBZr alloy achieves full saturation at a magnetic field of 17 kOe.

The magnetic properties of the nanostructured FeAlTiBZr alloy, influenced by milling duration, are subject to various factors such as particle size, shape, chemical composition, milling conditions, and the presence of impurities (Metidji et al., 2020). Previous studies have established correlations between observed magnetic behavior and structural defects, underscoring the pivotal role of prolonged milling times in phase transformation processes. Interestingly, despite the milling-induced alterations in other structural parameters, such as lattice strain and morphology, the crystallite size of the milled samples undergoes only marginal changes throughout the milling process (Jirásková et al., 2018). This observation suggests a nuanced interplay between milling-induced structural modifications and resultant magnetic properties, warranting further investigation into the underlying mechanisms governing the magnetic behavior of nanostructured alloys (Hsieh et al., 2011).

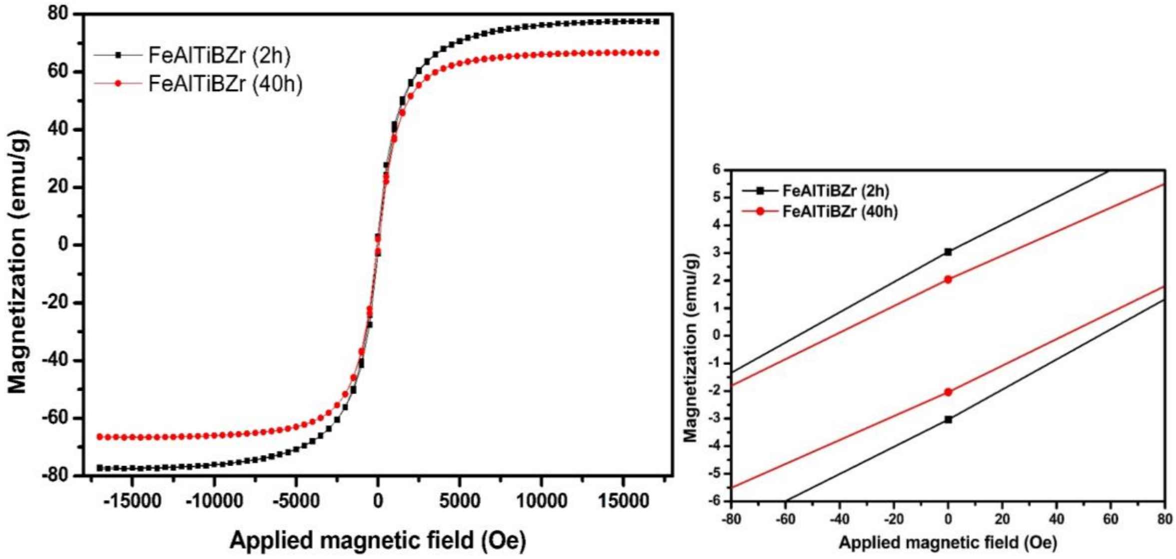

Figure 3**.** Hysteresis loops of nanostructured FeAlTiBZr alloy milled with different times

Table 2 presents the evolution of coercivity (Hc), saturation magnetization (Ms), remanence magnetization (Mr), and the ratio of remanence magnetization to saturation magnetization (Mr/Ms) over various milling durations. Coercivity (Hc) exhibits a consistent decrease over time, correlating with the reduction in average crystallite size. This relationship highlights the important influence of particle size variations on Hc. As particle size diminishes, the heightened surface-to-volume ratio increases surface reactivity, facilitating the formation of crystallographic defects such as dislocations and vacancies. These defects serve to effectively pin magnetic domain walls, thereby augmenting the coercivity of the nanostructured alloy (Rico et al., 2012; Meng et al., 2017).

Table 1. Magnetic parameters of nanostructured FeAlTiBZr alloy milled with different times

Milling time (h)	Hc (Oe)	Ms (emu/g)	Mr (emu/g)	Mr/Ms
2	55.78	77.35	3.08	0.039
40	42.83	66.39	2.04	0.030

The notable increase in both saturation magnetization (Ms), remanence magnetization (Mr), and the ratio Mr/Ms observed in the nanostructured FeAlTiBZr alloy during milling is closely tied to profound alterations in the magnetic domain structure. This phenomenon is further compounded by the heightened magnetic anisotropy resulting from concurrent processes of particle size reduction and defect formation (Strothers et al. 1991 and Strothers et al. 2023).

Conclusion

In conclusion, this study provides a comprehensive analysis of the structural and magnetic properties of nanostructured FeAlTiBZr alloy synthesized through mechanical milling. Through systematic investigation, it was observed that milling duration plays a crucial role in shaping the structural characteristics and magnetic behavior of the alloy. Notably, the reduction in crystallite size and increase in lattice strain during milling signify the progressive refinement and deformation of the alloy particles.

Furthermore, the observed trends in coercivity (Hc), saturation magnetization (Ms), and remanence magnetization (Mr) underscore the intricate interplay between particle size, defect formation, and magnetic domain structure. The decrease in Hc alongside the simultaneous rise in Ms, Mr, and the Mr/Ms ratio suggest a transition towards soft magnetic behavior, facilitated by the enhanced magnetic anisotropy resulting from particle size reduction and defect-induced pinning effects. These findings shed light on the fundamental mechanisms governing the magnetic properties of nanostructured alloys and hold significant implications for the design and optimization of magnetic materials for various technological applications. Future research endeavors may focus on further elucidating the underlying mechanisms driving the observed phenomena and exploring novel processing techniques to tailor the properties of nanostructured alloys for specific applications.

Recommendations

Future research should refine milling parameters and explore alloy composition adjustments to enhance control over magnetic properties. Advanced characterization and modeling would provide deeper insights; while environmental stability and application-specific testing, particularly for high-frequency devices, will help, confirm practical utility. Alternative synthesis methods and scalability assessments will also support transitioning FeAlTiBZr alloys from lab to industrial applications.

Scientific Ethics Declaration

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

Acknowledgements or Notes

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