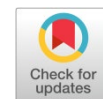


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Research article

Synthesis and microstructural characterization of nanostructured Fe₅₀Ni₅₀ alloy prepared by mechanical alloying

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ABSTRACT

In this comprehensive study, the synthesis, phase evolution, and microstructural characteristics of nanostructured Fe₅₀Ni₅₀ alloy powders produced via high-energy mechanical alloying (MA) were systematically investigated. Elemental iron and nickel powders were processed in a high-energy planetary ball mill under a controlled argon protective atmosphere to prevent oxidation, utilizing a constant ball-to-powder weight ratio (BPR) of 20:1 and a rotational speed of 350 rpm across cumulative milling durations of 0, 4, 8, 32, 48, and 96 hours. The structural transformations, crystallite size refinement, internal lattice strain accumulation, and lattice parameter expansion were evaluated using X-ray diffraction (XRD) data interpreted through Scherrer and Williamson–Hall analytical approaches. Furthermore, morphological alterations, particle size distributions, and microstructural transitions were examined using scanning electron microscopy (SEM) equipped with secondary electron detectors and Image Tool software. The XRD results indicate that continuous high-energy mechanical deformation up to 96 hours induces complete mechanical alloying, resulting in the formation of a random (Ni,Fe) solid solution with a face-centered cubic (fcc) crystal structure, as evidenced by the disappearance of the individual bcc iron diffraction peaks and significant broadening of the nickel-related peaks. The crystallite size exhibited a sharp initial decline followed by a relatively stable trend, decreasing from an initial value of 65 nm to a minimum of 12 nm after 96 hours. Simultaneously, the internal lattice strain increased progressively, reaching a maximum value of 0.58%. Morphological analyses revealed that initial microforging, cold welding, and particle flattening predominated during the early milling stages, whereas extended milling induced severe work hardening and fracture, ultimately achieving a dynamic steady-state balance at 96 hours, with refined, relatively uniform, and spherical particles possessing an average size of 10 μm.

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KEYWORDS

Mechanical alloying
Fe-Ni alloy
Nanostructured materials
Phase evolution
Microstructural characterization
X-ray diffraction



1. Introduction

Nanostructured materials can be produced through a variety of processing routes, which can generally be classified into vapor-phase, liquid-phase, and solid-state approaches. Vapor-phase techniques include processes such as inert gas condensation and related deposition methods, whereas liquid-phase routes include electrodeposition and

rapid solidification [1–3]. Solid-state processing methods, in contrast, provide an attractive alternative for producing nanostructured powders without requiring melting or vaporization of the constituent elements. Among these methods, mechanical alloying (MA) has emerged as a versatile and relatively simple powder-processing technique capable of producing metastable and equilibrium materials with refined microstructures. The process is particularly attractive because it can

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simultaneously promote alloying, grain refinement, defect generation, and microstructural homogenization during repeated mechanical deformation [4–6].

Mechanical alloying relies on repeated impacts and deformation of powder particles between milling media and the walls of a milling vessel. During high-energy milling, powder particles experience severe plastic deformation followed by flattening, cold welding, work hardening, fracture, and repeated re-welding [7, 8]. The competition between these mechanisms continuously modifies the particle morphology and internal structure. In the initial stages, ductile particles are generally flattened and cold-welded, which may increase particle size and develop layered or lamellar structures. With increasing milling time, progressive work hardening reduces the ability of the particles to undergo further plastic deformation, promoting fracture and refinement [9–11]. Eventually, the rates of cold welding and fracture may approach a dynamic balance, resulting in a relatively stable particle size and morphology. At the crystallographic level, severe deformation introduces high densities of dislocations, vacancies, stacking faults, and grain boundaries, while repeated fracture and re-welding promote intimate contact and chemical homogenization between different elemental constituents [12, 13].

One of the most important characteristics of mechanical alloying is its ability to induce solid-state reactions and form alloy phases that may be difficult to obtain through conventional equilibrium processing routes [14, 15]. During milling, the continuously generated lattice defects and highly refined crystallite structure provide numerous diffusion paths and active interfaces. As a result, elemental powders can progressively develop increasingly homogeneous compositions, eventually leading to the formation of extended solid solutions, metastable phases, amorphous structures, or intermetallic compounds depending on the composition and processing conditions [11, 16, 17]. The final microstructure is strongly influenced by milling parameters such as milling speed, milling time, ball-to-powder ratio, milling atmosphere, type and size of milling media, and the use of process control agents. Consequently, understanding the relationship between milling conditions and structural evolution is essential for controlling the properties of mechanically alloyed powders [18–20].

Among binary alloy systems, Fe–Ni alloys are of particular scientific and technological interest because their structural and functional properties are strongly dependent on composition, phase constitution, crystallite size, lattice defects, and processing history [21, 22]. Iron and nickel possess different crystal structures at ambient conditions, with Fe exhibiting a body-centered cubic structure and Ni exhibiting a face-centered cubic structure. Mechanical alloying of these two elemental powders therefore provides an interesting model system for investigating the gradual structural transformation associated with solid-state alloy formation [23, 24]. The severe plastic deformation introduced during milling can progressively modify the original crystalline structures, increase defect density, refine crystallite size, and promote atomic-scale mixing between Fe and Ni [25, 26]. The evolution of diffraction peaks during this process can consequently provide valuable information regarding phase transformation, crystallite refinement, lattice strain, and the development of the final alloy structure [27].

The structural evolution of mechanically alloyed Fe–Ni powders is also closely associated with changes in lattice parameters and microstrain [28, 29]. Continuous plastic deformation and defect

accumulation can produce significant distortions within the crystal lattice, while the incorporation of one elemental species into the crystal structure of the other can modify the lattice parameter [30]. At the same time, progressive crystallite refinement increases the contribution of grain boundaries and interfaces to the overall structure. X-ray diffraction (XRD) is therefore an effective technique for monitoring these changes because variations in peak position, peak width, and peak intensity provide information about phase evolution, crystallite size, and lattice strain [31, 32]. The Scherrer approach can be used to estimate crystallite size from diffraction-line broadening, while the Williamson–Hall method provides an additional means of distinguishing the contributions associated with crystallite-size broadening and lattice microstrain. Combining these structural analyses with morphological observations can provide a more comprehensive understanding of the mechanisms governing mechanical alloying [33–35].

The evolution of particle morphology is another important aspect of the mechanical alloying process. Unlike crystallite size, which describes coherent diffraction domains within particles, particle size and morphology are controlled primarily by the competition between plastic deformation, cold welding, work hardening, and fracture. Thus, substantial crystallite refinement may occur simultaneously with relatively large particle sizes during intermediate milling stages. Continued milling can subsequently promote particle fragmentation and morphological homogenization as work hardening increases [36, 37].

Despite the extensive application of mechanical alloying to different alloy systems, the progressive relationship between phase evolution, crystallite refinement, lattice strain, lattice-parameter changes, and particle morphology remains highly dependent on the specific elemental system and milling conditions [38, 39]. In particular, the simultaneous evaluation of crystallographic and morphological changes over extended milling periods can provide useful insight into the transition from elemental powders to a chemically and structurally homogenized nanostructured alloy [40, 41]. Such an approach is especially relevant for Fe–Ni powders, where the transformation from the initial coexistence of bcc-Fe and fcc-Ni phases toward a single fcc solid solution can be accompanied by substantial crystallite refinement, lattice distortion, and changes in particle morphology [42].

Therefore, the primary objective of the present study is to synthesize nanostructured Fe₅₀Ni₅₀ alloy powders through high-energy mechanical alloying and systematically investigate their structural and morphological evolution as a function of milling time. Particular attention is given to the progressive phase transformation from the initial Fe and Ni elemental powders toward an fcc Fe–Ni solid solution, together with the associated reduction in crystallite size, accumulation of internal lattice strain, variation in lattice parameter, and evolution of particle morphology and size. X-ray diffraction is employed to characterize the phase and crystallographic evolution, while scanning electron microscopy is used to examine changes in particle morphology and size distribution. By correlating these complementary observations, the study aims to clarify the dominant structural and morphological mechanisms operating during the mechanical alloying of equiatomic Fe–Ni powders and to identify the microstructural characteristics achieved after prolonged high-energy milling.

2. Experimental procedure

The experimental methodology was meticulously designed to investigate the synthesis and microstructural evolution of iron-nickel binary alloy powders under well-controlled processing parameters. High-purity elemental iron and nickel powders supplied by Merck (Germany), featuring a chemical purity exceeding 99% and an initial average particle size of approximately 10 μm , served as the starting raw materials. Analytical-grade ethanol with a purity of 99% was utilized as a process control agent (PCA) to mitigate excessive agglomeration and cold welding during early milling stages.

The mechanical alloying operations were executed utilizing a high-energy planetary ball mill (Sepahan 84 Mill D Mixer) equipped with a specially designed curved chamber that facilitates strict control over the internal milling atmosphere. A 15-gram powder charge corresponding to the equiatomic $\text{Fe}_{50}\text{Ni}_{50}$ composition was loaded into the milling vial alongside hardened steel balls to maintain a constant and rigorous ball-to-powder weight ratio (BPR) of 20:1. This optimized milling charge consisted of 7 large balls with an approximate individual weight of 32.6 g and 18 small balls with an individual weight of approximately 4 g each. Milling was conducted at a constant rotational speed of 350 rpm under a protective, high-purity argon atmosphere to preclude oxidation. To prevent excessive thermal buildup and potential thermal degradation within the vial, the system operated intermittently with a strict operational cycle consisting of 30 minutes of continuous milling followed by a 15-minute cooling rest period. Table 1 summarizes the detailed operational parameters utilized during the mechanical alloying process.

Following each cumulative milling interval (0, 4, 8, 32, 48, and 96 hours), the powder mixture and grinding balls were separated. Representative powder samples (2 to 3 grams for X-ray diffraction and 0.5 grams for scanning electron microscopy) were extracted for subsequent structural and morphological characterizations. Phase identification and detailed structural analysis were carried out using a Philips Xpert High Score X-ray diffractometer equipped with a copper anode ($\text{CuK}\alpha$ radiation, $\lambda = 0.154059$ nm), operating at an accelerating voltage of 40 kV and a current of 30 mA, utilizing a step size of 0.02 $^\circ/\text{s}$ across a continuous 2θ scanning range of 20 $^\circ$ to 110 $^\circ$. Crystallite size and internal lattice strain were evaluated using both the standard Scherrer formula and the Williamson-Hall analytical method

Table 1. Operating parameters of the $\text{Fe}_{50}\text{Ni}_{50}$ mechanical alloying process.

Mill type	Milling speed	Work-rest cycle	Milling atmosphere	Ball-to-powder weight ratio
Planetary ball mill	350 rpm	30 min work- 15 min rest	Argon (Ar)	1:20

to successfully separate crystallite size broadening contributions from non-uniform microstrain effects induced by heavy cold work. Lattice parameters were precisely determined using graphical extrapolation methods based on high-angle diffraction peaks and associated Miller indices. Finally, powder morphology, particle shape evolution, and size distributions were examined using a Vega@Tescan scanning electron microscope operating at an accelerating voltage of 20 kV with a Secondary Electron (SE) detector, where average particle sizes were statistically calculated from over 100 individual particles using Image Tool software.

3. Results and discussion

3.1. Phase evolution and XRD analysis

The phase transformations occurring progressively during mechanical alloying were evaluated by analyzing the X-ray diffraction patterns of both un-milled and milled powders. The XRD pattern of the unmilled pure nickel powder (Fig. 1a) exhibits distinct characteristic peaks corresponding exclusively to a face-centered cubic (fcc) crystal structure, whereas the pure iron powder pattern (Fig. 1b) displays sharp peaks matching a body-centered cubic (bcc) crystal structure.

In the unmilled $\text{Fe}_{50}\text{Ni}_{50}$ powder mixture before mechanical processing (Fig. 2), distinct coexistence peaks representing both bcc-Fe and fcc-Ni are clearly visible without any evidence of alloying. As the milling duration advances through 4, 8, 32, 48, and 96 hours (Fig. 3a through Fig. 3e, respectively), a progressive broadening and a significant attenuation in the intensity of the diffraction peaks are observed. A comprehensive comparison of these patterns is consolidated in Fig. 3f. Crucially, after 96 hours of high-energy mechanical alloying (Fig. 3e), all characteristic diffraction peaks

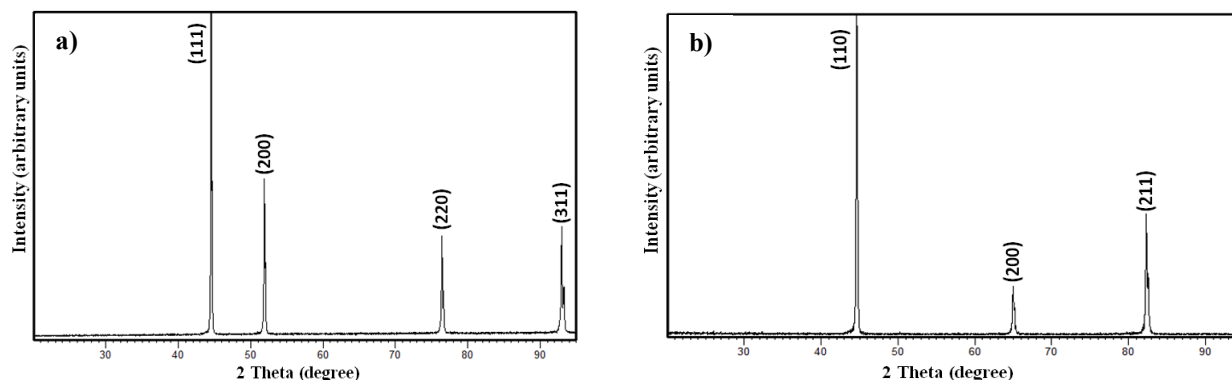


Fig. 1. XRD patterns of the starting powders: a) pure nickel (fcc) and b) pure iron (bcc).

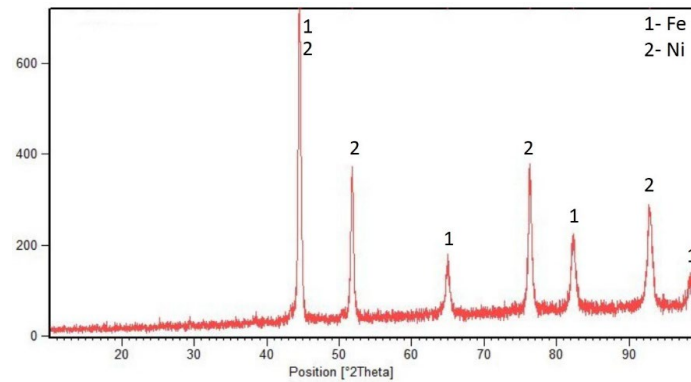


Fig. 2. XRD pattern of the unmilled $\text{Fe}_{50}\text{Ni}_{50}$ powder mixture.

corresponding to iron completely vanish. Due to the substantial accumulation of internal lattice strain and severe grain refinement, the remaining peaks undergo considerable broadening and angular shifting, confirming the successful completion of mechanical alloying and the

formation of a random solid solution designated as $\gamma\text{-(Ni,Fe)}$ possessing a single-phase fcc crystal structure.

This phase transformation is fundamentally driven by the severe plastic deformation experienced by the powder particles during high-energy

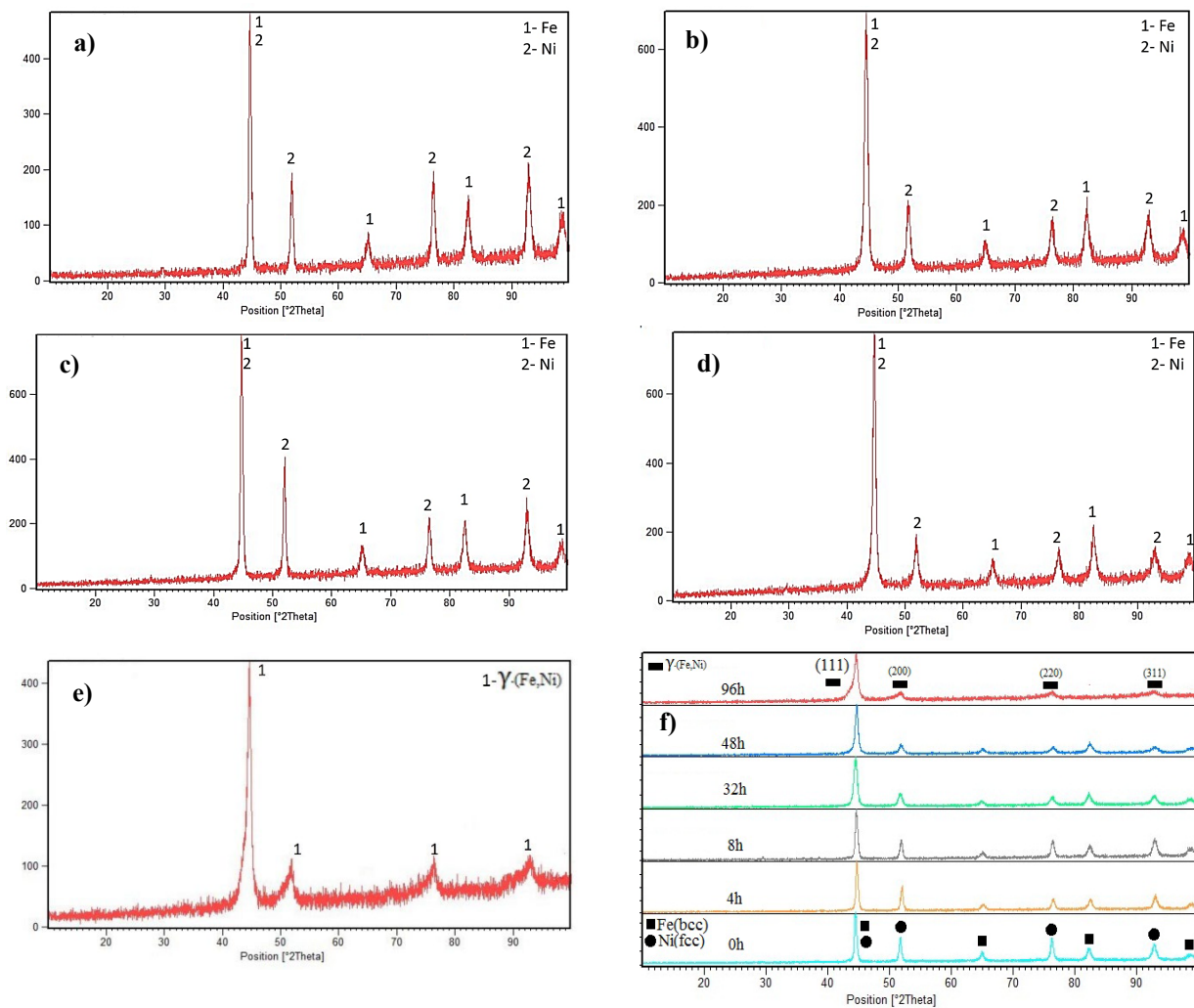


Fig. 3. XRD patterns of $\text{Fe}_{50}\text{Ni}_{50}$ powders milled for a) 4, b) 8, c) 32, d) 48, and e) 96 h, along with f) a comparative overview, confirming the formation of the $\gamma\text{-(Ni,Fe)}$ solid solution.

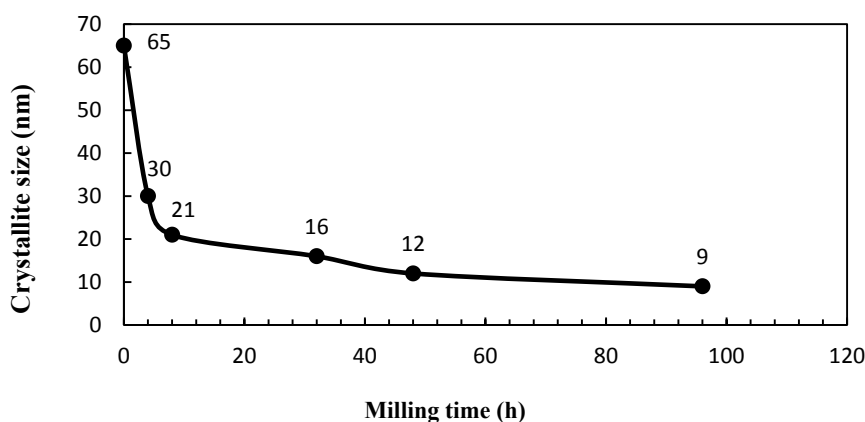


Fig. 4. Variation of crystallite size as a function of milling time for the Fe₅₀Ni₅₀ powder mixture.

ball milling, which introduces extremely high densities of crystalline defects such as dislocations, stacking faults, and a massive fraction of newly created grain boundaries. These structural defects dramatically enhance the atomic diffusion kinetics of solute atoms within the matrix. Furthermore, the localized transient temperature spikes generated during high-energy ball-to-ball and ball-to-vial collisions further assist atomic mobility, thereby promoting true solid-state alloying between the iron and nickel components.

3.2. Crystallite size and lattice strain kinetics

The structural parameters, specifically crystallite size and microstrain, were extracted from the XRD line broadening data. The initial crystallite size of the un-milled powder mixture is approximately 65 nm. Upon the initiation of mechanical alloying, the crystallite size exhibits a sharp downward trend (Fig. 4). During the first 8 hours of milling, a rapid and substantial decrease in crystallite size occurs, which is attributed to the drastic increase in dislocation density resulting from severe plastic deformation and the subsequent alignment of dislocations into dislocation sub-boundaries.

With further extension of milling time beyond 8 hours, the slope of the crystallite size curve tends to stabilize as these sub-boundaries progressively transform into high-angle grain boundaries, culminating in a minimum crystallite size of 12 nm after 96 hours of milling.

Concurrently, the internal lattice strain of the Fe-50% Ni system exhibits a continuous upward trend with increasing milling duration (Fig. 5), reaching a maximum value of 0.58% at 96 hours. The generation of these microstrains is directly linked to micro-stresses, the continuous multiplication and piling up of dislocations, and the creation of high densities of point defects (such as vacancies, which induce localized tensile stresses, and substitutional solute atoms, which generate compressive stresses within the crystal lattice).

3.3. Lattice parameter analysis

The lattice parameter represents another critical structural metric that reflects atomic-scale modifications during processing. The lattice parameter of the Fe₅₀Ni₅₀ alloy demonstrates a continuous upward trajectory as a function of milling time (Fig. 6), increasing from an

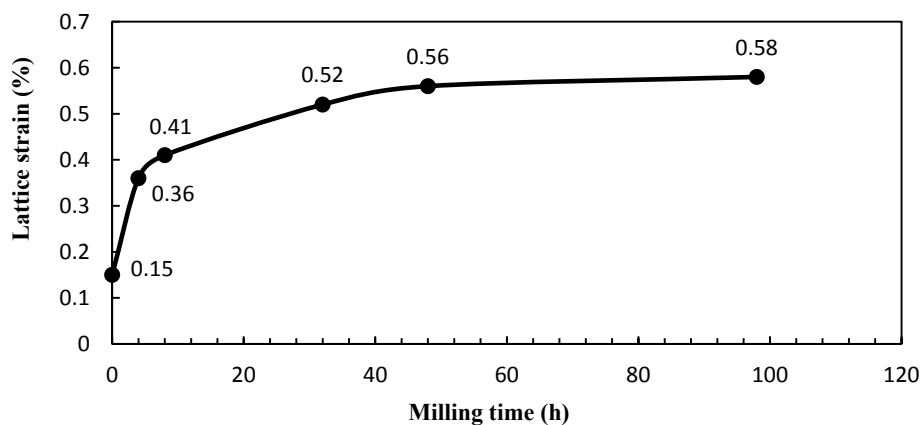


Fig. 5. Variation of internal lattice strain as a function of milling time for the Fe₅₀Ni₅₀ powder.

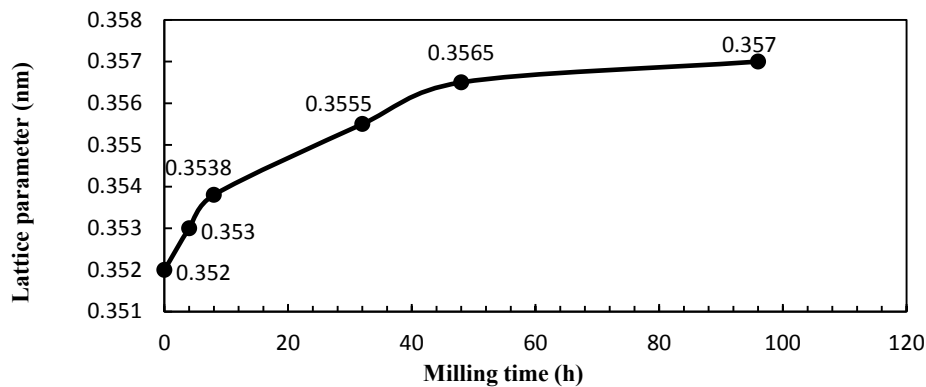


Fig. 6. Variation of the lattice parameter as a function of milling time for the $\text{Fe}_{50}\text{Ni}_{50}$ alloy.

initial value of 0.352 nm for the un-milled powder blend to a maximum value of 0.357 nm after 96 hours of milling.

This noticeable lattice expansion can be primarily attributed to the solid-state dissolution of iron atoms ($r_{\text{Fe}} = 0.126$ nm), which possess a larger atomic radius, into the nickel lattice ($r_{\text{Ni}} = 0.124$ nm), alongside the accumulation of high concentrations of lattice defects, grain boundaries, and vacancies generated during the repeated internal fracture, cold welding, and re-welding cycles inherent to mechanical alloying.

3.4. Morphological evolution and particle size dynamics

Scanning electron microscopy observations revealed distinct morphological transformations throughout the processing stages. As-received iron powders exhibit a spherical morphology (Fig. 7a), whereas nickel powders display rod-like shapes (Fig. 7b). The un-milled $\text{Fe}_{50}\text{Ni}_{50}$ powder mixture (Fig. 8a) consists of large, irregular agglomerates.

The progressive morphological transition of the powders from initial flattened structures to fine spherical particles is demonstrated from early to advanced stages of milling in Fig. 8b through Fig. 8f for 4 h, 8 h, 32 h, 48 h, and 96 h, respectively.

In the early stages of milling (e.g., 4 hours, Fig. 8b), the particles retain a relatively large size and continue to exhibit flattened morphologies. This initial stage is governed by high compressive forces generated by ball-powder and ball-ball collisions, where the soft ductile particles undergo microforging, leading to particle flattening, cold welding, and an initial increase in average particle size.

As milling proceeds up to 32 hours (Fig. 8d), the laminated, flake-like particles begin to crack and fracture, indicating that the fracture mechanism starts to predominate over cold welding as the particles undergo severe work-hardening. With further milling up to 48 hours (Fig. 8e), the size distribution becomes narrower, work-hardened particles become brittle, and the dominant fracture mechanism drives a morphological transition from flaky structures back toward more equiaxed, rounded shapes.

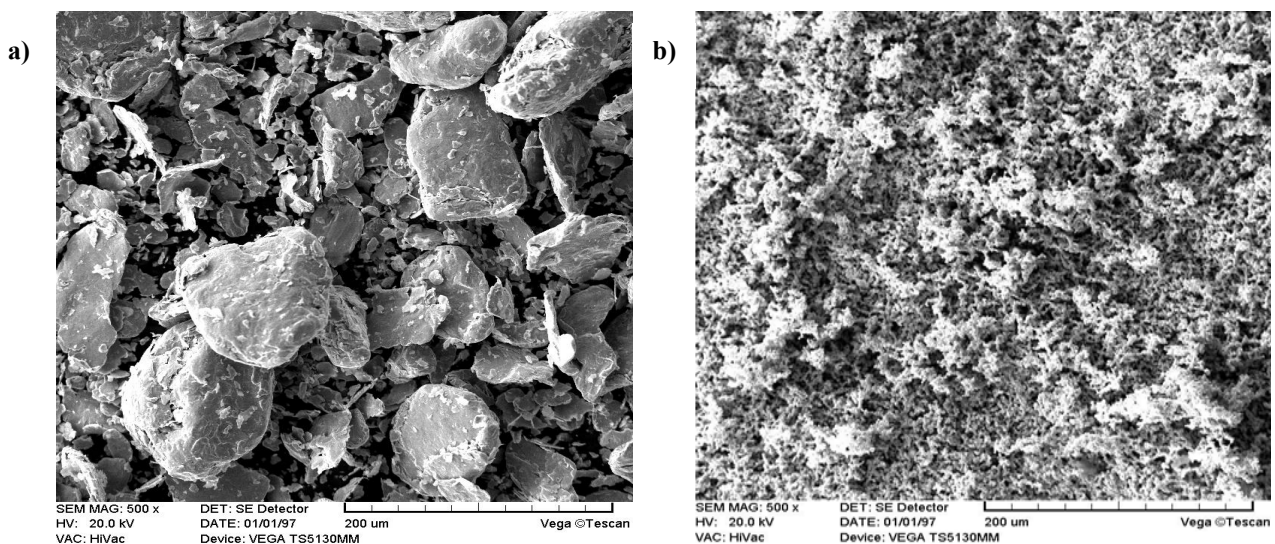


Fig. 7. SEM micrographs of the as-received powders: a) spherical iron powder and b) rod-like nickel powder.

Finally, after 96 hours of prolonged milling (Fig. 8f), the particle size distribution becomes highly uniform, and the particles achieve a fine, spherical morphology with a minimum average particle size of approximately 10 μm . At this stage, a dynamic equilibrium between the

competing processes of cold welding and particle fracture is established, representing the steady-state condition of mechanical alloying beyond which no significant alterations in particle size are observed (Fig. 9).

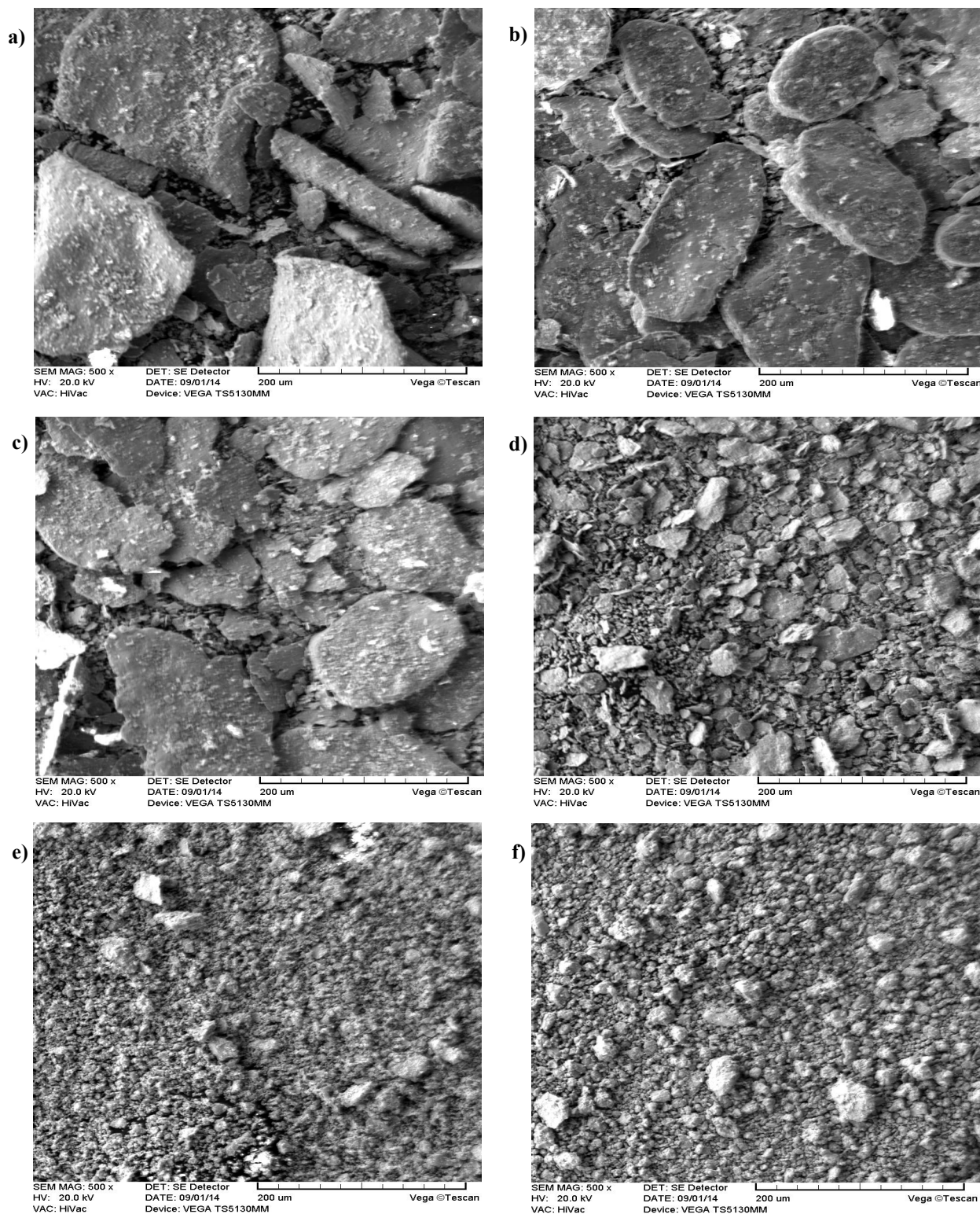


Fig. 8. SEM micrographs of $\text{Fe}_{30}\text{Ni}_{50}$ powders at different milling stages: a) unmilled large agglomerates, b) 4 h, c) 8 h, d) 32 h, e) 48 h, and f) 96 h, showing the morphological transition to fine spherical particles.

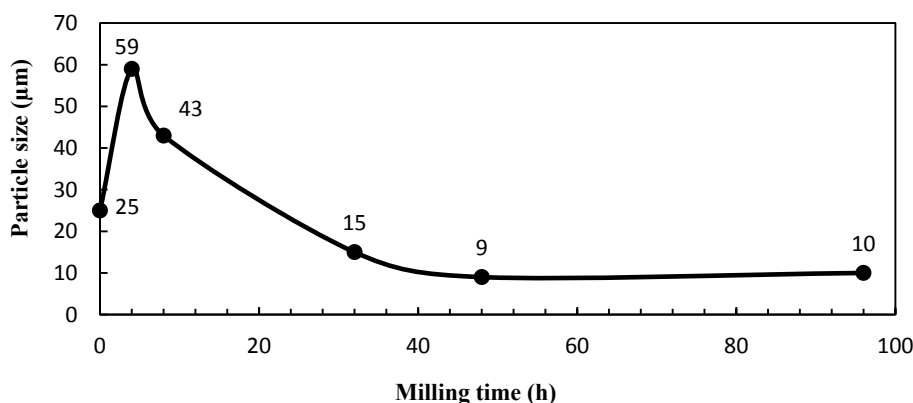


Fig. 9. Variation of average particle size as a function of milling time for the Fe₅₀Ni₅₀ powder mixture.

4. Conclusions

High-energy mechanical alloying of elemental iron and nickel powders at 350 rpm with a 20:1 ball-to-powder ratio successfully yielded a nanostructured γ -phase solid solution with an fcc crystal structure after 96 hours of processing. Prolonged milling induced severe plastic deformation, driving a dramatic reduction in crystallite size from an initial 65 nm down to 12 nm, accompanied by a continuous increase in internal lattice strain up to 0.58%. Furthermore, the lattice parameter expanded from 0.352 nm to 0.357 nm as a direct consequence of iron atom diffusion into the nickel lattice and the high-density accumulation of crystal defects and vacancies. Morphologically, the powder particles evolved progressively from initial spherical and rod-like geometries into flattened flakes via cold welding during intermediate stages, ultimately transforming into refined, highly uniform spherical particles with an average size of approximately 10 μm upon reaching a steady-state balance between fracture and cold-welding mechanisms at the end of the 96-hour milling period.

CRediT authorship contribution statement

Mohsen Abaei: Conceptualization, Methodology, Investigation, Writing – original draft, Writing – review & editing

Data availability

The data underlying this article will be shared on reasonable request to the corresponding author.

Declaration of competing interest

The authors declare no competing interests.

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Declaration of AI-assisted technologies in the writing process

During the preparation of this manuscript, the author used ChatGPT and Gemini to improve the manuscript's organization, grammatical

accuracy, and overall readability. All AI-assisted revisions were carefully reviewed, verified, and, where necessary, modified by the author. The author takes full responsibility for the content of the manuscript, including the accuracy of the data, the validity of the analyses, and the conclusions presented.

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