Original Article

Microstructure and magnetic properties of amorphous/nanocrystalline Ti50Fe50 alloys prepared by mechanical alloying

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\textbf{A B S T R A C T}

The effect of milling time on the microstructure evolution and soft magnetic properties of Ti50Fe50 alloy produced by mechanical alloying (MA) has been investigated. With the increase of milling time, the morphologies of Ti50Fe50 alloys experience a transition from lamellar structure to spherical structure, the α-Fe(Ti) solid solution containing a supersaturated Ti is formed, the grain sizes of Fe and Ti are decreased to nanoscale and their internal microstrains are increased. When the milling time is increased to 20 h, the nanocrystalline TiFe intermetallic phase appears and the Ti-rich amorphous phase is formed along with the complete disappearance of crystal Ti phase. Additionally, the saturation magnetization (M\textsubscript{s}) of the milled Ti50Fe50 alloys is reduced gradually, while the coercivity (H\textsubscript{c}) exhibits an increasing trend. This is attributed to the combination reactions of nanocrystalline, microstrain, grain boundaries, paramagnetic Ti-rich amorphous, paramagnetic nanocrystalline TiFe and ferromagnetic α-Fe(Ti) phases. The magnetic hysteresis loops, the field cooled (FC) and zero field cooled (ZFC) curves confirm that the amorphous/nanocrystalline Ti50Fe50 alloys produced by MA can serve as excellent soft magnetic materials.

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1. Introduction

Due to unique long-range atomic disorder and short-range atomic order, amorphous alloys called as metallic glasses exhibit excellent magnetic properties [1,2], good wear and corrosion resistance [3,4], high strength and super hardness [5]. Especially, since Chen et al. [6] used rapid continuous casting roller to prepare lots of amorphous ribbons which were

formally named as “metallic glass”, numerous researchers began to produce different metallic glasses by various methods. For instance, Botta et al. [3] prepared Fe-based amorphous ribbons by melt spinning and studied their corrosion resistance. The results showed that the presence of Cr in those ribbons resulted in a stable passive film which could improve the corrosion resistance. Shkodich et al. [7] produced Cu\textsubscript{63}Ti\textsubscript{50} alloy by high-energy ball milling and found that the alloy was composed of amorphous and nanocrystalline phases.

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Ti-based amorphous alloys with good mechanical properties [8], high specific strength [9,10], good glass forming ability (GFA) [11], high corrosion resistance, excellent biomedical compatibility [12,13] and hydrogen storage [14,15] can serve as promising structural materials and light-weight materials. For instance, Oak et al. [16] prepared Ti-based amorphous ribbons by melt spinning and found that the Ti-based amorphous alloy exhibited better bend ductility, higher strength as well as higher corrosion resistance than pure Ti and Ti–6Al–4V alloy. Deng et al. [17] prepared Ti47Cu28Zr7.5Fe2.5Sn1Ag2 alloy by selective laser melting (SLM) and found that the amorphous alloy with high density and strength can be obtained by the optimal processing parameters.

Mechanical alloying (MA) is considered as an advanced technique to induce solid-state reactions and therefore, can be used to produce a variety of nanostructures, metastable crystallines, quasicrystalline phases and amorphous alloys [18,19]. Compared to Fe-based or Ti-based amorphous alloys produced by melt spinning and casting, amorphous/nanocrystalline Ti–Fe alloys produced by MA have attracted significant attention. This is because mechanical alloying of Fe and Ti powder mixtures can induce the formation of amorphous or nanocrystalline TiFe and TiFe2 intermetallics [20]. Especially, the formation of amorphous or nanocrystalline TiFe phase by MA can exhibit favorable properties, such as the rapid kinetics for absorption/desorption and the high hydrogen absorbing ability with hydrogen atom (H) to metal atom (M) ratio H/M ~ 1 near ambient conditions at low cost [21]. Although it is believed that the TiFe phase has no magnetic properties [22], the mechanisms of microstructure evolution and magnetic of Ti–Fe alloys during MA has rarely dealt with. In this paper, the amorphous/nanocrystalline Ti50Fe50 alloys are prepared by mechanical alloying, the effect of milling time on the microstructure evolution and soft magnetic properties of Ti50Fe50 alloys are investigated.

2. Experimental

The raw mixture materials consisted of pure Fe powder (99.99%, particle size ~20 µm) and pure Ti powder (99.9%, particle size ~60 µm), whose morphologies were shown in Fig. 1. The atomic ratio of Ti to Fe was 1:1 in the raw mixture materials. Mechanical alloying was implemented with high-energy planetary ball milling at high purity argon atmosphere. The weight ratio of ball to powder was 10:1. The rotating speed of main disk (Ω) was 450 rpm and the milling time was in the range of 5–20 h. After each milling for 30 min, the operation was stopped for 10 min to avoid temperature rise in the milling vial.

The particle size distributions were measured by a laser scattering particle size analyzer (HORIBA LA-300, Japan). X-ray diffraction (XRD) (D/MAX-2500, Japan, Cu Kα radiation, 40 kV, 40 mA) was used to identify the phase constituents in the milled powders. The grain size and microstrain of the milled powders were calculated by employing Rietveld refinements yielding crystallographic data. The calculation formula can be described by Williamson–Hall equation [23]:

\[(\beta_1^2 - \beta_2^2)^{1/2} \cdot \cos \theta = 2\varepsilon \cdot \sin \theta + \frac{0.9\lambda}{D}\]

where \(\beta_1\) is the measured width, \(\beta_2\) is the width at half-maximum of the Si powder peaks used for calibration, \(\theta\) is the Bragg angle, \(\varepsilon\) is the internal microstrain, \(\lambda\) is the wavelength of the X-ray and \(D\) is the grain size. The microstructure of the unmilled and milled powders was characterized by scanning electron microscope (SEM, FEI Nova Nano SEM 400, Holland) equipped with X-ray energy dispersive spectrometer (EDS).

The thermal stability of the milled powders was executed by differential scanning calorimetry (DSC, SDT Q600, USA) with a heating rate of 20°C/min under argon atmosphere at 300–1473 K. The magnetic properties were examined repeatedly using a superconducting quantum interference magnetometer (SQUID, Quantum Design, USA) with a maximum applied of 20 kOe at room temperature. For zero field cooled (ZFC) and field cooled (FC) measurements, the samples were initially cooled to 2 K from 400 K in zero magnetic field and a field of 10 kOe, respectively.

3. Results

Fig. 2a shows the particle size distributions of Ti50Fe50 powders after the different milling time (5, 10, 15 and 20 h). At the early
milling time (5 h), the distribution range of the particle size is relatively larger and the average particle size is ~130 μm. When the milling time is increased to 15 h, the average particle size is decreased to ~51 μm. Especially, the average particle size can be reduced to ~32 μm significantly when the milling time is prolonged to 20 h (Fig. 2a). Moreover, the median particle diameter (D_{50}) and the specific surface of powders under the different milling time are shown in Fig. 2b. It is apparent that the median particle diameter D_{50} after milling for 5 h is the largest (~121 μm). With the increase of milling time, the D_{50} is decreased continuously, whereas the specific surface of powders is increased linearly, which is consistent with the result of particle size.

Fig. 3 shows the SEM images of Ti_{50}Fe_{50} powders after MA. At the early stage of milling, the primary powders are flattened and agglomerated severely (Fig. 3a and b) due to plastic deformation [21]. The average particle sizes are ~5.2 and ~1.7 times larger than those of pure Fe and Ti powders, respectively. With the increasing of milling time, the morphologies of Ti_{50}Fe_{50} powders experience a transition from the irregular shape to the spheroidal shape (Fig. 3c–e), showing that fracturing is the dominant mechanism. After milling for 20 h, large amounts of smaller powder particles are separated from larger powder particles and present a spherical shape (Fig. 3g and h), showing that cold welding and fracturing are the dominant mechanisms [24].

Fig. 4 shows the XRD patterns of Ti_{50}Fe_{50} powders after MA. The primary mixture powders are composed of relatively stronger diffraction peaks of body-centered-cubic (bcc) α-Fe, and many relative weaker peaks of hexagonal Ti. When the milling time is 5 h, the intensities of all diffraction peaks are reduced and their widths are increased. When the milling time is increased to 10 h and 15 h, some Ti peaks disappear, the diffraction widths of α-Fe phase are broadened and the diffraction patterns are coarsened, indicating the reduction of crystalline size and the formation of solid solution α-Fe(Ti). Eckert et al. [25] also found that the solid solution of Ti in α-Fe was ~20 at. % in Ti–Fe system after MA. Especially, when the milling time is further increased to 20 h, the diffraction peaks of hexagonal Ti phase disappear completely, the α-Fe(Ti) peaks are broadened significantly and the (1 1 0) plane of crystalline intermetallic TiFe phase with CsCl-structure is formed based on the result of EDS analysis (Table 1). Disappearance of Ti peaks during MA suggests that Ti is disordered rapidly and changes into an amorphous phase, which has been reported in Ref. [26].

Fig. 5 shows the grain size and internal microstrain of Fe and Ti phases after the different milling time. When the milling time increases from 5 h to 15 h, the grain sizes of α-Fe and Ti are decreased rapidly from ~379 nm to ~12 nm and from ~40 nm to ~8 nm, respectively. Especially, after milling for 20 h, the grain sizes of α-Fe and TiFe phases are only ~10 nm and ~5 nm, respectively. Furthermore, the internal microstrains of α-Fe and Ti phases are proportional with the milling time, but are inversely proportional with the grain sizes. The internal microstrain of Ti (~3.08%) is much larger than that of α-Fe (0.23%) after milling for 15 h. However, when the milling time is further increased to 20 h, the internal microstrain of α-Fe slightly increases to ~1.25%, which is still smaller than that of nanocrystalline TiFe phase (~5.03%).

Fig. 6a shows the magnetic hysteresis loops of Ti_{50}Fe_{50} powders prepared by MA. The saturation magnetization (M_s) of Ti_{50}Fe_{50} powder after milling for 5 h is ~87.9 emu/g. However, the saturation magnetization of Ti_{50}Fe_{50} powders is decreased dramatically with the increase of milling time. Especially, the M_s after milling for 20 h is decreased to ~34.6 emu/g, which is less than that of pure Fe (176 emu/g). Moreover, the coercivity (H_c) of Ti_{50}Fe_{50} powder after milling for 15 h can reach the maximum value of ~143.1 Oe, whereas the H_c is decreased abruptly to ~112.2 Oe when the milling time is increased to 20 h (Fig. 6b).

4. Discussion

Although mechanical alloying as a very versatile technique can produce nanocrystalline phases, amorphous alloys, intermetallic compounds and supersaturated solid solutions [27,28], the complicated mechanisms usually include solid-state interdiffusion, chemical reactions and amorphization [19,28]. At the early milling stage (5 h), large amounts of powder particles are captured by adjacent balls or vial wall, which results in impact, cold welding and plastic deformation of powder particles. Moreover, the newly generated surfaces of powder particles can induce the agglomeration of milled powder particles and generate larger powder particles during
MA. It is well-known that the Fe and Ti are ductile, leading to powder particles more likely to get together and an increase in particle size at the early stage of milling \cite{28,29}. Therefore, the powder particles are coarsened and flattened to form the laminar or irregular pancake structures (Fig. 3a and b). With the accumulation of milling time, the powder particles are consolidated and fractured into fragile flakes because the Ti$_{50}$Fe$_{50}$ powder particles suffer from severe plastic
deformation when balls and powder particles collide with each other. Fracturing is a more deterministic factor than cold welding at this stage of milling, leading to a decrease in powder particle size (Fig. 3c and d). When the milling time is increased from 15 h to 20 h, the particle size is decreased from \(~48\mu m\) to \(~32\mu m\) and the structure of powder particles is approximately spherical shape (Fig. 3e and g). It can be explained by the following reasons. Firstly, a balance of cold welding and fracturing is formed gradually with the prolonged milling time. Secondly, larger powder particles are stripped from multidimensional lamellar structure, while smaller powder particles are integrated into homogeneous framework (Fig. 3f–h). Finally, the minimization of Gibbs free energy helps powder particles present spherical structure during MA. Thus, the higher the milling time is, the more homogeneous the particle size is [29].

The microstructure evolution of milled powder particles has a close relationship with energy transferred from balls and vial to powder particles and temperature rise during MA.

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**Table 1 – EDS analysis of the amorphous/nanocrystalline Ti50Fe50 alloys produced by MA in Fig. 3.**

<table>
<thead>
<tr>
<th>Milling time (h)</th>
<th>Elements (at.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe</td>
</tr>
<tr>
<td>5</td>
<td>53.0</td>
</tr>
<tr>
<td>10</td>
<td>50.9</td>
</tr>
<tr>
<td>15</td>
<td>51.5</td>
</tr>
<tr>
<td>20</td>
<td>52.2</td>
</tr>
</tbody>
</table>

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**Fig. 4 – XRD patterns of Ti50Fe50 alloys after different milling time.**

**Fig. 5 – (a) Grains size and (b) internal microstrain of Ti50Fe50 alloys after different milling time.**

**Fig. 6 – (a) Magnetic hysteresis loops and (b) saturation magnetization and coercivity of Ti50Fe50 alloys after different milling time.**
(30). This is because the high rotation speed of main disk can produce large kinetic energy, which can in turn transform into thermal energy of powder particles and therefore, the temperature in the vial is raised during mechanical alloying. Generally, the temperature rise can accelerate the diffusion behavior of atoms by severe plastic deformation, which can produce large amounts of defects, such as vacancies, stacking faults, grain boundaries and dislocation densities (18,31). Moreover, the formation of grain boundaries can also induce more Ti atoms to diffuse into Fe-rich region, resulting in a supersaturated α-Fe(Ti) solid solution with ~25 at. % of Ti and the shift of α-Fe peaks to small diffraction angle. This is because the radius of Ti (0.147 nm) is much larger than that of Fe (0.126 nm) [4], resulting in lattice distortion of α-Fe. Furthermore, based on the coherent polycrystalline model, the volume fraction of grain boundaries \( f_{gb} \) is estimated by the following equation [38]:

\[
f_{gb} = 1 - \left(\frac{D - d}{D}\right)^3
\]  

(2)

where \( D \) is the grain size and \( d \) is the effective grain boundary thickness. The grain refinement causes an increase in volume fraction of grain boundaries, whereas the decrease of grain size is dependent on defects caused by deformation such as dislocation densities during MA. Generally, the dislocation densities \( \rho_D \) can be calculated as follows [32]:

\[
\rho_D = 2 \cdot \sqrt{\frac{\pi}{\pi}} \cdot \sigma(1/2) \frac{b}{D}
\]  

(3)

where \( b \) is the Burgers vector of dislocations, and \( \sigma \) is the microstrain. The microstrains of Fe and Ti grains are increased with the increase of milling time (Fig. 5b), which is attributed to the interdiffusion between Ti and Fe and the severe plastic deformation of powder particles during MA. According to the Eq. (3), with the increase of milling time, the grain sizes of Fe and Ti are decreased, whereas their microstrains are increased (Fig. 5b), indicating that the dislocation densities of Fe and Ti nanocrystallines are increased.

The microstructure of amorphous/nanocrystalline Ti50Fe50 alloys is also dependent on the temperature rise due to higher energy and exothermic processes during MA. Generally, a relative higher temperature can promote the formation of intermetallics due to relative higher atomic migration, whereas a relative lower temperature can induce amorphous or nanocrystalline phases due to large amounts of defects [28]. Therefore, the formation of the nanocrystalline TiFe phase after milling for 20 h indicates that the reaction between Fe and Ti occurs as follows:

\[
Ti + Fe \rightarrow TiFe
\]  

(4)

Generally, the adiabatic temperature rise during mechanical alloying can be estimated to be ~600 K [33]. According to the principle of minimization of the standard Gibbs free energy, using thermochemical data from Ref. [34], the Gibbs function increments of the probable reaction for the formation of the TiFe phase at ~600 K is calculated to be completely negative (~−54 kJ/mol), indicating that the reaction can take place spontaneously during MA. Moreover, the effective diffusion coefficient \( Q_{Ti} \) of Ti in Fe can be given as follows [35]:

\[
m_{Ti} = \frac{2}{\sqrt{\pi}} \cdot X_S \cdot \sqrt{Q_{Ti} \cdot t}
\]  

(5)

where \( m_{Ti} \) is the amount of Ti diffused into Fe, \( X_S \) is the concentration of Ti on the surface of Fe particles, \( S \) is the surface area of Ti powder particles and \( t \) is the time. The two separated layers of Ti and Fe are formed after milling for 5 h (Fig. 3a and b), but the interactions of these layers are produced after milling for 10–20 h (Fig. 3c–h). Moreover, it can also be seen that the content of Ti is decreased but the content of Fe is increased with the increase of milling time in Table 1. Additionally, the lattice parameter of Fe (0.2866 nm) is increased to 0.2889 nm after milling for 20 h, whereas the lattice parameters of Ti (\( a = 0.2951 \) nm, \( c = 0.4684 \) nm) is decreased (\( a = 0.2926 \) nm, \( c = 0.4670 \) nm) after milling for 15 h. These results further confirm that mass transfer process occurs during MA [24,35].

The formation of Ti-rich amorphous phase can be explained by the following reasons. Firstly, the mixing enthalpy of Fe–Ti system is ~−17 kJ/mol and there is a relatively large difference in atom size of Fe and Ti, which is sufficient for amorphization [25]. Secondly, according to Eqs. (2) and (3), large amounts of defects, such as dislocation densities \( \rho_D \), grain boundaries \( f_{gb} \) can induce more microstrains \( T_g \) in the powder particles, which can facilitate the diffusion of Ti into Fe. Thirdly, the refined microstructure can further decrease the diffusion distances. The temperature rise during MA can also accelerate the diffusion behavior and lead to easier amorphization [28]. Finally, the Gibbs free energy of amorphous phase is lower than that of crystallite phase [26,36]. Moreover, an endothermic peak and an exothermic peak are located in 973–990 K and in 973–1053 K (Fig. 7). The appearances of glass transformation temperature \( T_g \) (~893 K) and crystallization temperature \( T_c \) (~981 K) in the DSC curve, which further confirm that the Ti-rich amorphous phase is formed in the milled Ti50Fe50 powder. The similar results have been reported by Chu et al. [4] and Macri et al. [37].

Generally, the saturation magnetization \( M_s \) is determined by phase constituents, grains size and defects caused by those variables in Eqs. (2), (3) and (5). With the increase of milling time, the grains size \( D \) is decreased, the internal microstrain \( \sigma \) and the grain boundaries \( f_{gb} \) are increased, leading to a change in magnetic moment. Moreover, many Fe atoms are stuck on the Ti-rich region to form the Ti-rich amorphous phase and large amounts of Ti dissolve in Fe to form the
supersaturated α-Fe(Ti) solid solution, resulting in a decrease in magnetic α-Fe. Meanwhile, with the prolonged milling time, the amounts of the supersaturated α-Fe(Ti) solid solution are further decreased and the paramagnetic nanocrystalline TiFe is formed (Fig. 4). As a result, the Ms of the milled Ti50Fe50 powders is reduced with the increase of milling time (Fig. 6a).

Moreover, the coercivity of the milled Ti50Fe50 powders increases basically with the increase of milling time (Fig. 6b). This is because the increase of coercivity is mainly affected by powder particle size, grains size, internal microstrain, and grain boundaries produced during MA [18,38]. Generally, for a soft magnetic random anisotropy system with dislocation densities, the effective anisotropy constant $k_{eff}$ can be calculated as follows [39]:

$$k_{eff} = \frac{\sqrt{k_{s,m}^2 + (k_{a,ms})^2 + (k_1)^2}}{\sqrt{S_z}}$$

where $k_1$ is the magnetocrystalline anisotropy of the material, $S_z$ is the average particle size, $k_{s,m}$ and $k_{a,ms}$ are the average long-range and short-range magneto-elastic anisotropies, respectively. According to Eq. (6), the coercivity is increased as the decreasing of average particle size. When the grains size $D$ is less than the magnetic exchange length $L_{ex}$ ($L_{ex} = \sqrt{\frac{T}{k_1}}$), the coercivity $H_c$ can be given as follows [38]:

$$H_c = 3 \cdot \sqrt{\frac{k \cdot T_C \cdot k_1}{a \cdot M_s}} \frac{1}{D}$$

(7)

where $F$ is the exchange stiffness constant, $k$ is Boltzman constant, $T_C$ is Curie temperature and $a$ is lattice parameter. However, when the grain size $D$ is higher than the magnetic exchange length $L_{ex}$, the $H_c$ can be described as follows [40]:

$$H_c = 3 \cdot \sqrt{\frac{p_e \cdot k_1^2 \cdot D^6}{\mu_0 \cdot M_s \cdot F}}$$

(8)

where $F$ is the exchange stiffness constant, $p_e$ is a constant of the order of unity and $\mu_0$ is the permeability of free space. According to Eqs. (7) and (8), when the grain size $D$ (~10 nm) is larger than the $L_{ex}$, the coercivity $H_c$ is increased because the increase of the volume of grain boundaries can impede the domain walls movement. When the grain size $D$ (~10 nm) is lower than the $L_{ex}$, the coercivity $H_c$ is increased due to a decrease in the $M_s$. In addition, the coercivity $M_s$ reaches the maximum value after milling for 15 h (Fig. 6b), which may be attributed to the abrupt increase of microstrain (Fig. 5b) [38].

To further investigate the magnetic properties of the amorphous/nanocrystalline Ti50Fe50 alloys, the temperature dependence of magnetization curves after milling for 20 h are shown in Fig. 8. The splitting between field cooled (FC) and zero field cooled (ZFC) curves is clearly seen with the decrease of temperature. However, the abrupt change of magnetization is not found in the ZFC and FC curves at 2-400 K. The maximum magnetization is ~46.0 emu/g at ~2 K, which might be attributed to the ferromagnetic α-Fe(Ti) phase [40]. Especially, due to severe deformation during MA, the appearance of fine grains, microstrain and defects can further reinforce the randomness of Ti-rich amorphous phase. Therefore, the competition of paramagnetic Ti-rich amorphous phase, paramagnetic nanocrystalline TiFe phase and ferromagnetic α-Fe(Ti) phase leads to the occurrence of magnetic inhomogeneity. In other words, a high magnetization at room or low temperature can help amorphous/nanocrystalline Ti50Fe50 alloys sever as excellent soft magnetic materials.

5. Conclusions

(1) With the increase of milling time, the laminar or irregular pancake structures of primary Ti–Fe mixture powders are fractured, leading to the formation of near-sphere structures. Furthermore, the grain sizes of Ti50Fe50 alloys are reduced gradually. When the milling time is increased to 20 h, the nanocrystalline TiFe intermetallic phase with CsCl-structure is produced, the solid solution α-Fe(Ti) containing a supersaturated Ti is also formed, whereas the Ti-rich amorphous phase appears due to appearance of hexagonal Ti phase during MA.

(2) The saturated magnetization ($M_s$) is decreased and the coercivity ($H_c$) is increased, due to large amounts of grain boundaries, internal microstrain, dislocations, paramagnetic Ti-rich amorphous phase and nanocrystalline TiFe phase. Interestingly, the $M_s$ of the milled Ti50Fe50 alloy is ~34.6 emu/g and the $H_c$ is ~112.2 Oe when the milling time is increased to 20 h. The temperature dependence of magnetization curves also confirm that the amorphous/nanocrystalline TiFe alloys prepared by MA can exhibit good soft magnetic properties.

Conflicts of interest

The authors declare no conflicts of interest.

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