

Mechanical alloying and phase transformation in Fe-Si alloy

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Abstract: The Fe-Si mechanical alloying and its transformation are investigated to evaluate whether mechanical alloying is a useful process for producing Fe-Si alloy. The mechanical alloying process of Fe-Si powders is studied by SEM(scanning electron microscopy), EDS(energy dispersive spectrometer) and XRD(X-ray diffraction). The results show that the ball milling process first makes tough Fe powder a lump structure and brittle Si powder a small particle, and then as the mill power increases, the tough powder of iron with a lamellar structure forms and the Si particles lies on or between the Fe lamellas. Finally, the Fe and Si powders are mechanically alloyed through atom diffusion. So the Fe and Si powders can be alloyed by 15 h ball milling at a speed of 400 r/min and with a ball-to-powder ratio of 40 : 1. After heating at 1 243 K for 1 h, the milled powders transform to α -FeSi₂, and after heating at 1 243 K for 1 h, then cooling to 1 073 K for 1 h, the milled powders transform to β -FeSi₂. Therefore, the monophase α -FeSi₂ or β -FeSi₂ can be obtained by heat treatment of mechanically alloyed Fe-Si powders.

Key words: Fe-Si alloy; mechanical alloying; phase transformation; α -FeSi₂; β -FeSi₂

Owing to the combination of a low magnetostriction and a high saturation magnetization, Fe-Si alloys have been attracting much attention^[1-6]. The crystalline alloys of the Fe-Si system are suitable for the construction of transformers. Especially, the magnetic properties of β -FeSi₂ have semi-conducting transport properties. β -FeSi₂ has received considerable attention as a very attractive material for light detectors, photovoltaic applications, and thermoelectric applications^[7-11]. Unfortunately, due to its slow solidification rate, it usually takes a long time to obtain a simplex β -FeSi₂ with a coarse eutectic structure in the conventional casting process. The amorphous alloys of the Fe-Si system allow the control of magnetic anisotropy. Therefore, the high effective quenching rate of the melt spinning process is necessary for amorphizing the Fe-Si alloy^[12]. In this sense, mechanical alloying seems to be a promising method for producing an Fe-Si amorphous phase.

A considerable number of studies on various alloy systems by mechanical alloying have been carried out. It has been reported that metastable phases, amorphous phases, nanocrystalline phases and supersaturated solid solutions can be formed by milling elemental powders. To study whether mechanical alloying is a useful process for producing an Fe-Si alloy, the Fe-Si mechanical alloying and its transformation are investigated.

1 Experimental

Elemental powders of Fe and Si, used as the starting materials, were mixed together to form a nominal composition of Fe₆₇Si₃₃. The powders were loaded and sealed in a cylindrical stainless-steel container under an Ar atmosphere, and eight drops of methanol were used to prevent excessive welding in the chamber. The mechanical alloying was performed on a QM-BP planetary ball mill. The ball-to-powder ratio(BPR) was 40 : 1 or 80 : 1, and the speed of the ball mill was 300, 400 or 450 r/min. The milled powders were pressed on a CMT5350 press and heated in a VAF-5 annealing furnace. The scanning electron microanalyzing was obtained by SIRION 200 scanning electron microscopy (SEM). The qualitative analyzing for the milled powder was done by an EDAX energy dispersion spectrometer (EDS). The X-ray diffraction patterns were obtained using CuK α radiation with a graphite monochromator by an XD-3A X-ray diffractometer(XRD).

2 Results

In order to analyze the mechanical alloying process of the Fe-Si powders, SEM and EDS are used. The analysis results for Fe-Si powders are shown in Figs. 1 to 4. Fig. 1 shows that the SEM/EDS results for the powder sample milled for 5 h at a speed of 300 r/min (BPR 40 : 1). From the SEM metallograph and the EDS results, it can be seen that the powders are lumps and particles. The lumps are composed of Fe and the particles are composed of Si, so in this case, the powders are not alloyed. Fig. 2 shows analysis results for the powder sample milled for 5 h at a speed of 400 r/min(BPR 40 : 1). From the SEM metallograph, it can be seen that as the milling speed increases, the powders become smaller. The EDS results show that the powder is composed of Fe and Si, so the powder might be partly alloyed. The powder is magnified highly to observe the details as shown in Fig. 3, where the small particles lie on the layered particles. As the milling time increases from 5 to 10 h, the powders become smaller and the layered particles become smaller and thinner(see Fig. 4).

In order to study the mechanical alloying of the Fe-Si powders further, the X-ray diffractometer is used. Fig. 5 shows the X-ray diffraction pattern of the Fe-Si powders. It can be seen from Fig. 5(a) that when the powders are milled for 5 h at the speed of 300 r/min(BPR 40 : 1), the peaks of the elements Fe and Si can be observed, so in this condition the powders are not alloyed. As the milling speed reaches 400 r/min, the intensity of the elements Fe and Si peaks decreases and the peaks of α -FeSi₂, β -FeSi₂ and ϵ -FeSi are observed(see Fig. 5(b)). When the speed of the mill increases from 300 to 400 r/min, the Fe-Si powders are alloyed partly. From Fig. 5(c), it can be seen that as the milling time increases to 15 h, the peaks of the elements Fe and

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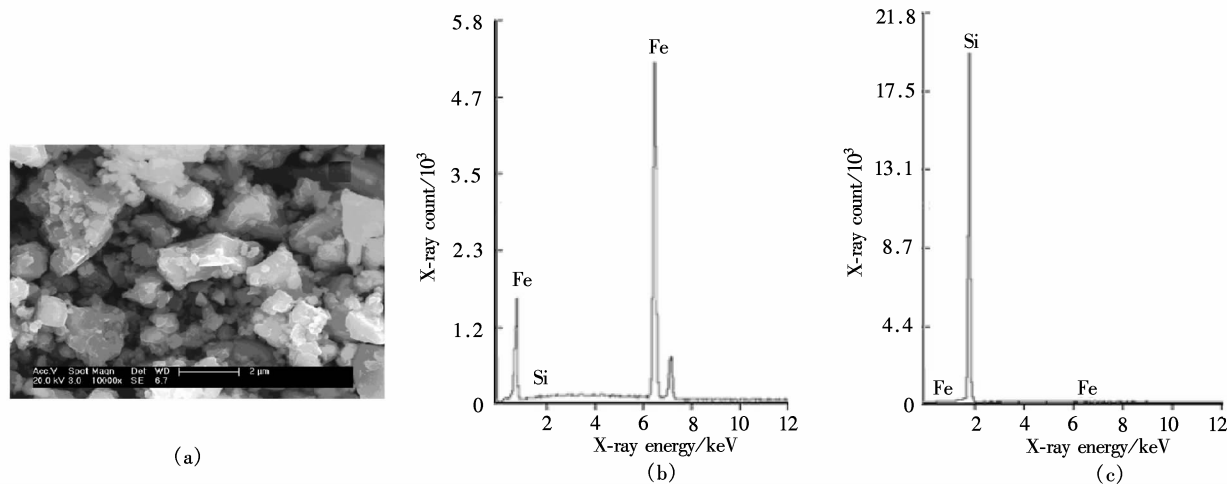


Fig. 1 SEM/EDS for the powder milled for 5 h at a speed of 300 r/min (BPR 40 : 1). (a) SEM metallograph; (b) EDS of the lump powder in (a); (c) EDS of the particle powder in (a)

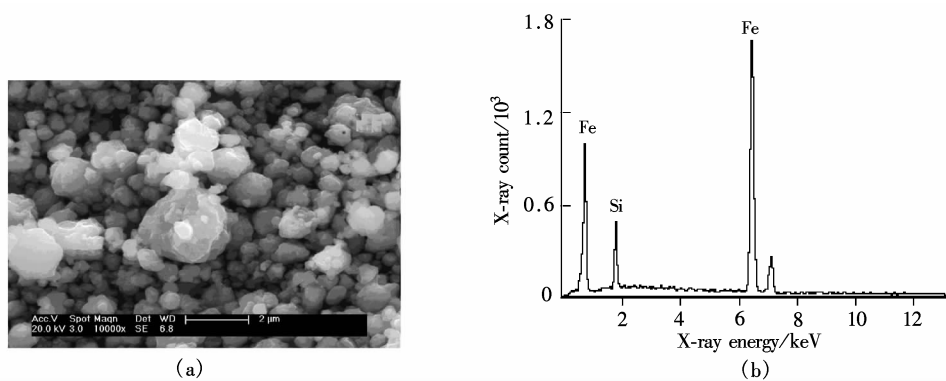


Fig. 2 SEM/EDS for the powder milled for 5 h at a speed of 400 r/min (BPR 40 : 1). (a) SEM metallograph; (b) EDS of the powder in (a)

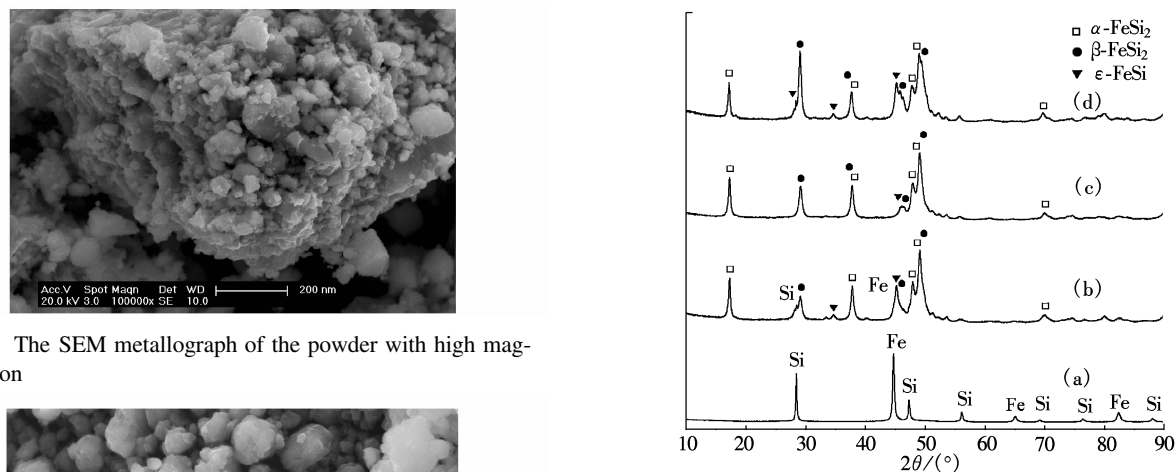


Fig. 3 The SEM metallograph of the powder with high magnification

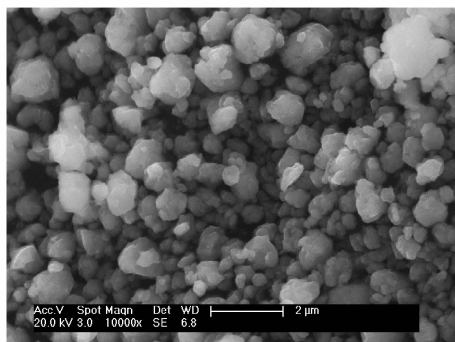


Fig. 4 The SEM metallograph of the powder milled in Fig. 2(a) for 15 h at a speed of 400 r/min (BPR 40 : 1)

Fig. 5 The X-ray diffraction analysis results of the powder samples for Fe-Si milled. (a) Milled for 5 h at 300 r/min (BPR 40 : 1); (b) Milled for 5 h at 400 r/min (BPR 40 : 1); (c) Milled for 15 h at 400 r/min (BPR 40 : 1); (d) Milled for 15 h at 450 r/min (BPR 80 : 1)

Si disappear and the intensity of the peaks of β -FeSi₂ increases, and the peaks of α -FeSi₂ and ϵ -FeSi can also be seen. Therefore, as the milling time increases from 5 to 15 h at 400 r/min (BPR 40 : 1), all the powders are fairly alloyed. Fig. 5(d) shows the X-ray diffraction analysis results for the powders milled for 15 h at a speed of 450 r/min (BPR 80 : 1). Compared with the XRD pattern of the powders milled for 15 h at a speed of 400 r/min (BPR 40 : 1),

the intensity of α -FeSi₂ peaks decreases, while the intensity of β -FeSi₂ and ε -FeSi peaks increases. Therefore, as the milling power increases, the powders are mechanically alloyed and the phase transformation occurs.

The alloyed Fe-Si powders are pressed to a pellet and then heated. Fig. 6 (a) shows the X-ray diffraction analysis results of the sample heated at 1 243 K for 1 h. It can be seen that only when the peaks of α -FeSi₂ are observed, the monophase α -FeSi₂ can be obtained. Fig. 6 (b) shows the X-ray diffraction analysis results of the sample heated to 1 243 K, cooled to 1073 K and then held for 1 h. It can be seen that only when the peaks of β -FeSi₂ are observed, can the monophase β -FeSi₂ be obtained.

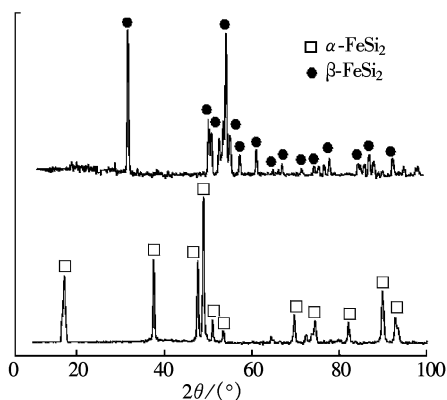


Fig. 6 The X-ray diffraction analysis results of the milled Fe-Si powder samples heated. (a) 1 243 K for 1 h; (b) 1 243 K for 1 h, and then 1 073 K for 1 h.

3 Discussion

Mechanical alloying is a novel solid alloying process. It is developed as a high-energy milling process for producing composite metallic powders with a fine, controlled structure from a mixture of pure elemental powders.

In this research, the mechanical alloying is performed in the ball mill. The Fe and the Si powder particles trapped by the colliding balls are subjected to severe mechanical deformation, cold weld and fracture. In this case, the ball milling process first produces tough powders of Fe with a lump structure and brittle powders of Si of small size. As a result of the ball collision, the tough powders of iron with a lamellar structure form; the brittle powders of silicon turn into fine particles and the Si particles lie on or between the Fe lamellas. So the nuclei of the Fe-Si alloy form between the Si particles and the Fe lamellas. The mechanical alloying of Fe-Si powders is completed by the diffusion of Fe and the Si atoms between the Fe lamellas and the Si particles. As the milling intensity increases, the Fe lamellas and Si particles refine and the size of the crystalline grain decreases. Thus, the new surface between the lamellas and the particles increases and the new grain boundaries increase. Further, the high intensity of the milling introduces severe plastic deformation into the particles, which generates many point defects and lattice defects (vacancies, interstitials and dislocations etc.). The large number of defects, the new surfaces between the lamellas and the particles and the new grain boundaries create many more paths for the diffusion of Fe and Si atoms and accelerate the dif-

fusion rate. Therefore, as the milling power increases, the true alloying and the phase transformation of Fe-Si powders occur. So the Fe-Si alloy composed of α -FeSi₂, β -FeSi₂ and ε -FeSi can be prepared by mechanical alloying, but no monophase Fe-Si alloy is obtained.

The metastable alloy is often obtained by mechanical alloying, so the designed composition of the Fe₆₇Si₃₃ alloy prepared by mechanical alloying is composed of α -FeSi₂, β -FeSi₂ and ε -FeSi. When the alloy is heated at 1 243 K for 1 h, the Fe and Si atoms in the alloy can sufficiently diffuse and the phase transformations occur. Then the monophase α -FeSi₂ is obtained. From the Fe-Si binary phase diagram, it can be seen that the α -FeSi₂ phase is a phase that only exists stably at high temperature, so the α -FeSi₂ phase is a metastable phase. The transformation from an α -FeSi₂ phase to a β -FeSi₂ phase depends on the atom diffusion through the β solid phase. Compared with the atom Fe, the size of the atom Si is not small enough to diffuse easily. The β phase is an intermediate phase, so it is difficult for the Fe and Si atoms to diffuse through the β solid phase. For the above reasons, the transformation from the α -FeSi₂ phase to the β -FeSi₂ phase must take a long time. Because the rate of the cooling from 1 243 K is rapid, the metastable phase of the α -FeSi₂ is kept at room temperature. When the anneal is held at 1 073 K for 1 h, there is an intrinsic thermodynamic driving force for the α -FeSi₂ phase to transform into the single β -FeSi₂ phase. Therefore, it transforms into the β -FeSi₂ phase.

4 Conclusions

The mechanical alloying of Fe, Si powders is investigated. SEM, EDS and XRD are used to analyze the mechanical alloying and phase transformation of the Fe-Si powders.

- 1) The Fe, Si mixed powders can be alloyed by milling for 15 h at a speed of 400 r/min (BPR 40 : 1);
- 2) The monophase α -FeSi₂ is obtained by treating the Fe-Si alloy at 1 243 K for 1 h;
- 3) The monophase β -FeSi₂ is obtained by heating the Fe-Si alloy to 1 243 K for 1 h, cooling to 1 073 K and then holding for 1 h.

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Fe-Si 合金的机械合金化及其相变研究

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摘要: 为了探讨机械合金化法制备 Fe-Si 合金的有效性, 研究了 Fe-Si 的机械合金化及其相变. 用扫描电子显微镜、X 射线能谱仪和 X 射线衍射仪研究了 Fe-Si 粉体的机械合金化过程, 结果表明: 球磨初期, 韧性的 Fe 粉体磨成块状, 脆性的 Si 粉体磨成小颗粒; 随着球磨强度的增加, 韧性的 Fe 粉体形成层片状而脆性的 Si 颗粒则分布在 Fe 的层片间或层片上; 最后, Fe-Si 粉体通过原子扩散实现机械合金化. 因此, Fe, Si 粉体在球磨机转速 400 r/min、球料比 40:1 的条件下, 球磨 15 h 可以合金化. 机械合金化后的 Fe-Si 合金经过 1 243 K 退火 1 h 可得到单相的 α -FeSi₂; 机械合金化后的 Fe-Si 合金经过 1 243 K 退火 1 h 再冷却到 1 073 K 保温 1 h 可得到单相的 β -FeSi₂. 因而, 单相的 α -FeSi₂ 或 β -FeSi₂ 可通过对机械合金化的 Fe-Si 合金进行热处理获得.

关键词: Fe-Si 合金; 机械合金化; 相变; α -FeSi₂; β -FeSi₂

中图分类号: TB34; TF124