Effects of Nb content on the microstructure and magnetic properties of Fe-6.5wt.%Si alloy

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The effects of adding different contents of Nb on microstructure and magnetic properties of Fe-6.5wt.%Si alloy were investigated. The results show that the addition of a suitable amount of Nb to Fe-6.5wt.%Si alloys has improved the ductility of the alloy by precipitating Nb-Fe-Si intermetallics. The precipitated Nb-Fe-Si particles have refined the grains and lowering the Si content in the matrix, the latter effect has resulted in the reduction of the ordered phases or the degree of long range order in the alloy. However, when the Nb content is 2.0wt.%, the numbers of precipitates are too high and starting to pin the movement of dislocations, which has an adverse effect on the processability of the alloy. Though the Fe-6.5Si-1.5Nb (wt.%) alloy showed better magnetic properties than the Fe-6.5Si-2.0Nb (wt.%) alloy, the addition of Nb would deteriorate the magnetic properties of Fe-6.5wt.%Si alloy. Thus, the addition of Nb should be less than 1.5 wt.% to balance the processability and magnetic properties of Fe-6.5wt.%Si alloy.

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1. Introduction

Soft magnetic materials, especially silicon steels, are widely used as core materials in transformers and motors. The magnetic properties of silicon steels are improved with the increase of Si content. When the Si content reaches 6.5wt.%, the silicon steel has the optimum combination of high magnetic permeability, low core loss, and near-zero magnetostriction [1,2]. However, when the silicon content is above 3.5wt.%, the room-temperature ductility of the steels drops significantly, and reaches to almost zero at about 6.5 wt.% Si. As a result, it is difficult to roll Fe-6.5wt.%Si steel into sheet form by conventional cold rolling [3].

In recent years, a number of methods have been developed to produce Fe-6.5wt.%Si alloy, such as Chemical Vapor Deposition (CVD) [4,5], Physical Vapor Deposition (PVD) [6], hot dipping and diffusion annealing [7], and rapid quenching [8,9] et al. Although all of these methods could be used to produce Fe-6.5wt.%Si alloy, they are all complex, time-consuming and uneconomical processes.

Therefore, the conventional rolling process is still the desirable method. In order to produce Fe-6.5wt.%Si sheet with conventional rolling process, the ductility of the alloy must be improved. A common approach to improve the ductility of Fe-6.5wt.%Si alloy is by alloying to either refine the grain size or to reduce the degree of the long-range order [10-12].

Turtelli et al [13] have studied the effect of Nb addition on the structural and magnetic properties of Fe–Si alloys, and found that Nb could prevent grain growth and increasing the micro-strain, the lattice constant, coercivity and the Curie temperature. It also demonstrated that the existence of a metastable hexagonal phase $NbFe_{2-x}Si_x$ in Fe-Si alloy after adding Nb. Miraghaei [14] studied the $Fe_{83.5}Si_{13.5}Nb_3$ (Fe-7.1wt.%Si-5.2wt.%Nb) nanocrystalline powders, and found that Nb prevents the grain growth during annealing but its presence in the Fe-Si alloys has adverse effects on the magnetic properties. Yang et al [15] indicated that the addition of micro-alloying Nb to Fe-6.5wt.%Si alloy could refine the grains and improve the compression strength. Others [16] reported that the addition of a small amount (≤1wt.%) of niobium could result in grain coarsening, while more Nb (≥1.5wt.%) addition would refine the grains. Therefore, the optimum amount of the Nb addition into Fe-6.5wt.%Si is still uncertain and controversial. Meanwhile, the effect of the Nb addition on the magnetic properties and the processability of the Fe-6.5wt.%Si alloy is still largely unknown.

In this paper, we have added different amounts of Nb into the Fe-6.5wt.%Si alloy and produced the Fe-6.5wt.%Si alloy sheet by conventional rolling process. The effects of Nb addition on the microstructure and magnetic properties of Fe-6.5wt.%Si alloy have been investigated, and the results have been discussed.

2. Experimental procedure

The alloys were produced by melting industrial purity of iron (99.5wt.% Fe), silicon (99.99wt.%) and niobium (99.9wt.%), and then refined and cast into ingots in a vacuum induction melt furnace at 1580~1600℃. Two Fe-6.5wt.%Si alloys were prepared containing either 1.5wt.%Nb or 2.0wt.%Nb. The as-cast ingots were forged into about 20mm thickness at approximately 1000~1150℃. The obtained slabs were then hot rolled to 0.8~1.0mm thickness at about 800~1000℃. The hot rolled sheets were finally warm rolled at a temperature about 450~750℃ to a thickness of 0.2~0.4mm.

Optical microscopy and scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS) were used to analyze the microstructure of the specimens. The core loss at 50Hz (1T) ($P_{10/50}$), 400 Hz (1T) ($P_{10/400}$) and the magnetic induction at 800 Am^{-1} (B_8) of the thin sheets produced were measured using an AC/DC magnetic measuring instrument (MPG-100D) from specimens with a size of 300 mm × 30 mm × 0.3 mm.

3. Results and discussion

3.1 Microstructure

Using the same rolling process, it was found that the Fe-6.5Si-1.5Nb (wt.%) alloy can be more easily warm-rolled than the Fe-6.5Si-2.0Nb (wt.%) alloy. In fact, the Fe-6.5Si-1.5Nb (wt.%) alloy can be warm-rolled to a thickness of 0.35 mm without forming any cracks. However, a large number of cracks formed at the edges of the Fe-6.5Si-2.0Nb (wt.%) alloy, when it is warm-rolled to the same thickness. The macrostructure of the warm-rolled sheets is shown in Fig. 1.

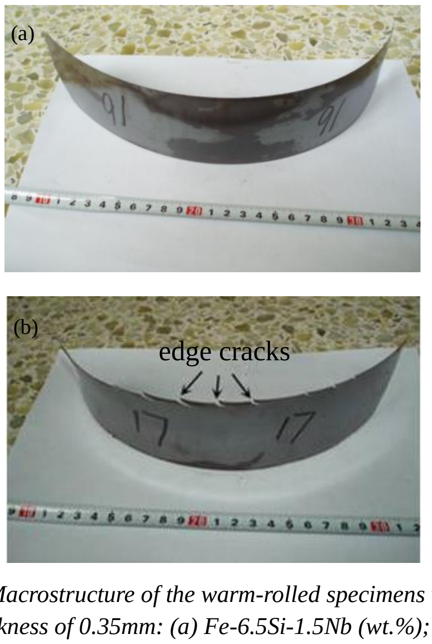

Fig. 1. Macrostructure of the warm-rolled specimens with the thickness of 0.35mm: (a) Fe-6.5Si-1.5Nb (wt.%); (b) Fe-6.5Si-2.0Nb (wt.%)

The metallographic microstructure of the forged specimens with different magnifications is shown in Fig. 2. Fig. 2 (a), (b) are lower magnification micrographs and (a'), (b') are the respective higher magnification micrographs. As seen in Fig. 2, there are many fine and uniform precipitates in both specimens, especially in the Fe-6.5Si-2.0Nb (wt.%) sample. Most of these precipitates are distributed around grain boundaries, and a little distributing within grains. This precipitation plays an important role in refining the grain size. The grain sizes of the forged specimens in this experiment with the addition of different content Nb are both around 30 μm, which is much smaller than that in the non-Nb containing Fe-6.5Si (wt.%) alloy [10]. The result is accordance with Yang et al [15]. However, the presence of a large number of precipitates suggests that the solid solubility of Nb in Fe-6.5Si (wt.%) alloy is likely to be smaller than 1.5wt.%.

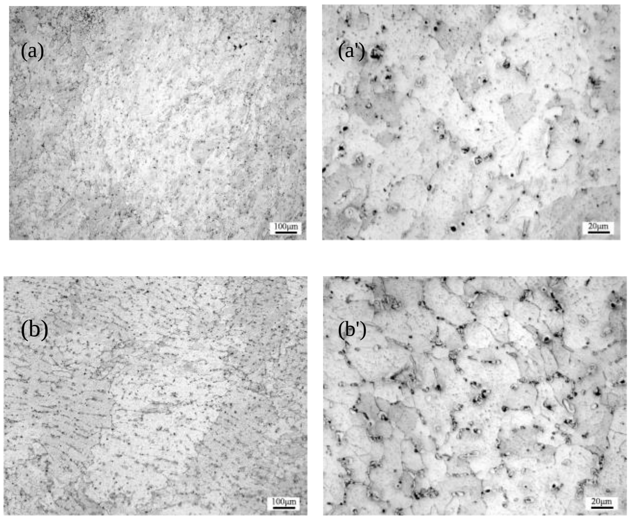

Fig. 2. Microstructure of the forged specimens: (a') Fe-6.5Si-1.5Nb (wt.%); (b) (b') Fe-6.5Si-2.0Nb (wt.%)

The microstructure of the hot-rolled specimens (Fig. 3(a) and (b)) is quite homogeneous through the thickness. This was because the rolling temperature (800-1000℃) was high enough to lead to dynamic recrystallization of the specimens. The grain sizes of the two specimens are both about 60 μm. As shown in Fig. 3 (a') and (b'), the precipitates were no longer distributed along the grain boundaries but uniformly distributed in the matrix. The amounts of precipitation in both hot-rolled specimens are much higher than those of the forged specimens, which suggests that the deformation has likely promoted the precipitation. Fe-6.5Si-1.5Nb (wt.%) specimen contained fewer and smaller precipitates than those in Fe-6.5Si-2.0Nb (wt.%).

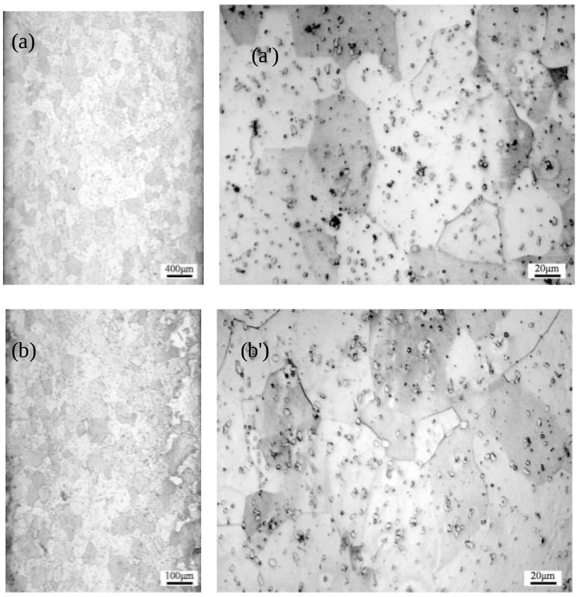

Fig. 3. Microstructure of the hot-rolled sheets: (a) (a'): Fe-6.5Si-1.5Nb (wt.%);(b'): Fe-6.5Si-2.0Nb (wt.%)

The microstructural examination of the warm-rolled specimens showed that the specimens consisted of elongated grains with some deformation band (see Fig. 4 (a) and (b)). Further examination under higher magnification revealed the uniformly distributed precipitates in the matrix as observed in the hot-rolled samples (see Fig. 4 (a') and (b')). Fe-6.5Si-1.5Nb (wt.%) specimen still contained fewer and smaller precipitates than that in the Fe-6.5Si-2.0Nb (wt.%) specimen. However, the amounts of the precipitation in both warm-rolled specimens are much higher than that of the hot-rolled specimens. This is likely due to the large amount of deformation during warm rolling, because deformation is well known to promote the formation of precipitation.

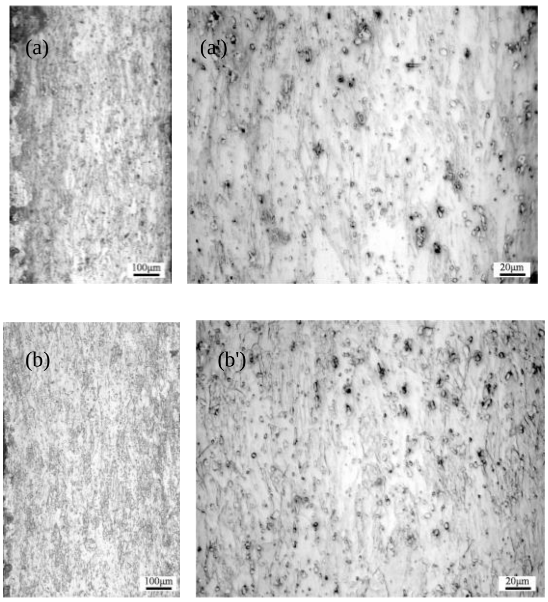

Fig. 4. Microstructure of the warm-rolled sheets: (a) (a'): Fe-6.5Si-1.5Nb (wt.%);(b) (b'): Fe-6.5Si-2.0Nb (wt.%)

According to the SEM microstructure of hot-rolled specimens and the corresponding EDS analysis (as shown in Fig. 5), the precipitates are Nb-Fe-Si intermetallics. Fig.6 summarized the statistical analysis results of the precipitates in the two specimens. The volume fraction of the precipitates in the Fe-6.5Si-1.5Nb (wt.%) specimen is about 3.1%, while the Fe-6.5Si-2.0Nb (wt.%) is about 4.7%. The average sizes of the precipitates are about 3.0μm in the Fe-6.5Si-1.5Nb (wt.%) and about 3.8μm in the Fe-6.5Si-2.0Nb (wt.%). Based on the above analyses, it can be concluded that the volume fraction and the size of the precipitates increased with the increase of Nb content, which is consistent with the microstructural observation under the optical microscopy.

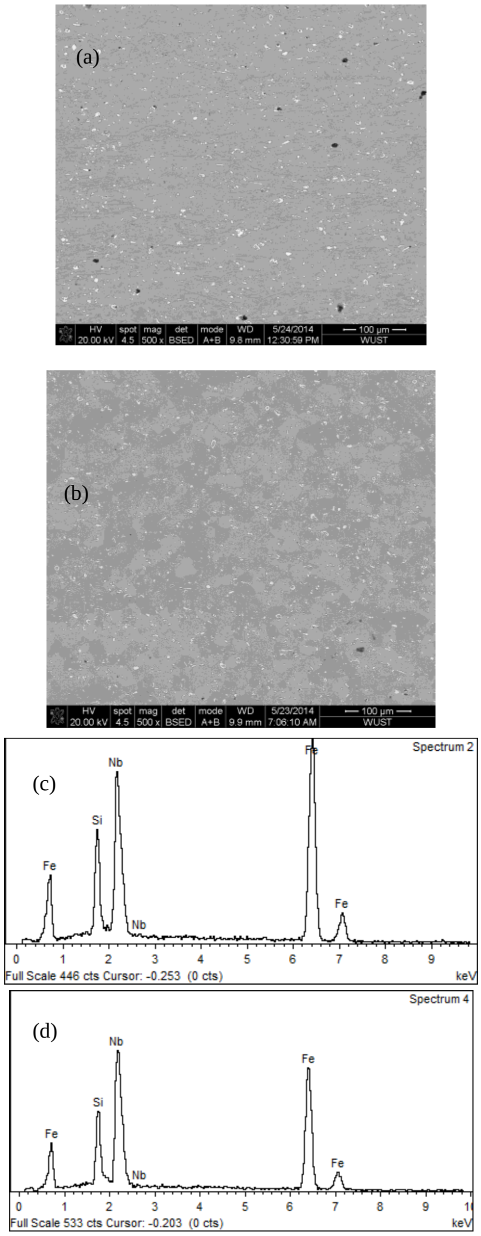

Fig. 5. SEM microstructure of the hot-rolled sheets: (a) Fe-6.5Si-1.5Nb (wt.%);(b) Fe-6.5Si-2.0Nb (wt.%); (c)(d) Corresponding EDS

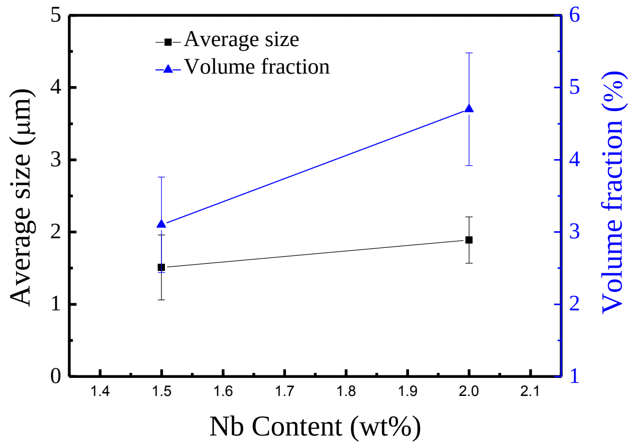

Fig. 6. Precipitation in the hot-rolled sheets

Microstructural examination showed that the addition of Nb has refined the grains of Fe-6.5wt.%Si alloy due to the precipitation in the alloy. Grain refinement would improve the ductility of the alloys. SEM and EDS analysis confirmed that the precipitated phase was Nb-Fe-Si intermetallics. As the precipitated phase contained Si, the Si content in matrix would be reduced. According to the Fe-Si binary phase diagram [17], at low temperature, the B2 ordered phase forms when the Si content is higher than 5.4wt.%, while the $D0_3$ ordered phase appears when the Si content is higher than 6.0wt.%. As mentioned in the introduction, these ordered phases are the main factors leading to the intrinsic brittleness of the Fe-6.5wt.%Si alloy. When the Si content of the matrix is reduced, the amount of the ordered phases or the degree of long range order in the alloy decreases. As a result, the ductility of the alloy improves. This is also believed to be the main reason why the Fe-6.5Si-1.5Nb (wt.%) alloy can be rolled to 0.35mm under relatively low temperature. However, the precipitation of the Nb added alloys will also strengthen the matrix, and the large numbers of precipitates will pin the movement of dislocations. According to the microstructural results of the two specimens with different amounts of Nb addition, the number of Nb-Fe-Si precipitates in the Fe-6.5Si-2.0Nb (wt.%) alloy is much higher than that of the Fe-6.5Si-1.5Nb (wt.%) alloy. Although the amount of the ordered phases or the degree of long range order in the Fe-6.5Si-2.0Nb (wt.%) alloy might also be decreased, the large numbers of precipitates would strongly pin the movement of dislocations during deformation process, and the adverse effect on the ductility was likely larger than the improvement. Therefore, when being rolled at relatively low temperature, many cracks would form along the edges of Fe-6.5Si-2.0Nb (wt.%) sheet. Based on the research results, it can be suggested that the addition of Nb in Fe-6.5wt.%Si alloy should be less than or equal to 1.5wt.% to achieve the optimum processability of Fe-6.5wt.%Si alloy.

3.2 Magnetic properties

The magnetic properties of the warm-rolled sheets with a thickness of 0.35mm were obtained after thermal annealing at 1150℃ for 1h (as shown in Table 1). The Fe-6.5Si-1.5Nb (wt.%) specimen have lower iron loss values and higher inductance value than the Fe-6.5Si-2.0Nb (wt.%) specimen. Though the inductance value of the listed Fe-6.5wt.%Si specimen produced by CVD is a little smaller than the Fe-6.5Si-1.5Nb (wt.%) specimen produced in this experiment, the Nb added specimens have much higher iron loss values than the Fe-6.5wt.%Si specimen produced by CVD. It is suggesting that the addition of such amount of Nb in this experiment may increase the iron loss values. The iron loss of silicon steels comes from three parts [1,19,20]: magnetic hysteresis loss (P_h), eddy-current loss (P_e) and abnormal loss (P_a), and the main iron loss for non-oriented silicon steel comes from the magnetic hysteresis loss (P_h). The main influence factor of P_h is the pinning of domain wall movement by grain boundaries and crystal defect (such as precipitates, inclusions, et al.) during the magnetization process [19]. Thus, the refined grains and a certain amount of precipitates in the Nb added Fe-6.5wt.%Si alloys will also pin the movement of domain wall, and consequently increase the iron loss values. Thus, the addition of Nb may deteriorate the magnetic properties of Fe-6.5wt.%Si alloy, especially when the Nb content is 2.0wt.%.

Table 1. Magnetic Properties of different specimens

Specimens	Thickness (mm)	Core loss (W/kg)		B_8 (T)
		$P_{10/50}$	$P_{10/400}$	
Fe-6.5Si-1.5Nb (wt.%)	0.35	1.815	19.845	1.294
Fe-6.5Si-2.0Nb (wt.%)	0.35	2.330	23.978	1.239
Fe-6.5wt.%Si (CVD)[18]	0.30	0.49	10.0	1.27

4. Conclusions

The addition of a suitable amount of microalloying element niobium to Fe-6.5wt.%Si alloys has improved the ductility of the alloy by precipitating Nb-Fe-Si intermetallics, which would reduce the Si content in the matrix and contribute to the reduction of the amount of the ordered phases or the degree of long range order in the alloys. The addition of Nb has also refined the grains of the alloys, which is beneficial to the processability of Fe-6.5wt.%Si alloy. However, when the Nb content is at 2.0wt.%, the amount of precipitates are too high and starting to pin the movement of dislocations, which has an adverse effect on the processability of the alloy. Though the Fe-6.5Si-1.5Nb (wt.%) alloy showed better magnetic properties than the Fe-6.5Si-2.0Nb (wt.%) alloy, the addition of Nb would deteriorate the magnetic properties of Fe-6.5wt.%Si alloy. Thus, the addition of Nb should be less than 1.5 wt.% to balance the processability and magnetic properties of Fe-6.5wt.%Si alloy.

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