Research Article

Fabrication of Fe-6.5wt%Si Ribbons by Melt Spinning Method on Large Scale

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Melt spinning method has been widely applied for fabrication of Fe-based amorphous/nanocrystalline ribbons in industry. Compared with Fe-based amorphous/nanocrystalline alloys, Fe-6.5wt%Si high silicon steel is of low cost and has comparable excellent soft magnetic properties. Due to higher melting point and absence of supercooled liquid region, fabrication of Fe-6.5wt%Si ribbons is very hard and is only on lab scale. In this paper, we report that large scale fabrication of Fe-6.5wt%Si ribbons was successful and microstructures, ordered structures, and mechanical and soft magnetic properties of the ribbons were investigated. Due to rapid solidification rate, the ribbons were of ultrafine grains, and low degree of order and exhibited some extent of bending and tensile ductility. After heat treatment, excellent soft magnetic properties were obtained. Due to near-zero magnetostriction, the ribbons are promising to be used in electric devices with high frequencies where low noises are required.

1. Introduction

Fe-6.5wt%Si alloy has excellent soft magnetic properties [1], but it is very brittle at room temperature due to formation of ordered structures [2–4]. It is very hard to fabricate this alloy into thin sheet by conventional cold rolling method as cracks are easy to take place [5]. Warm and cold rolling method was developed to improve the ductility step by step during the process, but the whole process route was complex [6–8]. There are several other methods developed to avoid the room-temperature brittleness area, such as chemical vapor deposition (CVD) [9], spray forming [10, 11], direct powder rolling (DPR) [12], and dipping and diffusion annealing [13]. These methods have not succeeded in industry except JFE Super Cores produced by CVD process in Japan.

Rapid quenching from melt using single-roller has also been applied to produce Fe-6.5wt%Si alloy ribbons [14–17]. Small samples with width 5–25 mm were successfully obtained. Microstructure and mechanical and magnetic properties were investigated. Due to the limited size of the ribbon, rapid quenching has not succeeded in industry. Compared with amorphous Fe-based ribbons, Fe-6.5wt%Si alloy is much more difficult to be rapidly quenched into

continuous ribbons. Limitation of both width and length of the ribbons hinders the application of the melt spinning method. In this paper, we report successful fabrication of wide and continuous ribbons fabricated by melt spinning from single-roller. Ordered structures have crucial effect on mechanical and magnetic properties of this alloy and are investigated thereby.

2. Experimental

Raw materials of Fe-6.5wt%Si alloy were fabricated by melting electrolytic iron and metallic silicon together in a medium induction furnace in vacuum. For melt spinning, the alloy ingot was loaded in a quartz tube with a nozzle 30 mm long and melted by high-frequency induction heating. The molten alloy was ejected to a copper wheel with the aid of high pressure of argon in the quartz tube and the wide ribbons were formed with rapid solidification. A set of experimental conditions such as roller rotation speed, ejection pressure, and melting temperature were adjusted for production of continuous ribbons. Surfaces of the as-quenched ribbons were observed by scanning electron microscopy (SEM, Zeiss

FIGURE 1: Melt spinning Fe-6.5wt%Si ribbons of 30 μm in thickness, with width of 30 mm, 20 mm, and 10 mm from left to right.

SUPRA 55). Bending ductility was estimated by bending the ribbons to 180° and the longitudinal microstructure of the ribbon after bending was observed by optical microscopy (OM, Ernst Leitz Wetzlar). Ordered structures of the as-quenched ribbons were investigated by transmission electron microscopy (TEM, JEM-2010), for which a twin jet polishing in a solution of 90% glacial acetic acid and 10% perchloric acid was applied. A four-high mill was used for cold rolling of the as-quenched ribbons. Tensile tests were carried out using Instron 5969 tensile tester at room temperature with a strain rate of 10^{-3} s^{-1} . AC magnetic properties were investigated followed by heat treatment of the as-quenched ribbon in vacuum and the MgO powder was used to prevent conglutination of the belt as reported previously [6].

3. Results and Discussion

The ribbons produced by single-roller melt spinning are shown in Figure 1, in which a continuous wound tape with 30 mm in height and 5 kg in weight is shown. The continuous ribbons have uniform edges, exhibiting good surface quality and metallic lustrous appearance. The thickness of the ribbons varies from 20 to 40 μm mainly controlled by the distance from the nozzle to the Cu wheel when other parameters are fixed, such as the wheel speed, the air pressure, and the liquid temperature.

Direct observation of the surfaces of the as-quenched ribbons by SEM is shown in Figure 2. Ultrafine grains are obtained in the as-quenched ribbons, with diameters ranging from 0.5 to 5 μm . Due to the fast cooling and solidification rate, the crystal nucleation rate from the liquid is large, while the lateral growth of the crystals is prohibited.

The ribbons can be bent into certain shapes. No cracks are found in the bending area even after bending into 180° as shown in Figure 3. The excellent bending ductility guarantees that the ribbons can be coiled into tapes.

The microstructure of the ribbons after quenching is shown in Figure 4. From the diffraction pattern, it is found that the B2 spots exist, although the intensity is faint, while the D0_3 spots hardly exist. According to Figure 4(a), we can see that the formation of B2 ordered structure is hindered but not completely prohibited by rapid quenching, while, for D0_3 , the ordering transformation is completely prohibited during the rapid solidification process. Due to the spallation from the Cu wheel after rapid quenching, the ribbons are bent and

deformation takes place. In Figure 4(b), we can see that the super dislocations tangled and aligned.

Due to excellent ductility after rapid quenching, the ribbons can be cold rolled and the ribbons as-quenched and after cold rolling are shown in Figure 5, including surfaces of the ribbon, that is, the surface contacting Cu wheel and the free surface. After cold rolling, the roughness of the surface is improved, which is helpful to improve the lamination factor and thus the soft magnetic properties can be improved.

For the ribbons as-quenched and after cold rolling, the tensile engineering stress-strain curves are shown in Figure 6. It is found that the as-quenched ribbon exhibited excellent tensile ductility of about 1%, which is very good for this brittle alloy. After cold rolling, the yield stress increases with increasing rolling reduction, while the ductility of the ribbon decreases. Due to work hardening, the yield stress increases and the ductility decreases drastically.

After heat treatment (1100°C/5 h, furnace cooling), AC magnetic properties of the ribbons are shown in Figure 7 in comparison with ultrathin sheet produced by cold rolling [6] and CVD [18] methods. It is found that, with the frequencies below 10 kHz, the core losses in this work are a little higher or as good as those produced by cold rolling and CVD process. With frequencies larger than 10 kHz, the iron losses are much lower than cold rolled or CVD sheet. The core losses of ribbons are crucial to the thickness of the ribbon and also to their microstructure. Thinner thickness is helpful to decrease eddy current loss at high frequencies, and the melt spinning ribbons are more suitable to be used in high frequencies.

With proper control of melt spinning process, continuous ribbons with uniform edges are fabricated, exhibiting good surface quality, and can be coiled into large tapes. The thickness of the ribbons varies from 20 to 40 μm , and the width of the ribbons is up to 30 mm. Due to rapid cooling rate and thus rapid solidification rate, melt spinning ribbons are of ultrafine grains in lateral, with diameters ranging from 0.5 to 5 μm as shown in Figure 2, while columnar grains are observed through the thickness of the ribbons, and the length could be 30 μm as its thickness, as shown in Figure 3(b). Compared with the grain diameter of 10 mm for the 25 kg ingot, the grain size is decreased drastically. The rapid cooling rate is also helpful to hinder ordering transformations in this alloy. Room brittleness of this alloy is ascribed to the appearance of B2 and D0_3 ordered phases in this alloy [3, 5]. At higher temperatures, the alloy is of A2 structure and is rather ductile to be hot rolled. Single-roller rapid quenching is helpful to hinder transformation from A2 to B2 and prohibit ordering transformation from B2 to D0_3 , for which transformations take place at 750°C and 600°C, respectively [19]. As the brittleness is originating from the ordered structures in this alloy, especially from the mixture of B2 and D0_3 ordered structures, rapid quenching is helpful to improve the ductility. Bending and tensile experiments as shown in Figures 3, 5, and 6 indicate that the as-quenched ribbons have excellent room-temperature ductility and that the ribbons could be bent to 180° without bending cracks, could be cold rolled to 35% in reduction, and could be plastically deformed by tension. The excellent ductility of the ribbons guarantees that the continuous rapidly

FIGURE 2: Surfaces of the ribbons after rapid quenching: (a) grain diameters of about 5 μm and (b) grain diameters of about 0.5 μm .

FIGURE 3: Rapidly quenched ribbons possess good bending ductility: (a) ribbons are bent into 180° and (b) no cracks are found after bending.

FIGURE 4: TEM of rapidly quenched ribbons: (a) bright field microstructure of the ribbon with diffraction pattern inset and (b) substructure of the ribbon showing super dislocations.

FIGURE 5: Rapidly quenched ribbons with and without cold rolling of 33% reduction: (a) surface contacting the single-roller and (b) free surface of the ribbon. AQ: as-quenched, CR: cold rolled.

FIGURE 6: Tensile engineering stress-strain curves of as-quenched ribbon and ribbons with different cold rolling reduction.

FIGURE 7: Core losses of rapidly quenched ribbons after heat treatment in comparison with cold rolled sheet and CVD sheet.

quenched ribbons could be coiled into tapes on large scale. After heat treatment, the rapidly quenched ribbons show as good soft magnetic properties as ultrathin sheet produced by cold rolling and CVD process and even better soft magnetic properties for higher frequencies up to 10 kHz.

4. Conclusions

- (1) Fabrication of Fe-6.5wt%Si ribbons with 30 mm in width was successful using single-roller rapid quenching method. The continuous ribbons have uniform edges, exhibit good surface quality, and can be coiled into large scale tapes.

- (2) Due to rapid solidification rate, ultrafine grains are obtained after melt spinning. Meanwhile, transformation of ordered structure B2 is hindered, and the ordered structure D0₃ is prohibited. The as-quenched ribbons exhibit excellent bending and tensile ductility.
- (3) After proper heat treatment, excellent AC magnetic properties can be obtained for as-quenched ribbons, comparable to ultrathin sheet fabricated by cold rolling or CVD process.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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