Effect of wheel speed on magnetic and mechanical properties of melt spun Fe-6.5 wt.% Si high silicon steel

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Fe-Si electric steel is the most widely used soft magnetic material in electric machines and transformers. Increasing the silicon content from 3.2 wt.% to 6.5 wt.% brings about large improvement in the magnetic and electrical properties. However, 6.5 wt.% silicon steel is inherited with brittleness owing to the formation of B2 and D0\textsubscript{3} ordered phase. To obtain ductility in Fe-6.5wt.% silicon steel, the ordered phase has to be bypassed with methods like rapid cooling. In present paper, the effect of cooling rate on magnetic and mechanical properties of Fe-6.5wt.% silicon steel is studied by tuning the wheel speed during melt spinning process. The cooling rate significantly alters the ordering and microstructure, and thus the mechanical and magnetic properties. X-ray diffraction data shows that D0\textsubscript{3} ordering was fully suppressed at high wheel speeds but starts to nucleate at 10m/s and below, which correlates with the increase of Young’s modulus towards low wheel speeds as tested by nanoindentation. The grain sizes of the ribbons on the wheel side decrease with increasing wheel speeds, ranging from \sim 100 \mu m at 1m/s to \sim 8 \mu m at 30m/s, which lead to changes in coercivity. © 2017 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/). https://doi.org/10.1063/1.5006481

INTRODUCTION

With increasing demand of higher energy efficient motors, generators and transformers, a new generation of soft magnetic materials is needed. Ever since Gumlich\textsuperscript{1} discovered the favorable effect of silicon on iron’s soft magnetic properties more than 100 years ago, Fe-Si has been the ideal choice of such applications. Currently, up to 3.5 wt.% silicon steels are predominately used in the market. They are hot rolled into 3 mm hot bands then cold rolled into thin sheets. The obtained thin sheets are further coated with polymer or oxide to form laminates. High silicon steel faces a challenge in processing due to its poor mechanical properties, but its high electrical resistivity makes it a good candidate for high frequency applications. The electrical resistivity increases from 48 \mu \Omega \cdot cm to 82 \mu \Omega \cdot cm when silicon content is increased from 3 wt.% to 6.5 wt.%, resulting in lower energy losses for 6.5 wt.% silicon steel.\textsuperscript{2} 6.5 wt.% silicon steel also offers superior magnetic properties, including low magnetocrystalline anisotropy, zero magnetostriction,\textsuperscript{3} and high permeability.

Fe-Si electric steel has a substitutional A2 body centered cubic (bcc) structure at low silicon concentration. When silicon concentration is increased to about 5.3 wt.%, B2 ordering starts to appear below 500°C according to the phase diagram.\textsuperscript{4,5} D0\textsubscript{3} ordering starts to appear when silicon content is further increased beyond 6 wt.%. A2 is in the disordered state, where the distribution of Fe and Si is random. The B2 and D0\textsubscript{3} phases result from the ordering of the nearest neighbor and the

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next-nearest-neighbor atoms, respectively. The ordering interacts with dislocations which results in a strengthening effect, adversely affecting the mechanical properties. The proposed superdislocation slip mechanisms for B2 and D0\textsubscript{3} also deteriorate the mechanical properties.

Before high silicon steel can be put into wide application, its brittleness problem has to be solved. A few techniques have enabled the processing of high silicon steel including thermomechanical processing (hot rolling and cold rolling),\textsuperscript{8–10} rapid solidification,\textsuperscript{11–14} and deposition/diffusion annealing.\textsuperscript{15–17} Cooling rate of the rapid solidification process has significant effect on the ordering and thus the physical properties of high silicon steel. In this study, 6.5 wt.% Si steel was produced using various wheel speeds during the melt spinning process. The effect of wheel speed on the ordering and physical properties of the melt-spun ribbons is systematically studied. The relation between the wheel speed and the solidification cooling rate is also being studied, where the results will be presented in a different paper.

**EXPERIMENT**

Fe-6.5 wt.% Si ingots were prepared by arc melting in an argon atmosphere. 6g charges were loaded into quartz melt spin tubes fitted with a 0.81 mm precision orifice. The charges were inductively melted at 1590°C in 250 Torr of ultra-high purity helium, then ejected onto a rotating copper wheel using an overhead pressure of 120 Torr He. The copper wheel is 2.5 cm wide and 25 cm in diameter. Melt-spinning was performed at seven different wheel speeds: 1 m/s, 3 m/s, 5 m/s, 7 m/s, 10 m/s, 20 m/s and 30 m/s.

To characterize the degree of the ordering, transmission X-ray diffraction (XRD) was performed on the ribbons using single crystal x-ray diffractometer (STOE IPDS, STOE & Cie GmbH) with Mo source. The incident beam and the image plate detector was fixed, while the sample was rotated from 0° to 180° ω angle to acquire the ring patterns, which were subsequently integrated to create the θ-2θ XRD pattern for evaluation of relative intensities. Ribbon thicknesses were measured on straight regions of the cross-section micrographs taken by optical microscope. The grain sizes were measured on the wheel side of the ribbon using the backscattered imaging mode of a Scanning Electron Microscopy (SEM) (Teneo, Fei Inc). The magnetic properties of the ribbons were measured using Vibrating Sample Magnetometer (VSM) (Versalab, Quantum Design, Inc.). Five ribbon pieces with the dimension of roughly 1mmX4mm were sampled from each wheel speed. The coercive force was measured with a maximum field of 8000 A/m, while the saturation magnetization was measured up to 80 kA/m. The mechanical properties of the ribbons were characterized using nanoindentation (NanoTest\textsuperscript{TM}, Micro Materials Ltd.) technique. The ribbons were mounted sideways in the epoxy mounts. The ribbon cross-sections were then polished to a final finish using 0.05 colloidal silica. Depth versus load method was used with a fixed loading rate of 5.0 mN/s and maximum load of 50 mN. 10 indentations were made on each sample using a 3 faceted Berkovich diamond indenter.

**RESULTS AND DISCUSSION**

Different wheel speeds result in various degrees of ordering as shown in Figure 1. Inside the fundamental (110) rings, an additional superlattice ring caused by D0\textsubscript{3} ordering can be clearly seen on samples with wheel speeds of 5 m/s and below. This D0\textsubscript{3} superlattice ring starts to disappear at intermediate wheel speeds of 7 m/s and 10 m/s, then completely disappears in the 20 m/s and 30 m/s samples. Integration and normalization of the rings allows relative intensity to be calculated. The increase in D0\textsubscript{3} relative intensity (Sample (I\textsubscript{s}/I\textsubscript{f})) towards low wheel speeds is clearly seen in Table I. Quantification of B2 using its superlattice (200) peak at around 14° is difficult due to its overlapping of the D0\textsubscript{3} peak and low relative intensity (53% of that of the D0\textsubscript{3} (111) peak at ~10°).

Long range order parameter (LROP) was used to quantify the relative amount of superlattice peak.\textsuperscript{18} LROP can be calculated using the following equation:

\[
\text{LROP} = \left( \frac{I_{s} / I_{0}}{I_{f} / I_{0}} \right)^{1/2}
\]
where $I_s$ and $I_f$ are the relative intensities of the superlattice peak and the fundamental peak of the sample, respectively, while $I_s^0$ and $I_f^0$ are the relative intensities of the superlattice peak and the fundamental peak of the ordered D0$_3$ reference according to International Center for Diffraction Data (ICDD) PDF-2 #45-1207. The (111) peak and (110) peak were used for the superlattice peak and the fundamental peak, respectively. As shown in Table I, LROP increases with decreasing wheel speeds, implying a higher degree of order in the samples melt-spun at lower wheel speeds.

The cooling rate of a similar alloy system (Fe (75 at.\%), Si (10 at.\%) and B (15 at.\%)) using the same instrument varied from $4.5 \times 10^5$ K/s to $5.2 \times 10^6$ K/s at 5m/s and 20m/s wheel speeds, respectively.\textsuperscript{19} It is generally accepted that faster cooling rates can be achieved by increasing wheel speed. Therefore, a direct relationship can be drawn between wheel speed and cooling rate, and the difference in the degree of ordering observed is a result of different cooling rates. The decrease in thickness with increasing wheel speed as shown in Table I also suggested a direct evidence of different cooling rates obtained at different wheel speeds.

The grain size information on the wheel side of the ribbon as a function of wheel speed is plotted in figure 2. An increase in grain size is observed when the wheel speed is decreased. The grain refinement of rapidly solidified alloys was discussed in detail by Greer.\textsuperscript{20} The microstructure of the melt spun ribbon is featured with a fine equiaxed chill zone on the wheel side of the ribbon where columnar grain grows. The initial grain size of the ribbon on the wheel side is dependent on crystal growth rate, heterogeneous nucleation frequency, and the time for solidification on the surface.\textsuperscript{20} With substantial initial undercooling available in the melt-spun ribbon, a simple equation can be derived,\textsuperscript{21} which indicates the grain size is inversely proportional to the wheel speed $V$ and the cooling rate $\frac{dT}{dt}$. Good linearity is observed when the grain size is plotted against the inverse of wheel speed with speeds between 7 m/s and 30 m/s with R-squared value of 0.99. The change in trend at wheel speeds of 5 m/s and lower is likely due to instabilities of the melt pool, evidenced by a large variation in

\begin{table}[h]
\centering
\begin{tabular}{|c|c|c|c|c|c|}
\hline
Wheel speed (m/s) & Sample ($I_s/I_f$) & D0$_3$ ref ($I_s^0/I_f^0$) & LROP & Thickness ($\mu$m) \\
\hline
1 & 0.054 & 0.06 & 0.94 & 384.9 ± 87.4 \\
3 & 0.012 & 0.06 & 0.45 & 180.0 ± 33.7 \\
5 & 0.065 & 0.06 & 1.04 & 139.1 ± 12.0 \\
7 & 0.007 & 0.06 & 0.34 & 101.7 ± 16.3 \\
10 & 0.002 & 0.06 & 0.17 & 41.7 ± 5.1 \\
20 & 0.002 & 0.06 & 0.18 & 28.8 ± 1.4 \\
30 & 0.002 & 0.06 & 0.20 & 22.5 ± 1.5 \\
\hline
\end{tabular}
\caption{Relative intensities (with background subtracted) of superlattice peak (111) versus fundamental peak (110), long range order parameter and ribbon thickness with change in wheel speed.}
\end{table}
ribbon thickness and width at lower wheel speeds. The increase in grain size with decrease in wheel speed is also reflected in the X-ray diffraction rings in Figure 1, where the fundamental rings, for example (110) rings, change from continuous to discrete due to fewer grains lying in the x-ray beam path for samples with larger grains.

Based on the loading and unloading curves from the nanoindentation experiment, power law fit pyramidal method were used for the analysis. Important parameters such as reduced modulus and hardness were thus calculated based on the analysis. Young’s modulus was calculated using the following equation referencing to Oliver and Pharr: \[\frac{1}{E_R} = \frac{1 - v_s^2}{E_s} + \frac{1 - v_i^2}{E_i}\] (2)

where \(v_s\) = Poisson’s ratio for the sample (0.3 in the case of silicon steel), \(v_i\) = Poisson’s ratio for the indenter (0.07), \(E_R\) = Reduced modulus of the sample, \(E_s\) = Young’s modulus for the sample, and \(E_i\) = Young’s modulus for the indenter (1141 GPa). Both the Young’s modulus and hardness as a function of wheel speed are plotted in Figure 3. A trend can be seen that Young’s modulus and hardness decrease with increasing wheel speed. This effect is more prominent when the speed was increased from 10 m/s to 30 m/s, while fluctuating up and down below the 10m/s range. It is
known that the ordering of Fe-Si results in brittleness, and fast quenching suppresses the disorder to order transition, which in turn leads to an increase in ductility. The reduction in modulus and hardness can be correlated to an increase in ductility. Nanoindentations were also applied to a sample melt spun at 30 m/s followed by an annealing treatment at 850°C and slow cooling. A Young’s modulus of 177.8 GPa and hardness of 5.9 GPa were measured. Due to the higher B2 and D03 phase concentrations in the annealed sample, higher modulus and hardness values are expected. The standard deviation for hardness measurements are high, probably due to its high sensitivity to microstructure.

The magnetic hysteresis loops of the melt-spun ribbons are shown in Figure 4. The saturation magnetization of the ribbons exhibits small variation, (1.74T to 1.78T) at 80 kA/m maximum applied field. It is likely that the saturation magnetization depends primarily on chemistry of the alloy, not quench rate. High wheel speed samples display high magnetic inductions at low field indicating higher permeability. The change in permeability is likely due to the varying degree of texture present in the samples. With <100> direction being the easy magnetization axis for body centered cubic crystals, higher permeability is a result of higher degree of <100> texture. The <100> out of plane fiber texture was observed on the 30m/s ribbon using the XRD pole figure technique, which resulted from the columnar grain growth along the heat extraction direction away from the wheel. The relative intensity of {200} planes was found to decrease with decreasing wheel speed when surface XRD was scanned on the sample free side, suggesting a decrease in out of plane <100> texture. Smaller grains and higher stress of the high wheel speed samples both lead to lower permeability. The fact that higher permeabilities are observed on the high wheel speed samples with smaller grains and higher stress further supported their higher degree of preferred texture. However, the coercivity of the ribbons are highly dependent on wheel speed. The coercive forces are 110.8A/m, 99.8 A/m, 60.5 A/m, 47.5 A/m, 33 A/m, 32.5 A/m, 30.0 A/m for wheel speed of 30m/s, 20m/s, 10m/s, 7m/s, 5m/s, 3m/s, 1m/s respectively. The decrease in coercive force correlates well with the decrease in wheel speed as shown in Figure 4, which can be explained by an increase in grain size at lower wheel speeds.

CONCLUSION

By altering the wheel speed during the melt spinning process, various degree of order, microstructure, mechanical and magnetic properties can be obtained. Faster wheel speeds result in lower degrees of ordering and smaller grain sizes. D03 ordering starts to appear at wheel speeds of 10m/s and lower, while it disappears completely once the wheel speed exceeded 20m/s. Grains with average size of 8 µm are observed on the 30m/s melt spun ribbons. The grains grow when the wheel speed is lowered, reaching 132 µm on the 1 m/s melt spun ribbons. The change in degree of ordering and grain size
significantly affects the mechanical and magnetic properties including Young’s modulus, texture and coercivity. To obtain ductile ribbon, it is suggested that the wheel speed to be 10m/s and above during the melt spinning process.

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