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The effect of precipitate size on magnetic domain behavior in grain-oriented electrical steels

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Precipitates in the form of grain growth inhibitors play an essential role in the production of grain-oriented electrical steels, as they promote the development of Goss texture during secondary recrystallization. However, the presence of precipitates in the final material can have a detrimental effect on loss and permeability, as they impede domain wall motion during the magnetization process. In previous work [K. Jenkins and M. Lindenmo, *J. Magn. Magn. Mater.* **320**, 2423 (2008)], a conventional grain-oriented electrical steel was presented that contained very fine precipitates, which did not damage the bulk magnetic properties. In this article the influence of precipitate size is investigated by comparing local Barkhausen noise measurements and electron backscatter diffraction analysis for a number of grain-oriented electrical steels, which are metallurgically similar except for the size and abundance of precipitates. © 2010 American Institute of Physics.

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I. INTRODUCTION

Electrical steels have seen a number of developments in which the links between metallurgy and magnetic properties have been exploited to both reduce power loss and increase permeability. One of the most significant advances has been the production of conventional grain-oriented (CGO) electrical steel, where polycrystalline silicon-iron strip undergoes a combination of cold rolling and heat treatments. This is designed to selectively grow grains with $\{110\}\langle 001 \rangle$ easy-axes parallel to the rolling direction, and thus develop what is commonly known as a Goss texture.

Grain growth inhibitors, which are introduced in the form of copper-manganese sulphides (CuMnS) precipitates, play a key role in producing a Goss texture by impeding normal grain growth and promoting the development of Goss grains during secondary recrystallization. However the presence of these precipitates is detrimental to the final product's performance, as they create nonmagnetic voids within the iron lattice that obstruct the movement of domain walls during the magnetization process. Therefore once the desired metallurgy is obtained by means of high-temperature annealing—at a temperature of approximately 1100 °C in a hydrogen atmosphere—it is beneficial to extend the annealing time and raise the temperature to 1200 °C to desulphurise the material, effectively removing the precipitates. When completed only fine Cu precipitate remains which, as this paper will show, have no discernible effect on the magnetization process.

Close inspection of the B–H loop of CGO reveals that flux density does not change continuously, but instead occurs as a sequence of discrete jumps, an effect known as Barkhausen noise (BN). These jumps are related to the way

domain walls interact with pinning sites, such as precipitates, as domains reorganize to align magnetic moments in the direction of the applied field.

In attempting to define the pinning strength of an inclusion Dijkstra and Wert extended Néel's dispersive field theory,^{1,2} proposing that within the body of a domain, magnetic dipoles are formed at the interface surrounding an inclusion. If a domain wall then bisects the inclusion, the dipole arrangement is split, creating a four-pole system and reducing the overall magnetostatic energy and pinning the domain wall as a result. This model was extended to incorporate a contribution from spike domains, which increase the effective volume of the pinning site. Intuitively it can therefore be stated that the number of Barkhausen jumps is to some extent determined by the number of inclusions, provided the volume of the inclusion and subsequent closure domains are sufficient to result in pinning. Hence the BN provides a useful tool for evaluating the scale of interaction between precipitates of varying size and magnetic domains.

In a previous study³ samples of CGO were prepared using a range of heat treatments and the microstructures were analyzed to investigate the role of precipitates during the manufacturing process. The paper presented bulk magnetic measurements and identified CGO samples containing fine Cu precipitate, which did not appear to affect the bulk magnetic properties. This work compares three types of uncoated CGO, using local BN measurements to assess the impact of precipitate size on domain behavior.

II. PRACTICAL EXPERIMENTATION

For the purpose of clarity the notation CGO_n is used to identify each batch of CGO, where the subscript n denotes the approximate precipitate size, which has been determined using TEM micrographs. The bulk magnetic properties, typi-

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TABLE I. Results of bulk magnetic measurements and metallurgical analysis.

Designation	CGO ₀	CGO ₁₀	CGO ₁₀₀
P at 1.5 T, 50 Hz (W kg ⁻¹)	0.81	0.84	1.50
B ₈₀₀ (T)	1.86	1.84	1.75
Precipitate type	N/A	Cu	CuMnS
Precipitate diameter (nm)	N/A	5–10	100–125
Sample length (mm)		150	
Sample width (mm)		30	

cal precipitate species, and sample geometry are given in Table I. Further details of both the method and results from this analysis are given in Ref. 3.

The metallurgy of both CGO₀ and CGO₁₀ should be considered fairly typical of a fully finished product. However, they were produced from different hot band sources. CGO₀ represents a control sample created from a hot band material containing no Cu, and thus leaving relatively no precipitate after desulphurisation. CGO₁₀ has similar properties to CGO₀, but has been alloyed with Cu, and so the final product contains a large number of fine Cu precipitates. Alternatively, CGO₁₀₀ contains a combination of fine Cu precipitates and coarse CuMnS precipitates as a consequence of interrupting the high-temperature annealing process before desulphurisation was completed.

When compared to CGO₀, the total power loss P and peak inductance B₈₀₀ (at a field strength of 800 Am⁻¹) for CGO₁₀ suggests that the Cu precipitates have had little effect on the magnetic properties. Alternatively, the coarse precipitates in CGO₁₀₀ have significantly reduced permeability and has almost doubled power loss. This drop in the magnetic performance of CGO₁₀₀, due to the increased number of pinning sites, has also been examined through measurements of local variations in BN.

III. BN MEASUREMENTS

A system has been developed,⁴ which uses a 3 mm diameter ferrite probe, with a relative permeability of approximately 900 ± 20%, to amplify BN contributions perpendicular to the surface of the sample. The subsequent variations in flux density within the probe are detected by a pick-up coil of 1000 turns wound around the probe. The coil's output emf, which is proportional to the BN, is measured using a data acquisition card (NI4552) at a sample frequency of 102.4 kHz.

The output of a second card (NI6731) is connected through a power amplifier and a shielded air-core isolation transformer to windings on two identical yokes made from electrical steel, which generated the applied field. The entire system was controlled using Labview to measure B via an 80 turn pick-up coil wrapped around the sample and apply a digital feedback algorithm⁴ designed to limit distortion of the 50 Hz B waveform and maintain a constant peak flux density of 1.0 T ± 0.1%.

A three-axis positioning system, consisting of linear stages driven by stepper motors, was used to automatically reposition the ferrite probe on the surface of the sample be-

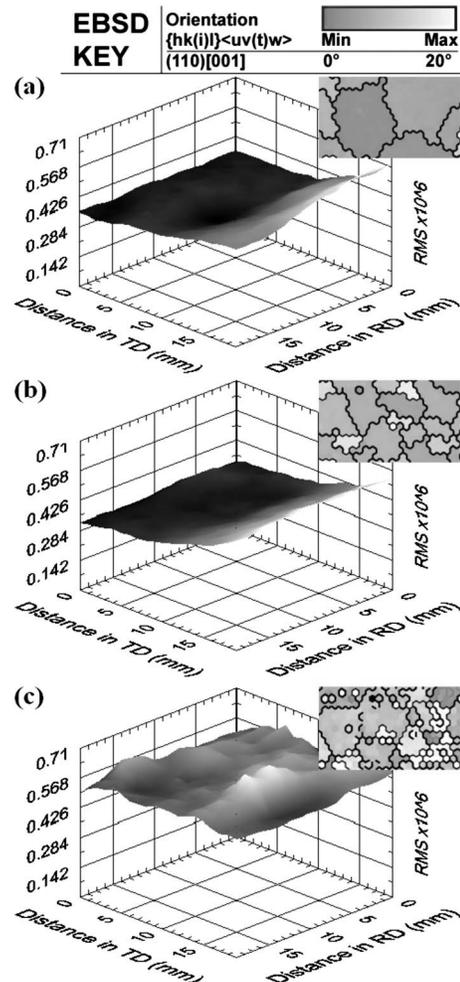


FIG. 1. Example of results from measuring local variations in BN and EBSD analysis of (a) CGO₀, (b) CGO₁₀, and (c) CGO₁₀₀ samples.

tween each measurement. Hence, two 20 × 20 mm areas on each of the three materials were raster scanned with an interval of 1 mm between measurements. The resulting BN at each point was rated, by calculating the root mean squared (rms) of the signal, and plotted as shown in Fig. 1.

IV. ELECTRON BACKSCATTER DIFFRACTION (EBSD)

The areas examined for BN were also cut into 30 × 30 mm squares and prepared for EBSD analysis. This involved mechanically grinding the samples, followed by several iterations of polishing and chemical etching using a 2% Nital solution.

Small 5 × 10 mm sections, representative of the resulting images, have been inserted into Fig. 1 next to the corresponding three-dimensional plot of BN rms. In these images the crystallographic orientation of each grain is represented using a gray scale, where the darker gray indicates a near Goss texture and regions of white signify a deviation of greater than ±20° from the {110}<001> direction.

V. RESULTS AND DISCUSSION

The EBSD analysis confirms that each sample in Fig. 1 has a predominantly Goss texture. However, CGO₁₀₀ also

contains a number of small grains with orientations that vary by more than $\pm 20^\circ$ from the Goss texture. Therefore, the reduced annealing time appears to have prematurely halted the development of the Goss texture within the CGO₁₀₀ material. This is apparent when comparing the BN plots, where Figs. 1(a) and 1(b) are relatively featureless, signifying the greater degree of homogeneity in the distribution of flux and pinning sites, such that the material presents a similarly uniform BN. Conversely, the BN distribution in Fig. 1(c) is uneven, with peaks and troughs sporadically distributed throughout the scan area and thus, further verifying the connection between BN and the metallurgy. It is proposed that the inhomogeneous distribution of BN is caused by the irregular distribution of flux within the specimens as a result of local variations in permeability.

With regard to whether an increase in the number of pinning sites occurs due to the presence of Cu and CuMnS precipitates, the BN plots appear to corroborate the findings from the bulk magnetic measurements. When the points for each plot are averaged, both CGO₀ and CGO₁₀ have a comparable overall magnitude, which is 26% lower than CGO₁₀₀. This suggests that although CGO₁₀ contains an abundance of Cu precipitates, they are not sufficient in size to impede the motion of domain walls. Thus they conform to the dispersive field theory, whereby inclusions that are relatively small compared to the width of the domain wall (approximately 179 nm, $\pm 10\%$ for 180 bloch wall⁵) are completely engulfed by the wall, and thus the dipole surrounding the inclusion is maintained.

The increased overall magnitude of BN demonstrated by CGO₁₀₀ implies that there is either an increase in the amplitude or the number of Barkhausen events, thus providing strong evidence that the number of pinning sites has increased due to the presence of the coarse CuMnS. The diameter of the CuMnS, and associated closure domains, must therefore be sufficient to bridge the width of the domain

walls, therefore providing the conditions necessary to create the four pole state, and the resulting decreased magnetostatic energy, that is responsible for pinning.

VI. CONCLUSIONS

The presence of precipitate, in the form of grain growth inhibitors, during the production of CGO electrical steel has a beneficial influence over the resulting microstructure.

However, this study has confirmed that those same precipitates (CuMnS), if not removed from final product, also have a significant detrimental effect on the magnetic properties of the final material.

Bulk magnetic measurements of loss and peak inductance have verified that the residual presence of fine Cu precipitates ($\sim 5\text{--}10$ nm) does not affect domain wall motion.

Local BN measurements have also been used to demonstrate that although these Cu precipitates do not influence the magnetic properties, coarse CuMnS precipitates do increase the rms BN.

It is proposed that this is due to the diameter of the Cu precipitates being relatively small compared to the domain wall thickness, whereas the CuMnS precipitates are of comparable size.

ACKNOWLEDGMENTS

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