Naturally occurring oxide on rapid solidified high silicon steel and its growth kinetics

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Fe-6.5Si, or high silicon steel, has been considered a favorable candidate for high-speed motors/transformers and high-frequency transformers. While keeping the saturation as high as 1.8T, Fe-6.5Si has improved resistivity (82 μΩ cm) than traditional silicon steel (52 μΩ cm), leading to a significantly lower eddy current loss when used at high frequencies (≥ 400 Hz). Rapid solidification by melt spinning is an effective route for producing high silicon to overcome its brittleness problem. However, for the thin melt-spun ribbons/tapes (25-40 μm thick), an extremely thin coating is needed to maximize the flux density of laminations for motor or transformation applications. The present study aims to elucidate the formation of naturally occurring oxide on high silicon steel and its growth mechanism and kinetics at high temperatures.

Keywords: high-silicon steel; coating; resistivity; surface techniques

1. Introduction

High silicon steel Fe-6.5Si (wt.%) is a candidate for high-speed motors and high-frequency transformers. With a high saturation of 1.8T, Fe-6.5Si has higher resistivity (82 μΩ cm) than traditional 3.2% silicon steel (52 μΩ cm), leading to significantly lower eddy current loss when used at high frequencies ((≥ 400 Hz)[1]. However, increased silicon content also makes Fe-6.5%Si too brittle to be processed using conventional processes, such as cold rolling. Rapid solidification by melt spinning/strip casting is a viable route for Fe-6.5%Si manufacturing. This process produces ribbons/tapes that are 25-40 μm thick with low losses (W10/400 of 6.1 W/kg when fully annealed[2]) that are extremely ductile[3]. However, coatings are needed to insulate the steel laminates for the intended application of motor laminates and transformer cores for eddy current loss control.

There have been some speculations in the field that an oxide film may have formed during the rapid solidification due to the observation that wound melt spun tape is fully insulating. This is reasonable considering the highly negative Gibbs energy of formation of silicon oxide. However, there is a lack of a clear understanding of the chemistry and depth of the oxide. Using the highly surface-sensitive analytical technique X-ray photon electron spectroscopy (XPS) coupled for depth profiling, the present study aims to elucidate the formation of naturally occurring oxide on high silicon steel. The oxide growth mechanism and kinetics at different temperatures will also be discussed.

2. Results and discussion

X-ray photon electron spectroscopy was scanned on the top surface from the free side of a high silicon steel ribbon that was melt-spun at 20 m/s. The XPS results show that the Si-O bond is present at the top of the surface instead of the elemental silicon bond. The surface is free of either metallic or oxidized Fe. It shows that the as-spun ribbon has a silicon oxide surface. To acquire a depth profile, ion sputtering/milling was conducted with a calibrated surface removing rate of 1 nm/s. From the series of patterns shown in Figure 1, the Si-O bond starts to disappear completely at 15 seconds (i.e., 15nm from the top surface), and the Si metallic bond dominates. This data confirms that a naturally occurring silicon dioxide forms on the melt-spun ribbons, and its thickness is ~15 nm, as shown in the schematic in Figure 1.

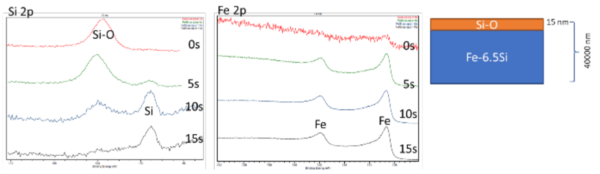

Figure 1. X-ray photon electron spectroscopy of a melt-spun ribbon showing the surface species as a function of depth from the top surface, and schematic showing the formation of 15 nm SiO_2 after melt spinning based on XPS result.

The Fe-6.5Si ribbon was oxidized at 1000°C, and the oxide growth kinetics are shown in Figure 2 (left panel). The TGA curve revealed that the ribbon follows parabolic oxide growth kinetics after a rapid mass gain. The XPS results of a ribbon oxidized at 1000°C for 1 hour showed that the surface scale is rather complex consisting of two distinct layers. The top layer consists of a mixed layer of silicon oxide and iron oxide that is 40 nm thick. The 2nd layer consists of pure silicon oxide that is 60 nm thick and free of iron oxide.

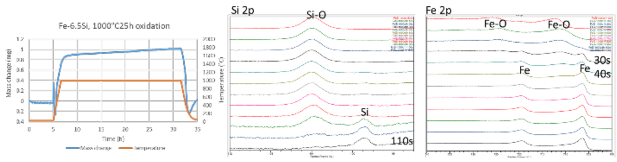

Figure 2. Thermal gravimetric analysis (TGA) of a Fe-6.5Si ribbon oxidized at 1000°C and XPS of a Fe-6.5Si ribbon oxidized in air at 1000°C for 1 hour.

References

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