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Oxidation Behavior of Fe-Ni Metal Amorphous Nanocomposite (MANC) for High Speed Motor (HSM) Applications.

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New interest in high performance soft magnetic materials (SMMs) have been pushed by the need to lower losses at higher operating frequencies while maintaining high flux density and tunable permeability in power conversion, electronic, and motor applications.[1–3] Conventional SMMs like electrical steels and Fe-based metal amorphous nanocomposite (MANC) alloys are dominated by eddy current losses at high frequencies and brittle mechanical properties, respectively. Recent breakthrough in high-performance Fe-Ni metal amorphous nanocomposite (MANC) SMMs have shown promise in reducing eddy current losses as compared to electrical steels while retaining excellent mechanical properties. Their intrinsic adherent native surface oxide layer provides sufficient electrical insulation to reduce interlaminate eddy current losses. While Co-based MANCs also show superior mechanical properties, the low cost of Ni compared to Co makes Fe-Ni MANC alloys a superior system with good saturation induction, tunable permeability, and low losses for the high frequency switching applications mentioned above.

Notwithstanding advances in MANCs, there exists a gap in the literature on investigations of the surface oxide layer responsible for a significant reduction of interlaminate eddy current losses in magnetic cores. This work examines the nature of the surface oxide, oxidation behavior, and relationship between oxide thickness and resistivity. Isochronal oxidation studies were performed between 350 ^{o}C and 600 ^{o}C for 1 h. Subsequently, isothermal kinetics oxidation study was carried out at 550 ^{o}C between thermal annealing times of 1 to 48 h. For characterization of oxide surface layer thickness and composition as a function of time, annealed FeNi alloy samples were analyzed by X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS). The morphology of the surface of the annealed samples were examined by scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS).

To determine the stability and order of the oxidation products, the relative affinity of oxygen for each constituent element in the FeNi alloy was evaluated by comparing the free energies of formation of the oxides per 1 mole of oxygen. The order of oxygen affinity is Si > B > Nb > Fe > Ni. Next, the XRD results presented in Fig. 1. show a broad amorphous peak at 350 ^{o}C which is indexed to the residual amorphous phase. At 440 ^{o}C, broad peaks are observed which can be indexed to a two-phase mixture of face-centered cubic (FCC) and body-centered cubic (BCC) nanocrystals overlapped by the broad peak of the residual amorphous phase. Peak shifting is also observed from 440 – 550 ^{o}C indicating strains at the growing metal-oxide interface. As oxidation temperatures approach 600 ^{o}C, peak sharpening is observed along with new spinel ferrites peaks. The sharpening of the peaks indicates an increase in the oxide thickness. The change in the oxidation behavior suggests ~ 440 ^{o}C as the transition from low temperature slow logarithmic kinetics to high temperature parabolic kinetics. Secondary crystallization of the amorphous phase at ~ 600 ^{o}C is deleterious to soft magnetic properties.

To support the XRD results, XPS depth profiling analyzes were carried out to determine the surface oxide composition, and thickness. From the XPS results, variations in oxide and metal as a function of depth indicate that Fe oxide is the predominant species at the top surface. As depth increases, Nb oxide content increased quickly. The oxygen content decreases at the beginning of sputtering but then increases just before the oxide/metal interface in conjunction with the appearance of oxidized B. Ni oxide was not observed at lower temperatures. At 550 ^{o}C, the interface is considerably broadened, and Ni was observed at the top surface. Table 1 shows an XPS depth profile of annealed ribbons as a function of oxide thickness. No significant change in oxide thickness was observed between 250 and 350 ^{o}C or as a function of annealing time. A slight increase of < 1 nm was observed at 400 ^{o}C, but again no change in thickness as a function of time. This is consistent with the slow logarithmic kinetics expected below the transition temperature. At and above the transition temperatures (440 ^{o}C), oxide thickness significantly increased as a function of time, consistent with expected parabolic kinetics at this temperature range. Logarithmic scaling of the measured oxide thickness on the air and wheel side as function of time of oxidation at 440 ^{o}C shows no significant difference in the rate of oxide growth on both sides. The slope of a plot of log of measured thickness vs log of time as a function of the ribbon thickness indicates either diffusion controlled (slope = 0.5) and or surface controlled (slope = 1) oxidations.[4] The slope for the annealed FeNi ribbon in this work was 0.65 indicating mainly parabolic rate law which is diffusion controlled oxidation.

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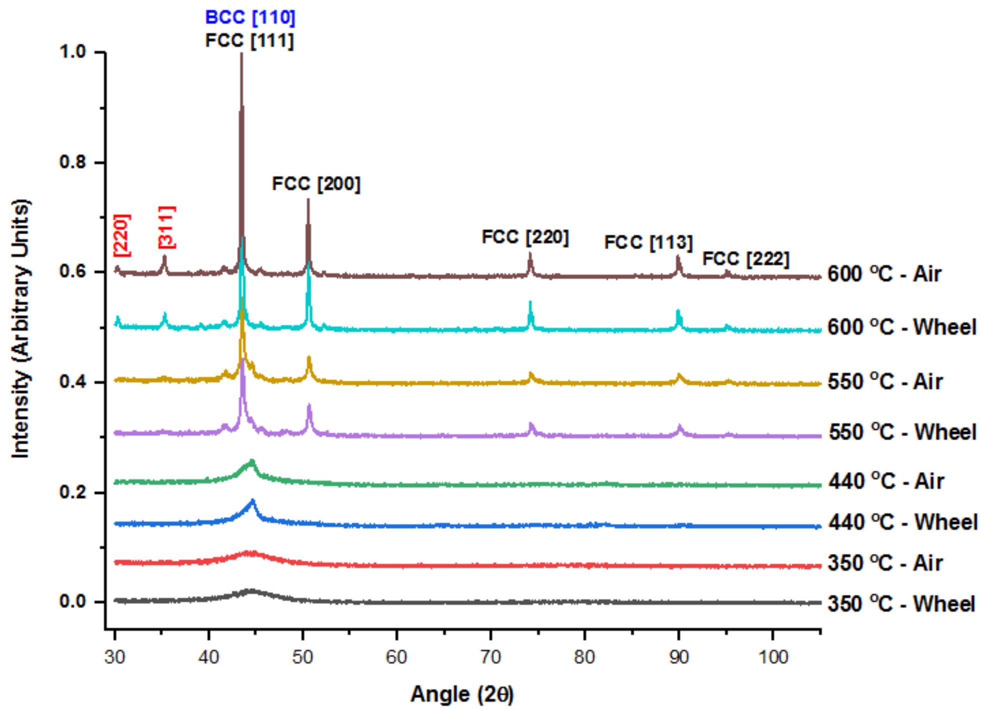

Fig. 1. XRD patterns of $(Fe_{70}Ni_{30})_{80}Nb_4B_{14}Si_2$ MANC ribbons, air and wheel sides annealed isochronally at 350, 440, 550, and 600 °C for 1 h. The spinel ferrites are labeled in red.

Temperature (°C)	Annealing time (hour)	Oxide thickness (nm)	Order of oxide present
250	1	9.8	B < Nb < Fe
	3	10.6	
	5	9.7	
350	1	10.0	
	3	9.4	
	9.5	9.9	
400	1	10.9	
	3	10.8	
	5	10.9	
440	1	11.7	
	4	29.6	
550	1	15.3	B < Nb < Fe < Ni
	3	54.4	

Table. 1. XPS depth profile results of annealed FeNi amorphous ribbon, air side, at 250, 350, 400, 440, and 550 °C.