

Corrosion behavior of $\text{Al}_{100-x}\text{Cu}_x$ ($15 \leq x \leq 45$) doped Nd–Fe–B magnets

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ABSTRACT

The corrosion behavior of $\text{Al}_{100-x}\text{Cu}_x$ ($x = 15, 25, 35, 45$ at.%) doped Nd–Fe–B magnets has been investigated in 0.005 M H_2SO_4 solution using potentiodynamic polarization and electrochemical impedance spectroscopy techniques. It was found that the corrosion resistance first improves with the increase of copper content, reaches a maximum at $x = 35$ and thereafter decreases. The existence of optimal corrosion resistance in the $\text{Al}_{65}\text{Cu}_{35}$ doped magnets is attributed to both the increase of chemical stabilization and the optimization of distribution/morphologies of (Pr, Nd)-rich intergranular phases. Addition of $\text{Al}_{55}\text{Cu}_{45}$ shows poorer corrosion resistance mainly due to coarsening of intergranular phases that, in turn, led to increasing the amount of active reaction channel at the triple junction of the matrix phase.

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1. Introduction

Nd–Fe–B sintered magnets have been developed over the last 25 years for advanced applications due to their excellent magnetic properties [1]. The technical difficulties in their applications are the poor corrosion resistance especially in corrosive environments and low thermal stability [2]. These drawbacks can be attributed in part to the intrinsic properties of the material, but corrosive nature is strongly affected by microstructural parameters such as the distribution and chemical composition of phases. In Nd–Fe–B sintered magnets, ferromagnetic $\text{Nd}_2\text{Fe}_{14}\text{B}$ matrix phase is basically surrounded by Nd-rich intergranular phase. The latter is more active than the matrix phase, which results in the preferential dissolution or corrosion of the Nd-rich intergranular phase [3,4]. Hence, the corrosion resistance of Nd–Fe–B sintered magnets can be enhanced through improving the chemical stability of Nd-rich phase by small additions of other alloying elements (Co, Al, Zr, Ga, Cr, Cu, Nb) prior to melting the alloy components [5–8]. Unfortunately, this method is likely accompanied by the deterioration of magnetic properties due to the dissolution of these elements in the matrix phase. Therefore, in order to obtain a balanced performance of Nd–Fe–B magnets, it is necessary to marginalize or avoid alloying elements entering the $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase. This would be attained if the additives were mixed with magnets in the form of powder during processing, as proved by recent investigations [9–12].

Recently, we have observed that the addition of $\text{Al}_{85}\text{Cu}_{15}$ (~1.2 wt%) into the intergranular regions of $(\text{Pr}, \text{Nd})_{14.8}\text{Fe}_{78.7}\text{B}_{6.5}$ alloy using a powder blending technique can effectively enhance the corrosion resistance without sacrificing magnetic performance of magnets [13]. The increase of corrosion resistance by Al–Cu addition is ascribed to both the chemical stability improvement of Nd-rich phase and its better distribution. For Al and Cu codoped Nd–Fe–B magnets, copper has more pronounced effect on the corrosion resistance than aluminum, because the standard electrode potential of 0.3419 V for Cu^{2+}/Cu is higher than -1.632 V for Al^{3+}/Al [14,15] and also because copper plays a more important role in modifying the grain boundary microstructure, particularly the distribution of Nd-rich intergranular phase [16,17]. Therefore, copper content of Al–Cu addition is expected to be a key factor to influence the corrosion resistance of Nd–Fe–B sintered magnets. In the present study, the effect of Cu content in $\text{Al}_{100-x}\text{Cu}_x$ ($15 \leq x \leq 45$) addition on the corrosion resistance of Nd–Fe–B sintered magnets was investigated.

2. Experimental

Cast ingots of $(\text{Pr}, \text{Nd})_{14.8}\text{Fe}_{78.7}\text{B}_{6.5}$ and $\text{Al}_{100-x}\text{Cu}_x$ ($x = 15, 25, 35, 45$ at.%) were produced by vacuum induction melting technique using Pr–Nd, Fe–B, and Fe, Al, Cu pure (99.9%) elements. The $(\text{Pr}, \text{Nd})_{14.8}\text{Fe}_{78.7}\text{B}_{6.5}$ ingot was crushed and pulverized to less than $250\text{ }\mu\text{m}$, and then milled to $\sim 4\text{ }\mu\text{m}$ powder through jet milling in nitrogen atmosphere. The $\text{Al}_{100-x}\text{Cu}_x$ ingot was crushed and pulverized to $\sim 4\text{ }\mu\text{m}$ powder through ball milling under argon atmosphere. The $(\text{Pr}, \text{Nd})_{14.8}\text{Fe}_{78.7}\text{B}_{6.5}$ alloy powders were blended with 1.2 wt% $\text{Al}_{100-x}\text{Cu}_x$ powder by ball milling for 1.5 h in nitrogen atmosphere. After blending, the mixed powders were compacted at a 6 MPa pressure in a magnetic field of 1.6 T. Compact powders were sintered for 2 h at $1100\text{ }^\circ\text{C}$ to achieve full density magnets ($\rho \geq 7.5\text{ g/cm}^3$). These sintered magnets were annealed at $890\text{ }^\circ\text{C}$ for 3 h and at $480\text{ }^\circ\text{C}$ for 2 h in two steps. Their magnetic properties are shown in Table 1.

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Table 1
Magnetic properties of $\text{Al}_{100-x}\text{Cu}_x$ doped Nd–Fe–B magnets.

$\text{Al}_{100-x}\text{Cu}_x$	B_r (T)	iH_c (kA/m)	$(BH)_{\text{max}}$ (kJ/m ³)
$x = 15$	1.263	1185	300
$x = 25$	1.275	1215	320
$x = 35$	1.285	1106	306
$x = 45$	1.290	621	242

Microstructure of the samples was observed under a field emission scanning electron microscopy (FESEM SIRION-100) equipped with an energy dispersive X-ray spectroscopy (EDS). Electrochemical tests were determined with a CHI604B electrochemistry analyzer and conducted at 25 ± 0.1 °C in 0.005 M H_2SO_4 (pH 2) aqueous solutions, which were purged with Ar for 1 h before the test. All experiments were performed in a standard three electrode cell consisting of Nd–Fe–B working electrode, saturated calomel reference electrode and Pt counter electrode. The working electrode with an operating surface area of $S = 0.1$ cm² was mechanically grounded with SiC-paper (grade 320–1000), polishing with diamond paste (0.5 μm). The open-circuit potential was monitored for 30 min to allow the working electrode to a steady state and the potentiodynamic polarization scan was commenced from the negative potential to the positive one with a scan rate of 2 mV/s. Electrochemical impedance spectroscopy (EIS) were measured in the frequency range from 10^5 to 10^{-2} Hz with a signal amplitude perturbation of 10 mV. Spectra analyses were performed using ZView software [18]. All electrochemical tests were performed three times in each case.

3. Results and discussion

Fig. 1a shows potentiodynamic polarization curves of $\text{Al}_{100-x}\text{Cu}_x$ ($15 \leq x \leq 45$) doped magnets in 0.005 M H_2SO_4 solution. All samples exhibit active dissolution without a transition to passivation up to 0 V. At potentials, a little more positive than E_{corr} , the anodic curves shift towards smaller current densities when Cu

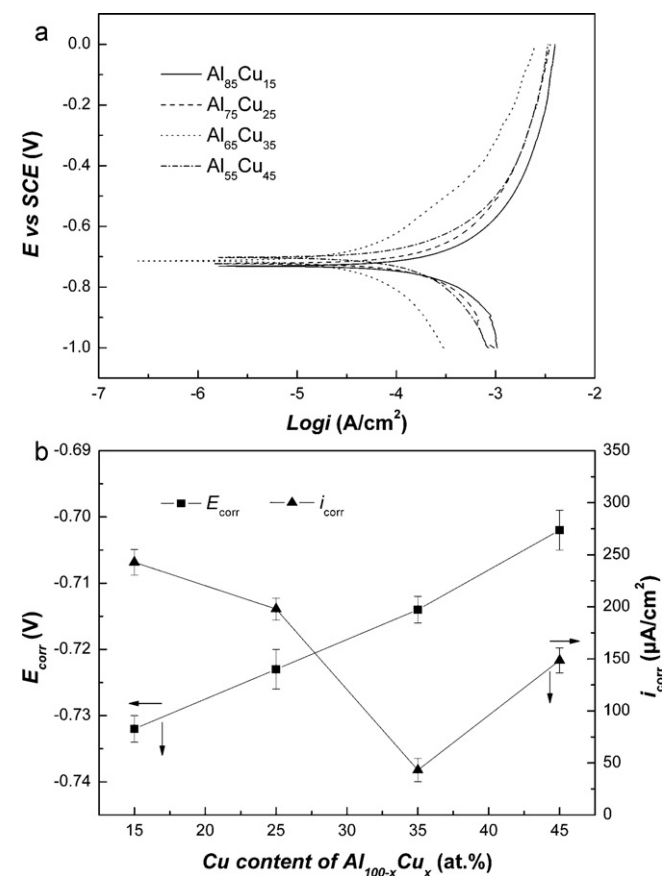


Fig. 1. (a) Potentiodynamic polarization curves of $\text{Al}_{100-x}\text{Cu}_x$ ($15 \leq x \leq 45$) doped magnets in Ar-purged 0.005 M H_2SO_4 solution, (b) Corrosion potentials and corrosion current densities corresponding to (a), with error bars.

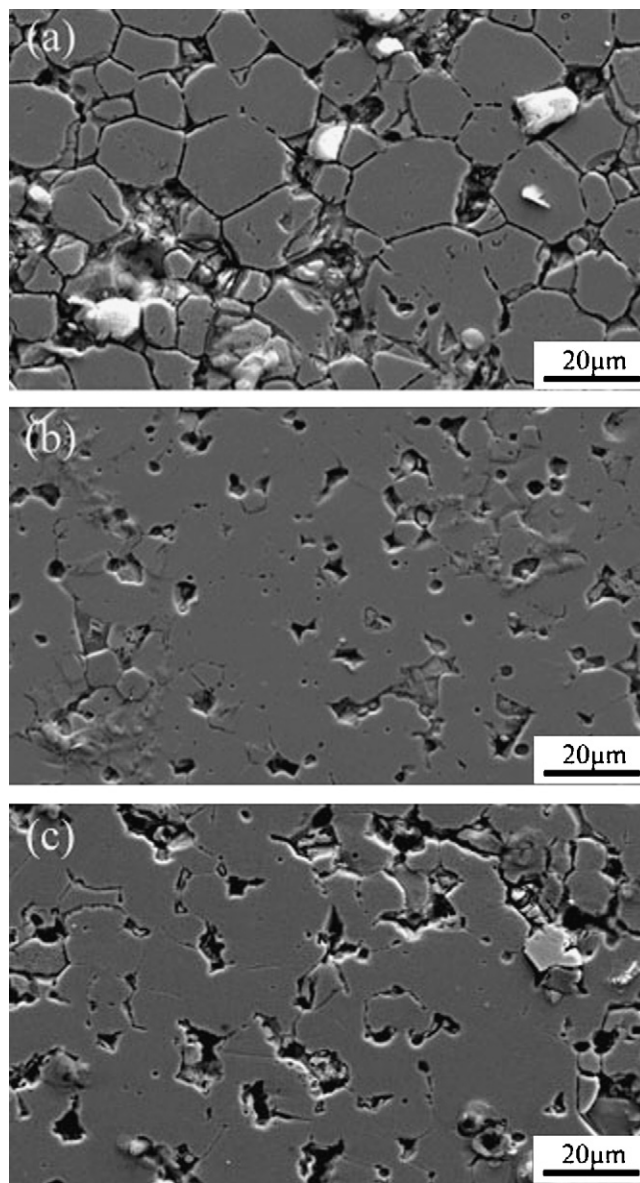


Fig. 2. SEM micrographs of corroded surfaces for the magnets doped with (a) $\text{Al}_{85}\text{Cu}_{15}$, (b) $\text{Al}_{65}\text{Cu}_{35}$, and (c) $\text{Al}_{55}\text{Cu}_{45}$ after potentiodynamic polarization scan in 0.005 M H_2SO_4 solution.

content increases from 15 to 35 at.%. Over 35 at.%, the anodic curves move towards larger current densities. These results indicate that the grain boundary phases of $\text{Al}_{65}\text{Cu}_{35}$ doped magnets dissolve at the slowest rate. The corrosion potential E_{corr} and corrosion current density i_{corr} for the magnets are shown in Fig. 1b. The value of i_{corr} was determined from the intersection of extrapolated cathodic and anodic Tafel lines at E_{corr} . As can be seen, E_{corr} of $\text{Al}_{100-x}\text{Cu}_x$ doped magnets increases from -0.731 V for $x = 15$ to -0.702 V for $x = 45$, while i_{corr} decreases from 242.8 $\mu\text{A}/\text{cm}^2$ for $x = 15$ –43.1 $\mu\text{A}/\text{cm}^2$ for $x = 35$ and then increases to 148.5 $\mu\text{A}/\text{cm}^2$ for $x = 45$. This fact suggests that the corrosion resistance of magnets increases as a function of Cu content in $\text{Al}_{100-x}\text{Cu}_x$ ($x < 35$ at.%), but it decreases for $x > 35$ at.%. It can be said that Cu content up to 35 at.% is optimal to enhance the corrosion resistance.

Fig. 2 presents scanning electron micrographs of the magnets doped with $\text{Al}_{85}\text{Cu}_{15}$, $\text{Al}_{65}\text{Cu}_{35}$, and $\text{Al}_{55}\text{Cu}_{45}$ after potentiodynamic polarization scan in 0.005 M H_2SO_4 solution. For these magnets, the intergranular phases intensively dissolve, thus causing grain boundary corrosion. A few etch pits are observed at the surface of

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