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Corrosion Mechanism of Nd-Fe-B Magnets in Humid Environments

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ABSTRACT: Although Nd-Fe-B magnets exhibit outstanding magnetic properties, they have suffered from poor corrosion resistance in humid air. Because of this, various problems occur such as degradation of magnetic properties and/or contamination of the magnetic circuits. The corrosion rate of Nd-Fe-B magnets was found to vary with O, C, and N contents and to reach its minimum when these elements are within the range of: O = 0.6 to 1.2%; C = 0.05 to 0.15%; and N = 0.05 to 0.10%. To understand the corrosion mechanism of Nd-Fe-B magnets and the effects of these elements on corrosion resistance, various Nd-Fe-B magnets were metallographically examined. The corrosion resistance of Nd-Fe-B magnets is closely related to the microstructure, which is determined by controlling the minor elements such as O, C, and N. When the oxygen or the carbon content is low, the alloy forms a thick Nd-rich phase (α -Nd or α -Nd and Nd-oxide) on the grain boundaries. As oxygen and carbon contents increase, the Nd-rich phase at the grain boundaries becomes thinner and agglomerates into the triple junction. As oxygen content increases, the α -Nd changes to the Nd-oxide. This microstructural change may be responsible for improving the corrosion resistance. A small increase in N content further improves corrosion resistance.

KEY WORDS: Nd-Fe-B magnet, corrosion, corrosion resistance, corrosion mechanism, humid environment, autoclave test, Nd-rich phase, α -Nd, NdO_x, Nd₂O₃, grain boundary, stabilization, carbon, nitrogen, oxygen, coating

Since rare earth permanent magnets were introduced in 1965, there has been significant improvement in magnetic properties by modification of alloy compositions and processes. Among these materials, Nd-Fe-B magnets have assumed an important position due to their outstanding magnetic properties [1,2]. However, corrosion has been a problem with Nd-Fe-B magnets because the phases rich in rare earth elements are easily oxidized in the air, especially in humid air [3,4]. Corrosion of Nd-Fe-B magnets in service can degrade their magnetic properties and also produce contaminants that can be detrimental to the magnetic circuits in which the magnets are used. Therefore, much effort has been made to improve the corrosion resistance of the Nd-Fe-B magnets. As a result, substantial improvements have been made. Although coating techniques and surface treatments have improved corrosion resistance to a certain degree, the corrosion problems of Nd-Fe-B magnets still remain. This is because the Nd-Fe-B magnet which serves as substrate for coating is prone to react with moisture. The coating or plating may be imperfect and allow the permeation of reacting species such as mois-

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ture. Nd-Fe-B magnets are protected from corrosion only when three conditions are satisfied: (1) a dense and protective coating and/or plating has been applied to the surface of the magnet; (2) a protective layer has been formed on the surface or the reactive Nd-rich phases have been otherwise removed from the surface by proper surface treatment; and (3) an improvement has been made to the intrinsic corrosion resistance of the substrate magnet. Consequently, consideration was given to the improvement of the intrinsic corrosion resistance of the uncoated magnets.

Narasimhan et al. [5] reported that raising the oxygen content to between 0.6 to 3.5% in Nd-Fe-B magnets significantly improved the corrosion resistance by reducing the disintegration of magnets in hot and humid environments. Kim and Jacobson [3] reported that the addition of Al and Dy or dysprosium oxide (Dy_2O_3) in Nd-Fe-B alloys improved the corrosion resistance in humid air. Sagawa et al. [6] reported that the addition of Co and Al improved the corrosion resistance of the Nd-Fe-B magnets. Nakamura et al. [7] reported that the substitution of Fe with Co and Zr improved the corrosion resistance of the Nd-Fe-B magnet by stabilizing the Nd-rich grain boundary. Sagawa et al. [8] and Hirosawa et al. [9] reported that the addition of V or Mo improved corrosion resistance by preventing formation of corrosion-prone $NdFe_4B_4$ phase. Although the corrosion resistance is improved by the alloy modifications mentioned above, the corrosion rate is still substantially affected by undefined factors. The factors affecting the corrosion rate and the microstructures of uncoated Nd-Fe-B magnets will be addressed in this study. The factors include the effect of the addition of O, C, and N.

Experimental Procedure

The Nd-Fe-B magnets used in this study were prepared by the conventional powder metallurgical sintering process. The content of oxygen or nitrogen in the alloy was controlled by introducing controlled amounts of air or nitrogen during the jet milling process. The carbon content was controlled by making carbon additions during the melting process. The prepared alloys were hydrided and jet milled to powders with average particle sizes of 1 to 4 μm by Fischer subsieve size (FSSS) measurement. The milled powder was magnetically aligned and isostatically pressed. The compacts were sintered under the vacuum atmosphere at 1030°C for 1 h. The sintered magnets were ground and sliced into cylindrical specimens.

Since previous studies [4] showed autoclave testing to be a viable accelerated method for predicting the long-term corrosion resistance of rare earth transition metal magnets in humid room temperature environments, this study used the autoclave test to measure the corrosion rate of the magnets. For comparison, a corrosion test of the magnet in the air at room temperature was also conducted.

The tested magnets were ground about 2 mm deep from the reacted surface and polished with diamond paste. The polished specimens were examined in the scanning electron microscope (SEM) with energy dispersive X-ray spectroscopy (EDX). The phases of the corrosion products were analyzed by X-ray diffractometer.

Results and Discussion

Effect of the O, C, and N Contents on the Corrosion Rate of Nd-Fe-B Magnets in an Autoclave Environment

The Nd-Fe-B magnets with various C, O, and N contents were made and ground into cylindrical specimens. Those specimens were tested in an autoclave at 5 to 10 psig (35 to 69 kPa) of steam pressure at 110 to 115°C for 40 and 96 h. After autoclave testing, the weight loss of the specimen was measured with a balance after brushing off the corrosion products from the

specimen. The weight loss per unit area of the specimen was plotted as a function of the content of O, C, and N. Since the weight loss of the magnet was measured per unit area of the specimen during the autoclave test, the weight loss may be used as a measurement of the corrosion rate of the magnet in autoclave environments. The corrosion rate is found to be significantly effected by the C, N, and O contents of the magnet.

As shown in Fig. 1, the weight loss of Fe-33.9Nd-1.15B-0.33O-0.024N magnet after 40-h exposure in an autoclave decreases rapidly as the carbon content increases from 0.06 to 0.09%, and then reaches its lowest value when the carbon content is between 0.09 and 0.11%. Further increases in the carbon content increase the weight loss. The weight loss of the same alloy with increased oxygen (0.5%) and nitrogen (0.05 to 0.09%) contents shows behavior similar to that of the alloy with low oxygen and nitrogen contents. The overall corrosion rate of the alloy, however, is reduced with increased oxygen and nitrogen contents. It is noted that as carbon content increases from 0.06 to 0.1%, the corrosion rate decreases less dramatically for the high O and N containing alloy. However, when the oxygen content is greater than 0.6%, the corrosion rate is much less sensitive to the carbon content in the range of 0.06 to 0.15%. As shown in Fig. 2, the corrosion rate of magnets with oxygen contents greater than 0.6% decreases rapidly as carbon content increases up to about 0.05% and then reaches the minimum at about 0.06 to 0.14% of carbon. If the oxygen content is about 0.7% and the carbon content exceeds 0.15%, the corrosion rate starts to increase. If the oxygen content is greater than 0.8%, then the minimum corrosion rate is maintained until the carbon content reaches about 0.2%. Therefore, when the oxygen content is greater than 0.6%, the proper range of carbon for the minimum corrosion rate is between 0.06 and 0.14%.

Since the corrosion rate reaches its lowest value when the carbon content is about 0.1%, an alloy, Fe-33.5Nd-1.1B-0.1C, was made and the corrosion rate measured as a function of O and N contents. Figures 3 and 4 show the weight loss of a Nd-Fe-B magnet after exposure in an autoclave at 5 to 10 psig (35 to 69 kPa) (110 to 115°C) for 96 h, as a function of oxygen content. As shown in Fig. 3, the corrosion rate of a Fe-33.5Nd-1.1B-0.1C-(0.05 to 0.15)N magnet decreases rapidly as the oxygen content increases from 0.2 to about 0.6% and then reaches the minimum when the oxygen content is between 0.6 and 1.0%. The minimum corrosion rate produced a weight loss of less than 1 mg/cm² and corrosion products that are hardly observable on the surface of the magnet.

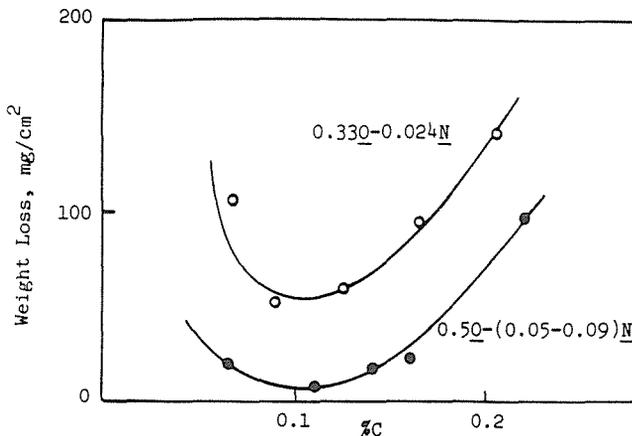


FIG. 1—Weight loss of Fe-33.9Nd-1.15B magnets after exposure in autoclave at 5 to 10 psig (35 to 69 kPa) for 40 h as a function of C, O, and N contents.

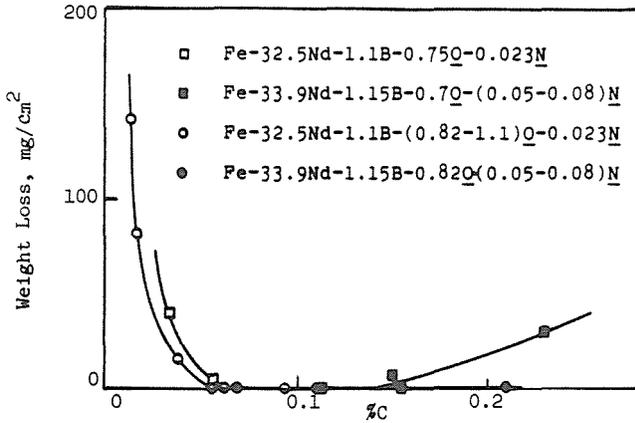


FIG. 2—Weight loss of Nd-Fe-B magnets after exposure in autoclave at 5 to 10 psig (35 to 69 kPa) for 96 h as a function of C content.

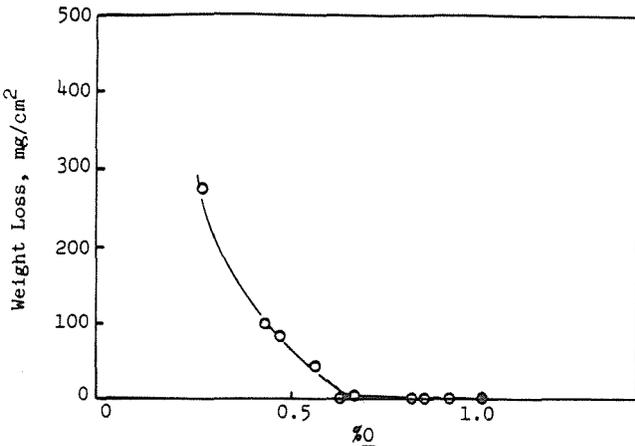


FIG. 3—Weight loss of Fe-33.5Nd-1.1B-0.1C-(0.05 to 0.15) N magnets after exposure in autoclave at 5 to 10 psig (35 to 69 kPa) for 96 h as a function of O content.

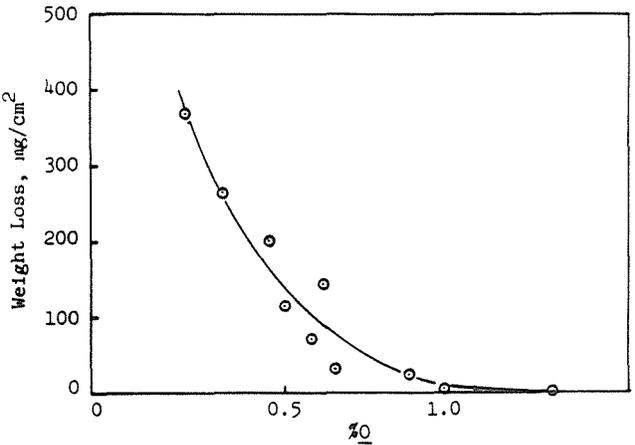


FIG. 4—Weight loss of Fe-33.5Nd-1.1B-0.1C-0.02N magnets after exposure in autoclave at 5 to 10 psig (35 to 69 kPa) for 96 h as a function of O content.

The oxygen content for the minimum corrosion rate varies depending upon the N content, although the corrosion rate decreases rapidly as the oxygen content increases up to about 0.6%. As shown in Fig. 4, the corrosion rate of the low N Fe-33.5Nd-1.1B-0.1C-0.02N magnets also decreases rapidly with increasing oxygen content from 0.2 to 0.6%, but does not reach the minimum when oxygen content is between 0.6 and 1.0%. Instead, it reaches the minimum at 1.2% oxygen content. From these results, it is noted that the corrosion rate decreases as nitrogen content increases from 0.02 to 0.05 to 0.15% at the given oxygen and carbon (0.1%) contents. This indicates that a certain range of nitrogen is beneficial in improving the corrosion resistance and shifts oxygen contents to a lower range for the minimum corrosion rate.

When the carbon content is relatively low ($\leq 0.06\%$), the effect of nitrogen content on the corrosion rate is adverse. As shown in Fig. 5, the corrosion rate of low carbon alloys increases slowly up to 0.1% N and then increases rapidly with a further increase in N content. Therefore, a nitrogen content exceeding 0.1% is detrimental to the corrosion resistance of low carbon Nd-Fe-B alloys, although somewhat higher nitrogen contents (0.05 to 0.15%) are beneficial to the corrosion resistance for high carbon Nd-Fe-B alloys. The proper nitrogen content for the minimum corrosion rate, therefore, ranges from 0.05 to 0.10% depending on the C and O contents.

These results indicate that the corrosion rate reaches a minimum when the nitrogen content is within the range of 0.05 to 0.10%, the carbon content is between 0.06 and 0.14%, and the oxygen content is between 0.6 and 1.2%.

Microstructural Study

The Nd-Fe-B magnets tested in autoclave were selected as shown in Tables 1 and 2. These were ground about 2 mm deep from the reacted surface and polished with diamond paste. The polished specimens were examined in the SEM with EDX.

Figure 6 shows the backscattered images of SEM micrographs of Specimens A (0.014%C-0.865%O) and B (0.055%C-0.815%O) at X1000 magnification. As shown in this figure, both Magnets A and B exhibit Nd-rich phases as mainly flat white phases with some raised white phases. EDX analyses on both flat and raised white phases of both Specimens A and B exhibit very high O and high Nd peaks. The raised white phases exhibit higher O and lower Nd peaks

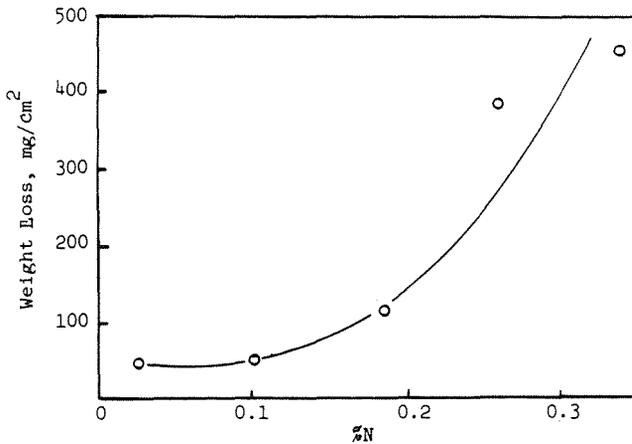


FIG. 5—Weight loss of Fe-34.2Nd-1.13B-0.55O-0.06C magnets after 40 h exposure in autoclave at 5 to 10 psig (35 to 69 kPa) as a function of N content.

TABLE 1—*Chemical compositions of the alloys examined in this study.*

Alloy No.	Chemical Composition (wt.%)					
	Fe	Nd	B	O	N	C
A	Bal	32.5	1.1	0.865	0.021	0.014
B	Bal	32.5	1.1	0.815	0.024	0.055
C	Bal	33.5	1.1	0.245	0.015	0.10
D	Bal	33.5	1.1	0.920	0.014	0.10
E	Bal	33.5	1.1	0.820	0.110	0.10

NOTE: Bal = balance.

than the flat white phases. This indicates that both white phases are Nd-rich phases containing oxygen or Nd-oxides [presumably, the flat white phases are NdO_x and the raised white phases are neodymium oxide (Nd_2O_3)]. From this result, it is noted that the α -Nd becomes Nd-oxide or a Nd-rich phase containing oxygen when the oxygen content is increased in a Nd-Fe-B magnet. The SEM micrographs show that both magnets exhibit similar microstructures, although the low carbon alloy (A) exhibited flat white phase both at triple junctions and at the grain boundaries, while the higher carbon alloy (B) exhibited the flat oxide mainly at the triple junctions. The agglomeration of flat white phase at the triple junctions may lead to thinning of the Nd-rich phase in the grain boundary. It is therefore believed that the increase in carbon content in a Nd-Fe-B magnet may cause the Nd-rich phase to agglomerate in the triple junctions and/or to stabilize the grain boundary Nd-rich phase against moisture attack. The distribution of the carbon in the magnet could not be determined due to equipment analytical limitations.

After microstructural examination, both Specimens A and B were removed from the SEM and exposed in the air at room temperature for 100 days. These specimens were again examined in the SEM.

Figure 7 shows the backscattered image of Specimens A and B after 100 days exposure in the air at room temperature. Magnet A exhibits a greatly increased number of raised corrosion products on the surface, while Magnet B exhibits few new raised corrosion products. It is rather similar to the microstructure of the freshly polished magnet (B) shown in Fig. 6. X-ray diffraction analyses of the corrosion products exhibit mainly $\text{Nd}(\text{OH})_3$ phase and $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase peaks. It is therefore assumed that the Nd-rich phases in the grain boundary and at the triple junctions react with moisture to form the neodymium hydroxide [$\text{Nd}(\text{OH})_3$] phase, which may be accompanied by a volume expansion of the Nd-rich phase at the triple junctions and grain boundaries and with grain boundary weakening. The simultaneous reaction of the grain

TABLE 2—*Weight loss of various Nd-Fe-B magnets after exposure in autoclave at 5 to 10 psig (35 to 69 kPa) (110 to 115°C) for 40 and 96 h, respectively.*

Alloy No.	Weight loss, mg/cm^2	
	40 h	96 h
A	31.8	142.0
B	1.0	0.5
C	93.0	368.0
D	0.4	6.9
E	0.3	0.4

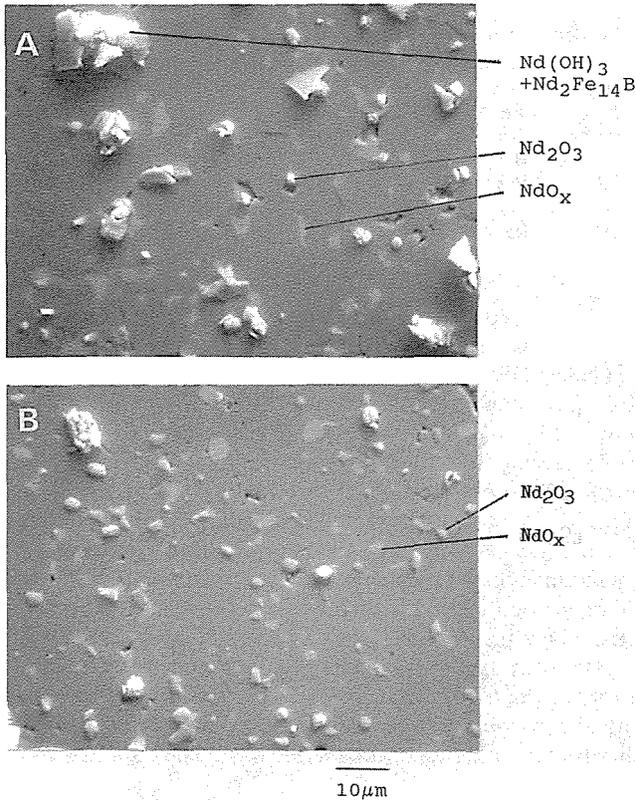


FIG. 6—SEM micrographs of Specimens A and B after polishing, back-scattered image (BSI), X1000.

boundary weakening and volume expansion of Nd-rich phase may separate a $\text{Nd}_2\text{Fe}_{14}\text{B}$ grain from the other grains and push it to the surface of the magnet.

These results indicate that carbon plays a very important role in improving the corrosion resistance of the Nd-Fe-B magnet in the ambient environment as well as in the accelerated corrosion environment of the autoclave.

Figure 8 shows the backscattered images of SEM micrographs of Specimens C (0.1%C-0.24%O-0.015%N), D (0.1%C-0.92%O-0.014%N), and E (0.1%C-0.82%O-0.11%N). Magnet C (low oxygen and low nitrogen) exhibits mainly flat white phases at the grain boundary and triple junctions and a few raised white phases near the triple junctions. The EDX analysis shows that this flat white phase exhibits only Nd peaks without oxygen peaks and that the raised white phase exhibits high O and Nd peaks. This indicates that the flat white phase of this magnet is the α -Nd phase and the raised phase is Nd-oxide (presumably Nd_2O_3). These results indicate that the magnet with low O, low N, and high C contents formed mainly α -Nd on the grain boundaries and triple junctions and some Nd-oxide near the triple junctions.

Magnet D (high oxygen and low nitrogen) exhibits mainly flat white phases at the triple junctions and some raised white phases near the triple junctions. The EDX analysis on both flat and raised white phases exhibit high O and Nd peaks. The raised phase exhibits an oxygen peak higher and a Nd peak lower than those of the flat white phase. This indicates that both

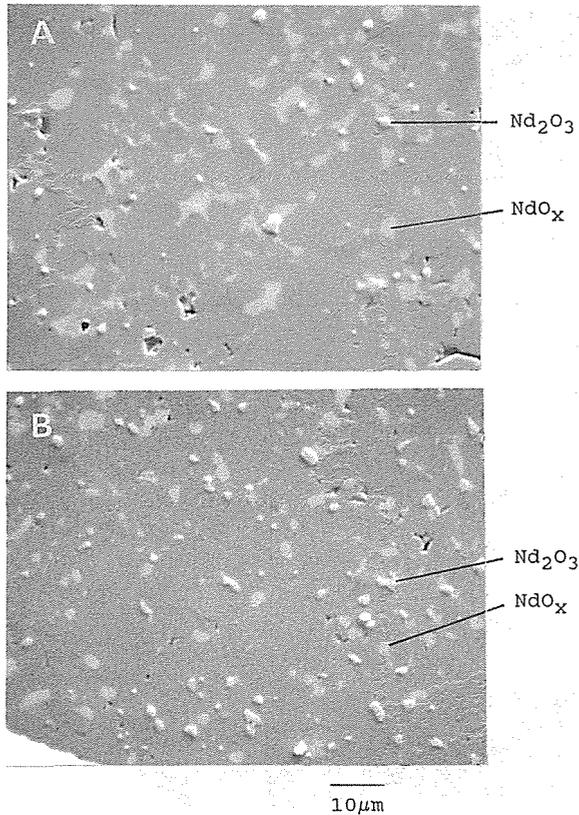


FIG. 7—SEM micrographs of Specimens A and B after 100-h exposure in air at room temperature, BSI, X1000.

flat and raised white phases of this magnet are Nd-oxides (presumably the flat phase is NdO_x and the raised phase is Nd_2O_3). These results indicate that the magnet with high O, low N, and high C formed Nd-rich phase as NdO_x mainly at the triple junctions and raised oxides (presumably Nd_2O_3) near the triple junctions.

Magnet E (high O, high N, and high C) exhibits mainly raised white phases and a few flat white phases near the triple junctions. The EDX analyses show that both raised and flat white phases exhibit high O and Nd peaks. The raised white phase exhibit an oxygen peak higher and an Nd peak lower than those of the flat white phase. This indicates that the magnet with high O, high N, and high C formed mainly raised oxides (presumably Nd_2O_3) near the triple junctions and a few flat oxides (presumably NdO_x) at the triple junctions.

From these results, it is found that the magnet with low O and low N contents forms mainly α -Nd as a Nd-rich phase both at grain boundaries and triple junctions. If only the oxygen content is increased in that alloy, the α -Nd is converted to an Nd-rich phase containing oxygen (presumably NdO_x) and the Nd-rich phase moves from the grain boundaries to the triple junctions. When both oxygen and nitrogen contents are high in the magnet, most of the flat Nd-oxide (presumably NdO_x) converts to the raised Nd-oxide (presumably Nd_2O_3). This indicates that small amounts of nitrogen addition helps to convert unstable Nd-oxide (presumably

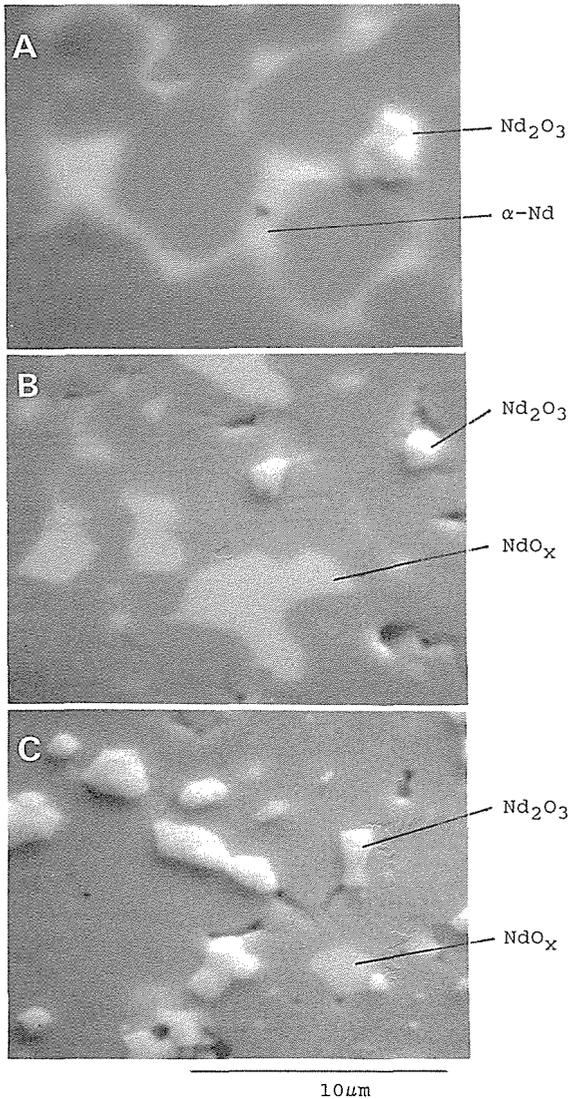
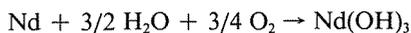


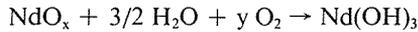
FIG. 8—SEM micrographs of Specimens C, D, and E, BSI, X5000.

NdO_x) to stable Nd-oxide (presumably Nd_2O_3). It is believed, therefore, that the change in microstructure is responsible for improving the corrosion resistance of the Nd-Fe-B magnets.

Based on the present study, the model for corrosion mechanism of Nd-Fe-B magnets can be suggested as follows: Since the Nd-rich phase ($\alpha\text{-Nd}$ or NdO_x) is much more reactive than the matrix $\text{Nd}_2\text{Fe}_{14}\text{B}$ phase, there will be a preferential corrosion reaction of the Nd-rich phase when there is enough moisture in the air. The reaction may initiate from the Nd-rich phase as follows:



or



where $y = 3/4 - X/2$.

When the grain boundary is coated with an Nd-rich phase of sufficient thickness and is exposed to moist air (Fig. 9A), then the corrosion reaction of the Nd-rich phase may proceed continuously along the grain boundary with an accompanying volume expansion of corrosion products as shown in Fig. 9C. The volume expansion of the corrosion products may lead to intergranular fracture or displacement of the grain, thus providing a path of moisturized air to the next Nd-rich boundary phase; thus the corrosion reaction may proceed continuously. The simultaneous corrosion reaction of the Nd-rich phase and volume expansion of corrosion products may lead to continuous corrosion. On the other hand, if the Nd-rich phase at the grain boundary is very thin or located only at the triple junctions (Fig. 9B), the continuous corrosion reaction may be confined to the triple junctions at the surface and, therefore, may not be self-propagating as shown in Fig. 9D. When the Nd-rich phase is stabilized against moisture by reducing its reactivity by the addition of effective elements such as O, C, and N, the corrosion reaction will be further restricted. The corrosion rate of the magnet is a function of the microstructure of the magnet, the reactivity of the Nd-rich phase (alloying composition), the humidity level at the interface between metal and environment, and temperature.

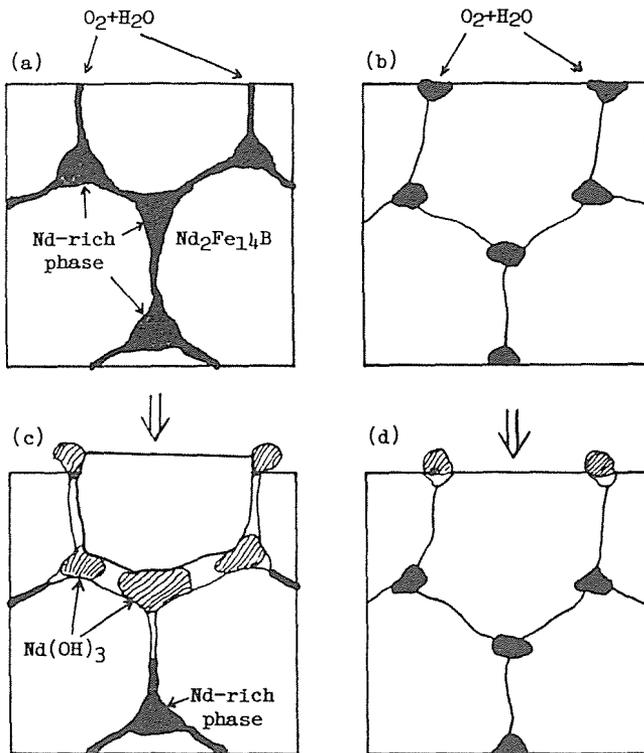


FIG. 9—Model for corrosion mechanism of Nd-Fe-B magnets in humid air: (a) Nd-Fe-B magnet with thick grain boundaries; (b) magnet with thin grain boundaries; (c) magnet (a) after exposure in humid air, and (d) magnet (b) after exposure in humid air.

Therefore, the corrosion resistance of the Nd-Fe-B magnet can be improved by modifying the microstructure, stabilizing the Nd-rich phase against humid air, and by reducing the moisture level at the metal-gas interface by forming a protective layer on the surface or by applying a dense and protective coating/plating layer on the surface of the magnet.

Summary

The corrosion resistance of the Nd-Fe-B magnets can be improved when three conditions are satisfied: (1) a protective coating has been applied to the surface of the magnet; (2) a protective layer has been formed on the surface and/or the reactive Nd-rich phases have been otherwise removed from the surface; and (3) an improvement has been made to the intrinsic corrosion resistance of the substrate magnet. The intrinsic corrosion resistance of the uncoated magnet is significantly affected by the O, C, and N contents of the alloy.

The corrosion rate rapidly decreases with increasing oxygen content and reaches the minimum when the oxygen content is between 0.6 and 1.2%. The corrosion rate rapidly decreases as the carbon content increases up to about 0.06% and then gradually decreases as the carbon content increases up to about 0.1%. A further increase in the carbon content up to about 0.15% slowly increases the corrosion rate and then increases the rate substantially with further carbon increase ($>0.15\%$). Therefore, the proper carbon content for the low corrosion rate is $0.10 \pm 0.04\%$. The effect of carbon content on the corrosion rate becomes smaller at higher oxygen contents in the alloy. The effect of nitrogen content on the corrosion rate is less dramatic when it is less than 0.1%, i.e., the corrosion rate decreases with increasing nitrogen content up to 0.10% when oxygen and carbon contents are properly high. The corrosion rate increases slowly with a nitrogen content increase when the carbon and oxygen contents are low. If the nitrogen content exceeds 0.1% in a low C and O alloy, the corrosion rate increases dramatically. The results of this investigation indicate that the corrosion rate reaches the minimum when: O = 0.6 to 1.2%; C = 0.06 to 0.14%; and N = 0.05 to 0.10%.

The corrosion resistance of the Nd-Fe-B magnet is closely related to the microstructure, which is determined by controlling the minor elements such as O, C, and N. When the oxygen or carbon content is low, the alloy forms a thick Nd-rich phase (α -Nd and/or Nd-oxide) on both grain boundaries and triple junctions of the grain boundary. As the oxygen and carbon contents increase, the Nd-rich coating on the boundaries becomes thinner and agglomerates into the triple junctions. As oxygen content increases, the α -Nd changes to the Nd-oxide. This microstructural change may be responsible for improving the corrosion resistance. A small increase in N further improves the corrosion resistance.

Acknowledgment

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