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ARTICLE

Influence of Phosphating Temperature on Microstructure and Corrosion Resistance of Phosphate Chemical Conversion Coating on Sintered Nd-Fe-B Permanent Magnets

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Abstract: The effect of phosphating temperature on formation, microstructure, and corrosion resistance of the phosphate chemical conversion (PCC) coatings and that on the magnetic property of the sintered Nd-Fe-B permanent magnets were investigated. The results show that the coating mass is increased slightly with increasing the phosphating temperature. The scanning electron microscope observation demonstrates that PCC coatings have blocky structure with the grain size of 5~10 μm . The analyses of energy dispersive spectra and Fourier transform infrared spectrometer spectra reveal that the coatings are mainly composed of neodymium phosphate hydrate, praseodymium phosphate hydrate, and a small amount of iron phosphate hydrate. The oxygen and phosphorous elements in PCC coatings are mainly distributed on the grain surface, while the iron element is mainly concentrated at the grain boundaries. The distribution of neodymium and praseodymium is relatively uniform. The electrochemical analysis and static immersion corrosion test show that PCC coatings prepared at different temperatures can effectively improve the corrosion resistance of the sintered Nd-Fe-B permanent magnets. The coatings prepared at 70 $^{\circ}\text{C}$ exhibit a better corrosion resistance due to the uniform and dense microstructure. Although the magnetic properties of the sintered Nd-Fe-B permanent magnets with PCC coatings are decreased, those with PCC coatings prepared at 70 $^{\circ}\text{C}$ are relatively fine. The optimal phosphating temperature of 70 $^{\circ}\text{C}$ for the sintered Nd-Fe-B permanent magnets is determined.

Key words: Nd-Fe-B permanent magnets; phosphate chemical conversion; phosphating temperature; microstructure; corrosion resistance; magnetic properties

Sintered Nd-Fe-B permanent magnet is a rare earth permanent magnetic material with high remanence (11 kG), high coercivity (1.03 MA/m)^[1], and maximum energy product (286~398 kJ/m³)^[2], which is widely used in the electrical instruments, communication, new energy vehicles, and other fields because of its excellent magnetic properties. However, the sintered Nd-Fe-B magnets easily suffer corrosion under the aggressive environment because of the high electrochemical activity of the Nd-rich phase and the porous structure. Therefore, a protective coating through the surface modification is crucial for Nd-Fe-B magnets. In recent years, various protective coatings have been prepared on the surface of sintered Nd-Fe-B permanent magnets, such as aluminum-sol coating^[3], diamond-like carbon coating^[4], magnetron

sputtering multilayer coating^[5], and amorphous SiO₂ coating^[6]. However, these coatings have the disadvantages of complex preparation, high cost, and weak adhesion.

The phosphate chemical conversion (PCC) is a surface treatment technique to form the non-conductive pyknotic phosphate coatings on the metal surface through the chemical and electrochemical reactions. PCC plays a significant role in the improvement of surface corrosion resistance owing to its low-cost, rapid coating formation, good adhesion, and easy availability for treatment of irregular surface^[7,8]. At present, PCC is mainly applied to conventional metal materials, such as steel materials^[9-11], magnesium alloys^[12-14], and aluminum alloy^[15,16]. However, the sintered Nd-Fe-B permanent magnets treated by PCC are relatively rarely reported.

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Furthermore, the researches mainly focus on the effect of additives and auxiliary means on the microstructure and corrosion resistance of PCC coatings. Saliba-Silva^[17] and Takeuchi^[18] et al investigated the effects of molybdate, tungstate, and other composites on the corrosion resistance of PCC coatings, and found that when the phosphating duration is 4 h, a small amount of molybdate can slightly improve the corrosion resistance of phosphate coatings. However, when the phosphating duration extends to 18 h, the addition of molybdate may in reverse reduce the corrosion resistance. The addition of tungstate can effectively improve the corrosion resistance of PCC coatings, because the tungstate can adhere to the substrate through the holes on the coatings, therefore repairing the defects of phosphate coatings on the magnet surface. Cerium can also increase the corrosion resistance^[19]. Kosobudskii et al^[20] studied the Nd-Fe-B magnets after ultrasonic phosphating, and found that the high-frequency phosphating can better enhance the growth rate and corrosion resistance of the coatings. Sun et al^[21] investigated the corrosion behavior of Nd-Fe-B magnet with and without ultrasonic phosphating coatings prepared at room temperature. The combination of phosphating treatment with ultrasonic improves the corrosion resistance of Nd-Fe-B magnet. The phosphate coating after ultrasonic treatment is more homogeneous and compact.

However, the effect of phosphating temperature is rarely investigated. For an endothermic reaction, raising the temperature benefits the chemical conversion. The coating formation speed and the coating mass can be increased if the temperature rises properly, because the surface activity of the material is enhanced. However, the precipitation may occur in the conversion solution at excessively high temperature, which will not only waste the coating-forming ions, but also coarsen the coating grains and reduce the coating corrosion resistance. If the conversion temperature is too low, the chemical conversion reaction cannot proceed sufficiently, resulting in the incomplete covering for the substrate surface or even the fact that PCC coatings cannot be formed^[22]. The corrosion resistance of PCC coatings is affected by several parameters, such as coating mass and structure homogeneity^[7,23]. Thus, the uniform coating with high coating mass and low porosity can improve the corrosion resistance, which is also the aim of the surface modification. In addition, the magnetic properties of sintered Nd-Fe-B permanent magnets are greatly affected by the temperature. Wang et al^[24] studied the effect of temperature on the magnetic properties of sintered 33EH Nd-Fe-B high-performance permanent magnet, and found that the magnetic remanence and the maximum magnetic energy product of the magnets are decreased obviously with increasing the temperature. When the Nd-Fe-B magnet is heated from room temperature to 100 °C, a reduction ratio of 7.6% and 14.9% for remanence and maximum energy product appears, respectively. When the temperature is further raised, these properties are decreased even obviously^[24]. Therefore, the temperature should be strictly controlled in the process of chemical conversion.

In this research, the microstructures and properties of phosphate coatings fabricated at different conversion temperatures were investigated, and the optimum temperature for PCC coating formation on the surface of the Nd-Fe-B permanent magnets was determined.

1 Experiment

The commercially available N40HCE Nd-Fe-B magnets were used as the substrate with the size of 10 mm×10 mm×5 mm, 25 mm×25 mm×2 mm, and Φ 10 mm×8 mm. Manganese dihydrogen phosphate was used as the main coating forming component in the phosphate solution with a small amount of accelerator. Based on Ref.[25], the pH value of the phosphate solution was 1.0.

The specimens were abraded using silicon carbide paper and then degreased in alkaline degreaser at 60 °C for 15 min. Then the pickling treatment with ultrasonic was conducted in the solution of 1.5vol% nitric acid at room temperature for 100 s. After cleaning by deionized water for several times, the specimens were immersed in the phosphate solution at 50~90 °C for 20 min. Finally, the specimens were washed with deionized water and dried by blowing air at room temperature. The flow diagram for preparation of PCC coatings is shown in Fig.1.

The TE214S analytical balance with the accuracy of ± 0.1 mg was used to measure the specimen mass. The coating mass M ($\text{g} \cdot \text{m}^{-2}$) can be calculated by Eq.(1), as follows:

$$M = \frac{m_1 - m_2}{A} \quad (1)$$

where m_1 and m_2 are the mass (g) of the specimens before and after the coatings are stripped, respectively; A is the area of the substrates (m^2). The coating stripping was conducted in a solution of 100 $\text{g} \cdot \text{L}^{-1}$ sodium hydroxide, 90 $\text{g} \cdot \text{L}^{-1}$ ethylene diamine tetraacetic acid (EDTA) tetrasodium, and 4 $\text{g} \cdot \text{L}^{-1}$ triethanolamine at 90 °C for 15 min. The process was conducted over and over until the mass change of the specimen was less than 0.1 mg. Each group was repeated five times to characterize the dispersion degree of the experiment results.

The microstructures and element distributions of PCC coatings and the sintered Nd-Fe-B substrates were observed using the SU-70 field emission scanning electron microscope (FE-SEM) coupled with the energy dispersive spectrometer (EDS). Furthermore, the Fourier transform infrared spectrometer (FTIR, BRUKER TENSOR 37) was used to characterize the functional groups of PCC coatings with the spectra range of 400~4000 cm^{-1} . The specimens for FTIR analysis were prepared with the powder mixture of KBr and PCC coatings scraped off from the Nd-Fe-B magnets.

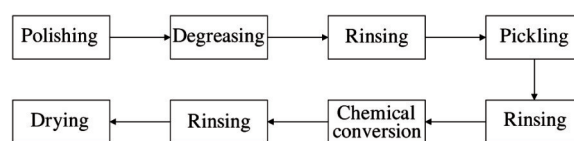


Fig.1 Flow diagram of preparation of PCC coatings

The corrosion resistance of the specimens was evaluated by the electrochemical tests with the classical three-electrode cell. The platinum was used as the counter electrode, the saturated calomel electrode (SCE) was used as the reference electrode, and the uncoated or coated specimens with an exposed area of 1 cm² were used as the working electrode. The corrosive medium was 3.5wt% sodium chloride solution. The open circuit potential (OCP) and the potentiodynamic polarization measurement were conducted using the electrochemical workstation (Parstat 2273) at room temperature. OCP was recorded as a function of time from 0 s to 1800 s for substrates and from 0 s to 1200 s for specimens with PCC coatings. The potentiodynamic polarization curves of the specimens were tested at a constant voltage scan rate of 5 mV·s⁻¹.

The static immersion corrosion test was also conducted to assess the corrosion resistance of the bare Nd-Fe-B substrate and the specimens after PCC treatment at different phosphating temperatures. The corrosive medium was 3.5wt% sodium chloride solution and the soak duration was 192 h. The corrosion rate v (mg·cm⁻²·h⁻¹) can be calculated by Eq.(2), as follows:

$$v = \frac{M_1 - M_2}{St} \quad (2)$$

where M_1 and M_2 are the mass (mg) of the specimens before and after immersion, respectively; S is the exposed area of the substrates (cm²); t is the soak duration.

The magnetic properties, including intrinsic coercivity, remanence, maximum energy product, and squareness, of Nd-Fe-B magnets with and without PCC coatings were measured at room temperature by NIM-2000 magnetic measuring instrument (National Institute of Metrology of China). At least five specimens were examined under each condition to acquire the mean value and standard deviation.

2 Results and Discussion

2.1 Coating mass

The coating mass is the prime factor to assess the quality of phosphating bath^[26]. The mass and thickness of PCC coatings prepared at different phosphating temperatures for 20 min are shown in Table 1. The coating thickness is obtained based on the relationship between thickness and mass according to Ref. [27]. Generally, the coating mass rather than the coating thickness is more commonly used for evaluation^[27].

Table 1 Mass and thickness of PCC coatings prepared at different phosphating temperatures

Phosphating temperature/°C	Coating mass/g·m ⁻²	Coating thickness/μm
50	3.93±0.07	3
60	4.14±0.07	3
70	4.16±0.08	3
80	4.21±0.18	3
90	4.37±0.22	3

It is clearly observed that the coating thickness of 3 μm barely changes, but the coating mass is increased slightly from 3.93±0.07 g·m⁻² to 4.37±0.22 g·m⁻² with increasing the phosphating temperature from 50 °C to 90 °C, because the low-energy points on the surface of sintered Nd-Fe-B permanent magnets can be activated through temperature rise^[22,28]. As a result, the number of nucleation points rises and then the coating mass increases. The higher temperature can benefit the formation of PCC coatings^[7].

2.2 Microstructure characterization

FE-SEM image and EDS analysis of the Nd-Fe-B substrate are shown in Fig.2. It can be seen that the sintered Nd-Fe-B magnets are mainly composed of Nd₂Fe₁₄B main phase (dark part) and Nd-rich grain boundary phase (bright part) with a few holes. Fig.2b reveals that the main constituent elements of the magnets are Fe, Pr, Nd, a little Al, and a little O. The aluminum is added to refine the grain and improve the intrinsic coercivity of the magnets. Oxygen exists because of the weak oxidation of Nd, Fe, and other elements.

Fig. 3 shows FE-SEM microstructures and corresponding EDS analyses of PCC coatings prepared at different phosphating temperatures for 20 min. It can be noticed that the morphologies are basically the same. PCC coatings are mainly composed of irregular block grains attached to the surface of sintered Nd-Fe-B matrix with a grain size of 5~10 μm. EDS results show that oxygen, phosphorus, neodymium, praseodymium, and iron are the main constituent elements of the coatings. It can be inferred that the main phases of PCC coatings are the neodymium phosphate hydrate, praseodymium phosphate hydrate, and a small amount of iron phosphate hydrate. The phosphating temperature has a little effect on the phase composition of PCC coatings on the

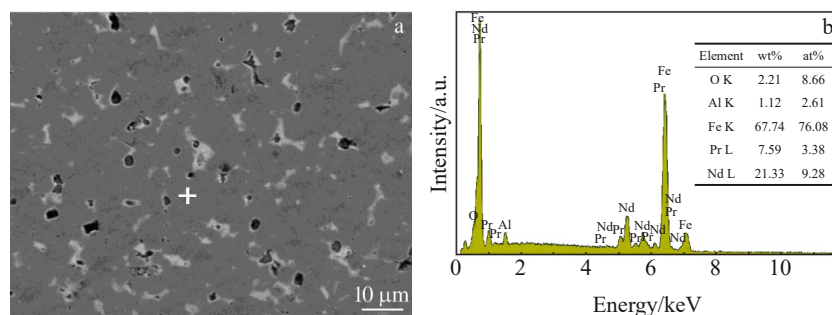


Fig.2 FE-SEM image (a) and EDS point scanning analysis (b) of sintered Nd-Fe-B substrate

surface of sintered Nd-Fe-B permanent magnets.

In addition, Fig. 3 indicates that the conversion coating prepared at the phosphating temperature of 50 °C has a relatively large grain spacing. The density of PCC coatings is

increased with increasing the phosphating temperature. However, when the temperature is elevated to 80 °C, the grains grow into different sizes and the surface roughness increases. This may be caused by the dissolution and

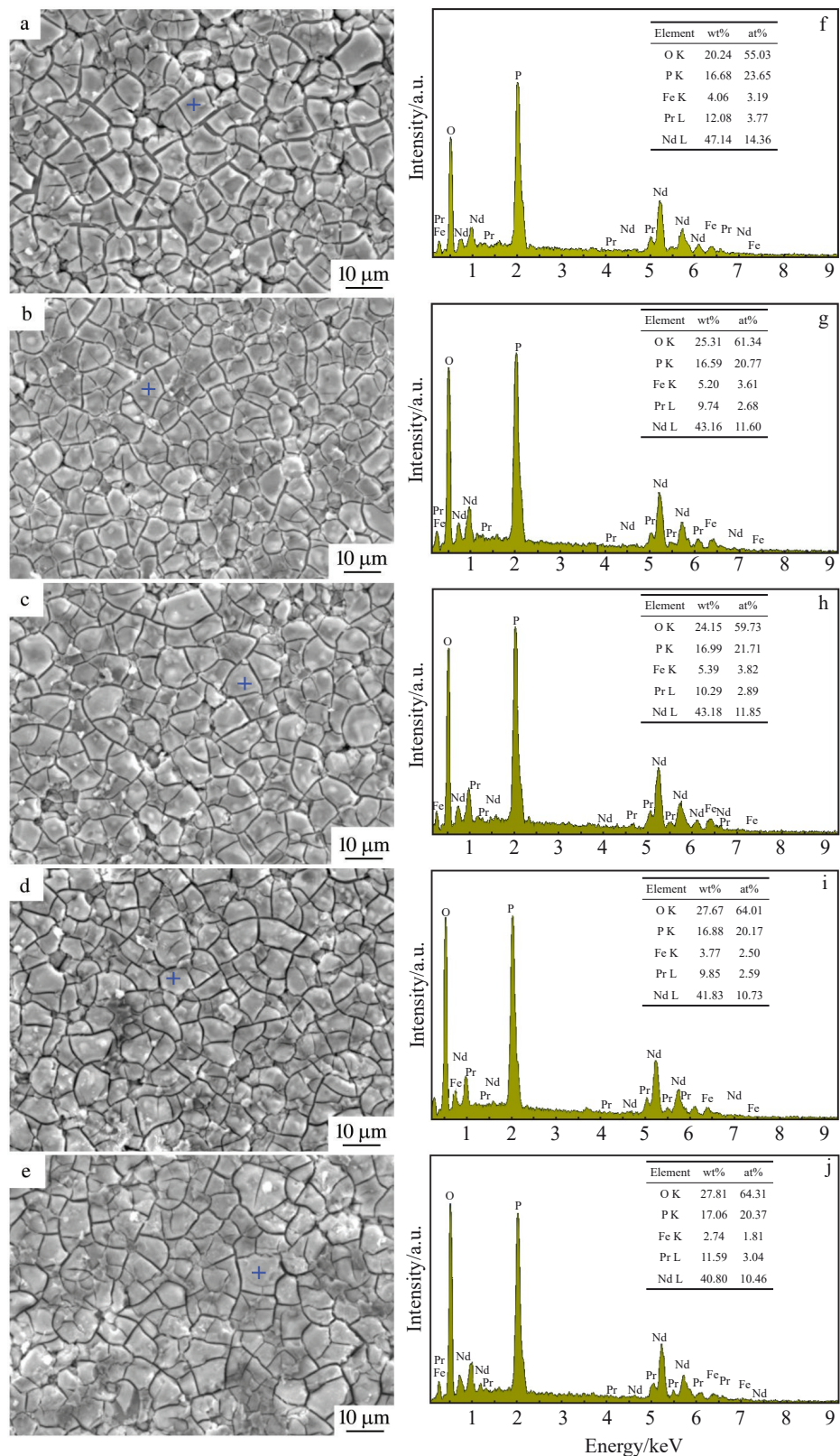


Fig.3 FE-SEM microstructures (a~e) and corresponding EDS point scanning analyses (f~j) of Nd-Fe-B magnets with PCC coatings prepared at different phosphating temperatures: (a, f) 50 °C, (b, g) 60 °C, (c, h) 70 °C, (d, i) 80 °C, and (e, j) 90 °C

recrystallization of the coatings in the acidic conversion solution at higher temperatures^[22,29]. The coatings prepared at 60 and 70 °C are more uniform and denser, respectively.

Fig. 4 shows the high-magnification FE-SEM images and element distribution of PCC coatings prepared at different phosphating temperatures. It can be inferred that the elements

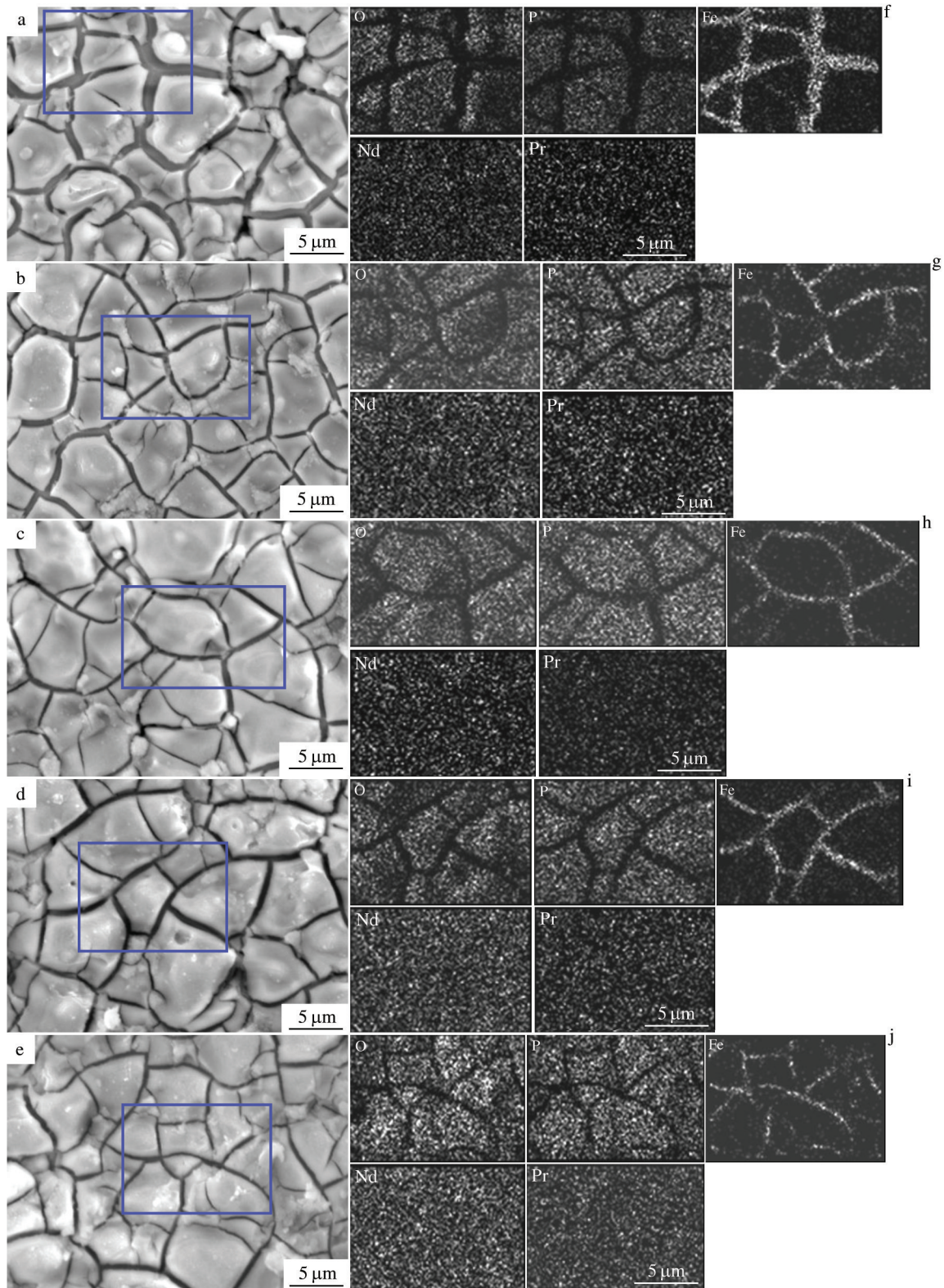


Fig.4 FE-SEM images at high magnification (a~e) and corresponding element distributions of rectangular areas in Fig.4a~4e (f~j) of Nd-Fe-B magnets with PCC coatings prepared at different phosphating temperatures: (a, f) 50 °C, (b, g) 60 °C, (c, h) 70 °C, (d, i) 80 °C, and (e, j) 90 °C

exhibit the similar distribution regularity. The oxygen and phosphorus are mainly distributed on the surface of the crystal grains. However, the iron element is mainly concentrated at the grain boundaries. The neodymium and praseodymium are slightly less at the grain boundaries, but exhibit a relatively uniform distribution.

2.3 Functional group characterization

FTIR spectra of PCC coatings obtained at different temperatures are shown in Fig.5. All FTIR spectra of coatings exhibit the typical absorption peak of PO_4^{3-} . The obvious bending vibration peaks and the stretching vibration peaks indicate that the coatings contain phosphate, which is consistent with EDS results. The stretching vibration peaks at $3600\sim 3300\text{ cm}^{-1}$ correspond to $-\text{OH}$ and those at about 1650 cm^{-1} correspond to H_2O , indicating the existence of hydrate in PCC coatings. In addition, the strong absorption peaks at about 1060 cm^{-1} correspond to PO_4^{3-} , while the absorption peaks at 520 and 540 cm^{-1} correspond to $\text{O}=\text{P}=\text{O}$.

2.4 Corrosion resistance evaluation

Fig. 6 shows the static immersion corrosion rates of Nd-Fe-B substrate and Nb-Fe-B magnets with PCC coatings prepared at different phosphating temperatures in 3.5wt% sodium chloride solution. It can be seen that the fastest corrosion rate of $0.0147\pm 0.0034\text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$ occurs for the

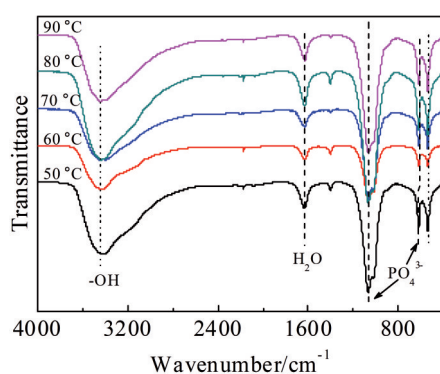


Fig.5 FTIR spectra of PCC coatings prepared at different phosphating temperatures

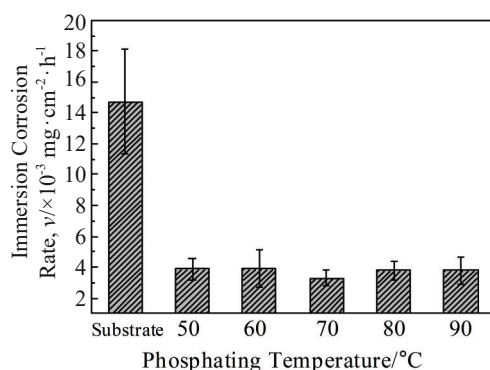


Fig.6 Static immersion corrosion rates of Nd-Fe-B substrate and Nb-Fe-B magnets with PCC coatings prepared at different phosphating temperatures

Nd-Fe-B substrate. When the phosphating temperature is $70\text{ }^{\circ}\text{C}$, the lowest corrosion rate can be acquired. The corrosion rates of other magnets with PCC coatings are similar, about $0.0038\text{ mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$, indicating that the temperature has a little effect on the corrosion resistance of the Nd-Fe-B magnets with PCC coatings.

The change trends of OCP of Nd-Fe-B substrate and Nb-Fe-B magnets with PCC coatings prepared at different phosphating temperatures in 3.5wt% sodium chloride solution are shown in Fig. 7. It can be seen that OCP of sintered Nd-Fe-B substrate is stable at around -0.955 V vs. SCE, while that of sintered Nd-Fe-B magnets with PCC coatings is relatively higher, suggesting that PCC coating can improve the corrosion resistance of the Nd-Fe-B magnets. Among them, the highest stable OCP value of PCC coating prepared at $70\text{ }^{\circ}\text{C}$ represents the optimal corrosion resistance. When the conversion temperature is $50\text{ }^{\circ}\text{C}$, the grain spacing of PCC coating is larger, i.e., the density of PCC coating is reduced, thereby weakening the corrosion resistance. When the phosphating temperature increases to 80 and $90\text{ }^{\circ}\text{C}$, OCP of the coatings fluctuates greatly, suggesting the poor stability of the coatings prepared under these conditions. According to Fig.3 and Fig.4, PCC coating prepared at $70\text{ }^{\circ}\text{C}$ is denser and more uniform in microstructure, which benefits the corrosion resistance. When the phosphating temperature increases to $80\text{ }^{\circ}\text{C}$, the grains of PCC coating are no longer uniform, and the surface dissolution leads to poor stability and inferior corrosion resistance.

The potentiodynamic polarization curves of Nd-Fe-B substrate and Nb-Fe-B magnets with PCC coatings prepared at different phosphating temperatures are presented in Fig. 8. The corrosion potential E_{corr} and the corrosion current density I_{corr} of the Nd-Fe-B substrate and Nd-Fe-B magnets with PCC coatings in the 3.5wt% sodium chloride solution are listed in Table 2, which are calculated based on the data in Fig.8 by the extrapolation method.

It can be found that E_{corr} determined by OCP is higher than that obtained by the polarization curves due to the scan rate effect on the Tafel slopes and the disturbance of the charging current^[30]. Fig. 8 and Table 2 show that for the Nd-Fe-B magnets with PCC coatings, their I_{corr} is decreased and E_{corr} is increased, compared with those of the Nd-Fe-B substrate, indicating that Nd-Fe-B magnets with PCC coatings have better corrosion resistance than Nd-Fe-B substrate does. The better corrosion resistance provided by PCC coatings is due to the chemical stability of phosphate. With increasing the phosphating temperature, the corrosion voltage of PCC coatings is firstly increased and then decreased, while the corrosion current is firstly decreased and then increased. This result indicates that the corrosion resistance of PCC coatings is increased firstly and then decreased with increasing the phosphating temperature. The best corrosion resistance is obtained when the phosphating temperature is $70\text{ }^{\circ}\text{C}$, which is in accordance with the tightly assembled crystal microstructure of PCC coating prepared at $70\text{ }^{\circ}\text{C}$. But the difference in corrosion resistance of PCC coatings prepared at

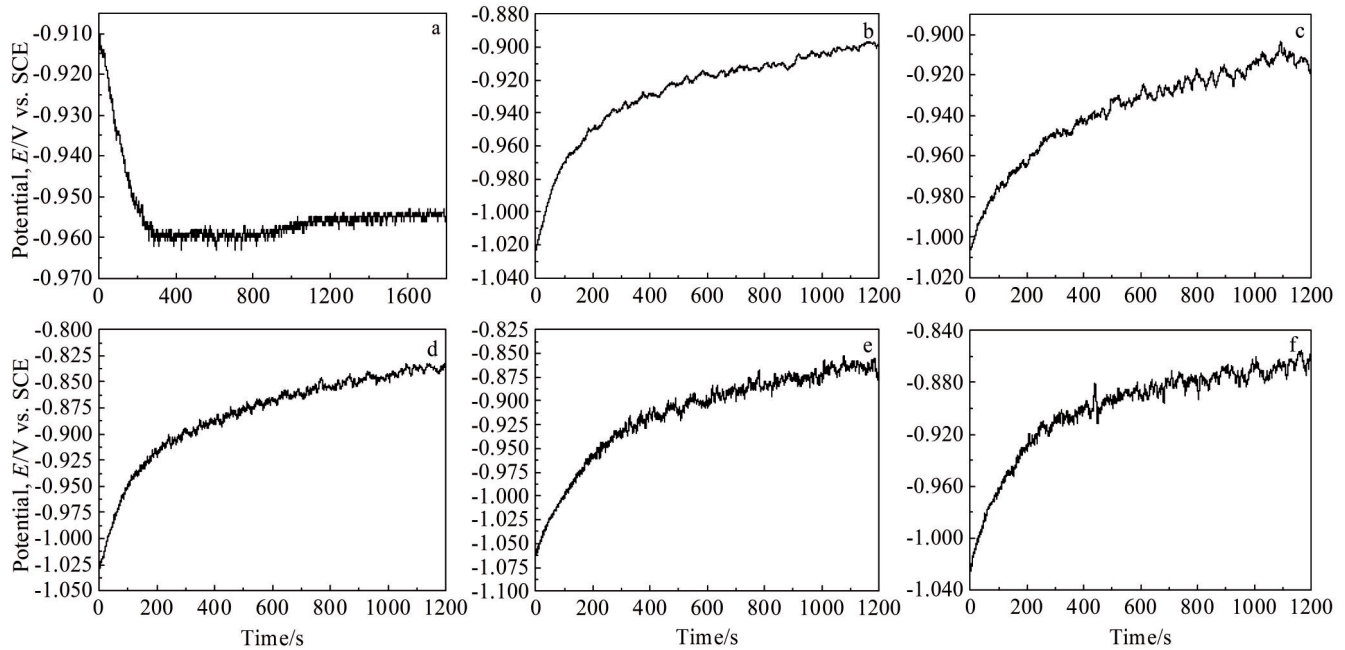


Fig.7 OCPs of Nd-Fe-B substrate (a) and Nd-Fe-B magnets with PCC coatings prepared at phosphating temperatures of 50 °C (b), 60 °C (c), 70 °C (d), 80 °C (e), and 90 °C (f)

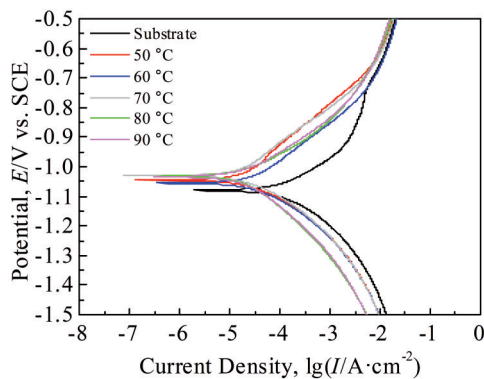


Fig.8 Potentiodynamic polarization curves of Nd-Fe-B substrate and Nb-Fe-B magnets with PCC coatings prepared at different phosphating temperatures

different temperatures is slight, which demonstrates that the phosphating temperature has a little effect on the corrosion resistance of PCC coating on the surface of the sintered Nd-Fe-B permanent magnets.

2.5 Magnetic properties

The intrinsic coercivity, remanence, maximum energy product, and squareness are the main technical indicators to assess the magnetic properties of sintered Nd-Fe-B permanent magnets. Fig.9 shows these magnetic properties of the Nd-Fe-B substrate and Nb-Fe-B magnets with PCC coatings prepared at different phosphating temperatures.

Owing to the corrosive action of the acidic conversion solution, the magnetic properties of all Nd-Fe-B magnets after PCC treatment are decreased to a certain degree. When the magnets are immersed in the acidic chemical conversion

Table 2 Corrosion potential E_{corr} and corrosion current density I_{corr} of Nd-Fe-B substrate and Nb-Fe-B magnets with PCC coatings prepared at different phosphating temperatures in 3.5wt% sodium chloride solution

Phosphating temperature/°C	$E_{\text{corr}}/\text{V vs. SCE}$	$I_{\text{corr}}/\mu\text{A}\cdot\text{cm}^{-2}$
Substrate	-1.079 ± 0.008	110.0 ± 16.7
50	-1.049 ± 0.021	28.35 ± 3.14
60	-1.063 ± 0.009	24.09 ± 4.44
70	-1.035 ± 0.003	20.24 ± 3.42
80	-1.049 ± 0.004	21.62 ± 2.49
90	-1.057 ± 0.010	28.64 ± 3.26

solution, the active Nd-rich phase will dissolve. The lamellar Nd-rich phase around the grain boundaries is beneficial to the high coercivity of the Nd-Fe-B permanent magnets^[31]. The dissolution of the Nd-rich phase leads to the decrease in intrinsic coercivity of the Nd-Fe-B magnets after PCC treatment. Furthermore, the dissolution of the Nd-rich phase results in the increase in porosity. The remanence and the maximum energy product of the magnets are mainly affected by the density^[32]. As a result, the decrease in remanence and maximum energy product is attributed to the diminution of the magnet density. Besides, the $\text{Nd}_2\text{Fe}_{14}\text{B}$ main crystal phase may also dissolve in or be corroded by the phosphating solution. The reduction in density and the loss of Nd-rich phase and $\text{Nd}_2\text{Fe}_{14}\text{B}$ main crystal phase lead to the decrease in magnetic properties when the Nd-Fe-B magnets are corroded. In addition, PCC treatment at different temperatures will generate non-magnetic PCC coatings on the specimen surface,

which can also decrease the magnetic properties of the sintered Nd-Fe-B permanent magnets.

It can also be seen from Fig.9 that there is slight change of the magnetic properties of the sintered Nd-Fe-B permanent magnets after PCC treatment at different temperatures. The magnetic properties of the Nd-Fe-B magnets are relatively fine when the phosphating temperature is 70 °C.

In general, PCC coatings prepared at 70 °C have relatively uniform microstructure and optimal corrosion resistance. Therefore, 70 °C is the optimal phosphating temperature to prepare PCC coatings on the sintered Nd-Fe-B permanent magnets when the pH value of the conversion solution is 1.0.

2.6 Formation mechanism

Because the formation mechanism of chemical conversion coatings is complex, there is no unified theory. According to the mechanism of steel phosphating^[7,33,34], the formation process of PCC coatings on the surface of sintered Nd-Fe-B permanent magnets should be divided into four stages (Fig. 10): (1) matrix dissolution; (2) formation and crystallization of the amorphous phase; (3) grain growth and coating formation; (4) dissolution and recrystallization of the grains.

When the Nd-Fe-B permanent magnets are immersed in the phosphating solution, the electrochemical corrosion occurs due to different potentials of different phases. Nd^{2+} , Pr^{2+} , and Fe^{2+} are generated by the anodic reactions of neodymium, praseodymium, and iron, respectively. Besides, the H^+ in the

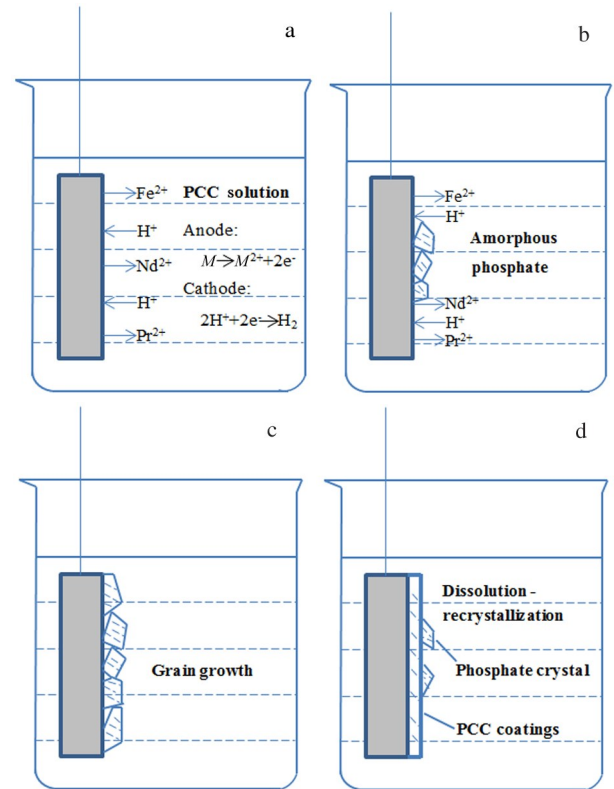


Fig.10 Schematic diagrams of coating formation on the surface of sintered Nd-Fe-B permanent magnets: (a) matrix dissolution; (b) formation and crystallization of the amorphous phase; (c) grain growth and coating formation; (d) dissolution and recrystallization of the grains

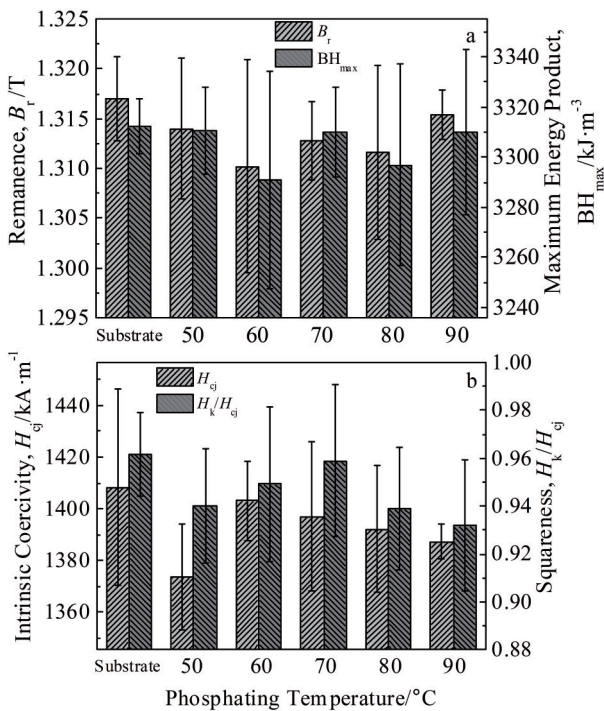


Fig.9 Magnetic properties of Nd-Fe-B substrate and Nb-Fe-B magnets with PCC coatings prepared at different phosphating temperatures: (a) remanence and maximum energy product; (b) intrinsic coercivity and squareness

solution is discharged at the cathode to generate hydrogen, which leads to the decrease in H^+ content. As the original dynamic balance is broken, a multistage hydrolysis of phosphate radical occurs to form PO_4^{3-} consequently. When the content of PO_4^{3-} , Nd^{2+} , Pr^{2+} , and other cations in the solution reaches the solubility product constant, the amorphous precipitate is formed and deposited on the surface of Nd-Fe-B substrate, as shown in Fig. 10a and 10b. Subsequently, PCC coating nucleates and crystallizes from the amorphous phase, which is the second stage of the coating forming process.

With the phosphating process further proceeding, the grains of PCC coatings continuously grow, and new crystal nuclei are generated continually due to the continuous hydrolysis of the phosphate radical in the conversion solution. Then PCC coating is formed by the close packing of a large number of grains, which is the stage of grain growth and coating formation.

The acidity of the phosphating solution changes during the continuous nucleation and growth of PCC coatings, resulting in the dissolution of the coating grains. Furthermore, the H^+ in the solution also corrodes the crystal faces with poor corrosion resistance, leading to the preferential growth of the PCC

coatings. Therefore, the new crystal nuclei are generated and crystallize at the grains in PCC coatings, which is the process of dissolution and recrystallization of the grains.

3 Conclusions

1) The coating mass is increased gradually with increasing the phosphating temperature, but the increment is not significant. All the phosphate chemical conversion (PCC) coatings prepared at different temperatures have a blocky structure, and the coatings prepared at 60 and 70 °C are relatively more uniform and denser. PCC coatings are mainly composed of neodymium phosphate hydrate, praseodymium phosphate hydrate, and a small amount of iron phosphate hydrate. The phosphating temperature has a little effect on the phase composition.

2) PCC coatings prepared at different temperatures can effectively improve the corrosion resistance of the sintered Nd-Fe-B permanent magnets, and the phosphating temperature has a little effect on the corrosion resistance. PCC coatings prepared at 70 °C exhibit the optimal corrosion resistance due to the relatively uniform and dense microstructure.

3) The magnetic properties of sintered Nd-Fe-B permanent magnets are decreased after phosphating treatment. The magnetic properties of the Nd-Fe-B magnets with PCC coatings prepared at 70 °C are relatively fine.

4) The optimal phosphating temperature for sintered Nd-Fe-B magnets to prepare PCC coatings is 70 °C when the pH value of the solution is 1.0.

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磷化温度对烧结Nd-Fe-B永磁合金表面磷酸盐化学转化膜 组织结构和耐蚀性能的影响

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摘 要: 研究了磷化温度对烧结钕铁硼永磁合金表面磷酸盐化学转化(PCC)膜的形成、微观结构和耐蚀性能以及对磁体磁性能的影响。结果表明,随着磷化温度的升高,转化膜膜重略有增加。扫描电镜观察结果表明,转化膜呈块状结构,晶粒尺寸为5~10 μm 。能量色散谱和傅里叶变换红外光谱测试表明,转化膜主要由钕磷酸盐水合物、镨磷酸盐水合物和少量铁磷酸盐水合物组成。转化膜中的氧、磷元素主要分布在晶粒表面,铁元素主要集中在晶界处,钕和镨的分布则比较均匀。电化学分析和静态全浸腐蚀试验表明,不同温度下制备的转化膜均可以提高烧结钕铁硼永磁合金的耐蚀性能。在70 $^{\circ}\text{C}$ 下制备的转化膜由于组织结构均匀致密,具有更好的耐蚀性能。磷化处理后,样品的磁性能均有所下降,但是70 $^{\circ}\text{C}$ 下处理的样品性能相对较好。因此,烧结Nd-Fe-B永磁合金的最佳磷化温度为70 $^{\circ}\text{C}$ 。

关键词: 钕铁硼永磁合金; 磷酸盐化学转化; 磷化温度; 组织结构; 耐蚀性能; 磁性能

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