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Fabrication and anti-corrosion performance of superhydrophobic silane film on sintered NdFeB

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Abstract: An eco-friendly superhydrophobic protective film (DTMS/TEOS silane film) was fabricated on sintered NdFeB substrate through the utilization of electrochemically assisted deposition technology. The structure, properties, and film-forming mechanism of dodecyltrime-thoxysilane (DTMS)/tetraethoxysilane (TEOS) silane films were comprehensively analyzed using Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS). Based on the test results, it can be determined that this film has a superhydrophobic property with a hydrophobicity angle of 152°. This special property can be attributed to the long alkyl chains in the DTMS molecule, the rough morphology, and the low surface energy of the DTMS/TEOS silane film. The surface of sintered NdFeB is coated with a layered three-dimensional network silane film that forms through the condensation of silanol substances. This film provides excellent corrosion resistance to the sintered NdFeB substrate, reducing its corrosion current density to 2.02×10^{-6} A/cm². Moreover, the impact of film on the magnetic characteristics of sintered NdFeB was assessed and found to be minimal.

Key words: NdFeB magnet; silane film; corrosion resistance; superhydrophobicity; electrochemically assisted deposition

1 Introduction

Sintered NdFeB magnets exhibit high saturation flux, coercivity, magnetic energy product $(BH)_{max}$, and favorable machining characteristics. They have been the focus of ongoing research and advancement in scientific investigation, manufacturing, and application [1–3]. However, practical exposure to corrosive environments renders the sintered NdFeB permanent magnet susceptible to deterioration. NI et al [4] have pinpointed the primary factors contributing to their poor corrosion resistance as their intrinsic

powder-sintered porous structure and multiphase composition, which significantly impedes and restricts their broader utility. Consequently, there is an imperative need to enhance the corrosion resistance of sintered NdFeB. At present, the surface protection methods employed in the sintered NdFeB manufacturing industry mainly include phosphating [5], electroplating [6]. electroless plating [7], organic coatings [8], and physical vapor deposition [9]. While phosphating treatment is commonly used as a surface pretreatment, it proves to be inadequate as a standalone method for safeguarding sintered NdFeB against corrosion. Electroplating offers cost-effective equipment and

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solutions; however, poor control often results in substantial environmental pollution. The electrophoretic coatings exhibit minimal environmental impact [10], while the organic coating is apt to abrasion in practical applications, prone to bubbling under hot and humid conditions, and unsuitable for the high temperature applications [11]. Consequently, there is an urgent need to develop a more environmentally friendly and efficient strategy to mitigate the corrosion of NdFeB magnets. In line with the growing emphasis on green environmental protection, silanization has garnered significant attention from researchers as a novel and eco-friendly technology for protecting metal materials [12,13].

Silane coupling agents, such as tetraethylorthosilicate (TEOS) [14], 1,2-bis-triethoxysilylethane (BTSE) [15], phenyltrimethoxysilane (PTMOS) [16], methyltrime-thoxy silane (MTMOS) [12] and dodecyl trimethoxy silane (DTMS) [17], are commonly employed for the creation of protective films featuring Si-O-metal bonds and Si-O-Si bonds. These bonds facilitate the formation of a uniform and dense three-dimensional (3D) network structure [14,15] on metal surfaces. The Si-Ometal covalent bonds enhance the adhesion of the silane film to the substrate. Due to the inherent instability of individual silane hydrolysis and the limitations associated with traditional dip coating methods [18], researchers [19,20] have turned to electrochemically assist deposition technology for the production of thick composite silanized films on various metal surfaces (e.g. steel [19], aluminum, and aluminum alloy [20]) as an anti-corrosion this electrochemically measure. In assisted deposition process, a negative potential is applied to generating abundant OH⁻ ions in the electrolyte near the metal surface, which promotes the sol-gel process and results in a more uniform, less flawed, and thicker silane film. This technique ensures the complete hydrolysis of silane monomers in an acidic solution and facilitates localized alkalization, providing sufficient impetus for silane film formation [14]. However, the application of electrochemically assisted deposition to create silane films for corrosion protection on sintered NdFeB has been relatively unexplored. Such film formation mechanisms and anti-corrosion performance also need further investigation.

TEOS is a widely employed silane monomer in the preparation of silane films on metal surfaces due to its rapid hydrolysis and uncomplicated molecular structure. Silane films prepared by TEOS exhibit the advantages of good chemical stability, high porosity, and certain roughness. However, the anti-corrosion property is weak [20]. Dodecyltrimethoxysilane (DTMS), featuring a long-chain alkyl group, can form hydrophobic silane films due to its low surface energy, but the chemical stability needs to be improved. For example, high temperature will result in the hydrophobicity degradation of the DTMS film [21]. To obtain a silane film with short hydrolysis time, robust chemical stability, and excellent hydrophobicity, we explored a combination of TEOS and DTMS to prepare a precursor solution. In this work, we prepared anti-corrosion DTMS/ TEOS silane films with superhydrophobicity on sintered NdFeB electrochemically substrate using assisted deposition technology, based on our previous We systematically work [22]. studied the morphology, structure and corrosion resistance of this film, and proposed a comprehensive mechanism for film formation and evaluated the magnetic properties of sintered NdFeB coated with the films to explore their application. Our goal was to develop an environmentally friendly silane film with exceptional corrosion resistance for sintered NdFeB.

2 Experimental

2.1 Pretreatment of sintered NdFeB substrate

The size of the prepared sintered NdFeB sample (obtained from China Hengdian Group Dongci Co., Ltd.) is $35.0 \text{ mm} \times 10.0 \text{ mm} \times 2.0 \text{ mm}$. Before the electrodeposition process, these samples underwent a series of surface treatments to eliminate any stains and oxide layer: (1) polish in sequence with abrasive papers of $500^{\#}$, $800^{\#}$, $1000^{\#}$ and $1200^{\#}$ to achieve a mirror-like finish; (2) thoroughly clean using ethanol, acetone, and deionized water; (3) dry using nitrogen and stored in glass drying dishes for subsequent utilization.

2.2 Fabrication of DTMS/TEOS silane films on sintered NdFeB substrate

The DTMS/TEOS silane films were prepared

via electrochemically assisted deposition using an Advanced Measurement PARSTAT2273 electrochemical workstation. This process was carried out in a three-electrode cell configuration, with the saturated calomel electrode (SCE) as the reference electrode, the pre-treated sintered NdFeB substrate as the working electrode, and a large platinum sheet as the counter electrode, respectively.

For the electrochemically assisted deposition, a precursor solution was prepared by combining 0.2 mol/L NaNO₃ and 5 mL of a TEOS+DTMS silane composite ($V_{\text{TEOS}}:V_{\text{DTMS}}=1:1$). This solution was agitated in a temperature-controlled water bath at 35 °C for 24 h. The pH of the silane precursor was adjusted to be 3.5 ± 0.1 . During the electrochemically assisted deposition, a deposition potential of -1.4 V was applied, with a deposition time of 300 s, and the deposition solution was maintained at a temperature of 35 °C. Finally, the obtained DTMS/TEOS silane films were rinsed with deionized water and then subjected to drying in an oven at 60 °C for 1.0 h.

2.3 Characterization

The surface micromorphology of the DTMS/TEOS silane film coated on the sintered NdFeB substrate was characterized using scanning electron microscopy (SEM, FEI Quanta 650) and a P-6 Step Profiler. The element distribution within the silane film was recorded via energy dispersive spectroscopy (EDS, Oxford EDS 7426). X-ray photoelectron spectroscopy (XPS, Thermo Scientific K-Alpha), Fourier transform infrared spectroscopy (FT-IR, Thermo Scientific Nicolet iS20), and nuclear magnetic resonance (NMR, Bruker AVANCE NEO 500) were performed to analyze the chemical composition, functional groups and chemical bonding of the prepared silane film. The water contact angle was measured on different areas of the deposited silane film using a contact angle meter (Shanghai Zhongchen Digital Technic Apparatus Co., Ltd., China). The binding force of the silane film was measured through a cross-hatch adhesion test, according to the ASTM D3359-09 standard. This involved cutting a $7.0 \text{ mm} \times 7.0 \text{ mm}$ area deeply into the substrate with a sharp blade, dividing it into 49 grids. Following this, the tape was applied and removed, and the degree of film detachment was evaluated, according to the standard scale ranging from 0B to

5B. Electrochemical tests were carried out to assess the anti-corrosion properties of the DTMS/TEOS silane films coated on sintered NdFeB substrates. The tests involved potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS). The experiments were carried out in a 3.5 wt.% NaCl solution with a pH of 6.5 at room temperature, using an electrochemical workstation (CHI660E) and a standard three-electrode cell configuration. The sintered NdFeB substrates with and without the DTMS/TEOS silane films were used as the working electrode, a large platinum plate was served as the counter electrode, and the SCE was employed as the reference electrode. Before data acquisition, the working electrodes were immersed in a 3.5 wt.% NaCl solution for 30.0 min to reach a stable condition. The exposed area of the working electrodes was standardized to be 1.0 cm². Potentiodynamic polarization curves were recorded within the potential range of φ_{ocp} (open circuit potential) ± 300 mV, with a scanning rate of 1.0 mV/s. EIS test was conducted over a frequency range from 100 kHz to 10 MHz with a 5 mV perturbation signal at the φ_{ocp} . The EIS data were analyzed using Z-View software.

The salt spray tests (SST) were performed to evaluate the anti-corrosion performance of the DTMS/TEOS silane films on sintered NdFeB substrate. During the experiments, 5.0 wt.% NaCl solution at a pH of 7.0 was atomized in a salt spray chamber (ERICHSEN606) at 35 °C. The test panels were positioned within the chamber at an angle of 45° and exposed to the salt fog for different testing time. Corrosion levels were assessed by observing changes in the surface morphology and color of the test samples. In addition, the impact of these silane films on the magnetic properties of sintered NdFeB substrates was examined using a magnetic material analyzer (PERM-REM C–300).

3 Results and discussion

3.1 Characteristics of DTMS/TEOS silane film

The surface micromorphology of the DTMS/ TEOS silane film on sintered NdFeB substrates is depicted in Fig. 1(a). The DTMS/TEOS silane film is uniformly distributed on sintered NdFeB substrates with no obvious cracks and defects. The hierarchical morphology with some discrete colloidal particles and micelles can be observed



Fig. 1 Surface characteristics of DTMS/TEOS silane film on sintered NdFeB substrate: (a) Micromorphology; (b) Water contact angle; (c) Film thickness; (d) 3D surface topography

under high magnifications. These constituents are interconnected, forming a three-dimensional networklike rough porous structure. The water droplets on the DTMS/TEOS silane film are shown in Fig. 1(b). The water contact angle was measured to be 152°, indicative of the superhydrophobic nature of the film. A superhydrophobic surface is defined as having a contact angle exceeding 150° and a sliding angle below 10° [23]. It is widely acknowledged that surface wettability is influenced by both chemical composition and microstructural geometry [24,25]. Based on this criterion, two primary approaches are employed to fabricate the superhydrophobic surfaces: one involves establishing the requisite roughness on the inherently hydrophobic materials, while the other entails modifying the rough microstructure with low surface energy compounds. Notably, the contact of metals with water can trigger corrosion reactions. Thus, reducing the contact area of water with metal surfaces is a well-established method for enhancing the corrosion resistance of metal structures [26]. In this study, we have successfully constructed a superhydrophobic silane film on sintered NdFeB substrates through an electrochemically assisted deposition method employing the DTMS/TEOS precursor solution. Figure 1(c) depicts the 2D cross-sectional image at the junction of the DTMS/TEOS silane film-coated and uncoated sintered NdFeB substrates. The thickness of the deposited silane film was measured to be approximately 11.1 µm, a significant increase compared to the dip-coating method (as reported in Ref. [18]). In Fig. 1(d), a 3D topographical image of the deposited silane film surface reveals its microscopically rough texture, with a roughness (R_a) of 0.897 µm. As explained in a previous study [27], this roughness, characterized by increased porosity and higher roughness, is attributed to the separation of the gelation process from the solvent evaporation during the electrochemically assisted deposition of the DTMS/TEOS silane films. The enhanced roughness of the DTMS/TEOS silane film plays a pivotal role in achieving superhydrophobic properties on the surface of the sintered NdFeB substrate.

Figure 2 shows the morphology and composition of the cross-section of the DTMS/TEOS silane film on sintered NdFeB substrates. A thin layer can be observed over the sintered NdFeB substrate with a thickness of 12.0 μ m (see Fig. 2(a)), which agrees well with the result of the P-6 step profiler (see Fig. 1(c)). The primary elements composing of the DTMS/TEOS silane film are silicon (Si), oxygen



Fig. 2 Cross-section characteristics of DTMS/TEOS silane film on sintered NdFeB substrate: (a) Micromorphology; (b–e) EDS maps of elements; (f) Corresponding contents of elements

(O), iron (Fe), and carbon(C). Si and O are the predominant elements within this silane film, while the presence of C can be attributed to the long-chain alkyl groups of DTMS molecules. The Si and C elements are distributed homogeneously throughout the entire cross-section of the silane film (see Figs. 2(c, d)). Notably, Si and C elements are more densely concentrated in the portion of the deposited silane film further from the sintered NdFeB substrate, indicating that the near-surface region of the silane film exhibits the highest densely connection. At the interface between the sintered NdFeB substrate and the deposited silane film, a minor amount of Fe element is detected within the silane film. This observation provides evidence of the diffusion of Fe element from the sintered

NdFeB substrate into the deposited silane film (see Figs. 2(e, f)).

The XPS spectra of the DTMS/TEOS silane films are presented in Fig. 3. The XPS analysis reveals the presence of C, Si and O elements on the surface of the sintered NdFeB substrate, which aligns well with previous EDS findings. In the high-resolution C 1s spectrum (Fig. 3(b)), two distinct peaks are observed at 284.5 and 285.2 eV, corresponding to C — C and C — Si bonds, respectively [28]. The O 1s spectrum (Fig. 3(c)) displays three discernible chemical states, which deconvoluted peaks at 530.2, 532.4 and 533.4 eV, and attributed to O—H, Si—O—Si and Si—O—Fe species, respectively [29,30]. The Si—O—Fe peak at 533.4 eV indicates a strong chemical bonding



Fig. 3 XPS spectra of DTMS/TEOS silane films on sintered NdFeB substrate: (a) Full spectral survey; (b) C 1s; (c) O 1s; (d) Si 2p

between the DTMS/TEOS silane films and the sintered NdFeB surface. Analyzing the Si 2p spectrum (Fig. 3(d)) reveals distinct peaks at 99.6, 101.4, 102.6 and 103.6 eV, representing Si-O-Fe, Si-C, Si-O-Si and SiO₂ species, respectively [31]. The above analysis confirms the successful covalent linkage between TEOS and DTMS, as well as between the DTMS/TEOS silane films and the sintered NdFeB substrate surface. The covalent bonds (Si - O - Si and Si - O - Fe)contribute to the formation of a densely connected DTMS/TEOS silane film. Theoretically, this film firmly attaches to the sintered NdFeB substrate surface and prevents the corrosive electrolyte from diffusion and penetration to the substrate surface, thereby efficiently safeguarding the sintered NdFeB substrate. However, the adhesive strength of the silane film, as determined by the cross-shade adhesion, is found to be 0B standard. Thus, further research is required to enhance the adhesion of functional silane films to the substrates [32].

Figure 4 illustrates the FT-IR spectra of

sintered NdFeB samples coated with the DTMS/ TEOS silane film with various hydrolysis durations. In the DTMS/TEOS silane film, five distinctive peaks at approximately 1467, 1382, 1065, 853 and 810 cm⁻¹ are identified, corresponding to the stretching vibration of $-[CH_2]_n - , -CH_3,$ Si - O - Si, Si - O - Fe, and Si - C bonds, respectively [15,33,34]. This observation aligns well with the earlier XPS analysis, providing the successful formation of the Si-O-Si network structure in this silane film, firmly attaching it to the surface of the NdFeB substrate via Si-O-Fe bonding. During hydrolysis time of 0 and 24 h, the characteristic peaks at 3450 and 940 cm⁻¹ signify the stretching vibrations of O-H and Si-OH, respectively [35,36]. Additionally, two peaks at 1085 and 1047 cm⁻¹ correspond to the vibrational mode of Si - O - C, indicating the incomplete hydrolytic reaction of the DTMS and TEOS molecules [37]. Extending the hydrolysis time to 48 h reveals the existence of the Si-O-Si bond at 1080 cm⁻¹, indicating that the condensation



Fig. 4 FT-IR spectra of DTMS/TEOS silane films and silane hydrolysates with different hydrolysis time

reaction of pre-hydrolyzed Si—OH group was dominant. Given that the electrochemically assisted deposition of silane films necessitates complete hydrolysis without the occurrence of the condensation reaction, 24 h was deemed the appropriate hydrolysis time in the silane film preparation process.

To further delve into the internal network structure of the DTMS/TEOS silane film, the specific chemical environment of silicon atoms was explored by nuclear magnetic resonance spectroscopy (NMR). Figure 5 presents the ²⁹Si-NMR spectrum of the DTMS/TEOS silane film. Notably, three distinct signal types emerge with the spectrum between -50×10^6 and -70×10^6 (corresponding to T₃-O₃SiR), between -80×10^6 and -100×10^6 (representing Q₃-O₃SiOH), and between -100×10^6 and -120×10^6 (indicative of Q₄-O₄Si) [38,39]. The presence of the T₃ structure signifies surface functionalization with hydrophobic



Fig. 5²⁹Si-NMR spectra of DTMS/TEOS silane film

dodecyl. Furthermore, the heightened intensity of peaks associated with the Q_3 and Q_4 structures is indicative of the polymerization of silanol groups and the formation of a 3D siloxane network [40].

3.2 Mechanism of film preparation

Based on the above experimental results, we can infer the mechanism underlying electrochemically assisted deposition of the DTMS/TEOS silane film on the sintered NdFeB substrate [17]. As illustrated in Fig. 6, this process begins with the formation of active DTMS and TEOS silanol groups derived from silane monomers within the precursor solution. This transformation occurs through a hydrolysis reaction taking place in an acidic environment, as described by Eqs. (1) and (2). Upon applying a negative potential, OH⁻ ions are generated near the surface of the sintered NdFeB substrate. These ions originate from the electrolysis of water, O_2 reduction, and NO_3^- reduction. Subsequently, the electrochemically generated OH⁻ ions catalyze the condensation reactions of the reactive sol ingredient (silanol component) as well as the reactions between silanols and the hydroxylated metallic substrate on the sintered NdFeB surface, as Eqs. (3) and (4) [41]. The condensation reactions are finalized with the assistance of subsequent heat treatment, resulting in the formation of a stable DTMS/TEOS silane film on the sintered NdFeB substrate.

These condensation reactions among reactive silanols lead to the creation of a compact crosslinking structure (Si-O-Si) within the DTMS/ TEOS silane film. Furthermore, the participation of dodecyl groups from the DTMS molecules in the condensation reactions enhances the hydrophobic properties of the deposited silane film, as shown in Eq. (5). In addition, the condensation reactions between silanols and the hydroxylated metallic substrates (Eq. (4)) further enhanced the adhesion of the DTMS/TEOS silane film to the sintered NdFeB surface through covalent binding (Si-O-Fe) at the interface. This covalent binding plays an important role in improving the corrosion resistance of the DTMS/TEOS silane film-coated sintered NdFeB substrate when exposed to a NaCl solution, resulting in a strongly adhered and compact protective layer on the sintered NdFeB substrate.



Fig. 6 Schematic illustration of electrochemically catalyzed condensation reaction involving DTMS and TEOS on surface of sintered NdFeB substrate

 $Si - OC_2H_5 + H_2O \rightarrow Si - OH + C_2H_5OH$ (1)

$$H_3C(H_2C)_{10}H_2C \longrightarrow Si(OCH_3)_3 + 3H_2O \longrightarrow$$

$$H_{3}C(H_{2}C)_{10}H_{2}C - Si - OH + 3CH_{3}OH$$
(2)

$$Si - OH + Si - OH \rightarrow Si - O - Si + H_2O$$
(3)

$$Si - OH + Fe - OH \rightarrow Si - O - Fe + H_2O$$
(4)

$$H_3C(H_2C)_{10}H_2C$$
—Si—OH+Si—OH→

$$H_3C(H_2C)_{10}H_2C - Si - O - Si + H_2O$$

$$(5)$$

3.3 Film performance

To investigate the corrosion protection properties of the DTMS/TEOS silane film on sintered NdFeB substrate, the corresponding electrochemical tests were carried out. Figure 7 illustrates the potentiodynamic polarization curves and EIS for both the DTMS/TEOS silane filmcoated and uncoated sintered NdFeB substrate in 3.5 wt.% NaCl solution. The electrochemical corrosion parameters are summarized in Tables 1 and 2. Comparing the coated and uncoated sintered NdFeB substrates, we observed a noticeable positive shift in the corrosion potential of the DTMS/TEOS silane film-coated NdFeB substrates, with φ_{corr} shifting from -0.779 to -0.682 V. This shift indicates that, from the thermodynamic perspective, the corrosion tendency is reduced in the presence of the DTMS/TEOS silane film [42]. Furthermore, the changes of the cathodic Tafel branch (β_c) and anodic Tafel branch (β_a) reveal that the DTMS/TEOS silane film can effectively inhabit both the anodic NdFeB dissolution reaction and the cathodic oxygen reduction processes. The corrosion current density (Jcorr) of the DTMS/TEOS silane film on NdFeB substrates was calculated to be 2.02×10^{-6} A/cm², which is nearly 28.5% that of the



Fig. 7 Potentiodynamic polarization curves (a) and EIS analysis, including Nyquist plot (b), Bode plot (c, d) for both uncoated and coated sintered NdFeB substrate immersed in 3.5 wt.% NaCl solution, corresponding equivalent electric circuits for fitting EIS data with subplots representing bare sintered NdFeB (e) and sintered NdFeB coated with DTMS/TEOS silane film (f) (The parameters in the circuits are R_s (electrolyte resistance), R_{ct} (charge transfer resistance), CPE_f (film capacity), R_f (film resistance), and CPE_{dl} (double layer capacity))

 Table 1 Electrochemical corrosion parameters associated with polarization curves of both uncoated and coated sintered

 NdFeB substrates in 3.5 wt.% NaCl solution

Sample	$arphi_{ m corr}$	(vs SCE)/V	J_{c}	$corr/(A \cdot cm^{-2})$	$-\beta_{\rm c}/({\rm m}$	V·dec ⁻	¹) $\beta_{a}/(m$	$V \cdot dec^{-1}$		
Bare sintered NdFeB		-0.779		7.09×10 ⁻⁶	2	.96		51.2		
Sintered NdFeB coated wi DTMS/TEOS silane film	th 1	-0.682		2.02×10 ⁻⁶		311		57.8		
Table 2 Fitting results of EIS for both uncoated and coated sintered NdFeB substrates in 3.5 wt.% NaCl solution										
Sample	$R_{\rm s}/(\Omega \cdot {\rm cm}^2)$	$CPE_{dl}\!/\mu F$	n_1	$R_{\rm ct}/(\Omega \cdot {\rm cm}^2)$	$CPE_{\rm f}\!/\mu F$	n_2	$R_{\rm f}/(\Omega \cdot {\rm cm}^2)$	$R_n/(\Omega \cdot \mathrm{cm}^2)$		
Bare sintered NdFeB	5.12	50.72	0.70	1867	-	-	-	1867		
Sintered NdFeB coated with	4.35	12.67	0.71	201.5	110.2	0.56	10891	11092.5		

 $R_n = R_t + R_{ct}$; R_1 is the value for DTMS/TEOS silane film-coated sintered NdFeB; R_2 is the value for bare sintered NdFeB

DTMS/TEOS silane film

bare sintered NdFeB substrate and significantly lower than that reported for DTMS film (J_{corr} = 1.299×10⁻⁴ A/cm²) and micro-arc oxidation (MAO) coating (J_{corr} =3.763×10⁻⁶ A/cm²) in the previous reports [11,17].

The EIS plots and corresponding analysis results, depicted in Figs. 7(b-d) and Table 2, demonstrated the profound impact of the DTMS/TEOS silane film on the corrosion behavior of sintered NdFeB substrate in 3.5 wt.% NaCl solution. Notably, the impedance modulus for DTMS/TEOS silane film-coated sintered NdFeB substrate far exceeded that of the bare counterpart. Specifically, the value of R_1 for the DTMS/TEOS silane film-coated sintered NdFeB substrates was five times larger than R_2 for the bare sintered NdFeB, as outlined in Table 2. This disparity strongly implies a remarkable reduction in the electrochemical corrosion rate of sintered NdFeB in 3.5 wt.% NaCl solution due to the presence of the DTMS/TEOS silane film. In the case of the bare sintered NdFeB substrate, a single capacitive loop was observed, whereas the DTMS/TEOS silane film-coated sintered NdFeB substrate exhibited two distinct semicircles. These two capacitive loops, appearing at high and middle frequencies, were attributed to the charge transfer process and the capacitance of the deposited silane film, respectively [43]. The impedance modulus for the DTMS/TEOS silane film at 0.01 Hz significantly surpassed that of the bare sintered NdFeB substrate, as illustrated in Fig. 7(c). This disparity indicates that the DTMS/TEOS silane film possesses good corrosion resistance. Additionally, the CPE_{dl} value of the sintered NdFeB substrate decreased upon coating with the DTMS/TEOS silane film. This reduction indicates a minimized contact area between the corrosive media and the sintered NdFeB substrate in the presence of the DTMS/TEOS silane film. In light of these electrochemical measurements, it can be deduced that the DTMS/TEOS silane film on sintered NdFeB represents an efficient and effective strategy for safeguarding the substrate against corrosion in NaCl solution.

The conventional salt spray test (SST) was also conducted to evaluate the corrosion protection effectiveness of the DTMS/TEOS silane film on the sintered NdFeB substrate against salt ions. The surface morphology and impedance spectra of NdFeB substrates covered with DTMS/TEOS silane films were examined at different time intervals during the SST, as illustrated in Figs. 8 and 9. For the sintered NdFeB coated with the DTMS/TEOS silane film, the surface remained intact and no obvious corrosion products even after 15 d of SST treatment. Furthermore, the corrosion resistance $R_{\rm f}$ (see Table 3) only decreased by 3.27%. This outcome provides additional evidence that the DTMS/



Fig. 8 Surface morphology of NdFeB substrates coated with DTMS/TEOS silane films in SST at 0 d (a) and 15 d (b)



Fig. 9 EIS analysis results of sintered NdFeB substrate coated with DTMS/TEOS silane film in SST at 0 d and 15 d (The corresponding equivalent electric circuit used for fitting EIS data is illustrated in Fig. 7(f))

Table 3	EIS	fitting	results	for	sintered	NdFeB	coated
with DTI	MS/T	'EOS si	lane fili	n du	ring SST	,	

Time/ d	$R_{\rm s}/$ ($\Omega \cdot {\rm cm}^2$)	CPE _{dl} / µF	n_1	$R_{ m ct}/$ ($\Omega \cdot m cm^2$)	CPE _f / µF	<i>n</i> ₂	$R_{\rm f}/$ ($\Omega \cdot { m cm}^2$)
0	7.84	6.37	0.43	275.41	16.02	0.55	15513
15	4.26	6.16	0.47	188.34	9.16	0.81	14782

TEOS silane film offers outstanding corrosion protection properties for sintered NdFeB substrates exposed to a NaCl solution, consistent with the results obtained from electrochemical tests.

The outstanding anti-corrosion performance of the DTMS/TEOS silane film on sintered NdFeB substrate in NaCl solution can be attributed to the following aspects. Firstly, the DTMS/TEOS silane film forms a robust bond with the sintered NdFeB substrate, as evidenced by cross-sectional SEM images. Additionally, analysis using XPS, FT-IR, and NMR techniques indicates the presence of a covalent bond (Si-O-Fe) at the interface of the deposited silane film and the sintered NdFeB substrate. This chemical bonding enhances the adhesion of the DTMS/TEOS silane film to the NdFeB substrate surface, enabling effective corrosion protection of the sintered NdFeB substrate in NaCl solution and ensuring long-term anti-corrosion performance. Secondly, the superhydrophobic nature of the DTMS/TEOS silane film on the sintered NdFeB substrate serves as a barrier, preventing water molecules and other corrosive species from direct contact with the substrate surface. The compact 3D network structure of the DTMS/TEOS silane film, formed by Si-O-Si siloxane bridges, acts as an excellent physical barrier, hindering the diffusion of corrosive media to reach the substrate surface.

As a practical permanent magnet material, it is essential to consider the impact of the DTMS/TEOS silane film on sintered NdFeB substrates for their magnetic properties. In Fig. 10, the hysteresis loops of both DTMS/TEOS silane film-coated and uncoated sintered NdFeB samples are presented,



Fig. 10 Magnetic results for uncoated and coated sintered NdFeB substrates

and the corresponding magnetic parameters extracted from Fig. 10 are listed in Table 4. Notably, the hysteresis loops of the DTMS/TEOS silane film-coated and uncoated NdFeB samples exhibit substantial overlap, indicating that the DTMS/ TEOS silane film minimally influences the inherent magnetic characteristics of the sintered NdFeB magnet. In comparison to the bare sintered NdFeB sample, the remanence (B_r), maximum energy product ((BH)_{max}), and the coercivity (H_{cb}) of the sintered NdFeB coated with the DTMS/TEOS silane film exhibit only marginal decrease of 1.17%, 1.52%, and 1.57%, respectively.

It is worth noting that other protective coatings that are applied to the NdFeB magnets, such as magnetron sputtering Al film [44], electroplated Al–Mn coating [45], and electroless plated Ni–P coating [46], have also demonstrated a reduction in magnetic properties (H_{cb} , B_r or (BH)_{max}) in previous reports (see Table 5). Therefore, the DTMS/TEOS silane film proves to be advantageous for the industrial utilization of sintered NdFeB magnets in real-world environments, as it exerts no significant adverse effects on the magnetic properties of NdFeB magnets.

Tabl	e 4	M	lagnetic	e properti	es of	uncoated	and	coated	l sintered	N	dl	FeB	3 substrates	5
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Sample	$B_{\rm r}/{\rm T}$	$(BH)_{\rm max}/({\rm kJ}\cdot{\rm m}^{-3})$	$H_{\rm cb}/({\rm kA}{\cdot}{ m m}^{-1})$
Bare sintered NdFeB	1.282	329	1016
Sintered NdFeB with DTMS/TEOS silane film	1.267	324	1000
Loss ratio/%	1.17	1.52	1.57

Plating material	Preparation method	Corrosion current density/(A·cm ⁻²)	Hydrophobicity	$H_{\rm cb}$ loss/% Source	
Ni coating	Electroplating	1.86×10^{-5}	_	_	[1]
Alumina ceramic coatings	Micro-arc oxidation (MAO)	2.17×10^{-6}	_	_	[47]
Alumina sol (AS) film	Electro-deposition and hydrothermal treatment	1.10×10^{-6}	_	-1.76	[7]
Al coating	Magnetron sputtering	1.90×10^{-5}	_	_	[9]
Ti-Al coating	Magnetron sputtering	7.90×10^{-8}	_	_	[9]
T-ZnOw/PDMS	Spray coating	2.80×10^{-6}	Superhydrophobic	-2.10	[11]
Al-Mn coating	Electroplating	1.57×10^{-8}	_	-3.20	[45]
Ni-P coating	Electroless plating	_	_	-24.00	[46]
KH-550 Silane film	Dip-coating	3.51×10 ⁻⁶	_	-0.20	[48]
DTMS/TEOS silane film	Electrochemically assisted deposition	2.02×10 ⁻⁶	Superhydrophobic	-1.57	This work

Table 5 Fabrication of anti-corrosion films on NdFeB magnets and associated properties

4 Conclusions

(1) The DTMS/TEOS silane film was successfully fabricated on the surface of the sintered NdFeB substrate using electrochemically assisted deposition technology, imparting superhydrophobicity and superior anti-corrosion properties.

(2) The film formation mechanism was proposed. Within the electrochemically assisted deposition process, local alkalization leads to the condensation of silanol groups from DTMS and TEOS onto the surface of the sintered NdFeB substrate. Hydrolyzed silane molecules form strong bonds through a siloxane bridge structure, creating a complex layered 3D network. This deposited silane film is anchored to the sintered NdFeB substrate via a chemical Si—O—Fe bond, enhancing the film's adhesion.

(3) According to the magnetic analysis result, it can be inferred that the presence of a DTMS/TEOS silane film has a minimal impact on the magnetic properties of sintered NdFeB. This merit underscores the significant potential of exploring composite silane film fabrication for practical applications in the transportation and storage of NdFeB magnets.

CRediT authorship contribution statement

Wen-tao JU: Validation, Formal analysis,

Investigation, Visualization, Writing – Original draft; Li JIANG: Conceptualization, Methodology, Writing – Review & editing, Resources, Supervision, Project administration, Funding acquisition; Yan-xia LIANG: Formal analysis, Investigation, Writing – Original draft; Shu-ting XU: Validation, Investigation; Ke WANG: Data curation; Yu-meng YANG: Funding acquisition; Ben-feng ZHU: Data curation, Formal analysis; Guo-ying WEI: Funding acquisition; Zhao ZHANG: Conceptualization, Methodology, Resources.

Data availability

Data will be made available on request.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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烧结钕铁硼表面超疏水硅烷膜的制备及其耐蚀性能

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摘 要:采用电化学辅助沉积技术在烧结钕铁硼表面制备一种新型环保的超疏水耐蚀膜(DTMS/TEOS 硅烷 膜)。通过红外光谱(FT-IR)、扫描电子显微镜(SEM)、X 射线光电子能谱(XPS)、动电位极化曲线和电化学阻抗 谱(EIS)等技术,研究十二烷基三甲氧基硅烷(DTMS)/四乙氧基硅烷(TEOS)硅烷膜的结构、性能和成膜机理。结果 表明,DTMS 分子中长烷基链、DTMS/TEOS 硅烷膜表面的粗糙形貌以及低表面能导致该膜具有超疏水性,疏水 角为 152°;烧结钕铁硼表面硅醇物质缩合形成的复杂层状三维网络硅烷膜具有较好的耐腐蚀性,可将烧结钕铁硼 基底的腐蚀电流密度降低至 2.02×10⁻⁶ A/cm²;此外,该膜对烧结钕铁硼的磁性能影响很小。 关键词:钕铁硼磁体;硅烷膜;耐蚀性;超疏水性;电化学辅助沉积

(Edited by Xiang-qun LI)