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# Multiple sheet layers super slippery surfaces based on AAO and its anticorrosion property

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| Complete List of Authors: | Song, Tingting; Harbin Engineering University, Key Laboratory of Superlight Material and Surface Technology, Ministry of Education<br>liu, qi; Harbin Engineering University,<br>Zhang, Meiling; Harbin Engineering University, Key Laboratory of Superlight Material and Surface Technology, Ministry of Education<br>Chen, Rongrong; Harbin Engineering University, Key Laboratory of Superlight Material and Surface Technology, Ministry of Education<br>Takahashi, Kazunobu; Harbin Engineering University, Institute of Advanced Marine Materials<br>jing, xiaoyan; Harbin Engineering University, Key Laboratory of Superlight Material and Surface Technology, Ministry of Education<br>Liu, Lianhe; Harbin Engineering University, Institute of Advanced Marine Materials<br>Wang, Jun; Harbin Engineering University, School of Material Science and Chemical Engineering |
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# Multiple sheet layers super slippery surfaces based on anodic aluminium oxide and its anticorrosion property

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Tingting Song,[a] Qi Liu,[a] Meiling Zhang,[a] Rongrong Chen,[b*] Kazunobu Takahashi,[b] Xiaoyao Jing,[a] Lianhe Liu,[b] Jun Wang,[ab*]

Inspired by the nature, there are many studies on surfaces, such as superhydrophobic surfaces, superolephobic surfaces and super slippery surfaces. In this paper, we grew compact $Co_3O_4$ layers on pure aluminium and anodic aluminium oxide (AAO), and then super slippery surfaces on two substrates were fabricated by immersing lubricating oil. In comparison with keeping super slippery time on the two $Co_3O_4$ super slippery surfaces and AAO super slippery surfaces, porosity of substrates by growing pink $Co_3O_4$ through hydrothermal method is a key factor in keeping super slippery time. The morphologies and compositions of surface are characterized by Scanning electron microscope (SEM) and X-ray photo electron spectroscopy. In addition, different pH liquids and anticorrosion property of multiple sheet layers super slippery surfaces was tested by water contact angles and Electrochemical Impedance Spectroscopy (EIS). Such high porosity and good anticorrosion property may be a good understanding for slippery liquid infused porous surfaces (SLIPS).

## Introduction

Surfaces inspired by natural plants and animals with special properties have been emerged for many years, such as superhydrophobic surfaces [1-2] and superolephobic surfaces [3]. Recently, slippery liquid infused porous surfaces (SLIPS) [4-6] have provided a new research orientation in bioinspired materials. This kind of material is prepared by infusing DuPont lubricating oil into functional substrates, which can repel various liquids such as organic liquids, crude oil and blood easily. Furthermore, these surfaces exhibit self-repairing [4], anti-ice and anti-frost [7] and antifouling [8] characteristics.

The original study about slippery liquid infused porous surfaces (SLIPS) is from Aizenberg team, its key of fabricating SLIPS is infusing fluorinated oil into porous structure. Many studies about super slippery surfaces have been reported on different substrates including glass, plastics or synthetic polymers [9-13]. SLIPS have emerged only recently; however, the above researches address some fundamental challenges regarding some respects of versatility and durability that superhydrophobic surfaces can not possess. Furthermore, there have been super slippery surfaces on the different types of substrates like the above mentioned, but there are a little super slippery reports about metal substrates. However, most of these reports were about improvements of properties [14-16]. The study about porosity of super slippery surfaces on metal substrates remains a challenge.

In this paper, multiple compact $Co_3O_4$ sheet layers super slippery surfaces on pure aluminium and anodic aluminium oxide substrates were prepared by hydrothermal method. In comparison with keeping super slippery time on the two super slippery surfaces, it demonstrated that porosity of substrates is a key factor in keeping super slippery time. In addition, different pH liquids and anticorrosion property of multiple sheet layers super slippery surfaces was tested by water contact angles and Electrochemical Impedance Spectroscopy (EIS). Such high porosity and good anticorrosion property may be a good understanding for slippery liquid infused porous surfaces (SLIPS).

## Experimental section

### Materials:

All reagents were of analytical grade and used as received without further purification. Aluminium sheets were purchased from Tianjin Guangfu Fine Chemical Research Institute, 0.2 mm thickness, they were used as the substrate (composition: 99.5wt% Al, 0.15wt% Si, 0.015wt% Cu, 0.015 wt% Fe, 0.005 wt% N), Perfluoropolyethers lubricating oil were purchased from America DuPont Krytox (PFPE), alcohol (Tianjin Fuyu Fine Chemical Co. Ltd), Co $(NO_3)_2 \cdot 6H_2O$ (Tianjin Zhiyuan Chemical Reagen Co. Ltd), $NH_3F$, $(NH_2)_2CO$ (Tianjin Zhiyuan Chemical Reagen Co. Ltd).

### Preparation of AAO (anodic aluminium oxide)

Pure aluminium plate (30*30*2mm) was ultrasonically cleaned in ethanol for 30 min and was then rinsed several times with distilled water. Second, 1mol/L NaOH solution was used for removing the oxide film of aluminium for 5min. After

[a.] Key Laboratory of Superlight Material and Surface Technology, Ministry of Education, Harbin Engineering University, 150001, PR Chin.
[b.] Institute of Advanced Marine Materials, Harbin Engineering University, 150001, PR China.
† E-mail: zhqw1888@sohu.com.
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washing with distilled water repeatedly and dried in the air, pure aluminium was electro-polished in the $HClO_4$:$CH_2CH_2OH$ (1:4 volume ratio) liquid obtaining bright surface. Next, two-step electrochemical anodizing process was introduced to prepare the ordered and sheet-layered aluminium oxide membrane in oxalic acid solution. The first step of electrochemical anodizing was performed in 0.3 M oxalic acid at voltage range of 40mV and temperature range of 5-10℃ for 2 h. The experimental conditions were the same as the first step, but the process was preceded for 4 h.

**Preparation of compact $Co_3O_4$ growth on AAO and pure aluminium**

0.3003g $(NH_2)_2CO$, 0.29103g $Co(NO_3)_2 \cdot 6H_2O$, 0.0741g $NH_3F$ was mixed in the 70ml distilled water after stirring for 30min at room temperature. The mixed solution and prepared AAO substrate were put into a 100mL Teflon-lined stainless-steel autoclave and reacted at 110 ℃ for 5h, and then cooled down to room temperature. Finally, the samples were rinsed several times with demonized water dried at 80 ℃ for 2h。The same process was performed on pure aluminium.

**Preparation of multiple sheet layer super slippery surfaces**

AAO and pure aluminium templates with compact $Co_3O_4$ were immersed in the PFPE lubricating oil for 1h obtaining multiple sheet layer super slippery surfaces, respectively.

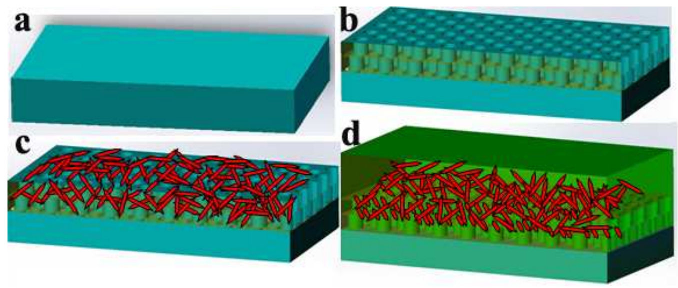
## Characterization

The surface morphologies and compositions are characterized by Scanning electron microscope (SEM) using a JEOL JSM-6480A microscope equipped with an energy-dispersive X-ray spectrum (EDS), X-ray photo electron spectroscopy (XPS, ESCALAB 250Xi, USA, Thermo) and X-ray photo electron spectroscopy, respectively. The phase composition and structure of $Co_3O_4$ growth on AAO and AAO are investigated by X-ray diffraction (XRD, Rigaku TTR-III, Cu Kα, λ = 0.15406 nm). Fourier Transform Infrared spectra were recorded as FT-IR Spectrum 100 Perkin-Elmer spectrophotometer. Static contact angles were measured on a FTA200 drop shape analysis system at room temperature. The corrosion properties of the samples were carried out by electrochemical workstation (IM6, German, Zahner) using 3.5 wt % aqueous solutions of NaCl at room temperature. A three-electrode system was used, in which the samples with an exposed area of 1 $cm^2$ acted as the working electrode, Ag/AgCl as reference electrode, and platinum as the counter electrode. The different pH liquids contact angles were measured at five points.

**Results and discussion**

**Morphology and mechanism of surfaces**

In this paper, we fabricated multiple compact $Co_3O_4$ sheet layers super slippery surfaces on pure aluminium and anodic aluminium oxide substrates were prepared by hydrothermal method. The mechanism of reaction process is shown in Sch.1. First of all, pure aluminium was prepared by electrochemical anodizing generating nano-micro sized porous anodic aluminium oxide (Sch.1a-b), micro-sized compact sheet-like $Co_3O_4$ were grown on the AAO (Sch. 1c), the super slippery surfaces was obtained after modifying perfluoropolyethers lubricating oil (Sch.1d). As shown in Fig.1a-b, the nano-micro sized sheet-layer pores are covered on pure aluminium by two-step anodic process, and sheet-like compact $Co_3O_4$ are grown on AAO through hydrothermal method (Fig.1c-d). $Co_3O_4$ are grown on pure aluminium directly via hydrothermal method in Fig.1 (2a-b).




Sch. 1. (a) pure aluminium substrate; (b) anodic aluminium oxide after two-step anodic process; (c) $Co_3O_4$ growth on AAO; (d) $Co_3O_4$ growth AAO super slippery surface

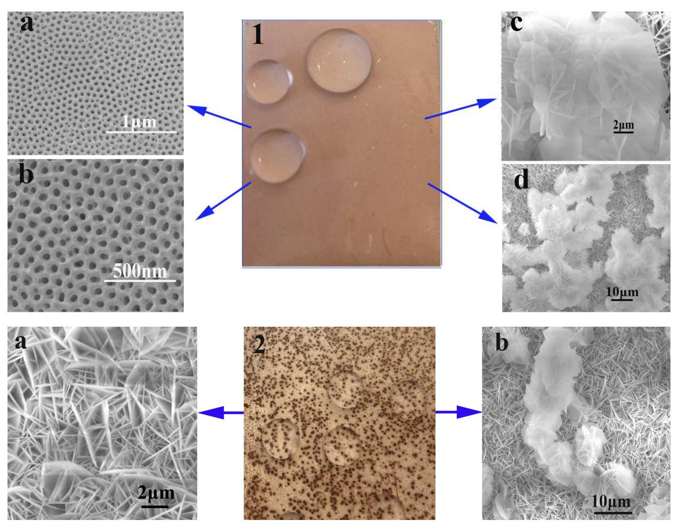



Fig. 1 SEM image of porous anodic aluminium oxide (1a-b); $Co_3O_4$ growth on AAO (1c-d) $Co_3O_4$ growth on pure aluminium (2a-b)

**Characterization of $Co_3O_4$ growth AAO super slippery surfaces**

The X-ray diffraction (XRD) of the sheet-layers-like $Co_3O_4$ growth AAO super slippery surfaces and AAO substrate is shown in Fig.2. The black line and red line refer to AAO substrate and sheet-layers-like $Co_3O_4$ growth on AAO, respectively. The three peaks of AAO substrate are corresponded to three peaks of sheet-layers-like $Co_3O_4$ growth on AAO. The other corresponding X-ray diffraction peaks are all indexed to spinel phase of $Co_3O_4$ (see standard card JCPDS-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431003). The results indicate that the $Co_3O_4$ grow on AAO substrate completely without any impurities.

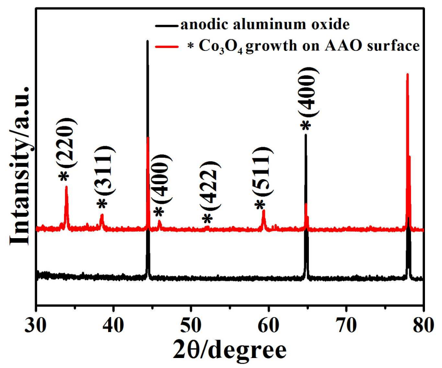


Fig. 2 X-ray diffraction of as-prepared AAO substrate and $Co_3O_4$ growth AAO super slippery surfaces

The chemical composition of $Co_3O_4$ growth AAO super slippery surfaces is characterized by X-ray photo electron spectroscopy and FT-IR. There are five obvious signals in the survey image: C1s, O1s, F1s, Al2p and Si2p (Fig.3a). It demonstrates that corresponding elements were existed on super slippery surfaces. The FT-IR data can further prove that super slippery surfaces are covered by compact $Co_3O_4$ and PFPE lubricating liquid. There are seven marked peaks that appear in the FT-IR spectrum of $Co_3O_4$ growth super slippery surface (Fig.3b). Two strong bands at 661 and 565 $cm^{-1}$ appeared in the low wavenumbers, the two peaks belong to the spinel structure of $Co_3O_4$ [17-21]. The 661 $cm^{-1}$ peak is attributed to the stretching vibration mode of Co-O in which Co is $Co^{2+}$ and is tetrahedrally coordinated. The other one at 565 $cm^{-1}$ can be assigned to Co-O of octahedrally coordinated $Co^{3+}$ [17-21]. The other five bands at 985, 1190, 1240, 1306 and 1884 $cm^{-1}$ appeared in the high wavenumbers, as shown in Fig.2b, the sharp peak exists at 985 $cm^{-1}$ , corresponding to the stretching vibration mode of C-O-C, the peaks at 1190, 1240, 1306 $cm^{-1}$ belong to the stretching vibration mode of –CF-, -$CF_2$-, -$CF_3$-, respectively. The peak at 1884 $cm^{-1}$ is assigned to the stretching vibration mode of –COF. In the previous reports about PFPE, the absorbent frequency of –CF-, C-O-C and –COF is range from 1400-1000 $cm^{-1}$, 900-1000 $cm^{-1}$ and 1884 $cm^{-1}$[22]. The above analyses completely prove that the super slippery surfaces possess $Co_3O_4$ and PFPE lubricating liquid existed.

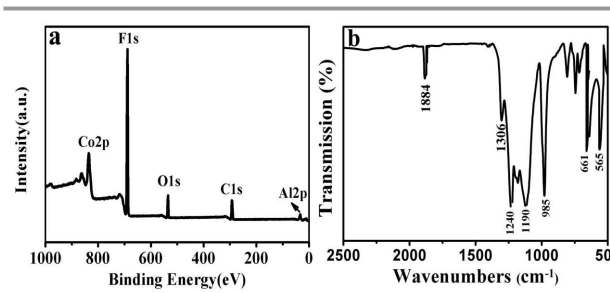


Fig. 3 XPS survey (a) and FT-IR (b) of $Co_3O_4$ AAO growth super slippery surfaces

## Super slippery Property of $Co_3O_4$ growth AAO super slippery surfaces

Schematic diagram porosity changes of $Co_3O_4$ growth on AAO and pure super slippery surfaces are shown in Fig.4. There are a lot of pores on AAO which can store lubricating oil (Fig.4a), even though this sample is placed for hours (Fig.4c), and water contact angles is still 115°(insets of Fig.4a,c). However, $Co_3O_4$ growth on pure aluminium super slippery surface keep few lubricating oil after placing several hours, the water contact angles become 145°(Fig.4b,d). The super slippery properties of $Co_3O_4$ growth AAO super slippery surfaces were also tested by macroscopic observation such as videos or pictures and contact angles. Fig.5 shows the contact angles of water and pH=2-12. From Fig.5a-h, there are no apparent difference between water and different pH liquids. The contact angles are range from 110° to 119°. Video S1-3 in the supporting information exhibit the movement of water, coffee and ink drops on $Co_3O_4$ growth AAO super slippery surfaces and $Co_3O_4$ growth pure aluminium super slippery surfaces after placing two months. It can be seen that that three drops adhere to $Co_3O_4$ growth AAO super slippery surfaces scarcely. In conclusion, the above mention demonstrate that acid and alkaline liquids have no effect on $Co_3O_4$ growth AAO super slippery surfaces and $Co_3O_4$ growth pure aluminium super slippery surfaces cannot keep super slippery properties due to low porosity.

The two super slippery surfaces are investigated after 24h of placing. The contact angles of $Co_3O_4$ growth super slippery surfaces varying with different pH and the existence forms of water on two super slippery surfaces are shown in the Fig.6. In the left of Fig.6a, the drops move by themselves on the $Co_3O_4$ growth AAO super slippery surfaces without sloping, two drops converged one big drop due to super slippery properties of $Co_3O_4$ growth AAO super slippery surfaces. However, the drops did not move and adhered to the $Co_3O_4$ growth pure aluminium super slippery surfaces in the right of Fig.6a. Furthermore, contact angles of two $Co_3O_4$ growth super slippery surfaces with different pH are exhibited in Fig.6b. Obviously, the contact angles and slide angles on $Co_3O_4$ growth pure aluminium super slippery surfaces are bigger than $Co_3O_4$ growth AAO super slippery surfaces. It demonstrated that the $Co_3O_4$ growth AAO super slippery surfaces can still keep super slippery property because porosity of $Co_3O_4$ growth AAO is higher than $Co_3O_4$ growth pure aluminium.

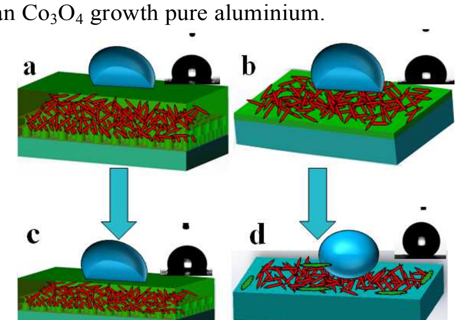


Fig. 4 Schematic showing porosity changes of $Co_3O_4$ growth AAO super slippery surfaces (a), $Co_3O_4$ growth pure aluminium super slippery surfaces (b)

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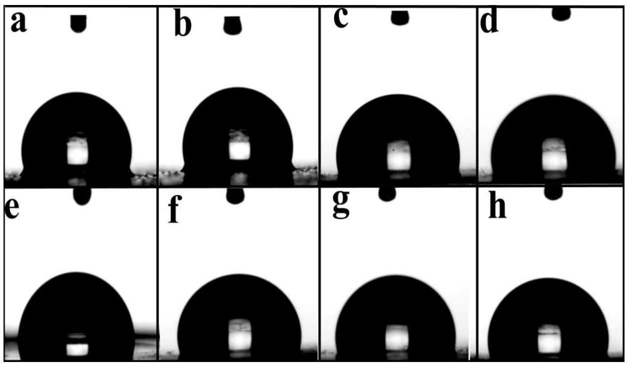


Fig. 5 The contact angles of water (a-b), pH=2 (c), pH=4 (d), pH=6 (e), pH=8 (f), pH=10 (g), pH=12 (h) liquid on $Co_3O_4$ growth AAO super slippery surfaces

**Anticorrosion Properties of $Co_3O_4$ growth AAO super slippery surfaces**

Corrosion resistance of two $Co_3O_4$ growth super slippery surfaces was investigated by electrochemical impedance spectroscopy (EIS).The measurement was performed on the standard three-electrode system with the 10mV disturbing value. The Nyquist and Bode plots of $Co_3O_4$ growth AAO super slippery surfaces are shown in the Fig.7. Fig.7a-b exhibits that $Co_3O_4$ growth AAO super slippery surfaces are immersing in 3.5% NaCl solution for 6h, 16h, 24h, 48h, 300h, 19d, 33d and 76d, respectively. From Fig.6a, the |Z| values of $Co_3O_4$ growth AAO super slippery surfaces are about 2.0E+005, 3.5E+005,4.0E+005 and 1.5E+005 after immersing in 3.5% NaCl solution for 6 h, 16h, 24h and 48 h, respectively. The above mentioned |Z| values have increased two orders of magnitude in comparison to those after immersing in 3.5% NaCl solution for 300h, 19d, 33d and 76d (Fig.7b). The sample is not corrosive after immersion of 76d, and the |Z| value of 76d is bigger than the |Z| values of 300h, 19d and 33d, the result is caused by the diffusion of film.

In addition, the above results demonstrate that super slippery surfaces have excellent anti-corrosion properties. It is related to the structure of surface, AAO with compact $Co_3O_4$ can store lubricating oil resulting in protecting this surface from eroding. Fig.8 shows Nyquist plots of $Co_3O_4$ growth AAO and pure aluminium super slippery surfaces after immersion of 16h in 3.5% NaCl solution, the |Z| values of $Co_3O_4$ growth AAO super slippery surfaces are about bigger a lot than $Co_3O_4$ growth AAO super slippery surfaces. So it proves that the anticorrosion property of $Co_3O_4$ growth AAO super slippery surfaces is better than $Co_3O_4$ growth pure aluminium super slippery surfaces. The electrochemical properties of $Co_3O_4$ growth AAO super slippery surfaces can also obtained from Bode plots (Fig.7$a_1$-7$a_2$, 7$b_1$-7$b_2$). From Fig.7a-7b, there is one loop at 6h, 16h, 24h, 48h, 300h, 19d, 33d and 76d, respectively. This result is in agreement with Bode plots (Fig.7$a_1$, 7$a_2$, 7$b_1$, 7$b_2$), which appear two corresponding peaks at 6h, 16h, 24h, 48h, 300h, 19d, 33d and 76d, respectively, each peak represents a time constant. The film of $Co_3O_4$ growth AAO super slippery surfaces is not corrosive without existing platform. According to the above analysis, $Co_3O_4$ growth pure aluminium super slippery surfaces possessed low anticorrosion property due to lower porosity.

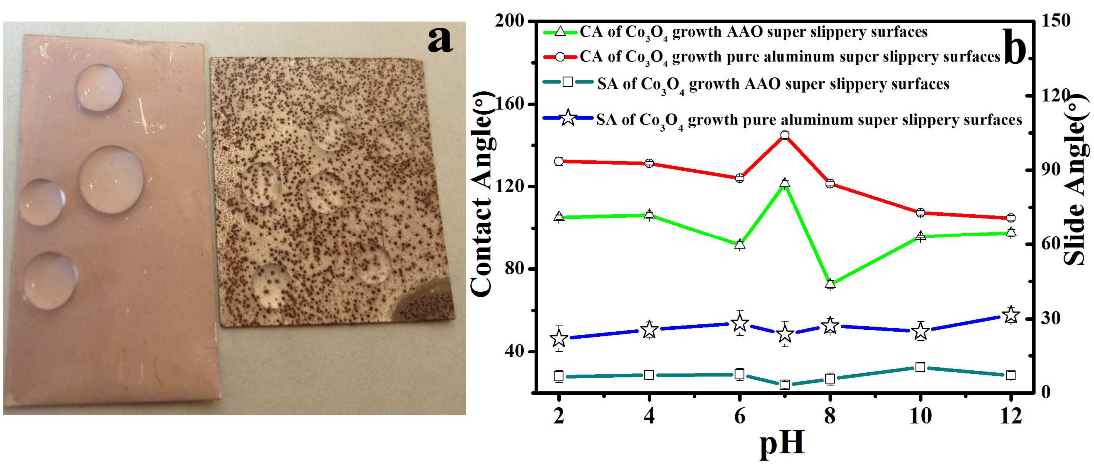


Fig. 6 (a) Water on $Co_3O_4$ growth AAO super slippery surfaces (left) and $Co_3O_4$ growth pure aluminium super slippery surfaces (right); (b) Variation of contact angles and slide angles of different pH

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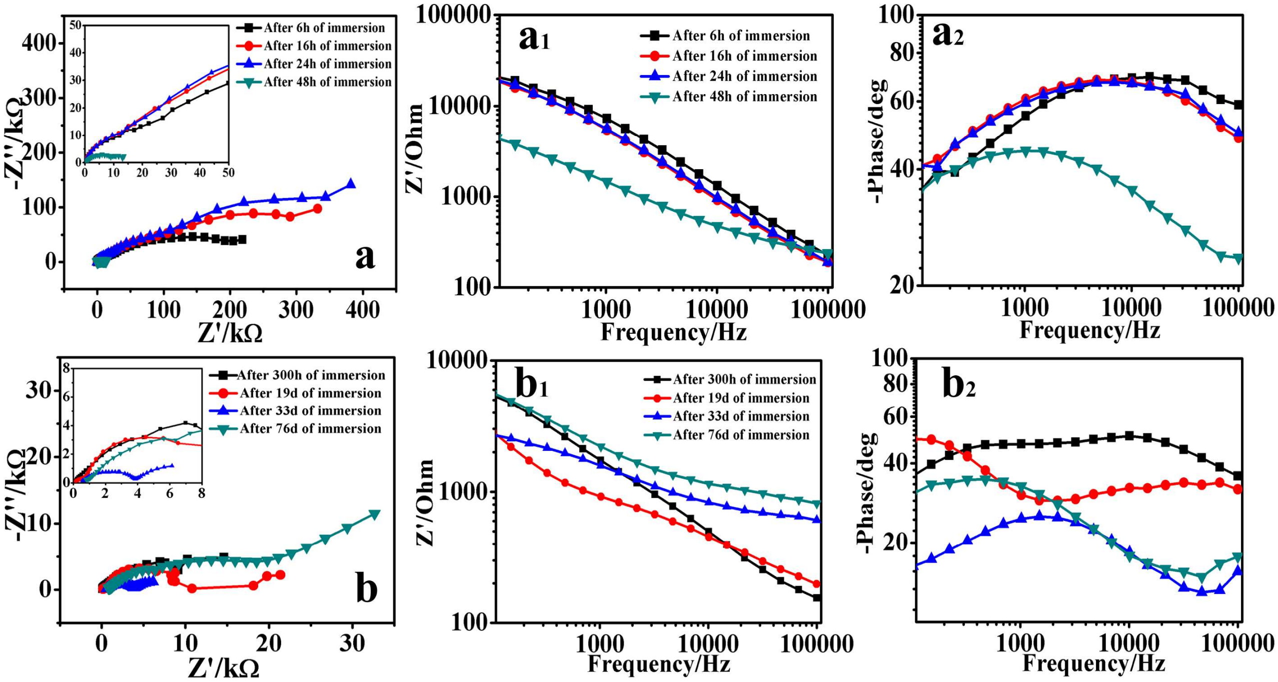


Fig. 7 (a)EIS results of $Co_3O_4$ growth AAO super slippery surfaces after immersing in 3.5% NaCl solution for 6 h, 16 h, 24 h, 48 h, respectively; (a1) Corresponding Bode plots of |Z| vs. frequency; (a2) Corresponding Bode plots of phase angle vs. frequency, (b) EIS results of $Co_3O_4$ growth AAO super slippery surfaces after immersing in 3.5% NaCl solution for 300h, 19d, 33d, 76d, respectively; (b1) Corresponding Bode plots of |Z| vs. frequency (b2) corresponding Bode plots of phase angle vs. frequency, respectively.

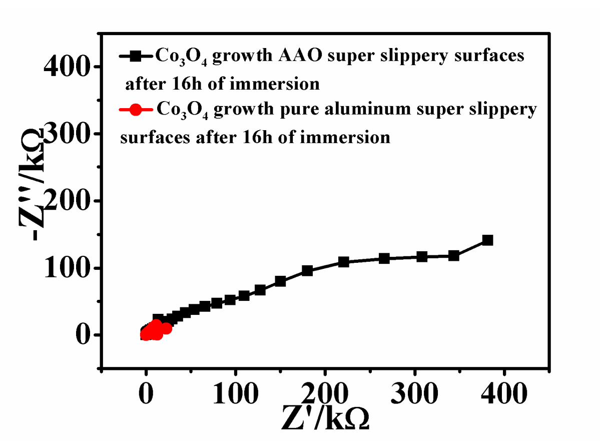


Fig.8 Nyquist plots of $Co_3O_4$ growth AAO and pure aluminium super slippery surfaces after immersion of 16h in 3.5% NaCl solution

In addition, the above results demonstrate that super slippery surfaces have excellent anti-corrosion properties. It is related to the structure of surface, AAO with compact $Co_3O_4$ can store lubricating oil resulting in protecting this surface from eroding. Fig.8 shows Nyquist plots of $Co_3O_4$ growth AAO and pure aluminium super slippery surfaces after immersion of 16h in 3.5% NaCl solution, the |Z| values of $Co_3O_4$ growth AAO super slippery surfaces are about bigger a lot than $Co_3O_4$ growth

AAO super slippery surfaces. So it proves that the anticorrosion property of $Co_3O_4$ growth AAO super slippery surfaces is better than $Co_3O_4$ growth pure aluminium super slippery surfaces. The electrochemical properties of $Co_3O_4$ growth AAO super slippery surfaces can also obtained from Bode plots (Fig.7a1-7a2, 7b1-7b2). From Fig.7a-7b, there is one loop at 6h, 16h, 24h, 48h, 300h, 19d, 33d and 76d, respectively. This result is in agreement with Bode plots (Fig.7a1, 7a2, 7b1, 7b2), which appear two corresponding peaks at 6h, 16h, 24h, 48h, 300h, 19d, 33d and 76d, respectively, each peak represents a time constant. The film of $Co_3O_4$ growth AAO super slippery surfaces is not corrosive without existing platform.

According to the above analysis, $Co_3O_4$ growth pure aluminium super slippery surfaces possessed low anticorrosion property due to lower porosity.

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## Conclusion

In summary, $Co_3O_4$ growth AAO and pure aluminium super slippery surfaces are fabricated by electrochemical method, hydrothermal method and immersing method. A layer of compact $Co_3O_4$ growth on the AAO and pure aluminium successfully is tested by SEM; $Co_3O_4$ growth AAO surfaces modified by lubricating oil are characterized by XPS and FI-IR. $Co_3O_4$ growth AAO super slippery surface repelled different pH liquids, coffee and ink via high porosity structure storing lubricating oil. Comparing with contact angles, slide angles and good anticorrosion property of two super slippery surfaces after placing two months, $Co_3O_4$ growth pure aluminium super slippery surfaces cannot keep lubricating oil as a result of low porosity. Therefore, this present report will provide a good understanding for slippery liquid infused porous surfaces (SLIPS) about porosity.

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