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Surface decoration of anodic aluminium oxide in synthesis of Nafion®-115 nanowire arrays

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Abstract

An extrusion method, using anodic aluminium oxide (AAO) membranes as templates, has been developed to fabricate Nafion®-115 nanowire arrays. Surface decoration of the templates plays an important role in the synthesis of the Nafion®-115 nanowire arrays. By using sodium dodecyl sulfate (SDS) as a surfactant in the surface decoration, the filling rate of the Nafion®-115 nanowires in the corresponding template exceeds 95%, compared with the filling rate of 42% in an undecorated template, while on using cetyltrimethylammonium bromide (CTAB) as a surfactant, the filling rate is only about 20%. Systematical investigations show that the effect of surface decoration is induced by the combination of surface tension and electrostatic force (radial component), of which the radial component of the electrostatic force is more important. This effect is significant in organic nanowire fabrication; furthermore, the as-synthesized organic nanowires would have potential application such as in organic electronics, optoelectronic devices, and nanoscale proton exchange membrane fuel cells.

1. Introduction

Since the discovery of carbon nanotubes in 1991 [1], one-dimensional (1D) materials have been attracting much attention due to their unique structures, properties and applications. In recent years, organic nanowires have been a subject of intensive research due to their peculiar properties such as electronic transport [2–4], optical properties [5–7], and excellent processing performance [8–10]. Some fabrication methods have also been developed for organic nanomaterials, such as electrospinning [11–13], evaporation [14], microemulsion [15], self-organization [16, 17], extrusion polymerization [18] and template synthesis [19].

Nafion® is one of the most common commercial organic materials, and is a registered trade mark by DuPont de Nemours (USA) for poly (perfluorosulfonicacid) membranes [20]. The greatest interest in Nafion® in recent years derives from its use as a proton conducting membrane in proton exchange membrane fuel cells (PEMFC), since hydrogen energy is considered as one of the most important new energy resources in the future. Thus, Nafion® nanowire arrays have potential application for nanoscale proton exchange membrane fuel cells which may be used as the power source of nanodevices such as nano-electromechanical systems (NEMS). But Nafion® nanowires had not been researched until we presented a method to synthesize Nafion®-115 nanowire arrays [21]. In this previous work [21], it was shown that, with surface decorated anodic aluminium oxide (AAO) as templates, Nafion®-115 nanowire arrays can

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be successfully fabricated with tunable length and diameter. However, the mechanism of surface decoration was not shown in the previous report [21]. Herein, the mechanism of surface decoration is investigated systematically. It is indicated that the effect of surface decoration is induced by the combination of surface tension and the radial component of electrostatic force, whose roles are discussed in detail in this paper. The results offer a wide base for the synthesis of organic nanowire arrays.

2. Experiment section

2.1. Materials

The Nafion®-115 membranes were purchased from DuPont Company (USA). The surfactants cetyltrimethylammonium bromide (CTAB) and sodium dodecyl sulfate (SDS) were purchased from Beijing Chemical Reagent Corporation, China, and the difunctional block copolymer surfactant P_{123} ($EO_{20}PO_{70}EO_{20}$) was purchased from BASF Company (Shanghai, China). Dimethyl sulfoxide (DMSO) reagent grade (AR), which was used as the solvent of Nafion®-115, was purchased from Beijing Chemical Reagent Corporation, China.

2.2. Sample preparation methods

2.2.1. The preparation of Nafion®-115 DMSO solution. In our experiment, the preparation of the Nafion®-115 DMSO precursor solution was as follows.

(a) A piece of Nafion®-115 membrane (1.10 g) was divided into several small pieces of about 2 mm^2.
(b) The small pieces of Nafion®-115 were immersed in 10% H_2O_2 at 303 K for 2 h in order to remove organic contamination. Then the sample surface was rinsed in distilled water several times quickly.
(c) The Nafion®-115 pieces were dipped into 10% sulfuric acid at 303 K for 24 h in order to remove metallic contamination. Subsequently, they were washed with distilled water.
(d) The small pieces of Nafion®-115 were dissolved in 100 ml dimethyl sulfoxide (DMSO) by refluxing at 450 K under N_2 for about 40 h to form a 1.0 wt% DMSO solution.

2.2.2. Surface decoration of anodic aluminium oxide. The AAO membrane was prepared by a two-step oxidation method [22, 23], with a diameter of about 85 nm, and a thickness of 30 μm. If the AAO template was undecorated, the filling rate of the Nafion®-115 nanowires in it was only about 42%. In order to improve the filling rate, surface decoration of AAO template was carried out in the study. First, a clean and dry AAO membrane with pore diameters about 85 nm was immersed in surfactant solution for 8 h after being pumped for 3 h. Subsequently, it was washed with distilled water quickly to remove the remnant surfactant solution on the AAO membrane surface, and then dried.

Three kinds of surfactant, cetyltrimethylammonium bromide (CTAB), sodium dodecyl sulfate (SDS) and poly (ethylene oxide)–poly (propylene oxide) (PEO–PPO) triblock surfactants P_{123}, were used. They also represent three different types of surfactant. CTAB is a sort of cationic surfactant [24], SDS is anionic surfactant [25] and P_{123} is a neutral surfactant [26].

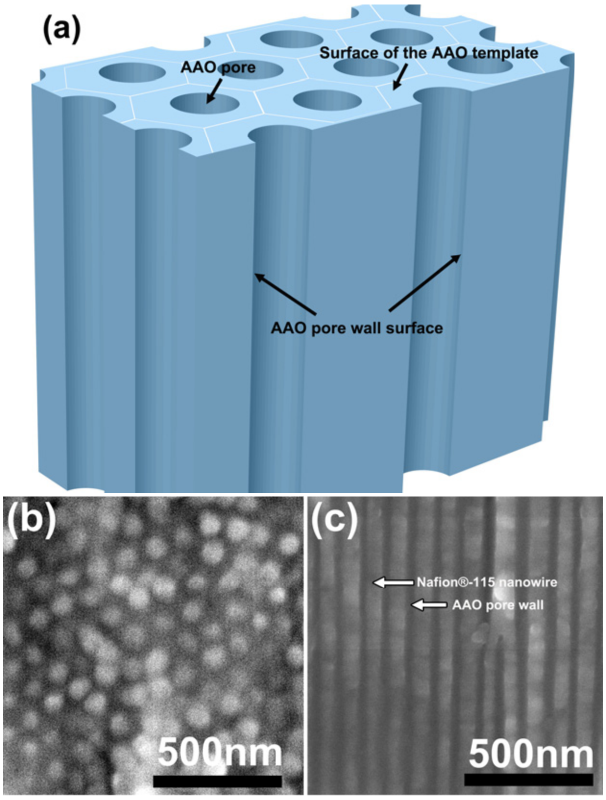

Figure 1. (a) A 3D model of an AAO template, and scanning electron microscope (SEM) images of: (b) the top view, (c) the cross-sectional view of an as-synthesized Nafion®-115 nanowire arrays in the AAO pores.

(This figure is in colour only in the electronic version)

2.2.3. The synthesis of Nafion®-115 nanowire arrays. The 0.1 wt% Nafion®-115 DMSO solution was extruded into the pores of the as-decorated AAO membrane under a mechanical pump pressure (0.1 MPa) in order to form the Nafion®-115 nanowire arrays. As its diameter and thickness can be tunable, nanowire arrays of different aspect ratio can be easily obtained. Details can be found in our previous report [21].

3. Results

For a clear distinction in the following text, two types of surface in an AAO template should be defined first. One is the surface of the AAO template and the other is the surface of the AAO pore wall (they will be called template surface and pore wall surface hereafter, respectively), which are shown in figure 1(a), a 3D model of an AAO template.

After an as-prepared Nafion®-115 dimethyl sulfoxide (DMSO) solution was extruded into the pores of an AAO template under a mechanical pump pressure (~0.1 MPa), nanowires were formed in the pores of the AAO template. A 5 M NaOH solution was used to etch the AAO template slightly at room temperature to reveal the top of the Nafion®-115 nanowire arrays, the morphology of which is shown in figure 1. It is shown in figure 1(b) that most of the AAO pores are filled with nanowires whose diameters are about 85 nm. Figure 1(c) shows that the nanowires (grey contrast) have fully

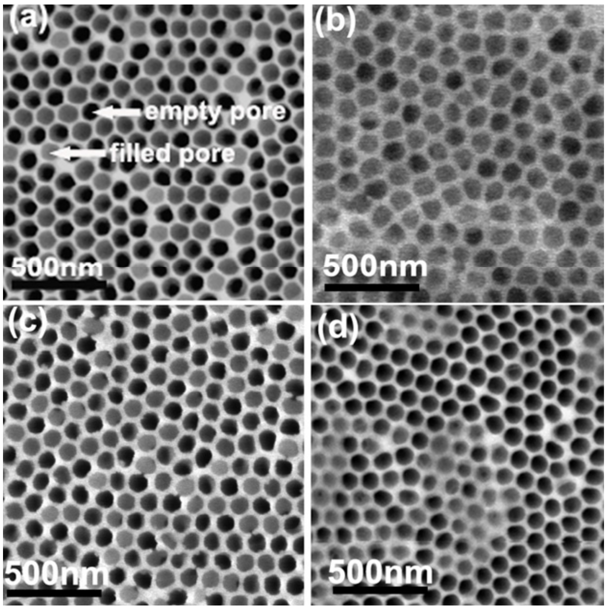

Figure 2. SEM images showing the filling rates of the Nafion®-115 nanowires in the AAO templates pretreated by using different surfactants: (a) no surfactants; (b) 1% SDS aqueous solution; (c) 1% P_{123} alcohol solution; (d) 1% CTAB aqueous solution.

Table 1. The filling rates of the Nafion®-115 nanowires in the AAO template, undecorated and decorated by different surfactants.

Case No.	Surfactant	Filling rate (%)
I	Undecorated	∼42
II	1% SDS aqueous solution	>95
III	1% P_{123} alcohol solution	∼40
IV	1% CTAB aqueous solution	∼20

filled the AAO pores (dark contrast) in the radial direction. Due to the confinement of the AAO template, the Nafion®-115 nanowires are well aligned. The diameter and the length of the Nafion®-115 nanowires can be controlled by changing the pore diameter and the thickness of the AAO templates. Near-IR Raman and IR spectrum studies show that the polymer nanowires are composed of pure Nafion®-115 only.

Figures 2(b)–(d) show the effects of surface decoration by the three different surfactants on the filling rate, comparing with the undecorated case (figure 2(a)). By measuring at least 20 areas, for the four cases which are assigned as case I, II, III, and IV respectively in table 1, the filling rates of the Nafion®-115 nanowires in the corresponding decorated template are shown in table 1.

It can be seen from figure 2 and table 1 that the surface decoration directly affects the filling rate. With different surface decorated templates, the filling rate of the Nafion®-115 nanowires varies from ∼20% to ∼95%. It is obvious that SDS is the best surfactant in our research. In this case (case II), over 95% of the AAO pores are filled with the Nafion®-115 nanowires. The filling rates of case I and case III are similar, while that of case IV is only about 20%. It is essential to investigate the mechanism of the surface decoration of the AAO templates in the synthesis of Nafion®-115 nanowires.

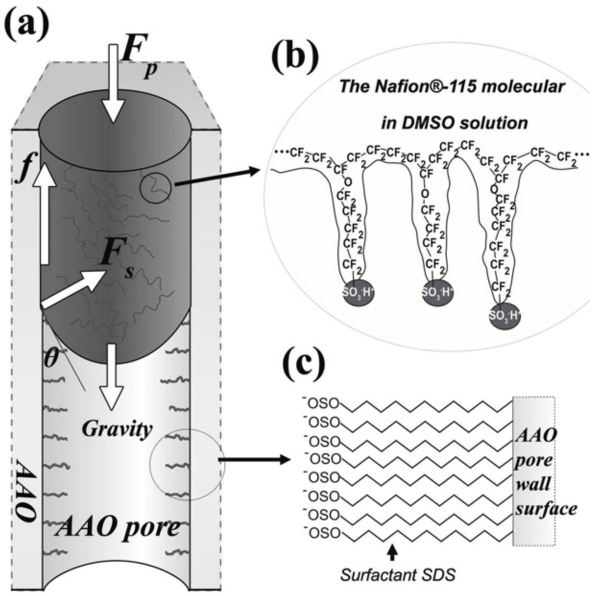

Figure 3. Schemes of: (a) the forces (except the electrostatic force) acting on the Nafion®-115 DMSO solution when it is forced through the AAO pores. (b) The Nafion®-115 molecule in the DMSO solution. (c) The AAO pore wall surface decorated by surfactants, where SDS is taken as an example. The electrostatic force is not shown here but in figure 4.

4. Discussion

To thoroughly understand the effect of surface decoration on the filling rate during the formation of the Nafion®-115 nanowires, theoretical considerations are necessary. In our previous report [21], transmission electron microscope (TEM) characterization of an isolated Nafion®-115 nanowire indicates that the Nafion®-115 nanowire has a rough surface, and we concluded that the Nafion®-115 nanowires were formed by an adsorption mechanism. That is to say, when a high pressure (usually is 0.1 MPa) was applied, the Nafion®-115 DMSO solution was forced through the AAO pores. During this process, the Nafion®-115 molecules were adsorbed onto the AAO pore wall, and then formed the Nafion®-115 nanowires by extruding several times. As a result, the smaller the velocity of Nafion® solution is, the more time would be needed for the Nafion® solution to go through the AAO pores, which creates more chance for the Nafion®-115 molecules to absorb onto the AAO pore walls, and to form the Nafion®-115 nanowires.

Based on the absorption mechanism in the synthesis of the Nafion®-115 nanowires, the forces acting on the Nafion®-115 molecules are considered. The Nafion®-115 DMSO solution in the AAO pores experiences several types of force: the surface tension F_s of the Nafion®-115 DMSO solution, the electrostatic force F_e (consisting of the radial component F_{e-r} and the axial component F_{e-a}), the friction force f between the Nafion®-115 DMSO solution and the pore wall surface, the pressure difference F_p between the two sides of the template and gravity; these are shown in figures 3 and 4. Under the action of these forces, the Nafion®-115 nanowires were formed in the AAO template. These forces will be discussed in detail in the following text.

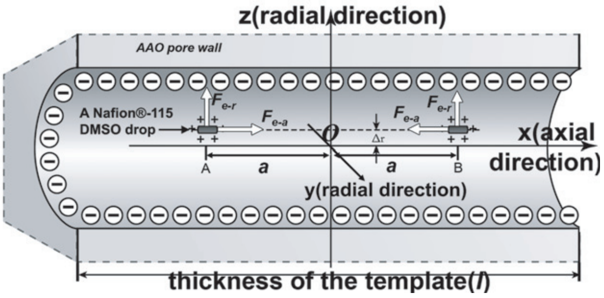

Figure 4. A scheme showing the model to study the electrostatic force in detail. For two Nafion®-115 DMSO drops that are marked by A and B, if they have the same distance from the original point O, F_{e-a} acting on one is equals to that on the other, but the two F_{e-a} have opposite directions.

Table 2. The contact angle (θ) and the surface tension coefficient γ of the Nafion®-115 DMSO solution on the template surface in the four cases.

Case No.[a]	Contact angle θ (degree)	Surface tension coefficient γ (mN m^{-1})
I	54 ± 1	35.14 ± 0.01
II	47 ± 1	34.59 ± 0.01
III	39 ± 1	34.50 ± 0.08
IV	18 ± 1	34.95 ± 0.01

[a] The case No. is the same as that in table 1.

4.1. The surface tension F_s of Nafion®-115 DMSO solution

We can calculate the surface tension F_s as [27]

$$F_s = \pi d \gamma \sin\theta \qquad (1)$$

where d is the diameter of the AAO pore, γ is the surface tension coefficient and θ is the contact angle of the Nafion®-115 DMSO solution on the pore wall surface. In order to calculate F_s, θ and γ should be measured first. The contact angle θ and the surface tension coefficient γ of the Nafion®-115 DMSO solution on the pore wall surface can be considered equal to those on the template surface when the pore diameter is small, according to Thompson and Yamamoto *et al*'s work [28, 29]. Hence, the contact angles θ and the surface tension coefficient γ of the Nafion®-115 DMSO solution on the pore wall surface can be measured on the template surface. The surface tension coefficient γ was measured on a Kruss-K12 surface tension instrument system. The results of measuring θ and γ are shown in table 2 and figures 5(a)–(d). It is indicated that the four different AAO surfaces in figures 5(a)–(d) are oleophylic. The contact angles θ of the four cases are in the order

$$\theta_{I} > \theta_{II} > \theta_{III} > \theta_{IV}.$$

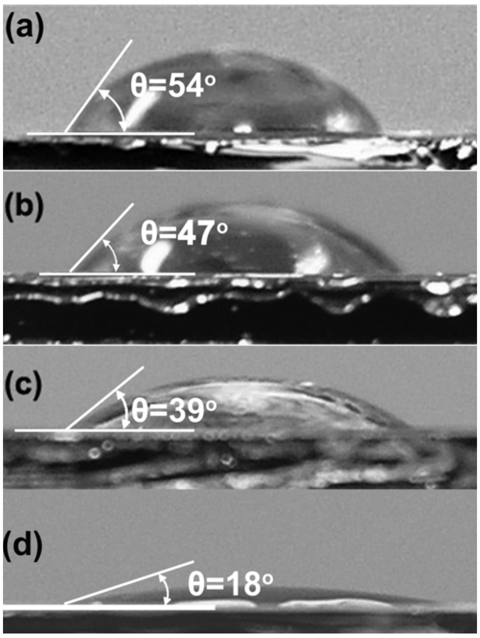

Figure 5. The contact angles (θ) of the Nafion®-115 DMSO solutions on the surface of the AAO template decorated by different surfactants: (a) no surfactant; (b) 1% SDS aqueous solution; (c) 1% P_{123} alcohol solution; (d) 1% CTAB aqueous solution.

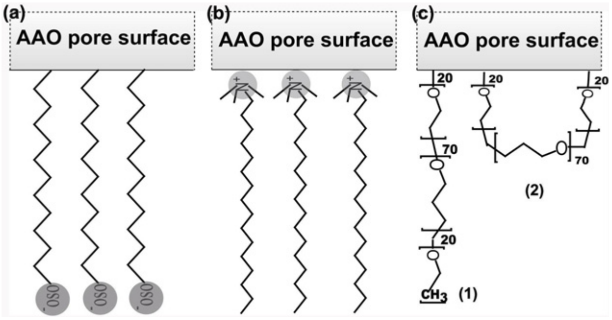

Figure 6. Schemes of the contact modes of surfactants on AAO pore wall surface: (a) SDS, (b) CTAB, (c) P123.

The AAO used in our research was prepared by a two-step oxidation method using 4% oxalic acid as electrolyte. The AAO pore wall surface is a little negatively charged due to the absorption of $C_2O_4^{2-}$ during the preparation process. The pore walls of the AAO were also reported to be negatively charged [30]. Due to the electrostatic effect, the three surfactants have different contact modes on the AAO pore wall surface in the decoration process. These are shown in figures 6(a)–(c). In case II (figure 6(a)), the anionic surfactant SDS is attached to the pore wall surface via an alkyl end with the sulfonic group outwards, so the contribution to lipophilicity in this case is a little better than that in the undecorated case. In case IV (figure 6(b)), the cationic surfactant CTAB is attached to the pore wall surface via an ammonium ion with the oleophylic cetyl head outwards. In case III (figure 6(c)), the neutral surfactant P123 can be attached to the pore wall surface by two modes (1) and (2), and the former is hydrophilic while the latter is oleophylic, so the lipophilicity of case III is between that of cases II and IV. As a result, the contact angles θ of the four cases are in the order shown above.

Thus, we can calculate the surface tensions F_s from equation (1); the results are presented in table 3. F_s are 7.59×10^{-9}, 6.76×10^{-9}, 5.80×10^{-9}, 2.88×10^{-9} N in cases I–IV, respectively, which are presented in table 3 for a further discussion.

Table 3. The filling rate versus F_s and F_{e-r}.

Case No.[a]	Filing rate (%)	F_s (N)	F_{e-r} (N)
I	∼42	7.59×10^{-9}	∼0
II	>95	6.76×10^{-9}	$\sim 10^{-8}$ (attractive force)
III	∼40	5.80×10^{-9}	∼0
IV	∼20	2.88×10^{-9}	$\sim 10^{-8}$ (repulsive force)

[a] The case No. is the same as that in table 1.

4.2. The radial component F_{e-r} of the electrostatic force

Take the surface decoration with the 1% SDS aqueous solution as an example. In order to study the electrostatic force, the charge remaining on a pore wall surface after surface decoration and the charge on a Nafion®-115 molecule must be estimated first.

After the AAO template was immersed in the SDS solution for 24 h, the amount of SDS remaining on one pore wall surface, n_0, can be calculated from

$$n_0 = \frac{\pi d^2 l \rho}{4 M_{\mathrm{SDS}}} \tag{2}$$

where d is the diameter of the AAO pore, l is the thickness of the template, ρ is the density of the 1% SDS aqueous solution, and M_{SDS} is the molecular weight of SDS.

As is known, each sulfonic group in an SDS molecule carries a unit of negative charge. At this point, the surface charge density σ_0 on a pore wall surface is

$$\sigma_0 = \frac{n_0 N_A e}{\pi d l} = \frac{d \rho N_A e}{4 M_{\mathrm{SDS}}} \tag{3}$$

where N_A is Avogadro's number and e is the unit charge, with the other parameters being the same as given above.

The Nafion® used in our study is Nafion®-115. A Nafion®-115 film has equivalent weight 1100 (the equivalent weight, EW, is the number of grams of dry Nafion per mole of sulfonic acid groups when the material is in the acid form), a nominal thickness of 0.005 inch and molecular weight (M_{Nafion}) about 25 000 g mol^{-1} [20]. Thus, the average amount n_1 of the sulfonic acid group on one Nafion®-115 molecule is

$$n_1 = \frac{M_{\mathrm{Nafion}}}{N_A \cdot \mathrm{EW}}. \tag{4}$$

As mentioned in section 2.2.1, the Nafion® used in the study has been immersed in 10% sulfuric acid at 303 K for 24 h. Thus, the material is in the acid form, and the H_3O^+ can be stored in the proton channel, which is formed by about ten sulfonic acid groups [20]. The positive charge Q carried on one Nafion® molecule can be obtained:

$$Q = \tfrac{1}{10} n_1 e. \tag{5}$$

According the mode demonstrated in figure 4, for the Nafion®-115 molecule at point A, the distance between it and the axle is Δr ($\Delta r \ll r$), and the distance from A to O is a; thus, we can calculate F_{e-r} as follows:

$$F_{e-r} = \frac{1}{2\pi\varepsilon_0} \int_{-\frac{l}{2}}^{\frac{l}{2}} \int_{-\frac{\pi}{2}}^{\frac{\pi}{2}} \times \frac{r^2 Q \sigma_0 \sin\alpha}{[(x+a)^2 + r^2 + (\Delta r)^2 - 2r\Delta r \cos\alpha]^{\frac{3}{2}}} \mathrm{d}\alpha \, \mathrm{d}x \tag{6}$$

where ε_0 is the vacuum dielectric constant, α is the angle between F_{e-r} and the Y axis, and the other parameters are the same as given above. In the present work, $d = 85$ nm, $l = 20$ μm, $\rho = 1.0$ g cm^{-3}, $M_{\mathrm{SDS}} = 288.38$ g mol^{-1}, $M_{\mathrm{Nafion}} = 25\,000$ g mol^{-1}, $N_A = 6.02 \times 10^{23}$, and $e = 1.6 \times 10^{-19}$ C. Thus, F_{e-r} in case II is about 10^{-8} N. F_{e-r} in case II is an attractive force because the pore wall surface is negative while the Nafion® molecule is positive. Since only a semi-quantitative estimation is needed in this study, only the order of the magnitude needs to be considered.

F_{e-r} in case IV can be obtained in the same way. The only difference is the molecular weight used in equations (2) and (3). With the molecular weight of CTAB, F_{e-r} is 10^{-8} N. Here, F_{e-r} is a repulsive force because the pore wall surface and the Nafion® molecule are both positive.

In cases I and III, F_{e-r} is approximately zero, since there is no external electric field acting on the Nafion® molecules for the neutral surfactant P_{123} or undecorated surface. These results are presented in table 3.

4.3. The other forces: F_{e-r}, f, gravity, F_p

For the axial component F_{e-a}, it can be seen in figure 4 that for two arbitrary Nafion®-115 DMSO solution drops, if they have the same distance from the original point O, the F_{e-a} acting on them are equal, but are in opposite directions. Thus, the contribution of F_{e-a} in the formation of Nafion®-115 nanowires can be considered to be negligible in cases II and IV.

The pore wall surface roughness in the four cases was similar before and after surface decoration, since all the AAO templates were fabricated by the same process and all the surfactants used in the experiment are small molecules [24–26], and have a molecular size less than that of Nafion®. Therefore, f is similar in the four cases and the difference in its contribution in the four cases can be considered to be negligible.

The gravity and the pressure difference F_p between the two sides of the template can be considered to be the same in the four cases, since all the studies were carried out under the same conditions. Thus, we can omit the difference of the affections of the gravity and F_p in the four cases.

4.4. F_s and F_{e-r} versus the filling rates

According to the above analyses, F_{e-r} and F_s contribute to the formation of Nafion®-115 nanowires, while the influences of F_{e-a}, f, gravity and F_p can be considered to be negligible. The final results are shown in table 3.

It can be seen from table 3 that the effect of surface decoration directly affects the filling rate and is induced by the combination of F_{e-r} and F_s.

In table 3, F_s in case I differs from that in case III, while the filling rate in case I is close to that in case III. This indicates that the effect of F_s on the formation of Nafion®-115 nanowires is small. Further, compared with cases I and III, F_{e-r} in case II is not zero and attractive, which induces the great improvement of the filling rate. Therefore, F_{e-r} is a crucial factor in the formation of Nafion®-115 nanowires. This is because, in case II, the Nafion®-115 molecules are intensely attracted by the attractive $F_{\mathrm{e-r}}$ in the AAO pores. This causes the Nafion®-115 molecules to have a greater chance to absorb onto the pore wall surface and to form Nafion®-115 nanowires. As a result, the filling rate in case II is much higher than those of cases I and III. For case IV, $F_{\mathrm{e-r}}$ is repulsive. Thus, once a Nafion®-115 molecule gets close to the pore wall surface, the repulsive $F_{\mathrm{e-r}}$ pushes the Nafion®-115 molecule away. Therefore, the filling rate in case IV is very poor.

5. Conclusion

Nafion®-115 nanowire arrays were fabricated with an extrusion method using AAO membranes as templates. The surface decoration is important in the synthesis of the Nafion®-115 nanowire arrays, and it was systematically investigated. With sodium dodecyl sulfate (SDS) as a surfactant in the surface decoration, the filling rate of the Nafion®-115 nanowires in the corresponding template exceeds 95%, compared with the filling rate of 42% in an undecorated template. It is shown that the effect of the surface decoration is induced by the combination of the surface tension and the radial component of the electrostatic force, of which the radial component of the electrostatic force is more important. That is to say, the charges carried on the SDS molecules are negative, while the charges on the Nafion®-115 molecules are positive. An electrostatic force exists between them, and this can attract the Nafion®-115 molecules onto the pore wall surface of the AAO templates. Due to the radial component of the electrostatic force, a high filling rate of the Nafion®-115 nanowires in the template is achieved. The results are expected to be significant in the fabrication of organic nanowires, which have potential application in some nanodevices, such as organic electronics, optoelectronic devices, and nanoscale proton exchange membrane fuel cells.

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