

Synthesis of SiO₂ nanoparticles self-assembled thin film by organic–inorganic hybrid method

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Abstract

Amphiphobic thin films for touched panel application was prepared by SiO₂ nanoparticles self-assembled nanostructure. Silicon dioxide nano spheres were prepared by sol-gel method and well dispersed in a solution with surfacants of low surface energy. Nanostructure thin films were obtained by spin coating technologies.

1. Introduction

Amphiphobic materials means that the materials has properties of hydrophobic and oleophobic simultaneously. Because of its character of cleaning with ease, amphiphobic materials receiving a great deal of attention. Many application of amphiphobic materials in our daily-life environment such as glass of automobile, tiles of multi-storey buildings, and panels of monitors.

The object of our work is making an amphiphobic thin film which applied to touch panel for anti-fingerprint. Silicon dioxide nano-spheres make the the roughness in nano dimension. The roughness will enhance the property of subsequent low - surface energy material about amphiphobic [1~4].

If the panel shows amphiphobic, the water and olein which on fingers will not adhere to the panel easily. It makes the panel to accomplish the property of anti-fingerprint.

There are many methods of synthesis amphiphobic thin films, such as plasma treatment [5], chemical vapor deposition [6], and sol-gel method [7]. Sol-gel technology is not only used in thin film materials area, but also in catalyst, coating,

hybrid materials. Since its advantages of high degree of product's purity, and simple synthesizing processes, sol-gel method is one of most popular synthesizing methods. In this paper, we also used this method to prepare inorganic nanoparticles and the procedure of hybrid.

2. Experimental

2.1 Synthesis of silicon dioxide nano-spheres

In this paper, silica particles were prepared using a sol-gel process with a sequential addition technique in an ultrasonication bath. First, ethanol (99.8%, Aldrich) 50ml was mixed with DI water 1ml (13.5 MΩ-cm) in a sonication bath. After 10 minutes, 2.5ml Tetraethyl orthosilicate (TEOS) (98%+, Aldrich) was added while sonicating. And then after 20 min, 2.5ml ammonium hydroxide (25%, Wako) was added as a catalyst to promote the condensation reaction. Sonication was continued for a further 20 min to get a blue semitranslucent suspension. Particle sizes were measured using scanning electron microscopy (SEM, JSM-5600).

2.2 Synthesis of low-surface energy solution

The low-surface energy material in this paper is a commercial product called "F4" (Tai-Fluorine Tech.). The composition of F4 is 1,1,2,2-Tetrahydroperfluoro-1-Decanol (chemical formula: C₈F₁₇CH₂CH₂OH).

We mixed F4, low-surface energy material, with solvent isopropanol (IPA) (J.T.Baker) to make

low-surface energy solution. The low-surface energy solution, 70wt% F4 solution, then mixed with SiO_2 nanoparticles of various volume fraction.

2.3 Synthesis of amphiphobic thin films by organic-inorganic hybrid method

In this work, the thin films are obtained from the nanoparticles and low-surface energy solution. We used surface modification for nano-spheres by combining silica particles with low-surface energy solution. This process will change surfaces property of nano-spheres from hydrophilic into hydrophobic.

In this procedure, we fixed the nanoparticles suspension volume as 10ml, mixed with low-surface energy solution (70wt% F4) 3, 5, 7, 9, 11ml respectively.

After that time, we spreaded modified nano-spheres on glass substrate via spin coating technique. Finally, we put the samples into the furnace and heated to 50°C for 10 hours for drying.

When the samples were drew out from furnace, we examined the water/oil contact angles of the thin films via Contact Angle Meter (Sindatek, 100 SB). The transparent ratio of the samples was conducted for light with wavelength 400 ~ 800 nm by UV/Visible spectrophotometer (Jasco, V-560). The morphology of the thin films was analyzed by field-emission scanning electron microscope (Hitachi, S-4100I).

3. Results and discussion

Silicon dioxide nano-spheres were prepared by sol-gel method. The nanoparticles' dimensional distribution is about 200 nm, as shown in figure 1.

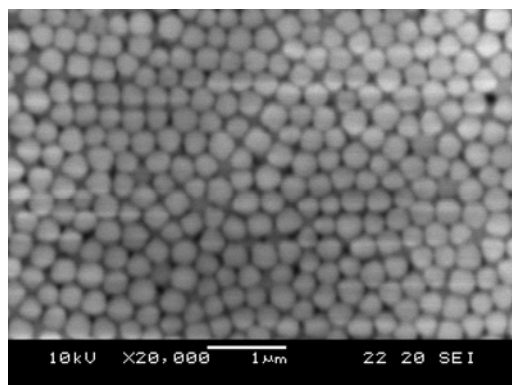


Fig. 1. SEM micrographs of SiO_2 nano-spheres

From figure 2, the analyzing of transparent ratio average in the range of visible light are from 75% to 45%. The decrease of transparent ratio of thin film in visible light range is caused by the increase of amount of 70wt% F4. When the amount of 70wt% F4 increased to 11ml, transparent ratio in visible light range of thin film down to around 45%. The reason is due to the increase of viscosity of the precursor [8]. Because of the thickness of thin films is influenced by viscosity, gravity, and centrifugal force. At the same condition of spin speed, the thickness is directly proportional to the square root of the thin-film solution viscosity.

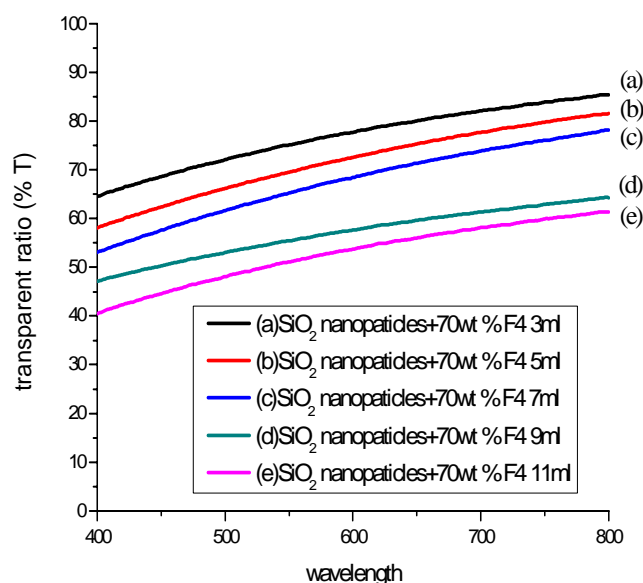


Fig. 2. Transparent ratio of thin film in the range of visible light : different amount of 70 wt% F4 (a)3ml (b) 5ml (c) 7ml (d) 9ml (e) 11ml

In the contact angle test, we chose DI water and glycerol to drop on the thin film. All of water/oil contact angles of the samples are greater than 90° . This shows the character of hydrophobic and oleophobic. (See table 1, figure 3, and figure 4)

Besides, it has been reported that the water/oil contact angles should increase with amount of low-surface energy solution increase [7]. However,

er, the water/oil contact angles of thin films are increase in lower amount of 70wt% F4 solution but not found in lower amount.

In the case of the amount of 70wt% F4 are 9ml and 11ml, congregation of nanoparticles (see figure 5) consequent on their decrease of water / oil contact angles in lower amount of 70wt% F4. From figure 5(d) (e), we can find island is made of silica nano-spheres. Therefore, the roughness degree is not in nano dimension anymore, it increase to micro level or bigger.

TABLE 1. water and oil contact angles of thin films with different amount of 70wt% F4

	3ml	5ml	7ml	9ml	11ml
WCA	114.6°	115°	118°	114.8°	113°
OCA	110°	111°	112.6°	108.2°	103°

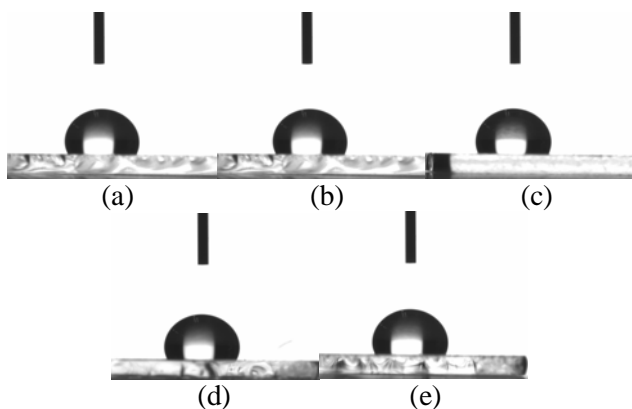


Fig. 3. water contact angles of thin film with different amount of 70wt% F4 (a)3ml (b) 5ml (c) 7ml (d) 9ml (e) 11ml

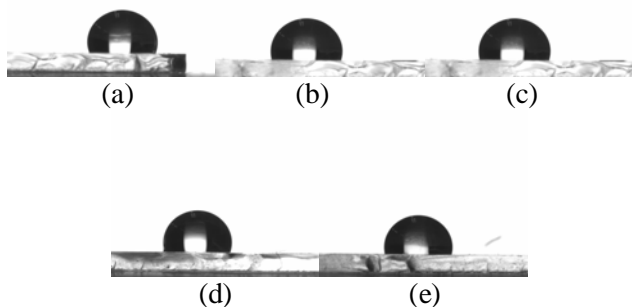


Fig. 4. oil contact angles of thin film with different amount of 70wt% F4 (a)3ml (b) 5ml (c) 7ml (d) 9ml (e) 11ml

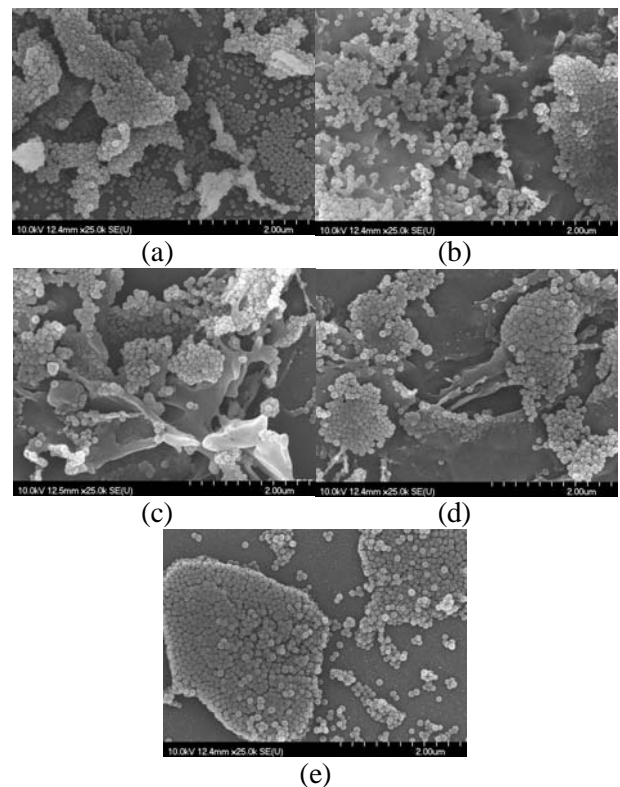


Fig. 5. SEM micrographs: morphology of the thin films with different amount of 70wt% F4 (a)3ml (b) 5ml (c) 7ml (d) 9ml (e) 11ml

4. Summary

In this paper, We have synthesize SiO₂ nanoparticles self-assembled thin film with character of amphiphobic. When the amount of low-surface energy solution increases at mixing, it will raise the thin film solution viscosity. For this reason, it leads transparent ratio of thin film in visible light range to decrease.

Moreover, water/oil contact angles of thin films were affected by congregation of nanoparticles, and caused water/oil contact angles of thin films can not increase further.

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5. References

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