Research on the effects of salt doping on the surface morphology of alumina thin films

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Abstract. We envisage that by the method of adding salt compounds, then washing them after the film sintered can produce holes inside and in the surface of the alumina film such as cheese-like, so that the organic matter can enter the aluminum oxide inside. Alumina can play a role in protecting internal organic matter from friction and prolong the use of hydrophobic film in the use. In the experiment, we found that the surface of the thin film with mixed magnesium acetate can form a good roughness.

1. Introduction
With the development of superhydrophobic materials, the superhydrophobic technology has been more widely used in life, which can be used to make self-cleaning glass, and In addition, the self-cleaning coating with low surface free energy, can prevent or reduce the adhesion of water vapor, ice and snow and other pollutants to solid surfaces, and also has important application value in the fields of aviation and aerospace [1-3]. However, the surface of hydrophobic film is mostly organic layer, the friction resistance is not strong, and there are few reports on the friction resistance of organic hydrophobic film.

In order to improve the friction resistance of the hydrophobic film, we assume that if the film can be made like cheese inside, there are multiple holes, so that the organic matter can enter the film, and the grafting amount is mostly increasing. After the wear of the organic layer on the surface of the film in the use, there is alumina as a hard support, the inner organic layer can be protected to play a good hydrophobic role, which can improve the friction resistance of hydrophobic film and prolong the service time.

According to the literature, there are two main methods to make the surface roughness of the film. the first one is the organic solvent volatilization, which is using the low boiling point of organic solvent can be preheated during the firing of the film, and the surface of the film will be have holes.
Polyethylene glycol doping is reported in this method. The second is the washing the inorganic ion, which is adding inorganic ions to the sol, then washing the surface of the film with deionized water to after firing to make the holes. This second is less reported. We envision Alumina thin films can be prepared by using miscellaneous inorganic salts or organic salts in alumina sol, and then the salt compounds were washed by hot water to make holes and surface roughness.

2. Experiment

2.1. Preparation of AlOOH Sol of Bom Stone
The secondary aluminium butanol (0.2mol, 49.264g) was added to 85 degrees water (20mol, 360g, the amount ratio of secondary aluminium to water was 1:100) and stirring with magnetic stirring apparatus for 30 min. The pH was adjusted to 2-3 with nitric acid and 85 degrees Celsius stirring for 30 min to obtain alumina sol. laying for 5 days.

2.2. Screening of compounds
As we all know, I_2 will volatile when it is preheating, we envision that if I_2 is mixed into the sol, during the firing, iodine preheat sublimation can form holes in the film. Similarly, we tried doping inorganic or organic salts such as AgNO_3, Ac_2Mg, NaCl, and MgCl_2 into sol to prepare the film, and then washed the inorganic salt compounds by hot water, hoping to make the desired cheese model.

We added the different compounds directly to the prepared sols at a ratio of 30%. The experimental results are as follows:

| Table 2-1. Film-forming results of added the different compounds |
|-----------------|----------------|--------|----------------|--------|----------------|
| No doping | Ac_2Mg | MgCl_2 | NaCl | I-IK | AgNO_3 |
| sol case | limpid | limpid | limpid | limpid | limpid | schlammbildung |
| Drying of the film | The film is smooth | The film is smooth | The film is smooth | The film is flat, the surface has sodium chloride needle crystal, the crystallization is even | Flat film with a small number of cracks in the middle | ——— |
| Sintering at 100°C | The film is smooth | The film is smooth | The film is smooth | The film is smooth | Film rupture | ——— |
| Sintering at 550°C | The film is smooth | The film is smooth | The film is smooth | Film rupture | ——— | ——— |

The experimental results show that the crystals of sodium chloride was precipitated during the drying process, and the crystals was destroy the continuity of the film, which caused the phenomenon of broken film. The colloidal charge of silver nitrate is opposite to that of alumina sol, and the precipitate is produced when the two sols are mixed. Alumina thin film made from sols mixed with magnesium acetate or magnesium chloride was not crack during the firing process, so we try to use different proportions of magnesium acetate, mixing, to find a suitable proportion to make the film, to improve the roughness and add internal holes.

2.3. Parametric Screening
The results of the experiments were as follows:
Table 2-2. Magnesium Acetate Mixed Sol

| Sol Sample No. | 1    | 2    | 3    | 4    | 5    |
|----------------|------|------|------|------|------|
| nAl: nMg       | 7:1  | 6:1  | 5:1  | 4:1  | 3:1  |
| Time of Gel    | >50d | >50d | 30d  | 20d  | 12d  |

2.4. Film preparation
We selected sol no.5 to coating on glass slides by coating, firing, and then boiled in hot water at 90°C for 30 min. get the sample.

2.5. Results Detection

2.5.1. Infrared Spectra of Thin Film (IR: Frontier)

![Figure 2-1a. IR spectrum of pure alumina film](image)

![Figure 2-1b. IR spectrum of magnesium acetate complex alumina](image)
The stretching vibration absorption peak of Al-OH bond is 3550–3000 cm\(^{-1}\), and three strong absorption peaks of 1000–1600 cm\(^{-1}\). There are certain absorption bands in the range of 400–1000 cm\(^{-1}\) [4], which is the characteristic absorption peak of nano-alumina. The enhancement of the three absorption bands in the 1000–1600 cm\(^{-1}\) interval in figure 2-1b is due to the addition of magnesium acetate, which makes the narrow particle size distribution of Al\(_2\)O\(_3\).

2.5.2. Thin film characterization. The films were characterized by AFM (AFM : SPA-400SPM unit; Scanning Probe:PPP-SEIHR-20).

![AFM on the surface of magnesium acetate complex alumina film](image)

Figure 2-2a. AFM on the surface of magnesium acetate complex alumina film

We can see after boiling water treatment, the surface of the magnesium Acetate mixed Film is net form, and there are obvious protrusions and pits in the net form, and the bulges are connected with each other by columnar structure to form a flower-like porous structure. The surface difference of peak value is about 200 nm. It shows that it has a certain surface roughness [5, 6].

3. Results and discussions

The surface roughness of magnesium acetate doped alumina film is controlled at 200 nm, which is more suitable for making hydrophobic film grafting. But I don't know if it formed the structure of the cheese model.

Acknowledgments

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