Surface Wettability of Silica Films Modified by UV/Ozone Treatment

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Abstract

Surface wettability of silica films was modified by UV/Ozone treatment. The silica films were prepared by dip-coating process using tetraethylorthosilicate (TEOS) as starting precursor. The TEOS was dissolved in mixing solution of absolute ethanol, deionized water and ammonia and then hydrochloric acid was added as the hydrolysis catalyst. The mixed solution was coated on glass substrates by dip coating process operated at 30 mm/min and heated at 100 °C. After that, the deposited films were exposed in UV/Ozone generator at different time treatment. The effects of time treatment on surface wettability, and optical properties of modified silica films were investigated by contact angle measurement, UV-Vis spectrophotometer. The contact angle of silica films was decreased after UV/Ozone treatment. These results implied that surface wettability of silica films was significantly improved by UV/Ozone treatment due to the oxidization its surface.

Keywords: UV/Ozone, silica films, surface wettability

1. Introduction

Silica (SiO_2) films have been widely used as anti-reflection, insulation, waveguides and corrosion protection layers in the integrated circuits [1]. Silica antireflective (AR) coating on optical equipment is one of effective method due to the decrease of light reflection in optoelectronic and photovoltaic devices. Sol-gel Silica coating is extensively prepared by tetraethylorthosilicate (TEOS) based on base/acid catalyst procedure via spin or dip coating method. For base/acid catalyst procedure, it can provide high transmittance and strong adhesive force on the substrates [2]. Due to its high specific surface areas by sol-gel silica AR coatings, it tends to adsorb water or polar organic molecules into the pore of silica film surface in environmental system. Poor adhesion and weather resistance are occurred after contaminated resulting in the inferiority of antireflection efficiency [3]. Therefore, surface wettability is a significant feature for solving this problem in optical equipment. It is well known that surface wettability is divided into 2 types of superhydrophobic and superhydrophilic properties. Superhydrophobic surface is the mechanism of

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unique water-repellent property with water contact angle greater than 150° and low contact angle hysteresis (lower than 5°). While, complete water spreading governed by both roughness of the surface and photo-induced surface reactions is named as superhydrophilic surfaces [4]. Various methods dealing with these problems have been proposed such as UV/Ozone treatment, oxygen plasma treatment and fabrications during deposited film [5]. UV/Ozone treatmentis a potential process because of short lifetime of modification and contaminated removability on surface film. Ozone is strong oxidant and environment friendly byoxygen decompositionresulting in surface cleaning. Moreover, the oxidation reaction by ozone treatmentis limited on top surface. Mechanical property in material was still stable and non-destructivestructure [6]. Based on recent report, surface modification by UV/Ozone with various time exposures on silica AR coating layer wasstudied. Moreover, the effective modified silica films were monitored by wettability property.

2. Materials and Methods

Silica films were deposited on glass substrates via sol-gel dip-coating method. A silica sol-gel precursor was prepared by two-step base/acid catalyst procedure using tetraethylorthosilicate (TEOS) as silica starting precursor. TEOS, ethanol, deionized water and ammonia with molar ratio of 1:37.6:3.25:0.17 were firstly stirred at 30°C for 2 h. After that, 1.02 molar of hydrochloric acid was added as a catalyst and continuously stirred at 30°C for 2 h. The mix solution was kept at room temperature for 9 days to obtain polysiloxane solution. Glass substrates were ultrasonically cleaned in detergent, DI water, acetone and ethanol, respectively. Before film deposition, the substrates were dried on hot plate. The precursor solution was coated on the glass substrates by a dip coater at speed 30 mm/min followed by curing process at 100°C for 2 h. Surface modification of the silica films was fabricated by UV/Ozone generator. The silica films were UV-irradiated in ozone at various exposure times.

Surface topographies and roughness were monitored by atomic force microscopy (AFM, Seiko Instruments, SPA400) using non-contact mode. Optical properties of the silica films were characterized by UV-Vis spectrophotometer. Water contact angles proving wettability on modified films were measured on model JYSP-360.

3. Results and Discussion

Surface wettablity of modified silica films by UV/Ozone with various exposure times monitored by contact angle measurementwas shown in Figure 1 (a)-(f). The contact angle of silica film before UV/Ozone treatment was aproximately 48.70° existing hydrophobic feature on the surface film. After UV/Ozone treatment, the contact angle of silica films graduallydecreased to 38.90°, 17.75° and 12.03° relating to exposure times at 5 min, 10 min and 15 min, respectively. Until exposure times up to 15 min, the contact angle was measured to 0° or flated on surface like water spreading onthin film. This result indicated that superhydrophilic property on silica film surface was distinctively obtained after UV/Ozone treatment with a short time exposure. This feature was good agreement with previous literature conducted by Lu, *et al.* [7] who reported that Si-OH group was key factor for complete water spreading with super hydrophilic mechanism. The possible phenomenon were proposed on the enhacement of surface modification of silica films via UV/Ozone treatment.

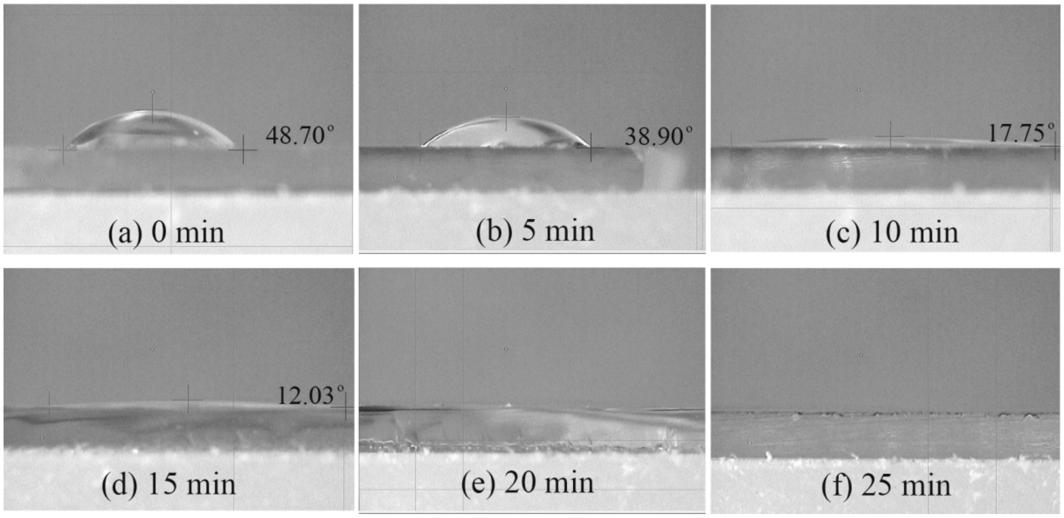

Figure 1. Contact angles between water droplets and silica films with various UV/Ozone exposure times (a) 0 min, (b) 5 min, (c) 10 min, (d) 15 min, (e) 20 min and (f) 25 min

During UV/Ozone operation, ozone molecule absorbed UV radiation and broke down in free oxygen atoms and ordinary oxygen molecules. Then the free oxygen atoms reacted with water molecule to generate hydroxyl radical in atmosphere [8]. This characteristic manner may be the higher –OH content in atmosphere and generating more Si-OH bonding on the surface films contribute to the increase of hydrophilicity property.

Surface to apography of silica films before and after UV/Ozone treatment were exhibited in Figure 2 (a)-(f). The surface topography of silica film without surface modification was rather ragged with root-mean-square (RMS) surface roughness of 0.42 nm. In contrast, the morphologies of silica films after UV/Ozone treatment were gradually smooth surface owing to Si-OH bonding in pore of silica film. Furthermore, water spreading was also facilitated by nanoporeson the surface filmvia wicking effect [9]. The decrease of RMS surface roughness was obviously obtained by increasing exposure times as illustrated in Figure 3. Smooth surface after UV/Ozone treatment was originated from the elimination of hydrocarbon contamination or organic residues on film surface [10]. Nevertheless, the RMS surface roughnesstend to a new heighten after exposure time up to 25 min was caused by the–OH groups fulfill in the pore of the silica film and then the bonding grow up into vertical plane.

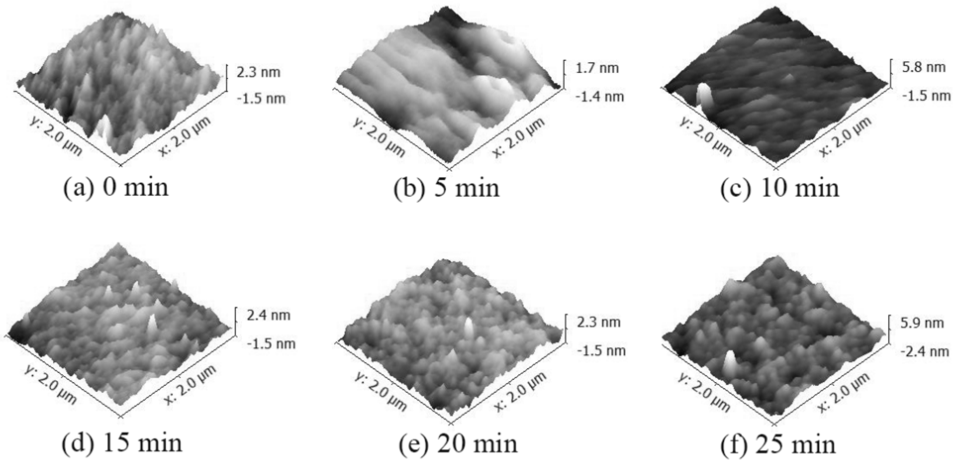

Figure 2. AFM images (2x2 μm) of silica films before and after UV/Ozone treatment with different time exposure (a) 0 min, (b) 5 min, (c) 10 min, (d) 15 min, (e) 20 min and (f) 25 min

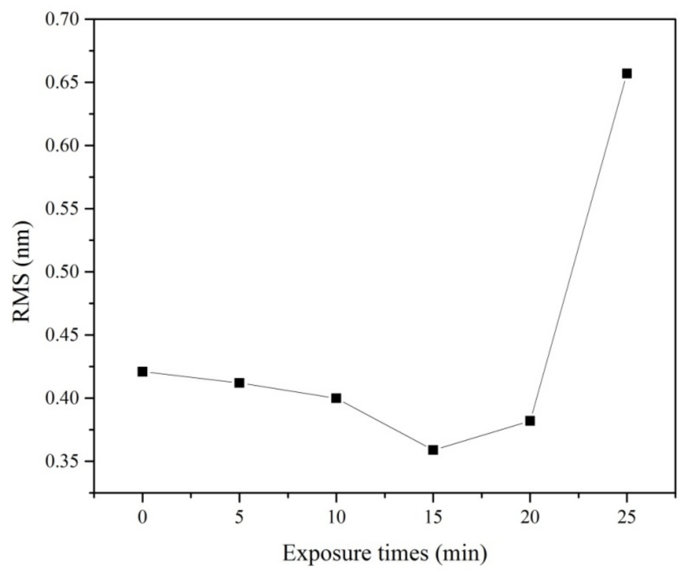

Figure 3. Root-mean-square surface roughness of silica film after UV/Ozone treatment

Transmittance spectra of silica films with various time exposures of UV/Ozone treatment in range of 250-800 nm were illustrated in Figure 4. High transparency of all samples was obviously occurred by modified silica film compared with pure glass substrate. This result can be interpreted that antireflection property on glass substrate was significantly improved after silica coating process. For modified silica film with UV/Ozone treatment, optical transmittance was identical to non-treatment spectrum. However, transmittance spectra of modified films slightly decreased under the increase of UV/Ozone exposure time due to the presence of –OH bonding on surface films.

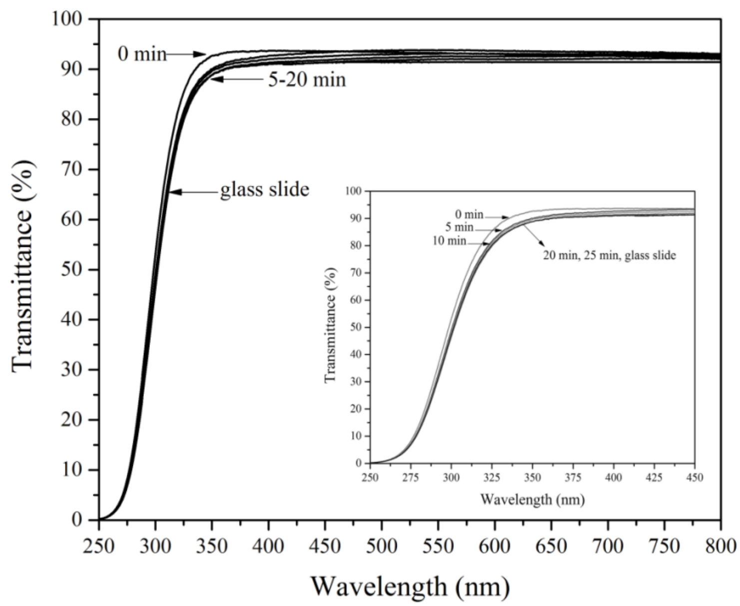

Figure 4. Transmittance spectra of Silica films at various time exposures.

4. Conclusions

The silica films were successfully prepared by sol-gel dip-coating method. The transmittance spectra of modified silica films by UV/Ozone treatment show insignificant alteration and remain high transparent comparing to pure glass substrate. Surface wettability of silica films identified by contact angle and AFM image revealed that complete waterspreading was obtained after UV-Ozone exposure times up to 20 min. Overall results indicated that superhydrophilicity feature on modified silica film was improved owing to strong Si-OH bonding on the surface, cleaning surface morphologies and wicking effect after UV/Ozone treatment.

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