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General Article

Hot Embossed TiO_2 Sol-Gel Wave Guide Films for Sensors Applications

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Abstract

A practical method to mass-produce gratings with limited lateral dimension is to emboss master grating into a Sol-gel waveguide film. Sol-gel titanium dioxide (TiO_2) thin film has been deposited on glass substrate using titanium precursors at low temperature environment. As-deposited films were amorphous. The morphology of the deposited film was analyzed by using high resolution SEM (HRSEM).The contact angle of water droplet on the film surface (hydrophilic properties) was measured by contact angle analysis. From the results; the formation of nano diffractive structure TiO_2 film obtained at low load and temperature environment was suggested for high quality structure.

Keywords: *Biosensor, Hot Embossing, Sol-gel Method, Waveguide film.*

1. Introduction

Sol-gel titanium dioxide (TiO_2) film with diffractive structure has numerous applications in optical waveguide light mode spectroscopy and sensors. In general, various functional coating layers (a high, medium or low refractive inorganic oxide) such as SiO_2, TiO_2, Al_2O_3 and ZrO_2 may be multi-coated on substrate are deposited by different methods to formed a layer capable of selectively absorbing/ reflecting light having certain wavelength ranges (Alam, *et al*, 2002), (Francioso, *et al*, 2003), (Gaur, *et al*, 2011), (Garzella, *et al*, 2000), (Mistry, *et al*, 2005), (Brinker, *et al*, 1990). Moreover, sol-gel titanium dioxide (TiO_2) has used in photovoltaic cells, oxygen sensors and biosensors application etc (Basu, *et al*, 2001), (Ebeoglugil, *et al*, 2005), (Sberveglieria, *et al*, 1995), (Zhenjiu, *et al*, 1999), (Crisan, *et al*, 2003), (Comini, *et al*, 2003). The controlling of the hardening process of the sol-gel thin films has led to a technology, which is capable to produce high precision replicated gratings in metal oxide waveguides as used for biosensors (Mistry, *et al*, 2005).
Sol-gel process has distinct benefits over other conventional techniques for preparing solid films with greater aspect ratio surface. A practical method to mass-produce gratings with limited lateral dimension is to emboss master grating into a sol-gel waveguide film. With proper optimization of the Sol-gel process, titanium dioxide (TiO_2) thin films have been deposited on glass substrate with grating structure formation by hot embossing using titanium precursors at low load and temperature environment.

2. Experimental details

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Coating solutions for deposition of titania were prepared by addition of titanium tetra-isopropoxide (20ml) to a pre-mix of ethanol (40ml), water (5ml) and acid. Obtained solution were generally left to react at room temperature for while and the mixture stirred vigorously (300 rpm) for 1hr until the suspension turned to a clear sol. Substrate surface is pre-treatment to ensure uniform wetting, was carried out by cleaning with alcohol and deionised water rinses and dried at room temperature. Titania coatings were applied to glass substrates by spin coating at 3000rpm. This process results in the deposition of a thin film of titania colloidal particles on the substrate surface.
The thickness of the coating can be varied by concentration of solution and spinning speed were undertaken to determine the effect of both these variables on coating thickness and uniformity. After spin coating, the substrates were left to dry for 10 minutes at room temperature and then placed in a pre-heated furnace at 150 to 250°C for 1hr. After the heat treatment, the samples were removed from the furnace and left in air to cool down.

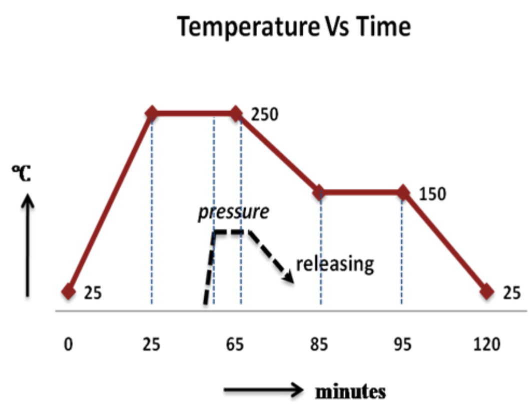

Fig.1 The sequence for heat treatment and hot embossing processes of the sol-gel TiO_2 coating on glass substrate.

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The master grating is placed over the sol-gel coated substrate and then, hot embossing carried out at 250°C for 20 minutes under applied load of 500kg. The sintering and hot embossing processes parameters are shown in Figure 1. The contact angle of the water particle on the film surface was analyzed by contact angle analysis. The surface morphology of grating structure was analyzed by using the high resolution scanning electron microscopy.

The cultivation of the bacteria on the TiO_2 film surface was analyzed by SEM. The TiO_2 with grating was immersed into the E.Coli bacteria for one week; the temperature was kept at 37℃ under controller. The bacteria solution with sample was stirred for one week. After that the surface morphology was observed by SEM analysis.

3. Results and discussions

It Uniformity of the film surface is good after sintering as compare to without sintering. As-deposited films were amorphous at lower temperature sintering process. The sequence for heat treatment and hot embossing processes of the sol-gel TiO_2 coating on glass substrate as shown in Figure 1. Critical load (500kg) is applied on master mold which is placed on the deposited coating for 20 minutes at 250℃ heat treatment stage (Figure1).Once pressing stage completed, gradually set the applied to zero. Slow cooling process helps to prevent the sticking of diffractive structure while demolding of the master piece. The thickness of TiO_2 sol-gel film was achieved up to 1 μm by spin coating method as shown in Figure 2. After embossed process, samples were allowed to analysis by High Resolution scanning electron microscope (HRSEM) as shown in Figure 3. We can see clear portion of a linear grating formed with an average line width of 200nm and a total length of 1.5mm.

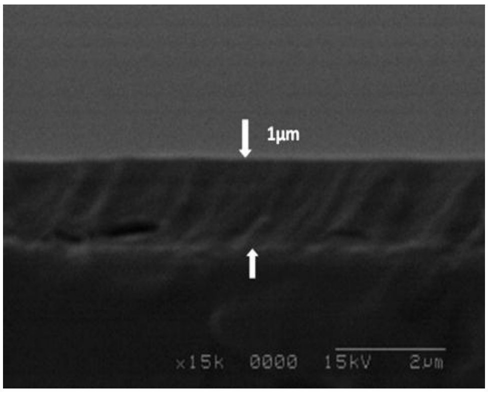

Fig.2 SEM micrographs of (a) thickness of the sol-gel TiO_2 coating (thickness less than 1μm) (b) side view of grating profile; Width of the grating structure: 200nm and height: 100nm.

Taking into account the important parameters affects the quality of the embossed grating film. Sol-gel TiO_2 film having refractive index is 1.745 under 60℃ drying and at 250℃ sintering temperatures.

Thickness of the film and side view of the grating profile was evaluated by HRSEM which is shown in Figure 3. We agreed that thinner coating more free from crack and internal stress than thicker coating. From the analysis results; after the deposited TiO_2 film on the glass substrate with sintering films having high contact angle than without sintering films.

The water contact angle on coated substrate and coated substrate with diffractive structure were measured by contact angle analysis as shown in Figure 4. In both case the films were shows hydrophilic properties. By using the plasma treatment, the contact angle can be modified under various power and gas flow conditions. As compare to film with grating having high contact angle than without grating structure. From our results; we concluded that the formation of grating on the TiO_2 film at low temperature sintering was suggested for high quality structure.

Absorb the interaction of bacteria with the film surface was investigated. Bacteria were not attached with the gratted TiO_2 film due to antibacterial properties of the titania. The photocatalytic activity of titania results in thin coatings of the material exhibiting self cleaning and disinfecting properties under exposure to UV radiation. These properties make the material a candidate for applications such as medical devices, food preparation surfaces, air conditioning filters, and sanitaryware surfaces.

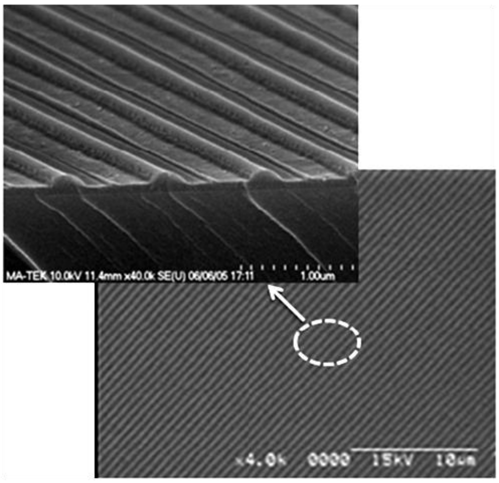

Fig.3 SEM micrographs of sol-gel TiO_2 coated substrate with diffractive structure obtained by hot embossing method.

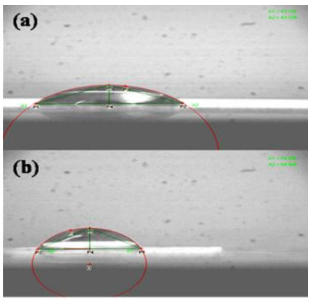

Fig.4 Photographs of (a) water contact angle on sol-gel TiO_2 coated substrate is 21°, (b) water contact angle on sol-gel TiO_2 coated substrate with diffractive structure is 53° obtained by hot embossing method.

Conclusions

The controlling of the hardening process of the sol-gel thin films has led to a technology, which is capable to produce high precision replicated gratings in metal oxide waveguides as used for biosensors. Sol-gel TiO_2 film having refractive index is 1.745 at 250℃ sintering temperature. Uniformity of the grating profile surface is good after sintering as compare to without sintering.

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