Preparation of Bilayer Doped Antireflection Film

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Abstract: TiO2-SiO2 film were prepared by sol-gel process. The prepared samples of these films have been characterized by FT-IR spectroscopy, TG-DTA, XRD and AFM. The transmittance of these films is tested by UV-VIS spectroscopy. The results show that the double doped glass antireflection film antireflection performance is good.

Introduction

Researchers to study long antireflection film, the recent emergence of many new breakthroughs [1]. Yu Bo prepared SiO2 by sol - gel film, in two-step acid-base catalysis. When TEOS: ethanol: hydrochloric acid: volume ratio of aqueous ammonia = 50: 146: 5.3: 2, the film at a wavelength of about 580nm light transmittance is better [2]. Peak transmission and other music Yueqin prepared on glass substrates using ammonia catalyzed SiO2 antireflection coating on both sides up to 99% [3]. XU Yao, etc. under acidic conditions in the glass K9 SiO2 antireflection films were prepared [4-5]. Shen Jun, TEOS, n-butyl titanate as raw material, sol - gel dip method successfully on a glass substrate plated with double antireflective film refractive index gradient, the bilayer glass in the 400-800nm wavelength transmittance increased by an average of nearly 6% [6-7]. Preparation antireflective film a lot, one of the most economic values is a sol - gel method. It process is simple, does not require vacuum equipment, low cost [8-10].

Experimental

Experimental materials and equipment. Furnace temperature controller, standard PH meter, 101-1A electric oven blast, SX24-12 box resistance furnace, magnetic stirrer, and vertical pulling machine: the equipment used.

Experimental procedure. The single-layer antireflection film preparation

N-butyl titanate (TPOT) as precursor, ethanol (EtOH) as the solvent, deionized water as reactants, glacial acetic acid (HAC) as the stabilizer, the volume ratio of the reactants is TPOT: EtOH: H2O : HAC = 5: 40: 1: 5. Preparation of the sol, in a state under stirring, the butyl titanate was dissolved in EtOH, sealed and stirred at room temperature 0.5 hours, ice dropwise acetic acid solution, stirred for 0.5 hours. After

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the solution was mixed until homogeneous, then was added dropwise a volume ratio of 1: 5 in aqueous ethanol, stirring was continued for 0.5 hours, was slowly added 1: Polyethylene glycol 400 (PEG400) solution of 5, stirring was continued for 0.5 hours, placed in a stable environment (20 ℃, relative humidity of 18% or less) standing to obtain a homogeneous, clear, transparent doped TiO2 sol. At room temperature and relative humidity of 45% -70% of the clean environment of the ordinary slides were coated by vertical pulling machine. After the slides were coated, dried in an oven at 40 ℃ for 1 hour in a muffle furnace and then were calcined at different temperatures by 1 hour.

Double layer antireflection film preparation:

TEOS (TEOS), EtOH, deionized water as the main raw material, adding an appropriate amount of HAC, the proportion of each component is TEOS: EtOH: H2O: HAC = 5: 40: 1: 5. At room temperature, mixed by a magnetic stirrer, was slowly added 1: PEG400 aqueous 5, stirring were continued for 0.5 hours, after standing to obtain PEG-doped SiO2 sol. At room temperature and relative humidity of 45% -70% of the clean environment of the monolayer film slides were coated by vertical pulling machine. After the second coated slides, dried in an oven at 40 ℃ for 1 hour and then in a muffle furnace with the different firing temperature for 1 hour.

Analysis and Testing:

Phase use Rigaku Corporation produced D-max-2500 / PCX ray diffraction analyzer. Russian morphology use sample NT-MDT company Solver P47 scanning probe microscopy.

Infrared Spectroscopy

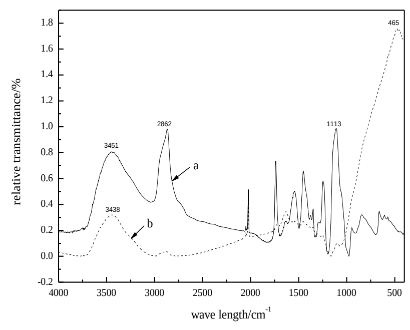

Fig. 1 FT-IR spectroscopy of PEG400/TiO_2 Gel (a: 100℃; b:

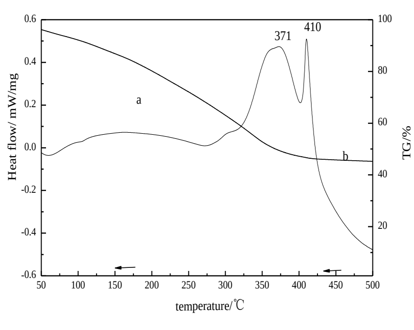

Fig. 2 DSC-TG curve for TiO2 PEG400/TiO2 Gel (a: TG; b: DSC)

Fig. 1 is an infrared PEG400 TiO2 sol was added and the analysis results. After the powder was calcined at 520 ℃ vibration peak near 2000cm-1-4000cm-1 is lower than 100 ℃ powder calcined at 2000cm-1-4000cm-1 near the peak of the vibration, which is due to the 100 ℃ of 520 ℃ calcining process, carbonization of organic matter decomposition and evaporation of free water caused.

DTA – TGA. Fig. 2 is a TiO2 xerogels DSC-TG analysis curve. TG curve from room temperature to 350 ℃ has been declining, this stage was about 52.27% weight loss, which is due to desorption of adsorbed water gel, ethanol release carbonized organic matter decomposition and organic polymers, and chemical bonding volatilization of water caused. In this process, the carbon in the film as the temperature is gradually increased stepwise becomes CO2.

From the DSC curve can be seen: there is a more gentle exothermic peak below 270 ℃, a clear exothermic peak at 340 ℃ to 430 ℃, which is the process of carbonation decomposition of organic matter caused by TiO2.

X-ray diffraction analysis. From the fig. 3 TiO2 powder XRD pattern can be seen: TiO2 anatase phase crystallization complete, did not find till 520 ℃ under rutile. It is formed at 25.192, 37.810, 47.861, 53.843, 54.912, 62.591, 68.705, 70.182, 75.008, 82.877 characteristic diffraction peaks at 2θ, and both showed significant anatase diffraction peaks. Therefore, in the heat treatment temperature of TiO2 thin films prepared anatase crystalline phase.

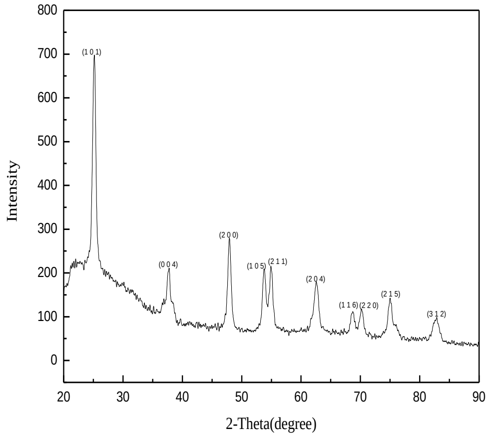

Fig. 3 XRD of TiO_2

Analysis of AFM. Fig. 4 is heat-treated after 1 hour 520 ℃ doped layer film of an atomic force microscope (AFM) in FIG. Film surface cracks and small gaps between particles, distribution is more uniform, no large aggregates, which indicates that the sol - gel method doped bilayer film uniform and dense. The film surface by a large number of columnar structures, surface columnar shape the rules, boundaries clear, uniform size, tightly packed. The thin film layer surface average roughness (Ra) of 3.531nm, rms surface roughness (RMS) is 4.491nm; the highest point of the film to the lowest point of the drop height (Rmax) of 34.710nm, the height is the average of all points 16.300 nm.

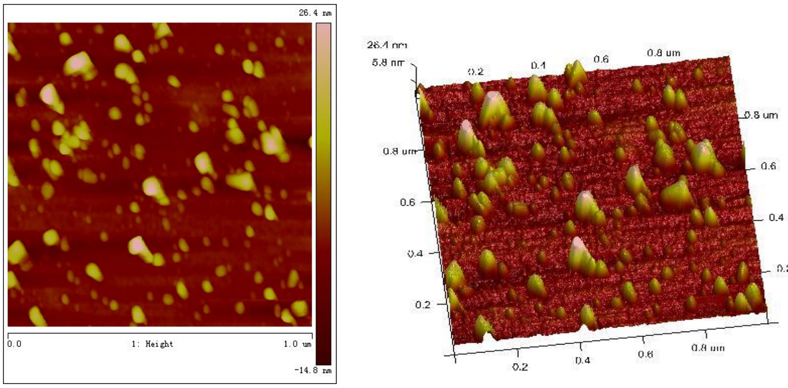

Fig. 4 AFM of TiO_2

Analysis of the light transmittance

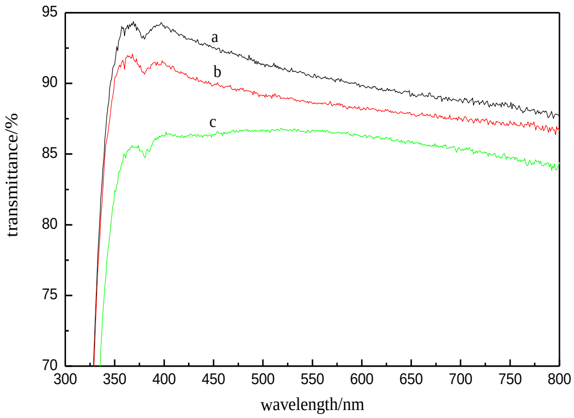

Fig. 5 Transmittance curve of antireflection thin film (a: TiO_2-SiO_2-PEG film; b: TiO_2 -PEG film; c: glass)

Fig. 5 is a graph showing a transmission line bilayer doped PEG, PEG transmission curve line b-doped monolayer film, c line transmission curve slide. PEG-doped bilayer best transmittance peak of 93.8%; PEG-doped monolayer film transmittance can reach 92.4%; blank slides transmittance peak was 86.7%. As shown, after the single-layer coating, slides in the 300-800nm wavelength transmittance increased by nearly 4 percent on average; after the double coating, slides in the 300-800nm wavelength transmittance by an average of nearly 8%.

Conclusions

Sol - gel method single dip-coating process for the preparation of the double doping antireflection film, uniform and compact, can effectively improve the light transmittance of the glass, especially double antireflection film doped glass transmittance rate more obvious.

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