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Original article

Sol-gel spin coating of dielectric reflector for energy harvesting and heat shielding applications

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ABSTRACT There are seasonal changes in the environment temperature which leads to additional usage of various devices like air-conditioners in the summer season and space heaters in the winter season. The usage of this equipment incurs increased consumption of electricity that can result in increased usage costs. High usage of air conditioners also releases large amounts of fluorocarbons that can contribute to global warming. Hence, to minimize the usage of air-conditioners and to protect from the seasonal temperature changes it is necessary to mold the light, which can be done by dielectric optical coatings. These coatings are derived from the thin films of varying refractive index that can be fabricated by many bottom-up techniques available in nanotechnology. Herein, we report one of the bottom-up technique i.e., sol-gel spin coating technique for the fabrication of a dielectric reflector that can serve as a window for the seasonal temperature changes by filtering the ultraviolet and near-infrared spectra. The fabricated reflector was investigated with FE-SEM for thickness and layer identification and UV-VIS-NIR spectroscopy analysis for the reflectance analysis.

KEYWORDS Ultraviolet and infrared radiation, sol-gel spin coating, dielectric reflector, smart window

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1. Introduction

Seasonally, the environment's temperature and meteorological conditions alter. The presence of UV and infrared radiations in the solar spectrum are principally responsible for the shift in temperatures and its repercussions, which can cause major harm to human organs [1, 2]. Due to their high exposure, skin, the most important organ of the human body, is prone to tanning and burning. UV radiation causes cataracts and retinal damage in the eyes, which can lead to blindness [3]. Humans are responding to the seasonal temperature variations by installing luxury items such as air-conditioners, space heaters, and air-coolers. Increased use of the aforesaid luxury equipment increases electrical consumption and emits chlorofluorocarbons (CFCs), which deplete the ozone layer by destroying hundreds of ozone molecules [4]. Protecting against these dangerous radiations can be accomplished by reflecting light back into the atmosphere through reflecting surfaces/reflectors. Reflectors are passive optical components that can be made from thin metal and dielectric sheets. Metal surfaces, on the other hand, are of little appeal because to their poor fading resistance and expensive maintenance costs [5]. Dielectric reflectors, which are made of dielectric materials with variable refractive index, are an alternative to metallic reflectors [6, 7], which can undergo constructive interference, resulting in a dazzling reflection pattern. TiO_2 with its refractive index 2.4 and SiO_2 with its refractive index 1.5 are the dielectric materials with a high refractive index contrast among dielectric materials which has the capability to result in a broad stop band for the desired wavelength spectrum [8].

Typically, dielectric reflectors are made via bottom-up nanotechnology processes, which include both expensive and inexpensive ways. It is necessary for the researcher to keep the reflector's fabrication costs low enough to cover the electrical costs associated with air conditioners and space heaters. Vapor based techniques such as chemical and physical vapor deposition are considered as the expensive techniques, whereas sol-gel dip-coating, spray pyrolysis and spin coating are considered as the inexpensive techniques. Out of the inexpensive bottom-up techniques, sol-gel spin coating technique is considered as the finest method due to its flexibility in tuning the process parameters (precursor, catalyst, spin speed and temperature) and its ease in operation [9]. The proper tuning of the process parameters can identify the reflectors in the mentioned spectral region with the minimal number of layers. Venkatesh et al. fabricated dielectric reflectors with TiO_2 and SiO_2 by varying the process parameters of sol-gel spin coating [10]. XRD, AFM and spectroscopic ellipsometry investigations on the fabricated thin films endorsed increase in precursor and catalyst concentration can increase the grain size and hence refractive index. This study identified a dielectric reflector for the near-infrared wavelength spectrum with only five layers of TiO_2/SiO_2 and also proved as a smart window by evidencing 105 °C temperature difference between in and out temperatures when studied with infrared source. Furthermore, the research identified 80 % reflection in the ultraviolet region along with the near-infrared region with the mentioned five layers. Hu et al. fabricated dielectric reflector with dense and porous films of TiO_2 by adding different amounts of polystyrene using sol-gel spin coating technique [11]. Ellipsometry investigations endorsed refractive indices of dense and porous TiO_2 as 2.17 and 1.49 with thicknesses about 24 and 115 nm respectively. The study evidenced the effect of number of pairs from in increasing the reflectance from 60 to 90 % by tuning the multilayer structures from 2.5 to 6.5. Further increase in the number of layers of the structure identified 100 % reflectance in the ultraviolet region. Ma et al. prepared a reflector with poly methyl methacrylate (PMMA) and TiO_2 using sol-gel spin coating technique by increasing the number of pairs from 2 to 6 [12]. Ellipsometry investigations identified refractive indices of PMMA and TiO_2 as 1.395 and 1.780 with thicknesses about 55 and 112 nm respectively. The study suggested increased number of pairs for the enhanced reflectance and shifting the stop-band towards higher wavelengths. Venkatesh et al. presented ultraviolet and near-infrared reflector with TiO_2/SiO_2 using sol-gel spin coating technique [13]. The study presented the influence of precursor and catalyst in shifting the stop band towards higher wavelengths. The cross-sectional FE-SEM investigations evidenced that increased precursor and catalyst concentrations can result in increased thickness of the multilayer structures. Finally, the change in thickness of the multilayer structures evidenced shift in the stop band from ultraviolet to infrared region. Romanova et al. deposited thin films of TiO_2/SiO_2 using sol-gel spin coating technique with thicknesses about 90 and 60 nm respectively [14]. The multilayer structure was fabricated with 12 layers and studied its reflectance through UV-vis spectroscopy technique. The as-fabricated reflector endorsed 100 % reflectance in the near-infrared region. Furthermore, the theoretical studies exactly correlated and supported the experimental studies.

The primary purpose of this study is to offer a low-cost method for producing a dielectric reflector capable of reflecting both harmful ultraviolet and infrared radiations utilising a multilayer $TiO_2/SiO_2/TiO_2$ structure. Section 2 presented below reports the experimental fabrication of the three layers along with the molar ratios of the various chemicals and spin coating parameters involved and further the Section 3 demonstrates the analysis on the fabricated reflector using cross-sectional field emission scanning electron microscopy (FESEM) and ultraviolet visible near-infrared (UV-VIS-NIR) spectroscopy. These structures can be used in office buildings, school buildings, and automobiles to shield people from the above-mentioned hazardous radiation.

2. Materials and methods

For the synthesis of TiO_2 sol solvent ethanol: precursor TTIP (Titanium isopropoxide): catalysts: acetic acid, HCl and H_2O in the molar ratios of 2.5:0.75:0.75:0.01:1 respectively. Similarly, SiO_2 sol was synthesized by considering solvent ethanol: precursor TEOS (Tetra ethyl orthosilicate): catalysts: acetic acid, HCl, and H_2O in the molar ratios of 2.5:0.75:0.75:0.01:1 respectively. The precursors TTIP and TEOS were purchased from sigma Aldrich, solvent from Changshu Hongsheng, HCl from Fischer scientific and acetic acid from Sisco research laboratories. All the chemicals were of analytical grade which doesn't require any further purification.

Figure 1(a,b) represents the flowchart and experimental fabrication of sol-gel spin coating process. Initially, the solvent was taken in a freshly cleaned beaker and in the next step catalyst, HCl, and acetic acid were added to the solvent on a magnetic stirrer as shown in Fig. 1(a). Finally, the precursor is added to the mixed solution of catalyst and solvent in a magnetically stirred beaker. The sol will be ready within very few seconds of magnetic stirring and requires no further aging. The procured sols were spin-coated on a thoroughly cleaned glass substrate by maintaining the spin rate of 2000 RPM for 15 seconds as shown in Fig. 1(b). The spin-coated films were sintered in a muffle furnace at 500 °C for 60 minutes to achieve crystallinity in the fabricated films. Multilayer ($TiO_2/SiO_2/TiO_2$) structure was obtained by following the same specifications periodically on the glass substrate.

The as-fabricated multilayer structures were characterized with cross sectional filed emission scanning electron microscopy (FESEM) for film thickness and layer identification (MIRA3 TESCAN) and ultraviolet visible and near-infrared (UV-VIS-NIR) spectrophotometer with a specular reflectance attachment (UV1800 Shimadzu, Japan) for reflectance analysis.

3. Results and discussion

Figure 2(a,b) shows the cross-sectional analysis and top-view of the multilayer ($TiO_2/SiO_2/TiO_2$) structure using field emission scanning electron microscopy (FE-SEM).

The cross-sectional FE-SEM investigation shown in Fig. 2 confirms the existence of TiO_2 and SiO_2 with their bright and dark appearance respectively. The thickness of the individual layers in the multilayer structure ($TiO_2/SiO_2/TiO_2$) is estimated at about 94/267/189 nm respectively. The study conveys that the films derived were very thick, and these thick films can shift the reflectance peak towards higher wavelengths due to the accumulation of a greater number of atoms on the surface [15, 16]. The increase in the film thickness can enhance the reflectance of the fabricated structure by increasing the photon absorption states [17]. It was also noted from the Fig. 2(b) that the multilayer ($TiO_2/SiO_2/TiO_2$) structure fabricated is uniform and free from cracks.

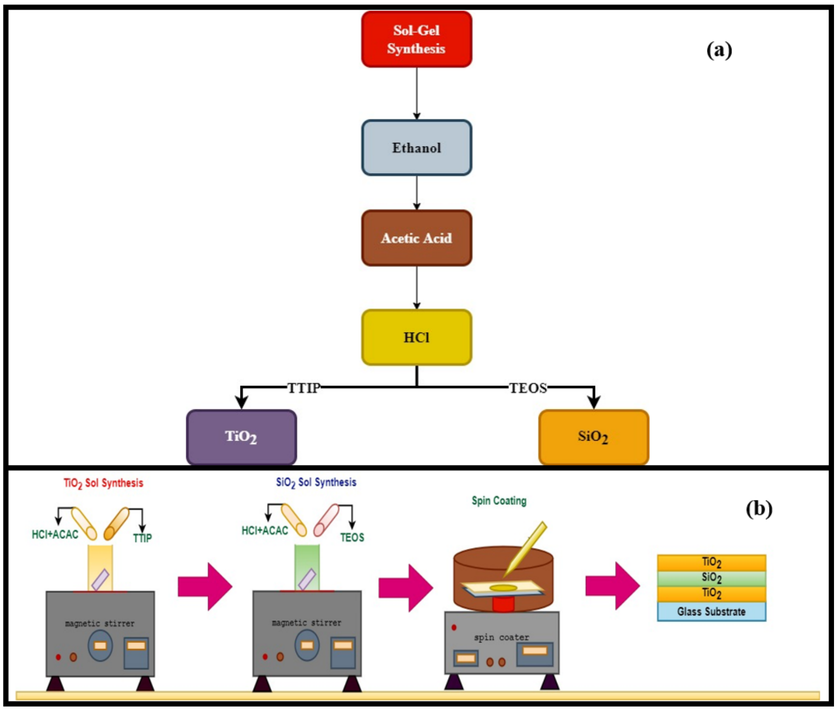

FIG. 1. Flow chart of sol-gel synthesis (a) and schematic of the experimental fabrication (b)

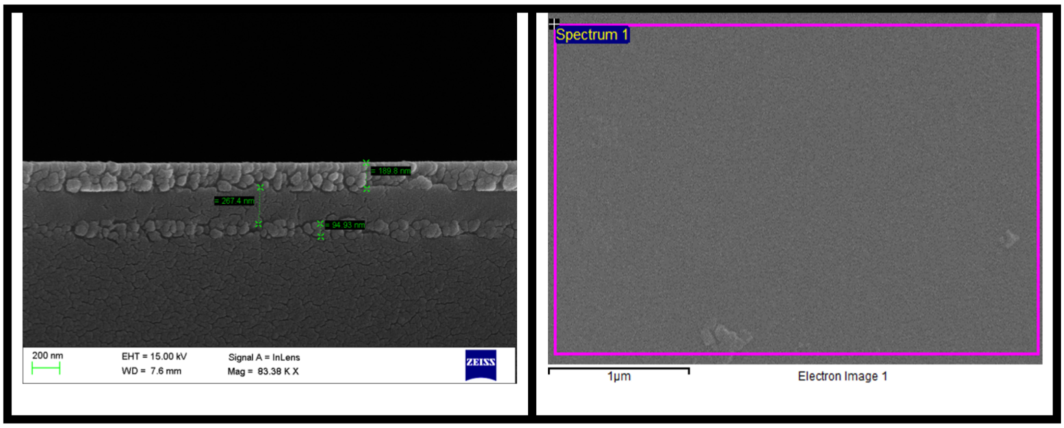

FIG. 2. Cross-sectional FE-SEM of the multilayer ($TiO_2/SiO_2/TiO_2$) structure (a) and top-view of the ($TiO_2/SiO_2/TiO_2$) structure (b)

The reflectance analysis on the multilayer ($TiO_2/SiO_2/TiO_2$) is performed using UV-vis spectrophotometer along with its digital image as inset and depicted in Fig. 3.

Figure 3 shows a multilayer ($TiO_2/SiO_2/TiO_2$) structure that clearly shows the presence of interfaces, which were interfered with to form a reflection pattern on the surface. The multilayer structure endorsed 60 % reflection in the infrared region with a central wavelength of 1000 nm and 72 % reflection in the ultraviolet zone, according to the reflectance analysis. This was a significant breakthrough in the field, reflecting both ultraviolet and infrared wavelengths in a single triple-layer construction. The as-reported structure can be applied in windows from protecting the seasonal temperature changes which reduce the usage of luxury equipment and also reduces the release of CFCs in the atmosphere.

4. Conclusions

An inexpensive method for the fabrication of ultraviolet and infrared reflector was proposed with only three layers of TiO_2 and SiO_2 by using sol-gel spin coating technique. The bright and dark bands shown in the cross-sectional FE-SEM analysis endorsed the presence of TiO_2, SiO_2, and TiO_2 films with their thicknesses about 94, 267, and 189 nm respectively. Furthermore, the reflectance analysis using UV-vis spectroscopy revealed that the thick films fabricated can undergo interference at the interfaces resulting 72 % reflectance in the ultraviolet region and 60 % reflectance in the infrared region. This kind of property made the multilayer promising for window application.

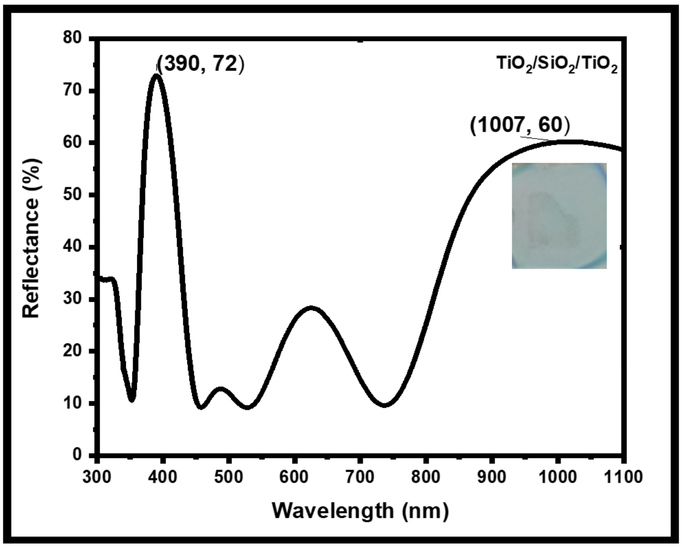

FIG. 3. Reflectance analysis of the multilayer ($TiO_2/SiO_2/TiO_2$) structure

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