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Mesoscale Surface Patterned Silica-Titania Sol-Gel Thin Film on Glass

Saswati Sarkar[1], Rimlee Deb Roy[2], Prasanta Kumar Biswas[1#], Shymal Kumar Bhadra[2] and Sunirmal Jana[1*]
[1]Sol-Gel Division, [2]Fibre Optics and Photonics Division
CSIR-Central Glass and Ceramic Research Institute (CSIR-CGCRI)
196 Raja S.C. Mullick Road, P.O. Jadavpur University, West Bengal, Kolkata 700032, India
[#]Presently at Physics Department, Indian Institute of Technology Roorkee,
Roorkee 247 667, Uttarakhand, India as Honorary Fellow
*Corresponding author: Tel.: +91 33 23223303, Fax: +91 33 2473 0957
E-mail address: sjana@cgcri.res.in, janasunirmal@hotmail.com (S. Jana)

Abstract. ***With the prior experience on fabrication of grating embossed sol-gel planar optical waveguide by soft lithography, we studied here the surface grating (mesoscale patterned) silica-titania thin film from 10 weight percent equivalent oxides containing precursor sol. An optimum viscosity, 1.98±0.5 cP was determined for obtaining high fidelity (with respect to structural uniformity) of patterns. Shrinkage of pattern height measured by atomic force microscope as a function of curing temperature was explained on the basis of thermo gravimetric weight loss of gel film as well as from volume percent porosity calculated using ellipsometrically measured film refractive index. Film surface morphology and crystallinity were also verified by FESEM and XRD measurements respectively. In addition to curing temperature, patterning also found to influence the percentage of UV-Vis transmission and reflection. This work would help on mesoscale surface patterning of other amorphous films of mixed oxides.***

Key words: **Sol-gel thin film, Soft lithography, Surface morphology, Thermo gravimetric analysis, Ultraviolet visible spectra**

I. INTRODUCTION

Studies on fabrication of grating based planar optical waveguide (GPOW) are continuously caring attention over the years [1]-[3] because the GPOW has numerous applications [1], [4]-[6] in the field of microelectronics and optoelectronics. Several techniques [7] including conventional photolithography are known for fabrication of structures such as laterally separated multiple gratings, stacked grating, bi-diffractive gratings, etc. These techniques have several limitations like material selection (applicable only for photosensitive materials), surface topography (curved and non-planer surface could not create acceptable results). Now-a-days, a cost effective unconventional soft lithography (SL) (e.g. capillary force lithography) is well known [7]-[9] over the conventional lithography for fabrication of nano/micro scales ordered structures (mesoscale patterns) on thin film surface. Also, the SL is a simple one for patterning single inorganic oxide from precursor sol of desired characteristics. However, for multi oxides many difficulties have to be faced to obtain defect free, high fidelity, structurally stable and chemically inert surface patterned films for application in devices [10], [11]. Therefore, it is very much necessary to know the precursor sol/solution characteristics that primarily depend upon many important factors, as for example, composition, viscosity, aging time, etc. This is because the factors strongly influence on the characteristics of thin film which would help to tune the quality of surface patterns. In this respect, a few reports [9], [12] are available in the literature. Further, several problems are to be faced to use soda lime silica glass (SLS) as substrate [13] for deposition of sol-gel thin films because it contains sodium and other diffusible ions, which can easily be penetrated through the porous network of low temperature sol-gel films. Due to the contamination problem, it is quite impossible to obtain the desired optical property (e.g. refractive index) of the films. We have already observed [1], [13, [14] the problem in patterned silica and silica-titania sol-gel films fabricated on soda lime silica glass by soft lithography technique. This could be solved if pure silica glass be used as substrate. Previously, we reported the theoretical aspects and a very little on fabrication of patterned silica-titania films. In this work, we prepared patterned silica-titania film on pure silica glass for fabrication of grating (mesoscale patterned) based planar optical waveguide (GOW) because for designing a GOW, an accurate value of film physical thickness (t) and refractive index (RI) are crucial factors. It is known [15] that the‘t’ and ‘RI’ of sol-gel films mostly depend upon the viscosity of precursor sol. In addition, the low temperature sol-gel film contains organics and the films are generally porous in nature. With increasing curing temperature, the removal of organics and mitigation of porosity would result shrinkage (densification) of film thickness. All these could effectively be influenced on the structural and optical properties of patterned silica-titania film.

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Volume 3, Issue 10, April 2014

However, these were not reported in our previous work [14]. Hence, in this work, a systematic study was performed to find out an optimum viscosity of precursor sol for obtaining perfectly surface patterned (order mesoscale structured) silica-titania film. An effect of change of sol viscosity on the formation and fidelity (especially height profiles, HP) of patterns was also made using AFM analysis. In addition, film microstructure and compositional analyses were made by FESEM and FESEM-EDS respectively. Moreover, the measurement of thermo gravimetric weight loss of gel film and volume percent porosity calculated using film refractive index were carried out to correlate the shrinkage behaviour of HP as a function of film curing temperature. Further, we studied the UV-Vis transmission and reflection spectral behaviour of patterned and non-patterned films.

II. EXPERIMENTAL

Fabrication of patterned silica-titania film

Precursor sol preparation to pattern fabrication in details has already been reported in our previous papers [1], [14]. Hence, a very brief for the same is given under this section.

A. Preparation of silica sol

All the chemicals were purchased from manufactures and used without their further purifications. The silica sol (10 wt% silica) was prepared from tetraethoxyorthosilicate (TEOS, Fluka Chemica, >98%) as silica source. TEOS was mixed with 1-propanol (E. Merck, ≥ 99%) and double distilled water in an acidic medium containing 1(N) HCl (E. Merck, India, GR Grade) as catalyst maintaining the molar ratio, water: TEOS : HCl = 2: 1 : 0.001.

B. Preparation of silica-titania sol

Titania sol was prepared by drop-wise and slow addition of tetraisopropylorthotitanate (TIOT as titania source, Puram grade, Fluka Chemica) in required amount of 2-propanol (E. Merck, GR) and acetyl acetone (acac, ≥98%, SRL) as complexing agent for controlling the fast hydrolysis rate of TIOT (acac: TIOT, molar ratio = 1 : 2) under stirring condition. After that, the as-prepared requisite amount of silica sol was added very slowly to the freshly prepared titania sol with vigorous stirring. Finally, required amount of 2-butanol was added to the aliquot to get the final sol (silica: titania, 50:50; total oxide content, 10 wt %). The mixed sol was kept further for ~3.5 h during stirring before film deposition.

C. Preparation of patterned silica-titania film on pure silica glass

The sols (as-prepared, 3h, 24h, 48h and 72h) were used for film deposition on pure silica substrate (Heraeus, Germany, Suprasil 2 Grade B, size: 75 mm x 25 mm x 1 mm) adopting dipping technique at a fixed lifting speed of 16 cm/min. As-prepared silica-titania film was then immediately embossed by using wet PDMS stamp. The PDMS stamp was prepared by Sylgard Silicone Elastomer 184 and Sylgard 184 curing agents (Dow Corning, Midland, MI). In our previous paper [1], [14] the detailed preparative method of PDMS stamp using commercially available compact disc (CD) having ~1.5 μm periodicity and 120 nm peak height for patterning has already been reported. In the present work, we slightly modified the embossing technique by applying pressure (0.826±0.12Pa) for obtaining relatively high profile height of patterns through enhancement of the capillary action. For this work, a scheme for the pattern fabrication is displayed in Fig. 1. The embossed samples were then desiccated for a day and left in an undisturbed condition. On the next day, after peeling off PDMS stamp, the samples were thermally cured at three different temperatures, 100^{o}, 300^{o} and 500^{o}C in pure oxygen.

III. CHARACTERIZATIONS

Viscosity of the sol was measured at room temperature (~30^{o}C) by software controlled Thermo Scientific HAKKE Rheostress (model RS6000), Germany using cone-plate (sensor system, C60/1) at a shear rate and time, 400 S^{-1} and 60 S respectively. Thermal characteristics (thermo gravimetric analysis, TGA; differential thermal analysis, DTA simultaneously) of the scratched off gel film were carried out by using a Netzsch STA 409 C/CD Thermo analyzer with Al_2O_3 as a reference material maintaining heating rate of 10 k/min in air. The TGA-DTA was measured up to 1000°C. Refractive index and physical thickness of non-patterned films were measured using an auto gain ellipsometer (Gaertner Auto–Gain Ellipsometer, L-116 B) equipped with a He-Ne red laser (wavelength, 632.8 nm). Patterned films were characterized by Atomic Force Microscope (AFM, Nanonics, Israel NSOM). AFM images and their respective height profiles were also recorded using the software of the AFM instrument. Amorphous nature of silica-titania film was confirmed by X-ray diffraction (XRD) study employing Bruker D8 Advance with DAVINCI design X-ray diffraction unit employed with nickel-filtered CuK_{α} radiation source (λ =1.5418 Å) in the 2θ range of 10^{o} to 80^{o}. Surface morphology and metal concentration (Si, Ti) mapping of patterned and non-patterned film was analyzed by FESEM and FESEM-EDS (ZEISS, SUPRA™ 35VP) respectively. UV-Visible transmission and reflection spectral study of the patterned and non-patterned films was measured by UV-Vis-NIR spectrophotometer (Shimadzu UV-PC-3100; photometric accuracy: transmission ±0.3%, wavelength resolution, 0.10 nm).

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IV. RESULT AND DISCUSION

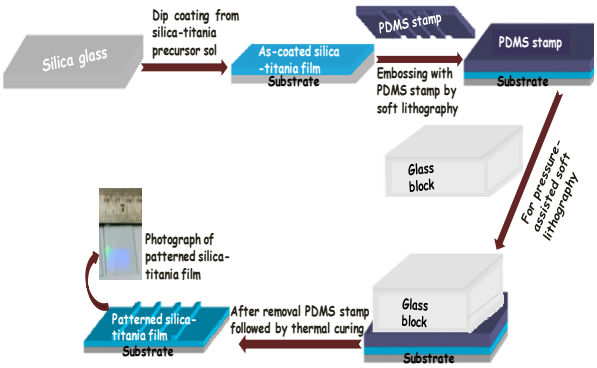

Fig.1. Scheme for fabrication of surface patterned sol-gel thin film by soft lithography.

Fig. 2 displays a plot constructed from viscosity versus ageing time of silica-titania precursor sol. The plot confirmed that the viscosity of the sol gradually increases with ageing time. This would be due to [14], [16], [17] the formation of homonuclear (≡Si-O-Si≡) and/or heteronuclear (≡Si-O-Ti≡) network [14] through condensation polymerization via hydrolysis of alkoxides (TEOS and TIOT) that were used in the precursor sol. It was noteworthy to mention that the use of acetyl acetone was to control the hydrolysis rate through partial complexation with Ti(IV)of TIOT. In capillary force lithography, the sol viscosity would be an essential factor for obtaining adequate capillary action through the grooves of PDMS stamp. Hence, an optimization of sol viscosity was very much essential for required capillary action which could generate defect free and high fidelity surface patterned (order mesoscale structure) film. It was observed that the sol viscosity increased rapidly after 3h of sol ageing.

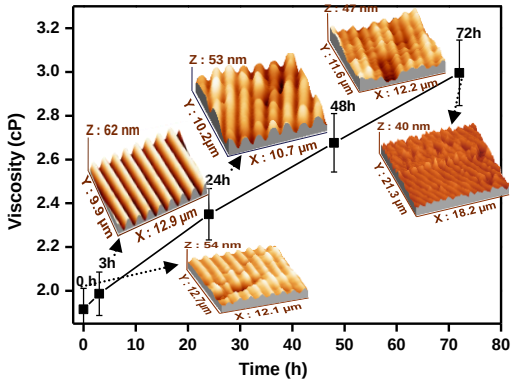

Fig. 2.Viscosity with ageing time of precursor sol. Insets show AFM images of 100^{0}C cured film deposited from the sol of different viscosities (aging times).

The film deposited from relatively high viscosity of sol would have less ability to undergo capillary action due to increased Gibbs's free energy [18]. Consequently, at this stage the formation of defect free patterned structure would be diminished. It was important to note that, at this condition, we were unable to fabricate the surface patterns on gel films even with the application of pressure.

During embossing process, we applied a pressure (0.826±0.12Pa) onto the as-deposited films for enhancing the capillary action (pressure assisted) which generated relatively high profile height of the pattern. This result strongly suggested that the formation of patterned structure was due to capillary rise of the sol through the groves of PDMS stamp. By AFM measurement, we verified the effect of changing sol viscosity on the fidelity of 100^{o}C cured patterned films deposited from the sol of different viscosities. From the AFM images (insets, Fig. 2) of the patterned films, a non-uniform and cracked patterned structures were resulted from the precursor sol of viscosity, >1.98 cP (sol ageing time >3h). In addition, the average height profile (HP) measured from the respective AFM image was found to be decreased. As for example, the HP obtained ~ 62 nm when the sol viscosity was 1.98±0.5 cP (aging time, 3 h) whereas it became ~40 nm for the sol viscosity, 2.98±0.5 cP (aging time, 72h). However, the as-prepared sol derived film generated irregular patterned structures with relatively low average HP (~54 nm).

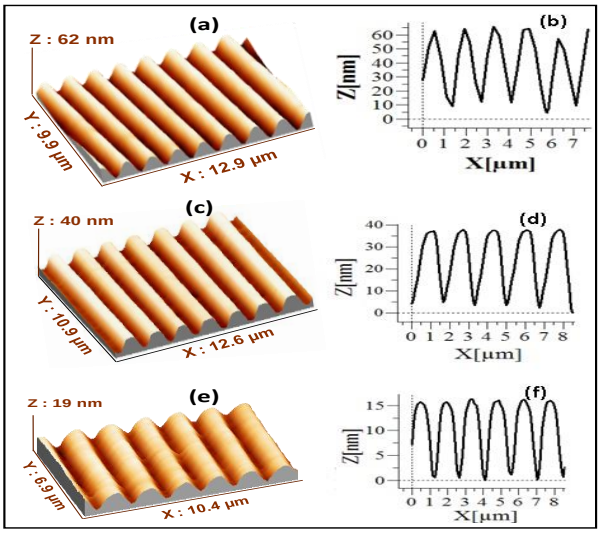

Fig. 3. AFM images, (a), (c) and (e) with their corresponding height profiles, (b), (d) and (f) of patterned films cured at 100^o, 300^o and 500^{o}C respectively.

The irregular pattern formation might be due to non-uniform (patch-like) film formation which would result from the low wettibility of precursor sol. Thus, our experimental results clearly demonstrated that an optimum viscosity (~1.98±0.5 cP) of the precursor sol could be a crucial and essential factor to obtain mesoscale order structure on the surface of silica-titania film through capillary force action [8], [19]. In addition, the importance of the optimum viscosity at a particular aging time was to obtain relatively high value of HP. It would

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an important factor for fabrication of efficient grating based optical waveguide sensor chip. Fig. 3 displays the AFM images (Fig. 3 a, c, e) along with the corresponding line profiles (Fig. 3 b, d, f) of silica-titania film (deposited from the optimum sol viscosity, 1.98±0.5 cP) at three different curing temperatures (100^o, 300^o and 500^{o}C). A uniform line profile of the patterns was formed onto the film surface. This would suggest that the technique is quite an efficient one for obtaining high fidelity surface patterns. However, the peak height of the patterns gradually diminished (shrunk) from 62 to 17 nm without appreciable change in original periodicity (~1.5 μm) on increasing curing temperature from 100^o to 500^{o}C. This would consider due to (a) the loss of waters and organics (used in precursor sol) and (b) film densification (as low temperature sol-gel film is generally porous in nature) [20]. However, the contribution of (a) and (b) would relate to the mass loss and the change of film porosity respectively. The effect of temperature on mass loss of materials could be obtained from the thermo gravimetric analysis (TGA) whereas the densification could be realised from the porosity calculation using measured refractive index of the films. This observation would be helpful for fabrication of grating based optical waveguide for sensor application.

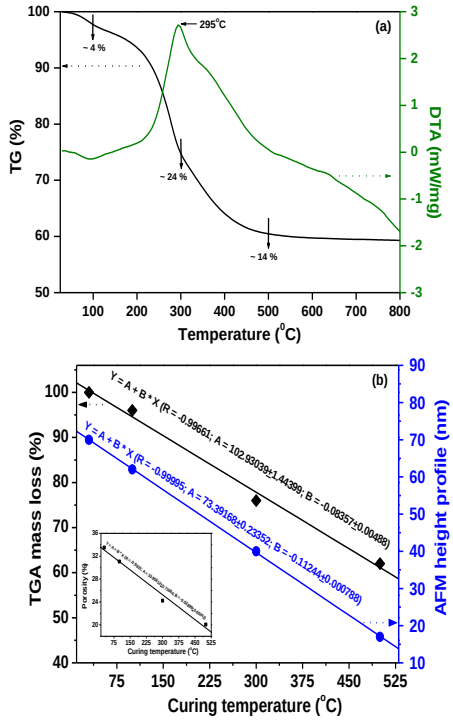

Fig.4. (a) TGA and DTA curves of the scratched off silica-titania gel film (STGF) deposited from the sol of viscosity, 1.98±0.5 cP. (b) Plots of TGA mass loss (%) of STGF and AFM height profile with curing temperature. Inset shows the change of calculated volume percent porosity as a function of curing temperature.

Fig. 4a shows the thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) curves of the scratched off gel film deposited from the sol of optimum viscosity 1.98 cP (aging time, 3h). It was noteworthy to mention that the sol generated perfectly surface patterned structure as evidenced from the AFM images (Fig. 3) of the thermally cured films. We cured the as-prepared gel film at 100^o, 300^o and 500^{o}C according to mass loss behaviour in TGA curve (Fig. 4a).The TGA curve shows the mass losses, ~4%, ~24% and ~14% at the curing temperatures, 100^o, 300^o and 500^{o}C respectively.

The low mass loss at 100^{o}C ascribed to removal of adsorbed water and alcohols (1-proponol, 2-proponol and 2-butanol) [21] used for sol preparation whereas the relatively high mass loss at 300^{o}C could be due to loss of certain extent of chemically bonded organics (mainly actylacetonate and alkoxide moieties). The presence of an intense exothermic peak appeared at 295^{o}C in the DTA curve could support the loss for chemically bonded organics. The major mass loss would consider the main pyrolysis reaction of organics occurred at ~300^{o}C. However, above 300^{o}C there was a gradual loss up to ~500^{o}C. The mass loss would be due to removal of carboneous materials through oxidation in air atmosphere during the measurement of TGA/DTA data. It should be mentioned that the formation of actylacetonate complex with the metal ion, especially Ti (IV) has already been confirmed and reported [14] in our previous work. Fig. 4b shows the mass loss (Fig. 4a) of silica-titania gel film from TGA measurement and the decrease of AFM height profile (Fig. 3b, d and f) of the patterned films on increasing curing temperature (30^o to 500^{o}C). In Fig. 4b, the decrease of height profile is found to be more linear than the thermal mass loss which might be related to the thermal decomposition behaviour of the organics (discussed earlier) present in the gel film. Further, the film density/refractive index would be related to film porosity. We already reported in our previous report [14] that the refractive index of the film increased with curing temperature. Hence, from the measured refractive index, it is possible to calculate the volume porosity, P (in vol %) of the silica-titania film using Lorentz-Lorenz (L-L) relationship [20],

$$1-(P/100) = (n^2-1)/(N^2-1) \times (N^2+2)/(n^2+2) \quad \text{-----} \quad (1),$$

Where 'n' is the observed RI of the film and 'N' corresponds to the RI of dense material [20]. Yasushi Murakami et al. [20] reported that the crystalline TiO_2 has a greater RI value 'than the amorphous TiO_2 and the TiO_2 in anatase and rutile form exhibited RI at 2.54 and 2.75 respectively. However, the amorphous dense titania film showed the value at 2.064. In the present work, the silica-titania film was amorphous in nature as evidenced from the XRD (inset, Fig. 5b). Thus, in this work, we considered the RI, 2.064 [22] for the amorphous dense titania film for porosity calculation by using the equation 1. The calculated porosity (in vol %) found to decrease from 33.52 to 20.05±0.5% on increasing curing

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temperature from 100° to 500°C respectively. However, the change (inset, Fig. 4b) of the calculated porosity as a function of film curing temperature was approximately linear in nature. Moreover, the decrease of film porosity found relatively rapid up to 300°C than at higher curing temperatures. Thus, the film cured at 500°C would not appear to be a fully densified amorphous film. Presence of surface patterns of films was distinctly visualized by FESEM images (Fig. 5). A uniform mesoscale surface pattern (appeared as periodic hills and valleys) over the non-patterned zone of the film was observed from the FESEM image (Fig. 5a) of the film cured at 300°C. However, the patterns found to change into separate beam-like structure when the film was cured at 500°C.

The appearance of film morphology (separate beam-like structure) might be due to different level of shrinkages resulted from unequal thicknesses of the hills and valleys. However, the periodicity (~1.5 μm) of the patterns in all the films approximately matched with the PDMS stamp used.

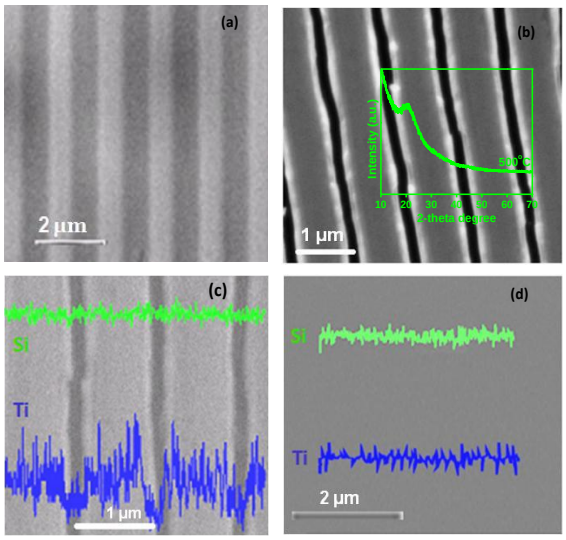

Fig. 5. FESEM images of patterned (a, b, c) and no patterned (d) films: (a) cured at 300°C; (b, c, d) cured at 500°C. Inset of (b), XRD curve of the film cured at 500°C. Insets, (c) and (d) indicate concentration mapping of Si and Ti from FESEM-EDS analysis.

A distinct change was seen in the concentration mapping (obtained from FESEM-EDS) of surface elements (particularly Si and Ti) present in patterned and non-patterned films cured at 300 and 500°C. Apparently, no change (Fig. 5 c, d) of Si concentration mapping was noticed in the patterned and non-patterned films. Moreover, the Ti concentration mapping (Fig. 5d) in non-patterned film found identical like Si mapping. However, approximately wave like periodic hills and valleys was observed in the Ti concentration mapping of the film cured at 500°C where the content of Ti differs due to the presence of separate beam-like structure. It should recall that the silica-titania film was deposited on pure silica glass. Thus, the Si concentration mapping on the patterned film would expect to remain same due to the silica glass substrate. This could further authenticate the existence of separate beam-like patterns in 500°C cured film. The beam-like morphology of the patterned film might be useful [14], [23] for some specific applications including waveguide sensors.

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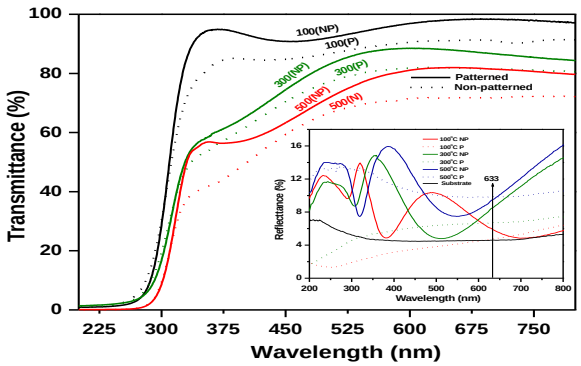

Fig. 6. UV-Vis transmission and specular reflection (inset) spectra of patterned (P) and non patterned (NP) films cured at 100°, 300° and 500°C.

This could further authenticate the existence of separate beam-like patterns in 500°C cured film. The beam-like morphology of the patterned film might be useful [14], [23] for some specific applications including waveguide sensors.UV-Vis transmission spectra of the patterned and non-patterned films are shown in Fig. 6. Percent transmission of patterned films was found always lower than that of nonpatterned films. The lower transmission might be considered due to scattering loss from the mesoscale surface patterns [24]. Also, comparatively low percent transmission was noticed in the film cured at higher temperatures while the lowest percent transmission was found in the film cured at

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500°C. This could be attributed to an enhancement of film refractive index (at 632.8 nm) which would induce a higher reflectivity. This proposition could approximately be justified from the observation of increased percent reflectance at 633 nm (inset, Fig. 6) on increasing curing temperature (identical to the change of refractive index [14] measured ellipsometrically). Moreover, the interference like wavy nature (WAN) was found in both the reflectance and transmittance spectra of the films but a very low value of amplitude of interference was found in the patterned films. The diminishing amplitude of WAN might be caused due to the presence of order mesoscale surface patterns [9].

V. CONCLUSION

A study was performed on soft lithography based surface patterned silica-titania thin film from the precursor sol of different viscosities. Defect free and high fidelity surface patterns could be achieved from an optimum sol viscosity of 1.98±0.5 cP. Shrinkage of pattern height as a function of curing temperature found dependent on thermo gravimetric weight loss of gel film as well as on volume percent porosity calculated from film refractive index. Presence of surface patterns also found to influence the percentage of UV-Vis transmission and reflection. This work would help on mesoscale surface patterning of other amorphous films of mixed oxides.

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AUTHOR'S PROFILE

Saswati Sarkar is working as Junior Research Fellow at CSIR-Central Glass and Ceramic Research Institute, Kolkata, India under the guidance of Dr. Sunirmal Jana, Principal Scientist for her PhD (Sci) degree. Nano metal/alloy coated patterned dielectric thin films for sensor applications is her present field of research. Ms Sarkar completed her M.Sc (Chemistry) from Nagpur University, India. She has published 4 research papers in SCI journals and 6 conference articles based on the fabrication of grating based optical waveguide sol-gel thin films by soft lithography and nanomaterials.

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Rimlee Deb Roy is a Senior Research Fellow at CSIR-Central Glass & Ceramic Research Institute and currently working towards the completion of her thesis. Having completed M.Sc (Electronics) from Jadavpur University in 2005 respectively joined CSIR-CGCRI in 2006 as a research fellow. She has worked in fiber lasers and amplifiers using rare earth doped fibers. She began her studies in planar waveguides under her supervisor Dr. S. K. Bhadra in 2008 and currently working toward the completion of her thesis. Her research interests include nanophotonics and plasmonics, integrated optics, soft patterning of surfaces.

Prasanta Kumar Biswas did his M. Sc. in 1977 from the Indian Institute of Technology (IIT), Kharagpur, India and obtained his Ph. D. (Sc.) degree in 1982 from Calcutta University, India. He did his dissertation work in the Indian Association for the Cultivation of Science (IACS), Kolkata, India. In 1984 Dr. Biswas joined CSIR-CGCRI and started work on the development of optical coatings by sol-gel processing, a technique based on soft chemistry. He has expertise on the development of dielectric / semiconductor thin films for optics and photonics having different functional properties such as high visible transmissivity with electrical conductivity and low thermal emissivity, QD based excitonic transitions, ferromagnetism in dilute magnetic semiconductor (DMS), and grating pattern. Moreover, he has also contributed to the development of foam/spongy semiconductor, a promising material as radar absorbing. He was involved with 20 national/ international projects leading 14 major projects during his tenure at CSIR-CGCRI. He has published >70 publications in SCI journals with citations >1000. He is the author/co-author of 3 monographs, 18 book chapters in edited Proc. vol, 45 contributory papers (abstracts of which have been published in Conference Proceedings) and also co-inventor of six Indian patents (three utilized). Presently he is the life member of different academic societies such as Materials Research Society of India (MRSI), Optical Society of India (OSI), Indian Association for the Cultivation of Science (IACS), Indian Ceramic Society (ICS), Indian Thermal Analysis Society (ITAS), but he was the regular member of American Chemical Society (ACS), American Ceramic Society (ACerS), Optical Society of America (OSA), Optical Society of Korea (OSK) and Society for Applied Spectroscopy (SAS) during his tenure at CSIR-CGCRI. Dr. Biswas is the recipient of Career Award (MRSI Medal) from MRSI (Material Research Society of India) in 2010 for his significant contribution in Materials Science. He has also been elected as a Fellow [F.A.Sc.T.] of West Bengal Academic Society of Science and Technology in 2010. Presently, Dr. Biswas is in the Physics Department of IITR, Roorkee, India as Honorary Fellow after the retirement from CSIR-CGCRI and acting as the adviser of different on-going projects giving thrust on the development of organic photovoltaics.

Shyamal Kumar Bhadra is a Chief Scientist at Fiber Optics and Photonic Division, CSIR-Central Glass and Ceramic Research Institute, Kolkata, India, where he is currently involved in the advanced areas of fiber optics. He has coordinated various national and international research projects and published a number of papers in international reputed journals. He is the holder of five national and international patents. He is the Honorary Editor of the Journal of Transaction Indian Ceramic Society. His research interests include fiber laser, photonic crystal fiber & super continuum generation, nonlinear optics & ultra fast pulse dynamics in guided medium, nonlinear solution dynamics in guided medium, plasmonics & metamaterials.

Sunirmal Jana did his M.Sc. (Chemistry) in 1991 from Kalyani University, Nadia, WB, and India and obtained his Ph. D. (Sc.) degree in 1998 from Jadavpur University, Kolkata, India. In December 1997, Dr. Jana joined CSIR-Central Glass and Ceramic Research Institute, Kolkata, India as Junior Scientist and presently working as Principal Scientist at the same Institute. His current areas research is dielectric / semiconductor oxides based sol-gel thin films and nanomaterials for optics and photonics applications (such as sensor, photo catalytic activity, radar absorbing etc.). Dr Jana has actively involved/coordinated various national and international research projects. At present, he published 27 research articles in SCI journals, 59 conference articles, 4 Book Chapters and 3 Indian patents. He is the life members of Materials Research Society of India, Indian Ceramic Society, and Indian Association for the Cultivation of Science and NCE Bengal & Jadavpur University, Kolkata, India. Dr. Jana availed Brain Pool Fellowship from Korean Federation of Science & Technology Societies (KOFST) at Korea Research Institute of Chemical Technology (KRICT), Daejeon, South Korea. He also worked as visiting scientists with foreign research Institutes (Slovenia and Portugal).