Synthesis And Characterization Of Thin Sol Gel Films Enhanced With UV Curing Technique

Mohd Hamzah Harun[1*], Norfazlinayati Othman[2], Nik Ghazali Nik Salleh[1], Zainal Abidin Talib[2], Mohd Sofian Alias[1], Mahathir Mohamed[1], Mek Zah Salleh[1] and Khairul Azhar Abdul Halim[1]

[1]*RadTech Malaysia, c/o Malaysian Nuclear Society, Radiation Processing Technology Division, Malaysian Nuclear Agency, Bangi, 43000, Kajang, Selangor, Malaysia*

[2]*Physics Department, Faculty of Science, Universiti Putra Malaysia,43400, Serdang, Selangor*

Corresponding author: hamzah@nm.gov.my

ABSTRACT

The sol gel films that were coated on glass microslide surface was prepared by facile sol-gel method and enhanced with UV curing. The UV curing is an alternative step instead of solely rely on heat treatment which is industrially unfriendly due to time consuming that is encountered during annealing process. The hydrophobicity phenomenon is governed by synergistic effect of roughness filler created by random distribution of nanosilica as well as low surface energy attributed by long chain fluoroalkylsilane. The contact angle obtained is above 150^o and the degree of transparency is in the range of 85-90% which was measured by UV-Vis spectroscopy. The chemical composition was evaluated by FTIR spectroscopy and it shows the presence of Si-O-C peak that indicates the fluoroalkylsilane peak is successfully attached to nanosilica thus created hydrophobic surface. The surface topography was analyzed with AFM that gives confirmation that uniform distribution of nanosilica on microslide surface.

ABSTRACT

Filem sol gel yang telah disalutkan pada permukaan slaid mikro gelas disediakan dengan kaedah sol gel yang mudah dan dipertingkatkan dengan pematangan UV. Pematangan UV merupakan satu kaedah alternatif selain daripada pergantungan sepenuhnya kepada rawatan haba yang kurang mesra industri disebabkan masa proses dijalani yang lebih lama semasa proses sepuhlindap. Fenomena kekalisan air ditentukan oleh kesan sinergistik pengisi kekasaran tercipta oleh taburan rawak nanosilika serta tenaga permukaan yang rendah disumbangkan oleh fluoroalkilsilana berantai panjang. Sudut sentuh diperoleh atas daripada 150^o dan darjah lutsinar diperoleh di antara 85-90% yang diukur dengan spektroskopi UV-Vis. Komposisi kimia diukur dengan spektroskopi FTIR dan ia menunjukkan kehadiran puncak Si-O-C yang membuktikan puncak fluoroalkilsilana berjaya tercangkuk dengan nanosilica lalu menghasilkan permukaan kalis air. Topografi permukaan sampel dianalisis dengan AFM dan ia memberikan pengesahan yang taburan nanosilika yang uniform pada permukaan slaid mikro.

Keywords: UV curing, sol gel, thin film, superhydrophobicity

INTRODUCTION

In the past few decades, thin sol gel hybrid coating films have become a focus in functional coating materials research owing to their unique and significant properties. These materials combine the flexibility of organic precursor with the hard and thermally stable of ceramic fillers (Kesmez, 2019). The sol gel process is relevant method to synthesize organic-inorganic hybrid films and overcome the difficulty of thermodynamic low affinity of its phase. Thus, the coating hybrid films that distinguish either hydrophobic, oleophobic, self-cleaning and antistatic characteristics can be utilized at various surface materials (Harun et al., 2018).

The research of thin sol gel films cured with ultraviolet radiation without requiring high thermal hardening has become the centre of attention (Cakir et al., 2018). It has various advantages include high chemical stability, less harmful to the environment, lower processing cost and higher rates of hardening. These type of coating films are applicable on different substrates such as paper, metal, wood and the most important is on plastic (Wang and Guo, 2019). It is because plastic is easily deformed when subjected to high annealing temperature.

In this study, the thin sol gel film was synthesized, coated on glass substrate and annealed with UV ray before being annealed using thermal oven. The performance for several characterizations of the sample was compared with the samples at different UV exposure.

MATERIALS AND METHOD

The chemicals used in this study were tetraethoxysilane, TEOS (Merck), fluroalkylsilane, FAS namely, heptadecafluorodecytrimethoxysilane (Fluorochem), silica, (Sigma), ethanol (J. Kollins), hydrochloric acid (Merck) and distilled water. All chemicals were used as received. The detail of the synthesis can be referred to the literature (Harun et al., 2018). For curing process, the sample was annealed with UV irradiator (IST, Germany) at the speed of 5 m/min and then to complete the annealing process it was further annealed in vacuum oven. Several characterizations were carried out and the results were compared with conventional annealed sample (without UV curing).

RESULTS AND DISCUSSION

FTIR study

The chemical composition of thin sol gel films for UV annealed and thermal annealed was investigated by ATR technique via transmission mode. As depicted in Figure 1, the absorption peak can be observed in the range of 4000-500 cm^{-1}. The most important peak is in the range of 750-800 cm^{-1} because it shows the successful fluorination of fluoroalkylsilane onto the silica nanoparticles. Both samples show the same trend and it indicates that either with UV annealed or thermally annealing alone is sufficient to undergo this reaction.

Water Contact Angle Study

Figure 2 shows the water contact angle (WCA) measurement for thin sol gel films at different UV exposure. For the samples at 1 and 5 passes, the calculated WCA value is in the region of hydrophobic. However, when the UV exposure was increased up to 10 passes, the WCA value is increased surpassing the super-hydrophobicity value in which it indicates that UV annealing is capable to enhance the water repellency of the thin sol gel films.

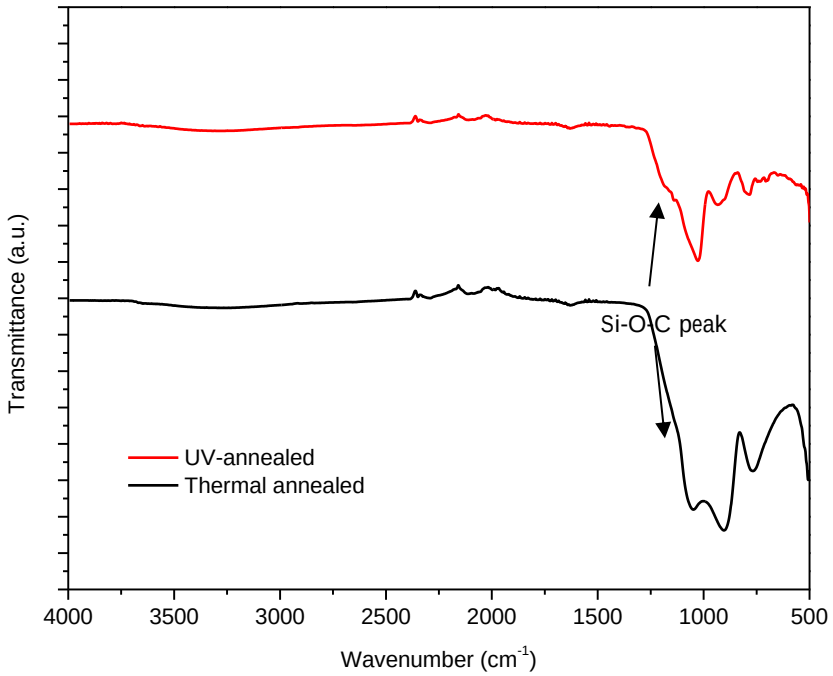


Figure 1: FTIR spectra for UV-annealed and thermal annealed thin sol gel films

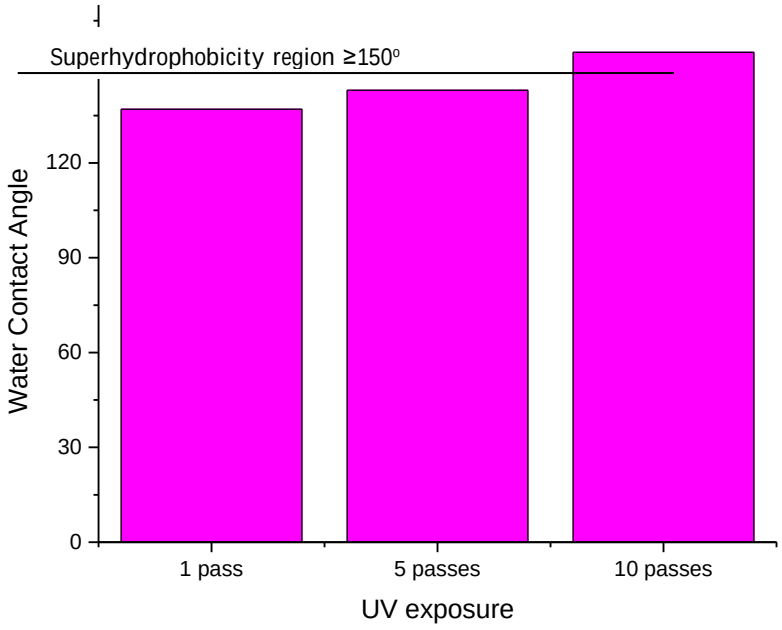


Figure 2: Water contact angle for the samples at different UV exposure

UV-Visible Spectroscopy

Optical transmission of thin sol gel films at different UV exposure was measured and the spectra can be referred at Figure 3. All samples show high transmittance degree which is above 80%. The transmittance of the bare glass is also measured as a control sample. The sample with 10 UV passes shows the lowest transparency which can be deduced to high crosslinking of silica in the sol gel network and tend to change to whitish color imitating the original color of silica. Transparency and super-hydrophobicity are two competitive properties. Therefore, the parameter to obtain both characteristics should be crucially check in order to retain both properties (Li and Amirfazli, 2005).

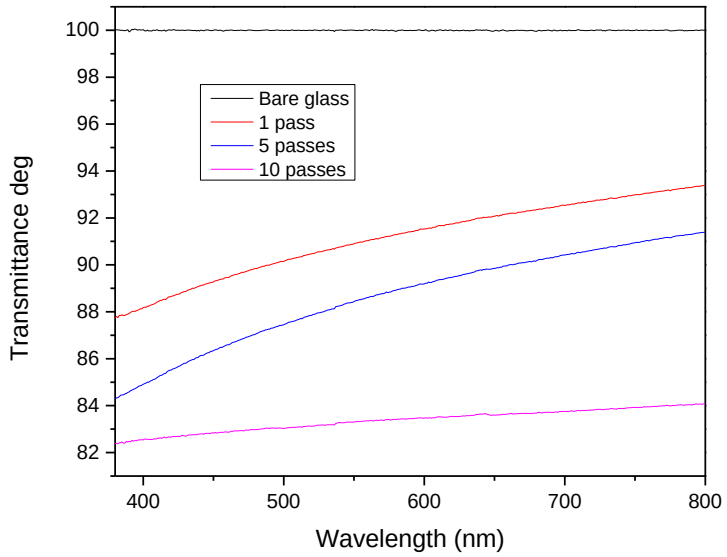
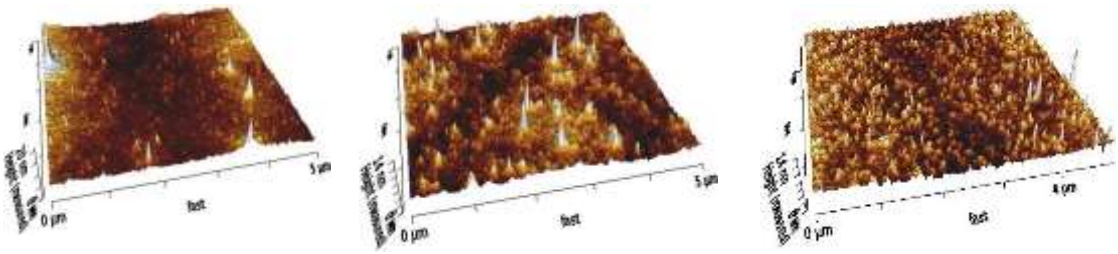


Figure 3: UV-Visible spectra for the samples at different UV exposure

Atomic Force Microscope

The surface topography of the samples at different UV exposure is shown in Figure 4. Three different images can be found. Sample (a) represent a single UV pass consist of low roughness in which only a few silica island can be found at the bottom of the image. On the other hand, sample (b) shows a better roughness which is almost 70% of the surface possess island peak and some of them are very sharp. From the roughness obtained, it shows that this sample possess a better hydrophobicity. Interesting topography can be found for the sample (c) that was undergone ten UV passes. The silica islands distributed evenly at almost every corner. This observation with outstanding roughness supports the contact angle in which it distinguishes the best contact angle which is above 150° (Mahadik et al., 2010). This indicates that it has reached superhydrophobicity level.



(a) 1 UV pass (b) 5 UV passes (c) 10 UV passes

Figure 4: AFM images for the samples at different UV exposure

CONCLUSION

The thin sol gel film enhanced with UV curing having water repellency ability is successfully obtained. The water repellency is greatly improved after UV exposure and it even reached superhydrophobicity value. It shows that UV ray is capable to improve the water repellency of the thin sol gel films and has better potential for commercialization purpose as compared to the treatment of thermal annealing alone.

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