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SPUTTER DEPOSITED TIN OXIDE THIN FILM PROPERTIES AND THEIR APPLICATION FOR RADIO WAVE PROPAGATION

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ABSTRACT

A major disadvantage of energy saving glass using tin oxide (SnO_2) thin film is that SnO_2 attenuates the mobile signal from passing through it. In order to improve such signal transmission, a frequency selective surface (FSS) structures are designed on SnO_2 thin film. In this paper, SnO_2 thin film with FSS structure was fabricated using combination of printed circuit board technology and reactive magnetron sputtering deposition. SnO_2 thin films were deposited at various rf discharge powers and O_2 flow ratios and their physical, electrical and optical properties were analyzed. Experimental results reveal that the transmission of microwave signal improved with the reduction of SnO_2 sheet resistance. The grain size of SnO_2 thin film significantly influences the value of sheet resistance. The SnO_2 sheet resistance was lower at larger grain size. Therefore, a clear correlation was found between SnO_2 thin films properties and transmission of microwave signal through SnO_2 coated glass with FSS structure. In addition, sufficient amount of O_2 gas was required to deposit transparent and functional SnO_2 thin film. Optimum condition to deposit SnO_2 thin film for energy saving glass with improved microwave transmission has been demonstrated.

Keywords: tin oxide thin film, frequency selective surface, magnetron sputter deposition.

INTRODUCTION

The use of low-emissivity or energy saving glass has become very popular in the modern day building design. This energy saving property is achieved by applying a thin metal oxide coating on one side of the glass. Materials used for the coating are indium tin oxide, zinc oxide doped with aluminum and tin oxide (Granqvist, 2012; J.-W. Lim, Na, & Kim, 2012; Parker, Antonopoulos, Simpson, & Kingdom, 1997; Tsakonas *et al.* 2001). Among those, tin oxide (SnO_2) is the cheapest and cost efficient material. Typically, SnO_2 is a transparent n-type semiconductor. SnO_2 thin film has low electrical resistance and high optical transparency in the visible range of the electromagnetic spectrum. Over past decades, SnO_2 thin film has been widely investigated owing to its good optical and electrical properties (Batzill & Diebold, 2005; Kasar, Deshpande, Gudage, Vyas, & Sharma, 2008). Energy saving glass with SnO_2 coated thin film has the ability of rejects the heat from outside at summer while kept warmer at the winter. This was done by blocking the infrared going into the building. However, the disadvantage is that the SnO_2 coated thin film attenuates the useful microwave frequencies through it. The microwave frequencies ranging from 0.8 – 2.2 GHz such as GSM mobile phones, GPS and 3G are very much critical in this issue. To overcome this problem, frequency selective surface (FSS) has being introduced to improve the microwave transmission through the SnO_2 coated glass (Chen & Chou, 2012; Gustafsson, Karlsson, Rebelo, & Widenberg, 2006; Liu *et al.* 2014; Ullah, Habibi, & Kiani, 2011; Zhang, Ouslimani, Letestu, Le Bayon, & Darvil, 2012). The FSS structure etched on the SnO_2 coated glass is design to reflects the infrared signal while improve the transmission through it. The performance of microwave transmission depends on the FSS structures, electrical properties and physical properties of SnO_2 coated thin film (Costa, 2013; G. I. Kiani, Karlsson, Olsson, & Esselle, 2007; Sohail, Esselle, & Kiani, 2012). Although several works on the effect of indium tin oxide conductivity on the performance of FSS have been reported, the details of the physical thin film properties were not presented (Parker *et al.* 1997; Tsakonas *et al.* 2001). In addition, we found no experimental report on the properties of SnO_2 thin films for FSS applications. In many FSS modeling studies, the important parameters such as thin film conductivity and glass dielectric constant have been based on assumptions (Gustafsson *et al.* 2006; G. Kiani, Olsson, Karlsson, & Esselle, 2008).

SnO_2 thin films deposited using rf magnetron sputtering is the most attractive deposition technique from the industrial point of view. This is because of its high deposition rate, good reproducibility and applicable for large-scale deposition system. Depending on the deposition parameters and the chamber environments, different structured of SnO_2 will take shape and it will affect the properties of SnO_2 thin films. In present work, structural and electrical and properties of SnO_2 thin films deposited on glass substrates by 13.56 MHz radio frequency (rf)-magnetron sputtering have been investigated. The effect of O_2 flow ratio and sputtering rf discharge power on SnO_2 thin film properties were studied here. In addition, the microwave transmission properties through SnO_2 coated glass with FSS structure were analyzed. Particular attentions have been paid to

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investigate the correlation between the SnO_2 thin film properties and the microwave transmission through SnO_2 coated glass with FSS structures.

EXPERIMENTAL DETAILS

SnO_2 thin film deposition using rf magnetron sputtering

SnO_2 thin films were deposited by rf-magnetron sputtering on glass substrates using 99.99% purity SnO_2 ceramic target. The background vacuum chamber was evacuated to less than 1.0 x 10-5 Torr by turbo molecular pump and rotary vane pump. Prior to the deposition, glass substrates were cleaned ultrasonically using acetone for 5 minutes and rinse under de-ionized water and dry it using nitrogen gas. During deposition, high purity O_2 was used as a reactive gas while argon as a sputtering gas. They were injected into the chamber via mass flow controller. For all deposition steps, the rf sputtering discharge was turned on 10 minutes for pre-sputtering at chosen sputtering condition. This is to clean the target surface. The distance between target and substrate was 13 cm. The sputtering plasma was produced by 13.56 MHz rf-magnetron discharges with an auto-matching network.

Series of samples were prepared at rf sputtering powers ranging from 150 to 300 W. While the O_2 flow ratio were varied from 0% (0 sccm), 14% (4 sccm), 24% (8 sccm) and 39% (16 sccm). Argon flow rate and total gas pressure were fixed at 25 sccm and 8.25 mTorr, respectively. The deposition time was at 20 minutes for all samples. During deposition, the total gas pressure was controlled by butterfly valve in front of the turbo molecular pump. The deposition process was done at room temperature where no additional heating was introduced to the substrate holder.

SnO_2 thin film characterizations

Thickness of SnO_2 thin film was analyzed by Alpha Step IQ Surface Profiler. Surface morphology of the films was characterized by field emission - scanning electron microscope (FE-SEM, JEOL, JSM-7600F Series). A probe voltage and current of 5 kV and 7 mA, respectively were used when analyzed using FESEM. Magnification of 100,000 was used to evaluate the changes of the grain size with different deposition parameters. The electrical properties of SnO_2 thin film was measured using Keithley 2400 current-voltage (I-V) measurement setup. Two probes system being used to measure the SnO_2 thin film resistivity between the gap of probes at 1 mm. The sheet resistance was evaluated from the I-V curve results and the resistivity was evaluated from the sheet resistance and thin film thickness.

FSS structure fabrication and evaluation of microwave transmission through SnO_2 thin film coated glass

FSS structure was fabricated on sample glass using simple printed circuit board technique. The combination of cross-dipoles and circle shapes were used as shown at lower inset of Figure 1. The length and width of cross-dipole were 44 mm and 4 mm, respectively. The diameter of circle at the center was 10 mm in diameter. The combination structure was choose because of our previous simulation works showing that this structure meets the criterion for optimum transmission at GSM mobile signal frequency range. The structures were created using laminated film before the SnO_2 deposition using rf magnetron sputtering. After the deposition, the structures were then remove using sodium hydroxide. Using this simple technique, we fabricated FSS structures on SnO_2 coated glass. Size of the glass used was 10 cm × 10 cm and thickness was 0.6 cm. The SnO_2 thin films were fabricated at various parameters as in section 2.1. Then the microwave transmission through various SnO_2 coated glasses with FSS structures were evaluated.

The microwave transmission measurement setup is shown in Figure-1. Hyperlog 7060, log-periodic antennas from Aeriona company having a frequency range of 800 MHz to 2.4 GHz were used for transmission measurements. The antennas were connected to network analyzer (Rohde & Schwarz ZVB4) through 50Ω standard cables. The measurement was carried out in an anechoic chamber to prevent reflection of microwave signal. An aluminum sheet was also used to cover the transmitter antenna from unnecessary leakage and to make sure that the microwave signal pass across the glass window shown in Figure-1. The SnO_2 coated glass with FSS structure was placed at the center window of steel plate with a size of 50 cm × 50 cm. Thickness of the metal sheet was 3 cm. Distance between the two antennas was 12 cm.

RESULTS AND DISCUSSION

Surface morphologies of SnO_2 thin films

SnO_2 thin films were deposited at various rf discharge powers and O_2 flow ratios. In general, the thickness and deposition rate increased monotonically with rf discharge power. This is consistent with the fact that increasing sputtering power results in an increase in current and bias-voltage of the target, which attributes to higher sputtering yield and deposition rate. On the other hand, the thickness and deposition rate of SnO_2 thin film decreases with the O_2 flow ratio. The main reason of this phenomena is due to the addition of electronegative O_2 that is highly affinitive to electrons in the discharge. Thus it reduces the plasma density and the volume of bombarding argon ions. Therefore, increasing the O_2 flow ratio results in more negative O ions and fewer argon ions.

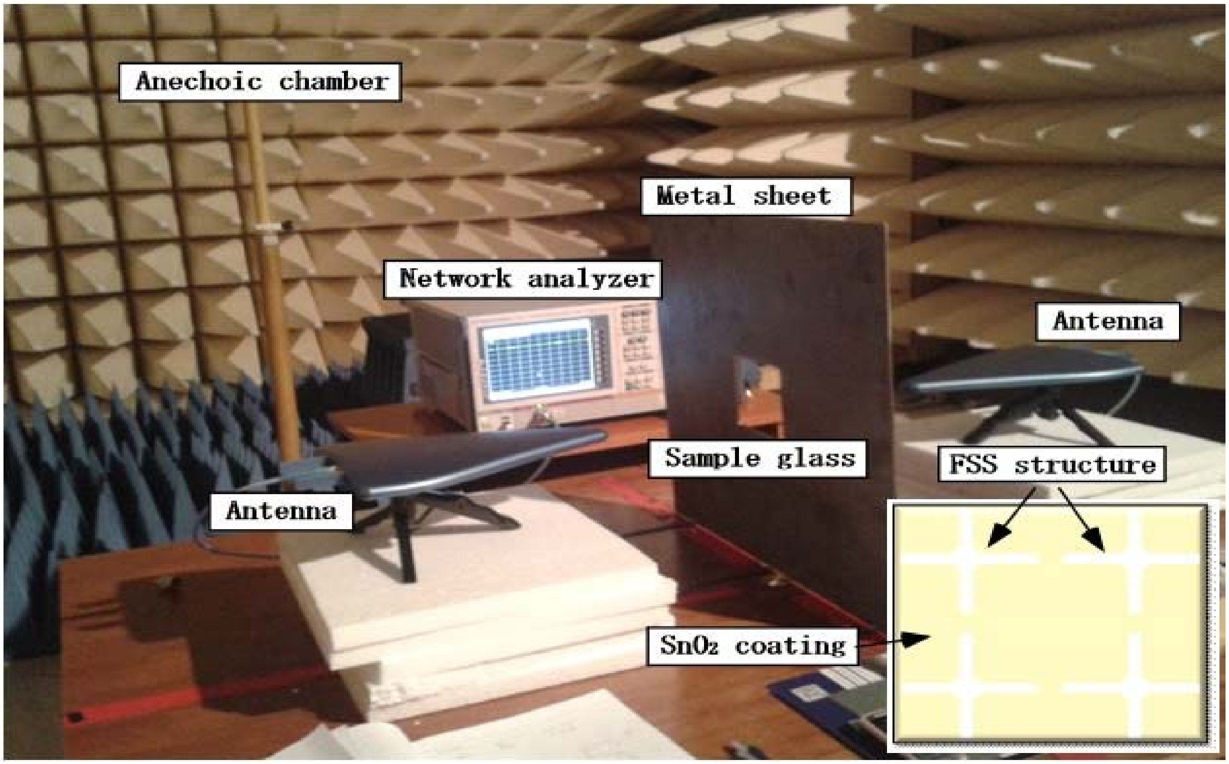

Figure-1. Photograph showing the microwave transmission experimental setup using network analyzer and log-periodic antennas in anechoic chamber. SnO_2 coated glass with FSS is placed at the center of metal sheet. Design of FSS structure on glass substrate shown at lower inset.

Figure-2 shows the FE-SEM images of as-deposited SnO_2 thin film at various sputtering discharge powers. The O_2 flow ratio was fixed at 24%, which is 8 sccm. From the surface images, the grains in all thin films are regular in shape, indicating a good crystallinity on the thin film. The sputtering discharge power strongly influences the grain size. Figure 2(a) and 2(b) clearly show that the grain size of SnO_2 thin film was smaller than 100 nm. However, at 300 W of rf discharge power, the grain size of SnO_2 thin film was larger than 100 nm as shown in Figure 2(d). These images indicate that sputtering ion energy could increase the grain size of SnO_2 thin film deposited under energetic condition. Note that at high sputtering discharge power, deposition rate improved and thus increase the nucleation rate. However, if the deposition rate was too fast, the film compactness become poor and it will increase defect and then obtain grain with many pores defect. It have been also confirmed with atomic force microscope analysis that as the sputtering discharge power increased, the grain size become larger and the surface roughness increased. Similarly, the rough surface of SnO_2 thin film may be due to the ion bombardment effect at high discharge power. As mentioned, at high discharge power, energetic argon ions increased so thus it will bombard and alter the SnO_2 thin film surface. This phenomena also known as re-sputtering (Vozniy, Duday, Lejars, & Wirtz, 2011).

Figure-3 shows the FE-SEM images of as-deposited SnO_2 thin film at various O_2 flow ratios. The grain size of SnO_2 thin film decreased when the O_2 flow ratio increased. At 0%, the grain size was approximately 100 nm. When the O_2 flow ratio increased to 39%, the grain size was approximately 10-20 nm. It was also found that the SnO_2 surface roughness decreased with the O_2 flow ratio. Weak sputtering plasma at high concentration of O_2 had reduce the density of energetic argon ions and results in decrease of the thin film grain size and surface roughness.

Sheet resistance of SnO_2 thin films

As mentioned, sheet resistance of coated thin film is very important for microwave transmission applications. High conductivity and low sheet resistance are required for energy saving glass applications. The sheet resistance of SnO_2 can be altered by modifying the micro- and nano-structure of SnO_2 grain, roughness and crystality (Khan, Mehmood, Rana, & Bhatti, 2009; Wang, Li, & Yang, 2006). The crystality was confirmed by XRD pattern of as-deposited SnO_2 thin films where strong peak at (101) and (110) plane was observed. We found no significant change on XRD peak as a function of rf discharge power. Only the (101) peak decreased when the O_2 flow ratio increased. However, as will be shown later, the transmission of microwave signal was not affected by the reduction peak of SnO_2 plane.

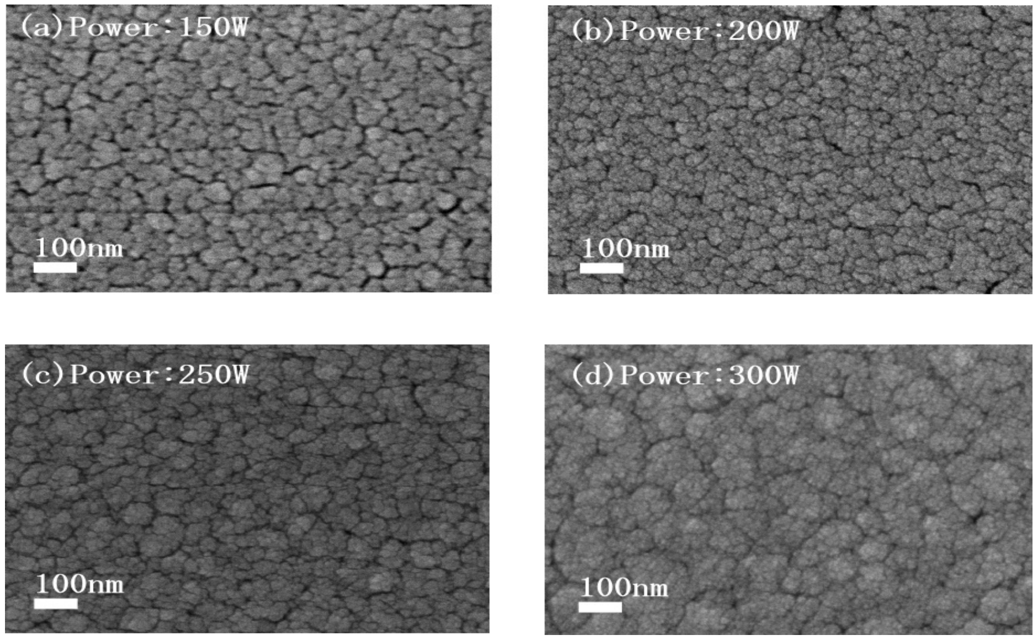

Figure-2. FE-SEM image of SnO_2 thin film surface deposited at various discharge powers.

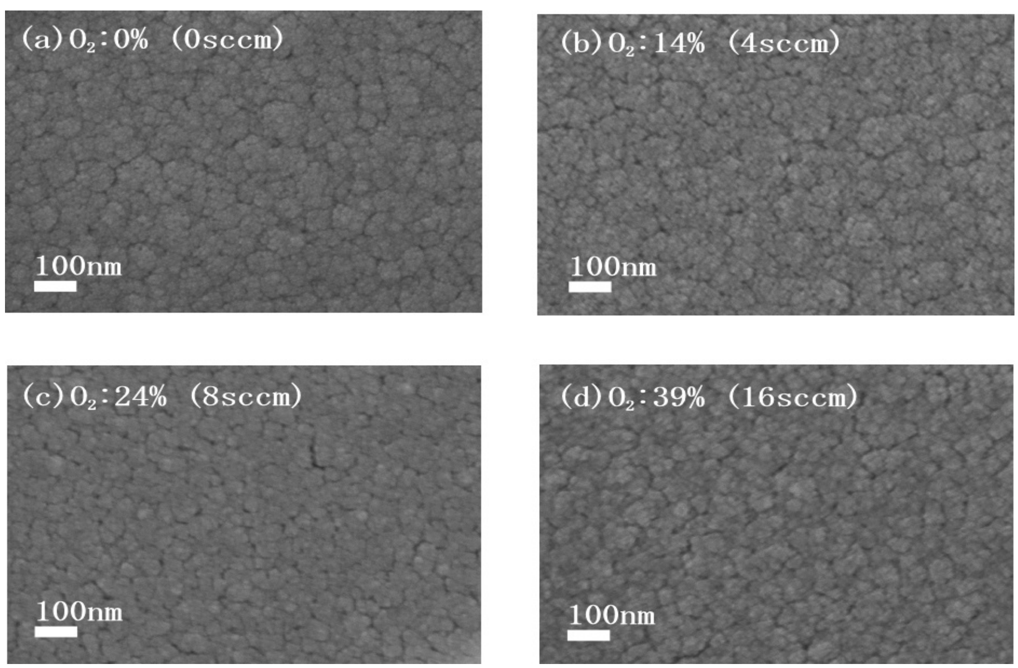

Figure-3. FE-SEM image of SnO_2 thin film surface deposited at various O_2 flow ratios.

Figure-4 shows the relationship between sheet resistance and resistivity of SnO_2 thin films as a function of rf discharge power and O_2 flow ratio. As shown in Figure-4(a), the sheet resistance of SnO_2 is at the range of 10~20 ohm/square. This is consistent with the value used by researchers in their FSS modeling analysis (Parker *et al*. 1997). Figure-4(a) shows that the sheet resistance of SnO_2 thin film decreased with rf discharge power. This may be due to leakage current in between the crack of larger grain size at higher rf discharge power. However, since the thickness and deposition rate increased with the rf discharge power, the resistivity of SnO_2 thin film was almost the same, as shown in Figure-4(a).

On the other hand, Figure-4(b) shows the sheet resistance and resistivity of SnO_2 thin films as a function of O_2 flow ratio. The sheet resistance increased with the O_2 flow ratio. This is common when the O_2 content was increased in the metal oxide thin films. By comparing FE-

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SEM images in Figure-3 and sheet resistance in Figure-4(b), we clearly understood that sheet resistance increased when the grain size of SnO_2 decreased.

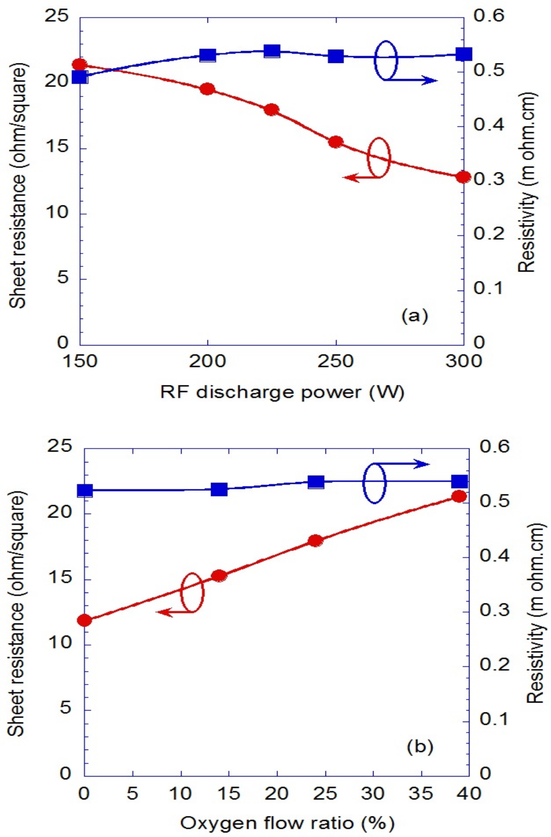

Figure-4. The relationships between sheet resistance and resistivity as a function of (a) rf power and (b) O2 flow ratio.

Microwave transmission through SnO_2 thin film with FSS structures

Network analysis measurement using two antennas was carried out to investigate the performance of microwave transmission through SnO_2 thin film coated glass with FSS structure. Figure-5(a) shows the transmission respond of microwave signal through glass coated with SnO_2 thin film. The SnO_2 thin film was sputter deposited at various rf discharge powers. The O_2 flow ratio was 24%. For comparison, the transmission through an uncoated glass is also shown in Figure-5(a). Our particular attention is the microwave frequency at 0.9 GHz, which is for GSM mobile signal applications. As shown in Figure-5(a), the transmission loss at range 0.8 to 1.2 GHz decreased for the sample prepared at higher rf discharge powers. This result is consistent with the sheet resistance result since the sheet resistance decreased with the rf discharge power. The sheet resistance decreased due to larger grain size of SnO_2 thin film at higher rf discharge power. Therefore, good correlation between microwave transmission, sheet resistance and grain size was obtained. High conductivity of SnO_2 thin film is essential in order to improve the microwave transmission through energy saving glass with FSS structure (Gustafsson *et al.* 2006; Yu, Xu, Liu, & Gao, 2014).

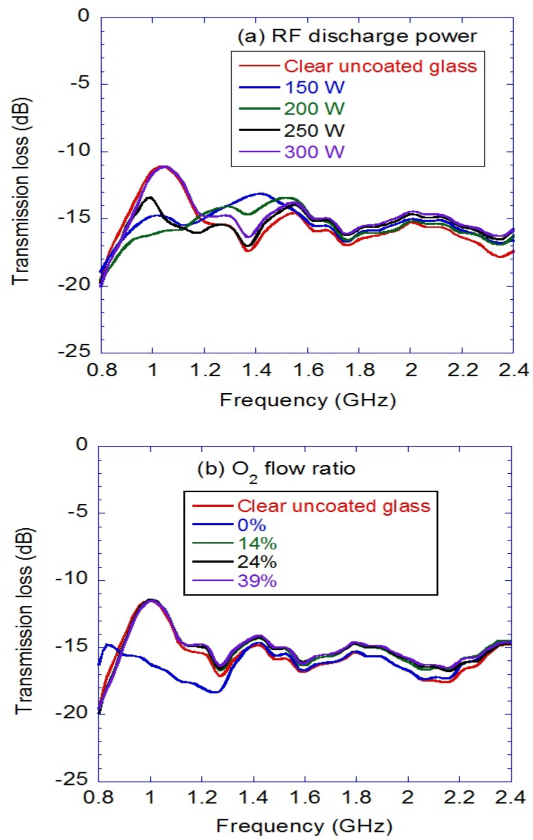

Figure-5. Transmission respond of rf signal through SnO_2 coated glass with FSS structure. SnO_2 thin films were coated at various (a) rf powers and (b) O_2 flow ratios.

Figure-5(a) shows that SnO_2 thin film deposited at lower rf discharge power has a peak at 1.4 GHz. Although this peak is not preferably for mobile signal application, this result indicates that one could also minorly alter the peak position from the deposition parameters and grain size. In our previous work, we had simulated using computer simulated technology software showing that the peak of microwave transmission was strongly influenced by the shape of the FSS structure (H. S. Lim *et al.* 2014). Figure-5(b) shows the transmission respond of microwave signal through glass coated with SnO_2 thin film deposited at various O_2 flow ratios. The rf discharge power was fixed at 225 W. The transmission respond shows that only the sample prepared at 0% of O_2 flow ratio had low transmission across the range of GSM mobile signal. The result is consistent with the UV-Vis analysis since the SnO_2 thin film prepared at 0% of O_2 flow ratio was not transparent and not applicable for energy saving glass applications. Therefore, sufficient amount of O_2 gas need to be introduced into the sputtering plasma in order to deposit stoichiometric and transparent SnO_2 thin film.

Finally, we had also did a simple test on temperature dependent through the SnO_2 coated glass with and without the FSS structures. This is to make sure that the uncoated FSS area will not weaken the infrared blocking property of SnO_2 coated glass. We found that the FSS structures will not affect the thermal insulation properties of SnO_2 coated glass.

CONCLUSIONS

In conclusion, we had successfully fabricated SnO_2 thin film with FSS structure on glass substrate using combination of printed circuit board technology and magnetron sputtering deposition employing SnO_2 ceramic target. Comparative study on the physical properties of SnO_2 thin films on sheet resistance and microwave transmission have been demonstrated. Large grain size with low sheet resistance are essential to transmit the GSM signal at 0.9 GHz effectively. In addition, sufficient amount of O_2 gas is required to deposit transparent and functional SnO_2 thin film. Parameters used to deposit SnO_2 thin films in this experiment will be a guideline to develop and optimized SnO_2 thin film for energy saving glass applications with improved microwave transmission. To further investigate the microwave transmittance through SnO2 coated glass with FSS structures, theoretical simulation using current experimental results will be beneficial.

ACKNOWLEDGEMENTS

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