THE PREPARATION OF TIN OXIDE FILM HEAT MIRRORS BY A SOL GEL PROCESS

Jongjit Hirunlabh, Sarayuth Lorsrichandr, Krissanapong Kirtikara and Ralph.D. Pynn

Energy Technology Division
School of Energy and Materials
King Mongkut's University of Technology Thonburi
Bangmod, Thungkur, Bangkok 10140, Thailand

ABSTRACT

This paper describes the development of a process for the preparation of heat mirrors by a sol gel process, based on the coating of glass substrate with a thin film of the oxide. In Thailand, a sol gel process has been used for the preparation of fiber, and for the creation of multi layers for electronic device research. A Sol gel process for heat mirror preparation was developed by a committee of research workers at King Mongkut's University of Technology Thonburi (KMUTT). This research studied the effects of process parameters on the preparation of tin oxide films by the sol gel process, namely, (i) effect of viscosity of sol gel solution, (ii) effect of speed of withdrawal, (iii) effect of sintering temperature, and (iv) effect of doping tin oxide with fluorine.

1. INTRODUCTION

Transparent heat mirrors can be divided into different classes characterised by their application or construction. Considering applications, two groups exist, namely, (i) mirrors which transmit visible light and, in the ideal case, reflect over the total infrared heat spectrum (class 1) and (ii) mirrors transmissive to visible and near infrared light and reflective only beyond the near infrared transmission cut off (class 2). Class 1 contains all present applications related to energy conservation and personal protection and thus includes architectural glass coatings to reduce air conditioning requirements, light bulb envelopes, furnace windows, welder and laser goggles and astronaut helmets. Class 2 heat mirrors comprise solar heat collector covers and window coatings for the passive solar heating of buildings. Some of these applications are still in an early development stage.

Herve G. Floch and Jean-Jacques Priotton[1] reported sol gel optical coatings for laser design for advanced fusion experiments. For example a porous silica sol gel coating having a median optical index of 1.22 would require a thickness of approximately 218 nm for 1064 nm wavelength.

A. Maddalena, R. Dal Maschio, S. Dire and A. Raccanelli[2] studied electrical conductivity of tin oxide films prepared by sol gel process. Films could be doped by zirconium and titanium oxides and it was found that titanium oxide dopant have the highest conductivity.

Doughlass M. Mattox[3] demonstrated that SnO_2 doped In_2O_3 films can be obtained by baking sol gel derived coatings, found that In_2O_3 dopant having the highest sheet resistance. C.J.Brinker, G.C. Frye, A.J.Hurd and C.S. Ashley[4] studied fundamentals of sol gel dip coating and shown parameters relation.

2. EXPERIMENTAL PROCEDURE

2.1 Preparation of sol gel solution

200 g of $SnCl_2.2H_2O$ was dissolved in 200 ml of absolute ethanol. The solution was well stirred and underwent reflux distillation at 80°C, for 6 hours and ageing in an open vessel for about 5 days.

2.2 Coating technique

Coating of glass substrates by sol gel solution was done in the clean room. Glass substrates were immersed in sol gel solution by a withdrawal machine, where the withdrawal speed was controlled in the range of 10-210 cm/min.

2.3 Sintering process

After the coating process, samples were sintered in normal atmosphere at 400-600°C for 20 minutes.

3. RESULTS

3.1 Effect of viscosity of sol gel solution

The tin oxide film thickness depends on viscosity of the sol gel solution, which should vary in the range 1.4-5.0 mPa.s. The speed of withdrawal was fixed 12 cm/min. This experiment was carried out while room temperature was 30°C and relative humidity was about 70 %. The results are shown in Figures 3.1.1 to 3.1.3.

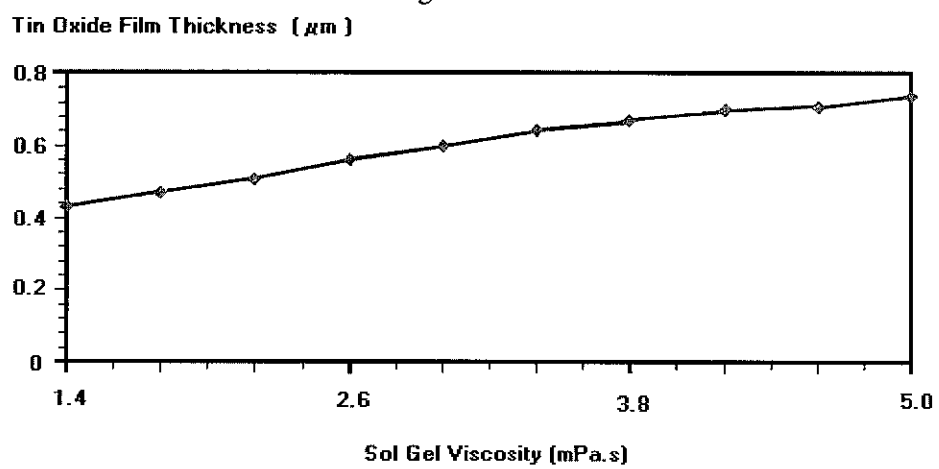

Figure 3.1.1. Tin oxide film thickness vs sol gel viscosity.

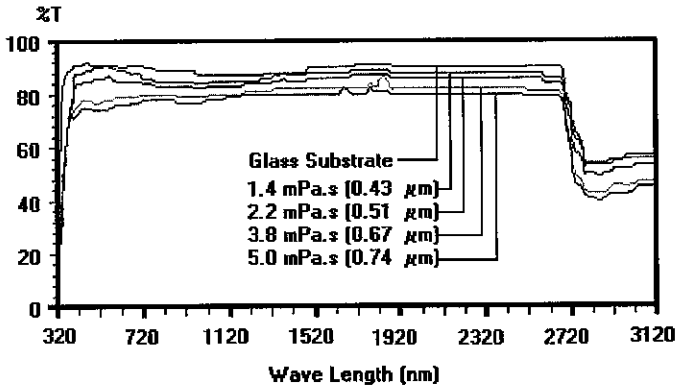

Figure 3.1.2 Light transmission in percent vs sol gel viscosity.

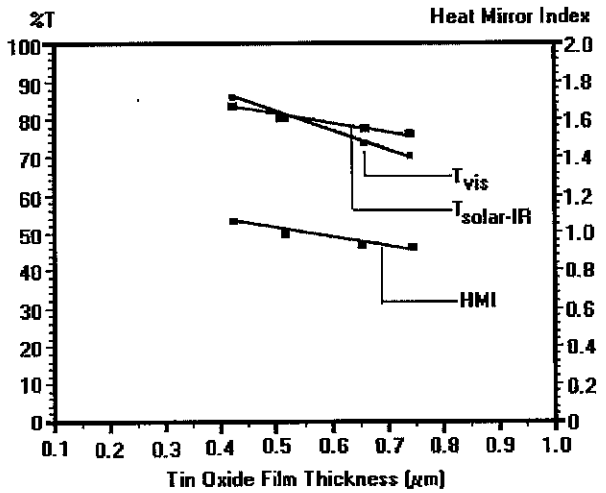

Figure 3.1.3 Average light transmission in percent and heat mirror index.

3.2 Effect of speed of withdrawal

The tin oxide film thickness is dependent on the speed of withdrawal. That was shown by varying the speed of withdrawal in the range of 10-210 cm/min with the viscosity of sol gel fixed at 5.0, 3.0 and 1.4 mPa.s. This experiment was done while the room temperature was 30°C and relative humidity was about 70%. The results are shown in Figures 3.2.1 to 3.2.7.

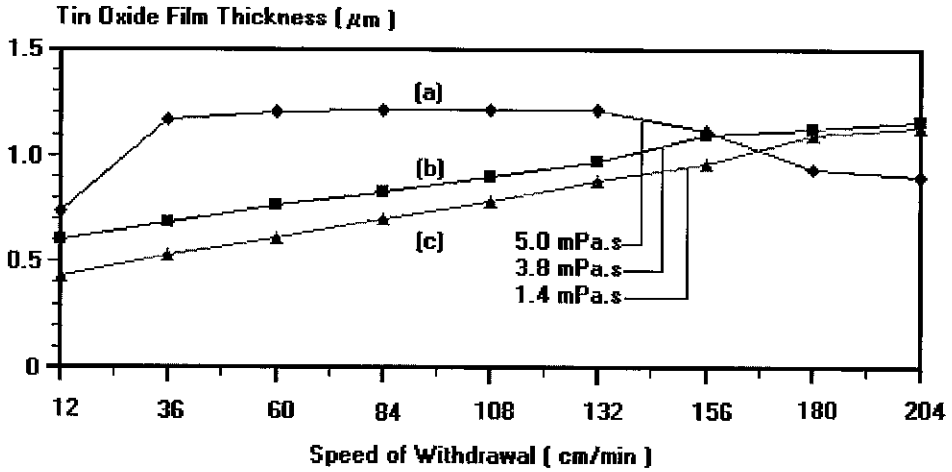

Figure 3.2.1 The speed of withdrawal vs tin oxide film thickness.

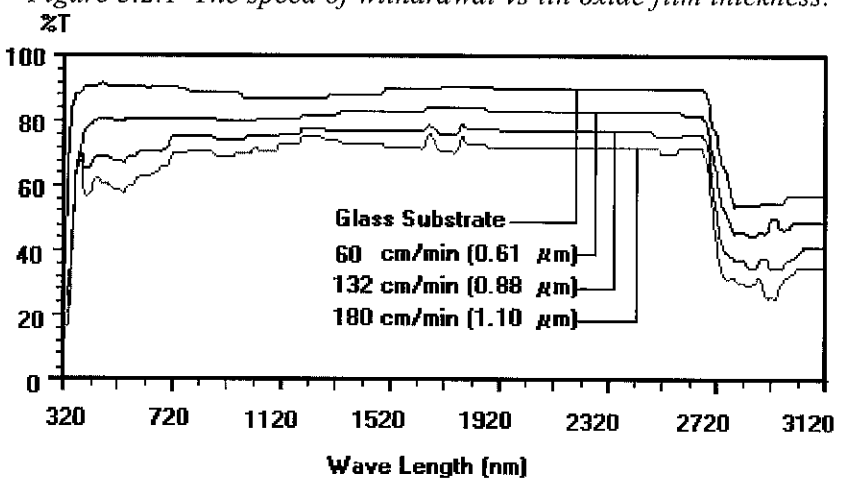

Figure 3.2.2 Light transmission in percent, sol gel viscosity is fixed 1.4 mPa.s.

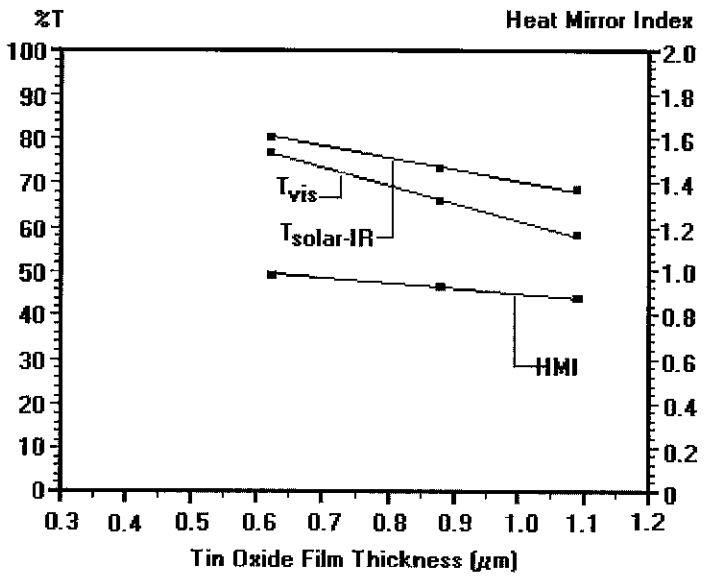

Figure 3.2.3 Average light transmission in percent and heat mirror index.

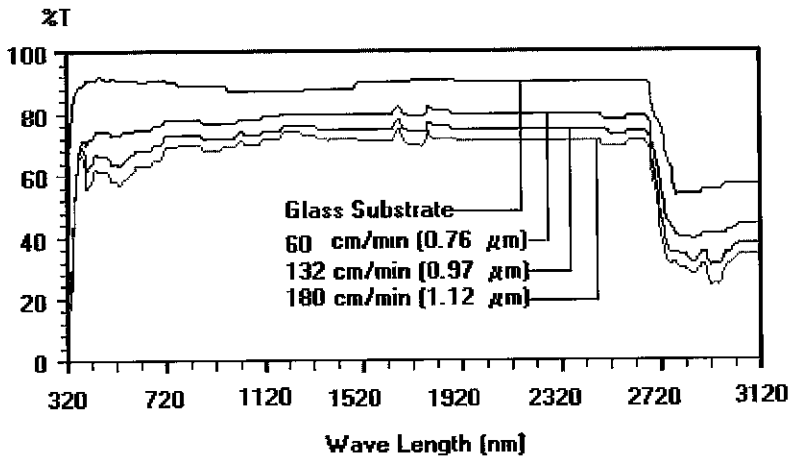

Figure 3.2.4 Light transmission in percent, sol gel viscosity is fixed 3.8 mPa.s.

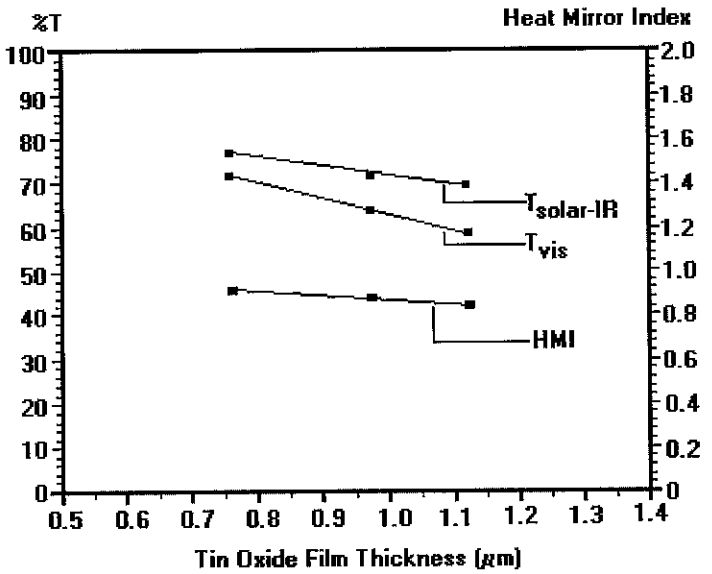

Figure 3.2.5 Average light transmission in percent and heat mirror index.

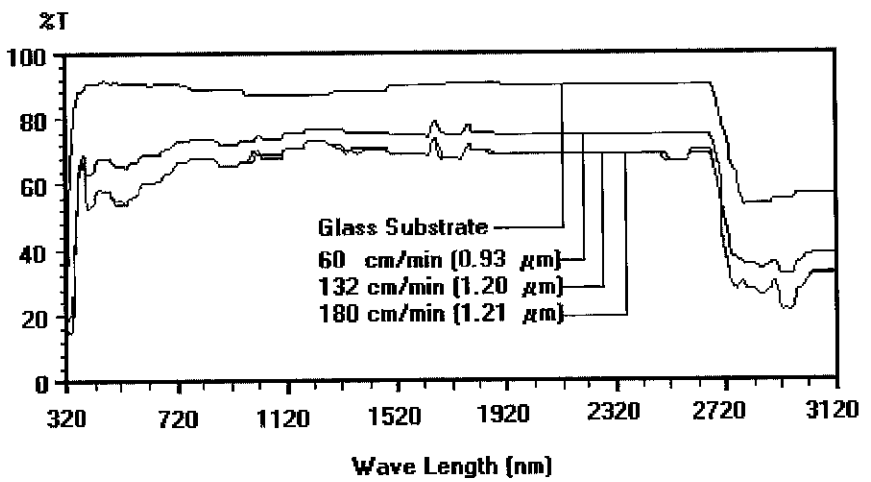

Figure 3.2.6 Light transmission in percent, sol gel viscosity is fixed 5.0 mPa.s.

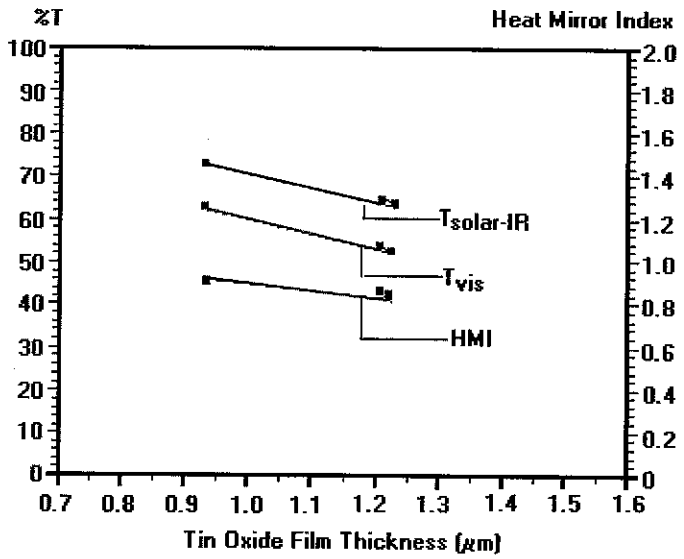

Figure 3.2.7 Average light transmission in percent and heat mirror index.

3.3 Effect of sintering temperature

We studied the effect of sintering temperature on tin oxide film structures, by varying sintering temperature in the range 400-600°C and non-sintering. This experiment was operated under normal atmospheric pressure and at the relative humidity about 70%, and using sol gel viscosity of 5.0 mPa.s, a speed of withdrawal fixed at 12 cm/ min, and tin oxide film thickness of 0.74 μm. The samples were tested by X-Ray diffraction. The results are shown in Figure 3.3.1.

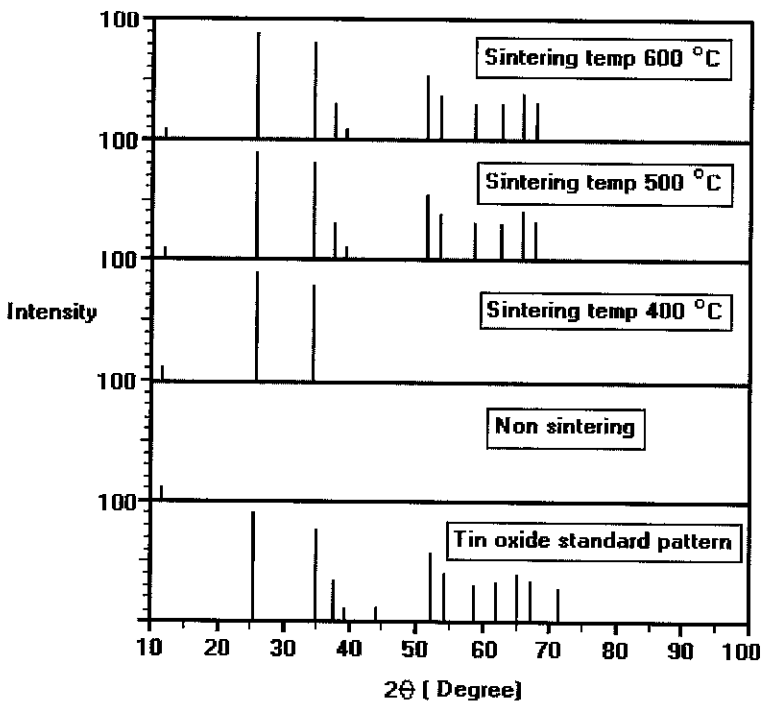

Figure 3.3.1 X-ray diffraction patterns for tin oxide films formed at different temperatures.

3.4 Effect of fluorine doping

Fluorine doping was studied as the last parameter. The heat mirror properties depend on fluorine doping. This process used NH_4F/tin-alkoxide additive to the sol gel solution, with ratios of F:Sn of 1:1, 1:10, 1:100 and 1:1 000 by atoms. The samples were tested for light transmission percent on wave length 320-3200 nm.

The results are shown in Figures 3.4.1 to 3.4.2.

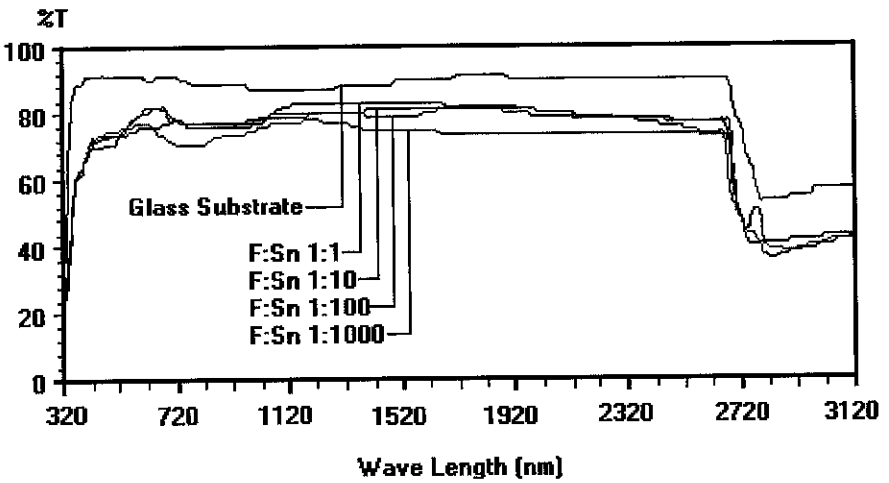

Figure 3.4.1 Light transmission in percent vs fluorine doping ratios.

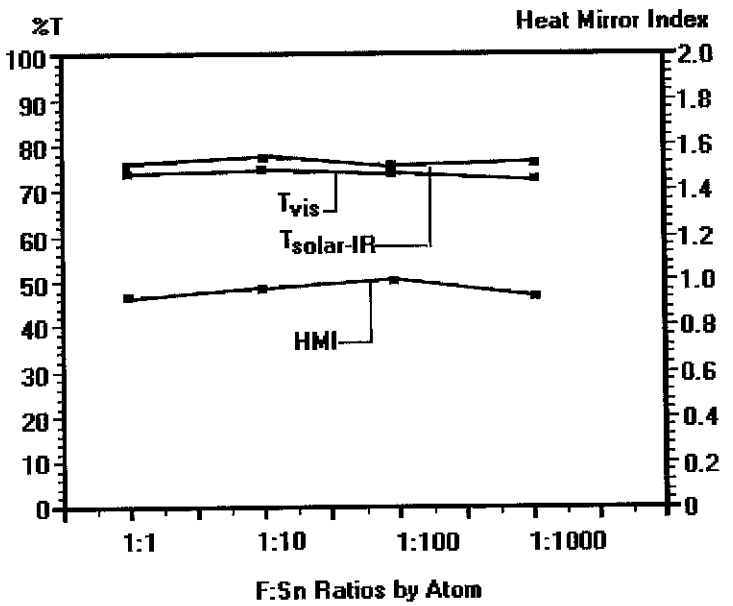

Figure 3.4.2 Average light transmission in percent and heat mirror index.

4. CONCLUSION

A sol gel process is selected for tin oxide heat mirror film preparation in this research work. Film thickness and optical properties are focused as points of interest in this work.

(1) Tin oxide film thickness can be controlled by varying the viscosity of sol gel solution and the speed of withdrawal. We found that thickness increased with increasing viscosity of sol gel solution and speed of withdrawal. A tin oxide films thickness in the range 0.43-0.74 μm could be prepared by varying viscosity of sol gel solution and thickness in the range 0.74-1.2 μm by varying speed of withdrawal while viscosity of sol gel solution are 5.0, 3.8 and 1.4 mPa.s.

(2) Light transmission of films in the range of 320-3200 nm were tested. We found that it decreased when tin oxide film thickness is increased. T_{VIS} is in the range 58.41%-86.68% and $T_{SOLAR\text{-}IR}$ is in the range 69.22%-85.67%.

(3) A heat mirror index in the wavelength of 320-3200 nm of a solar radiation air mass 1.5 as reference was measured. We found that it decreased while tin oxide film thickness increased. The heat mirrors indices in the range 0.84-1.01 are obtained.

(4) With direct fluorine doping using NH_4F, we found small variations in the optical properties.

(5) When sintering under a normal atmospheric pressure, we found that tin oxide film forming occurred in the range of 500-600°C.

(6) X-Ray diffraction yielded no detectable peaks of fluorine compounds in films. This indicates that either there was no fluorine in the films or fluorine compounds were present in negligible amount and could not be detected.

5. ACKNOWLEDGMENT

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6. REFERENCES

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