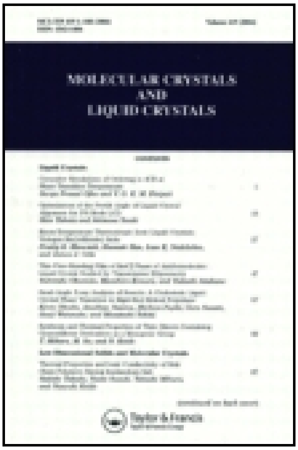

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ZrO_2-SiO_2 Coatings for Wavelength-Selective Reflection Filter

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Using ZrO_2 and SiO_2 sol as coating materials, a multilayer wavelength-selective high reflective (HR) coating was deposited by the way of sol-gel spinning technique. A new kind of ZrO_2 sol was prepared to lower the layer number and to increase the laser damage threshold of HR filter. The adding of Polyvinylpyrrolidone (PVP) was added into ZrO_2 sol to decrease the porosity and then to enhance the refractive index of ZrO_2layer as well as to increase the coating strength. At the peak position, the reflection more than 98% can be easily achieved with 21 layers. The laser damage threshold was as high as 19.9J/cm^2 (3 ns, 1064 nm).

Keywords: sol-gel; ZrO_2-SiO_2; reflection filter; laser damage threshold

INTRODUCTION

Sol-gel technique shows more and more advantages in the production of optical thin films and high quality ceramics. They are widely used to improve the property of optical elements and therefore considered to be one of the main techniques for the production of thin film optics[1]. Using metal alkoxides as starting materials, molecular networks of oxide can be produced by means of their hydrolysis and polycondensation.

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EXPERIMENTAL

Preparation of Sols

SiO_2 sol was prepared by mixing tetraethyl orthosilicate (TEOS), polyethylene glycol (PEG) and ethanol, and then slowly adding water; the reaction was catalyzed with $NH_3 \cdot H_2O$. The stirring of the sol continued for 4h. ZrO_2 sol was prepared by processing zirconium n-propoxide in ethanol. For ZrO_2 sol-gel process, the reaction control is usually difficult and therefore Diethylene glycol (DEG)[2] was used as a chelating agent to stabilize ZrO_2 sol. DEG and PVP were added to a stirred solution of zirconium n-propoxide and ethanol. After being stirred for 10 min, the mixture was hydrolyzed with a certain amount of water dissolved in ethanol.

Coating and Optical Measurement

The coatings were deposited on well-cleaned K9 glass discs by spin-coating technique, the thickness of which was controlled by both the viscosity and the spinning rate. The transmissions of the coatings were measured using UV-VIS spectrophotometer.

RESULTS AND DISCUSSION

Results

Figure 1 shows the thickness range of the two oxides produced by spin-coating technique at different spinning rates. The variation of optical thickness (nd) with spinning rate (ω) was found to follow the equation as nd = $a\,\omega^{-1/2} + b$, in which both a and b is constant related with sol characteristics, ambient temperature etc.[3].

Figure 2 shows that the transmission at the peak position decreased with the layer number. The reflectance decreased slowly when the layer number was over 13. More than 20 layers were needed if a transmission less than 2% was obtained. The UV-VIS spectrum of the $\lambda/4$ alternative stackings, 21-layer reflector is shown in Figure 3.

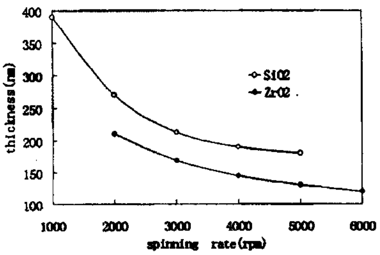

FIGURE 1 Effect of spinning rate on the coating thickness.

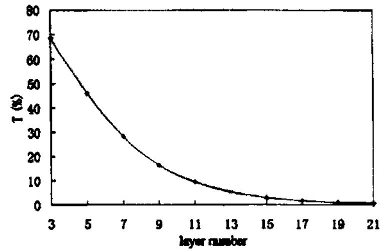

FIGURE 2 Change of the transmission with the number of layers.

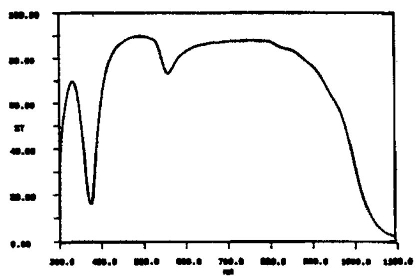

FIGURE 3 Transmission spectrum of a reflector with 21 layers.

Discussion

PVP was used not only to decrease the porosity and then to enhance the refractive index of ZrO_2 layers, but also to increase the coating strength. In addition, the addition of PVP increased the viscosity of sol. The thickness of thin film was related to spinning rate and sol viscosity, and in turn, the viscosity was dependent on its concentration. If the concentration was very high, the sol flew unstably on the spinning substrate, and the film surface would be very rough. On the other hand, if the concentration was too low, the lower spinning rate was required, which implied ineffective wetting of substrate and formation of a "trouble zone" round the boundary of the circular substrate. As a result, the concentration of the ZrO_2 sol was chosen to be 0.2 M and the SiO_2 sol 0.02 M, which led to homogeneous precipitate-free and craze-free coatings.

The addition of PEG into SiO_2 sol could inhibit the growth of particles and led to the cross-linking network of regular ring-like structure in short order[4], which increased the laser damage threshold of SiO_2 layers[5].

CONCLUSIONS

A wavelength-selective (1064 nm), 21-layer reflector was prepared by alternative stackings of alkoxide-derived SiO_2 and ZrO_2 sols. PVP was used to increase the refractive index and laser damage threshold of ZrO_2 layers. As an additive, PEG could change the structure of SiO_2 sol and then increased the laser damage threshold of SiO_2 layers. The transmission at reflective peak position of the filter went down to 1.9% and the laser damage threshold was as high as 19.9 J/cm^2 (3 ns, 1064 nm).

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