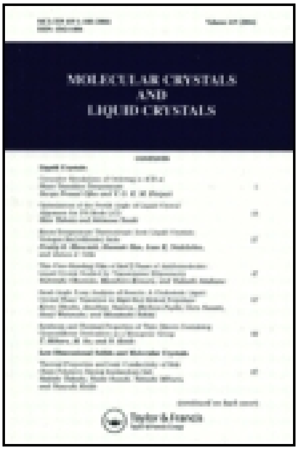

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Nano-Sized SiO_2 Sol-Gel for Structure-Controlled Optical Coatings

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Sol-gel process of TEOS in the presence of amphiphilic polyethylene oxide (AEO) was investigated by the particle size distribution (SDP) and transmission electron microscopy (TEM). The results indicated that AEO influenced seriously both hydrolysis and polycondensation of TEOS, leading to the formation of the network in short order. With such kind of SiO_2 sols, Two-layer SiO_2 anti-reflective (AR) coatings were prepared by spin-coating, which showed high laser damage threshold.

Keywords: Sol-gel; SiO_2 sol; AEO; AR Optical coatings

INTRODUCTION

The development of designed materials has been attracting much attention at molecular level. The careful control of the preparation parameters, especially the use of template molecules to control the structure and properties of the final products was shown to be very effective. So-produced (or "tailor-made") materials at a molecular or nanotechnological level play a key role in the development of high technology[1]. Here the chemical modification of SiO_2 network with the surfactant AEO was explored in order to produce structure-controlled anti-reflective coating.

EXPERIMENTAL PROCEDURE

TEOS (Tetraethoxysilane, analytical grade, distillated) and ethanol (analytical grade, distillated) were mixed in a flask by stirring for 20 min in a water bath (solution A); the basic ethanol solution (solution B) in which 6M ammonium hydroxide aqueous and a suitable amount of AEO were mixed with the same volume of ethanol was added slowly to solution A. After 240 min stirring the solution was moved to small polyethylene bottle and sealed. It was kept at different temperature in the oven until the gelation[2].

The AR coatings on BK7 glass substrates (35 mm diameter, 3mm thickness) were prepared by spin-coating at 2000 rpm. The shape and size of primary particles was traced by transmission electron microscope (H-600. Japan). The size distribution of the clusters/aggregates of the sol particles was estimated by SDP (Coulter N4 Plus. USA). The transmittances of coatings were measured by the UV/VIS/near-IR spectrophotometer (PC2501. USA).

RESULTS AND DISCUSSION

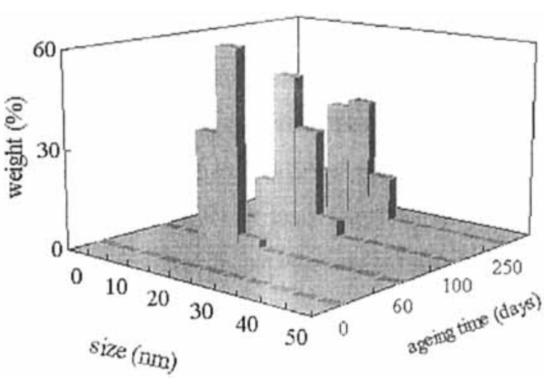

FIGURE 1 Relationship between particle size distribution and aging time in AEO-SiO_2 sols

Fig. 1 clearly shows that the particles grew with the aging time. Obviously, the size distribution of sol particles was in the range of 10-30nm and therefore the presence of AEO inhabited the growth of sol particles. But, the particle growth of SiO_2 sol was faster in speed and larger in size in the absence of AEO. Our these results also showed that the effects of AEO controlled the degree of hydrolysis and subsequent condensation, and leaded to uniform of particle size distribution of sol than that without AEO, because particles in surfaces were wrapped with (EO) hydrophilic groups through hydrogen bonding. This implied that AEO would play an important role in the stability of the sol-gel.

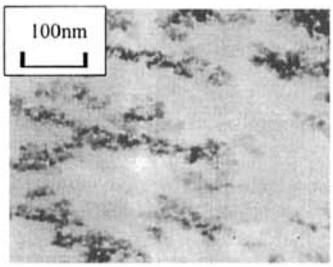

FIGURE 2 TEM micrograph of AEO_6-SiO_2 sols

TEOS-derived SiO_2 sols showed to be a highly cross-linked network with a typical of rod-like structure in the presence of AEO_6 (see Fig.2). TEM revealed the significant differences in both network structure and particle size distribution of the sols. With AEO_6, the sols appeared the dense rod-like networks with a uniform particle size distribution (10-20nm in diameter), and in the presence of AEO_3 or AEO_9, the sol system also showed rod-like networks consisted of sol particles (10-20nm in diameters). However, without AEO, the sols formed cross-linked network in the presence of small and disordered ring-like with sol particles (20-30 nm diameter) and the rod-like structure was not observed in the sol solution, which led to an irregular size distribution of sol cluster in the same aging conditions. It is obvious from fig.2 that SiO_2 sol system was consisted of ordered in short scale, uniform distribution of three-dimension network due to hydrolysis-polycondensation controlled and growth of particles limited in the presence of AEO.

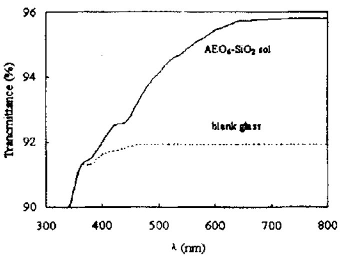

FIGURE 3 Transmittances of the coating by AEO_6-SiO_2 sol

By spin-coating method, the homogeneous AEO-SiO_2 sol-gel coatings were prepared and their optical properties determined (see Fig.3). For two-layer AEO-SiO_2 single-coatings, transmission almost reached 96% and the coatings with AEO-SiO_2 sol displayed a better laser induced damage resistance than those prepared without AEO.

CONCLUSION

The introduction of AEO into SiO_2 sols led to the formation of the secondary particles with rod-like structure in short-order, which significantly influenced the transmission of SiO_2 AR coatings. The interaction between AEO and sols showed to be complicated very much, and its nature is still under investigation.

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