



TITLE:

Preparation of Ag/TiO Composite Films by the Sol-Gel Method (Commemoration Issue Dedicated to Professor Sumio Sakka On the Occasion of His Retirement)

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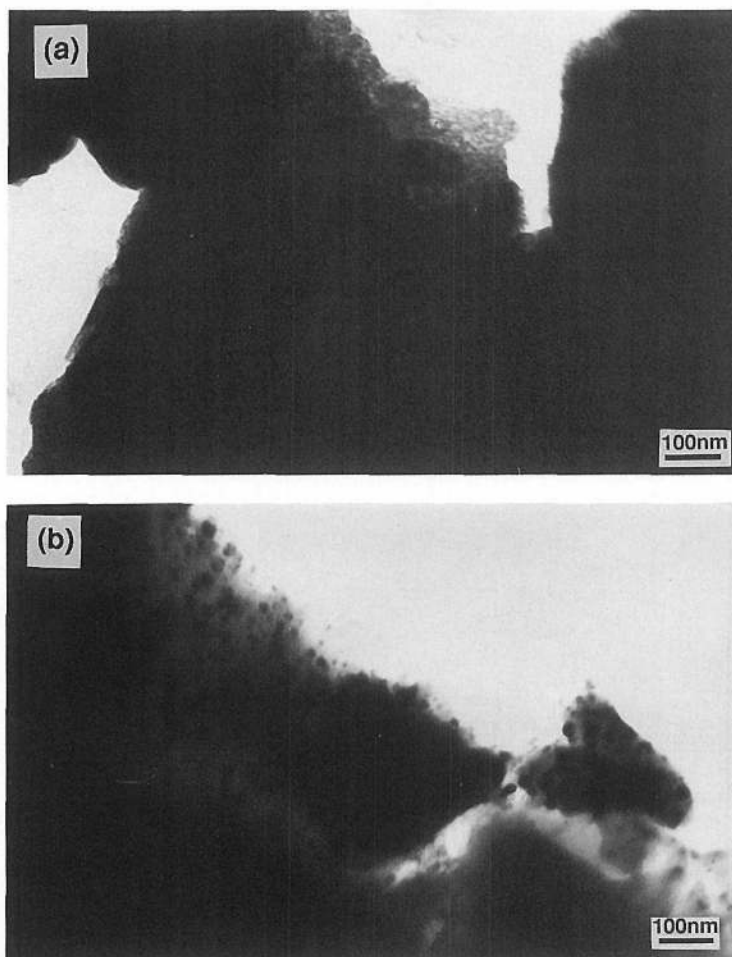


Fig. 10. TEM pictures of the gel coatings films before (a) and after (b) formalin vapor exposure.

exposed to formalin vapor (Fig. 10(a)).

Fig. 11 shows TEM pictures of the samples heated at 600°C. Fig. 11(a) and (b) show TEM pictures of the samples prepared without and with formalin vapor exposure, respectively. The sample shown in Fig. 11(c) was prepared by heating the formalin vapor exposed gel film at 600°C for 30 min with 5 times repeated coatings. Namely, the samples shown in Fig. 11(a), (b) and (c) correspond to those whose optical absorption spectra have been shown in Fig. 7(d), Fig. 8(d) and Fig. 9, respectively. Ag metal particles of 5~25 nm were observed in these samples. Negligible difference was found between the samples prepared with and without formalin vapor exposure.

Fig. 12 shows TEM pictures of the samples heated at 800°C without and with formalin vapor exposure. Ag particles of 13~41 nm and 13~45 nm were observed in the samples prepared without and with the formalin vapor exposure, respectively. It should be noted that both Ag particles and TiO₂ crystals are larger in size in the samples heated at 800°C than those at 600°C.

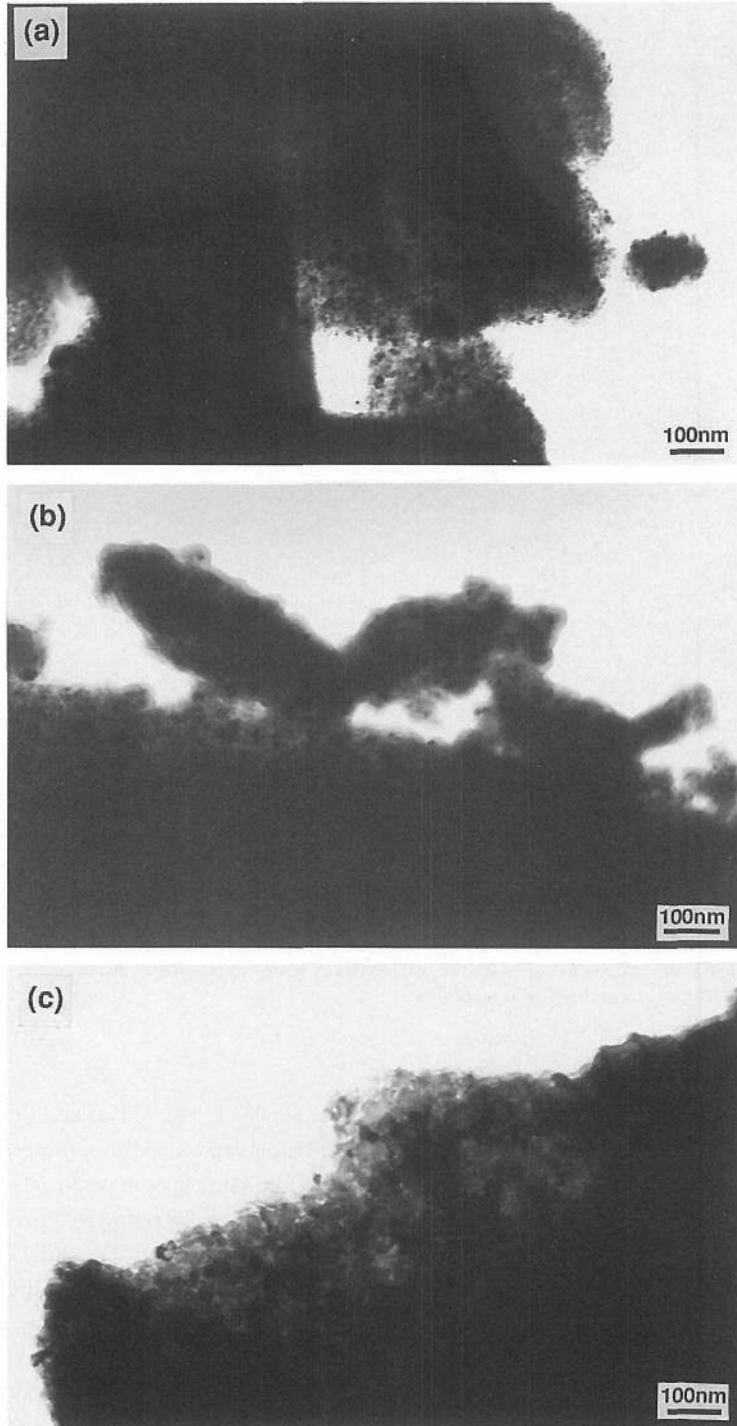


Fig. 11. TEM pictures of the coating films heat-treated at 600°C. (a) Without formalin vapor exposure. (b) With formalin vapor exposure. (c) Corresponding to the sample shown in Fig. 9.

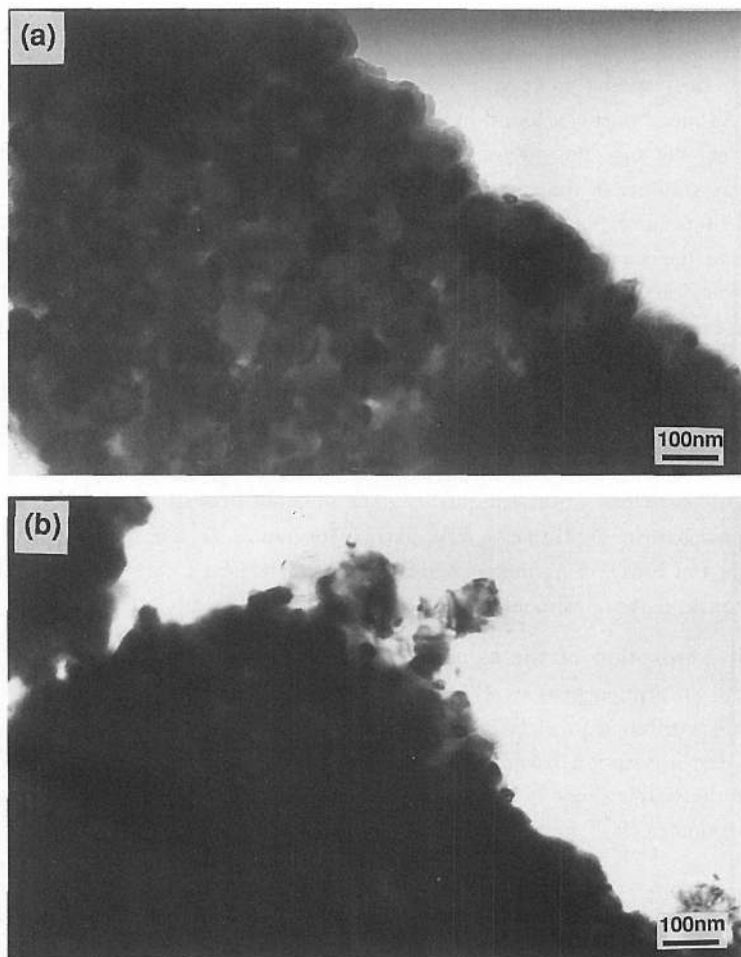


Fig. 12. TEM pictures of the coating films heat-treated at 800°C. (a) Without formalin vapor exposure. (b) With formalin vapor exposure.

4. DISCUSSION

4.1 Formation of Ag Metal Particles in the Coating Films

Exposure to formalin vapor caused reduction of Ag⁺ ions and formation of nanometer-sized Ag metal particles in the gel films. Considering the high concentration of Ag⁺ ions in the starting solution, 0.034 mol/l, formation of nanometer-sized Ag metal particles cannot be expected if reduction is carried out in solutions; instead aggregation of Ag particles would occur, and formation of much larger Ag particles and precipitation would take place in solutions. It is thought that Ag⁺ ions trapped in the titania gel pores were reduced and Ag metal particles were formed within the gel pores, where migration of Ag ions, atoms or particles is much more limited than in solutions.

It has been found that temperatures as high as 600°C were needed for formation of Ag metal particles when the gel films that were not exposed to formalin vapor were heat-treated in air. In

