Synthesis and modification of indium tin oxide particles in supercritical water
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1. Introduction
Indium tin oxide (ITO) is an important transparent conducting oxide material, which has already been used in industry as screen panel, transparent electrode for watch, LCD, and is also expected to use as selectively permeable membrane and transparent conductive material for solar cell. Currently, the process to produce ITO plate needs two major steps. One is the synthesis of ITO crystal and the other is the coating on the glass plates. ITO particle was usually synthesized with such methods as spray pyrolysis, chemical vapor deposition (CVD), sol-gel method, co-precipitation, hydrothermal synthesis so far. However, these methods were complicated and/or usually need long time period, furthermore, most of them need post-anneal at higher temperature so that it is time and consuming process. Additionally, in the present coating process, e.g., the spattering process, the utilization ratio of ITO is lower than 10%. A new high efficiency process is definitely necessary for environmentally benign industry. In our research, we have tried to utilize the unique properties of supercritical water to synthesize the ITO particles with excellent conductivity, small diameter, and surface modification with an organic molecule. The modified ITO nanoparticles can be dispersed in solvents excellently so that it can be used as ITO coating ink. According to this procedure, we could increase the utilization ratio of ITO drastically.

2. Experimental section
The experiments were conducted with a batch reactor at the lower temperature than 450 °C for 5-30min. under 21-40 MPa in the pH range of 5.5~11.7. The co-precipitated sol of In/Sn(OH), prepared from In(NO3)3 and Sn(CH3CH2OO)4 was used as precursor and a carboxyl acid was used as the modifiers. Certain reducing agents were used as additives. The synthesized particles were characterized by way of XRD, TEM, EDX, TG-DTA, UV-VIS and FT-IR analysis.

3. Results and Discussion
A series of experimental results showed that ITO particles were successfully synthesized in the appropriable pH range without much dependent on the other factors. It was confirmed that the ratio of In:Sn was near 9:1 by EDX analysis. From the XRD analysis, it was indicated that the particles were well crystalized into cubical ITO with the size range from 10 um to 50 nm. It was also confirmed that ITO nanoparticles were modified with a carboxyl acid and dispersed into a organic solvent. Figure 1 shows the TEM images of the particles synthesized in the study. In addition to these small particles, it was observed that large beautiful cubic shaped ITO crystals were formed. It could be produced through Ostwald ripening mechanisms1).

The series of experimental results suggested that pressure, pH and the reduction agent additives played the important roles in the supercritical hydrothermal synthesis of ITO. The addition of reducing agents led to the formation of ITO particle even at lower temperature and shorter reaction time. This research could open a high efficient route for synthesis of ITO nanoparticles.

Fig.1 TEM images of ITO particles synthesized at low temperature and pressure with adding reduction agents.


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