초임계유체를 이용한 ITO 나노입자 제조에 있어서 공급유속의 영향

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The effects of feed flow rate of solution on manufacture of the ITO nanoparticle using supercritical fluids

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Introduction

Supercritical Fluid is defined as a fluid exists above critical temperature and critical pressure, and frequently used supercritical fluid are supercritical water(Tc=374 ℃, Pc =221 bar), supercritical carbon dioxide.(Tc=31 ℃, Pc=73.8 bar)

Among them, nanoparticle manufacturing using supercritical carbon dioxide are RESS(Rapid Expansion of Supercritical Solutions), SAS(Supercritical Anti-Solvent) and PGSS(Particle from Gas Saturated Solutions).

SAS is the technique applied to the materials which are hard to grind or recrystallize because of the properties sensitive to temperature or pressure and using the principle to extract the solute dissolved in solution by spraying the supercritical fluid used as antisolvent with the solution.

ITO is ceramic material with many eletronic and optical applications due to its high electrical conductivity and transparency to light in the visible region[4].

In this work, ITO particles were synthesized by a SAS method in supercritical CO_2 to enhance productivity, uniformity and various properties for electronic and optical applications and the aim is to produce nanometric ITO particles and to control the particle size(PS) and types of particle shape by modulating the feed flowrate of solution as the process parameter.

Experiment

A typical SAS experiment was started by delivering supercritical CO_2 to the precipitation chamber up to 150 bar at 50 ℃ with 20 % concentration of solution determined by the preliminary experiments as the optimum operating conditions. Then, the flowrates of ethanol used the solvent in this study and the supercritical CO_2 was regulated at 1 ml/min and 10 ml/min respectively to obtain the steady-state operating condition and to avoid the clog of the nozzle[6]. As soon as the injection of the pure liquid solvent was stopped, to investigate the influence of the solution flowrates on the morphology of the precipitated particles, the solution was dilivered through the nozzle at the flowrate of 0.2, 0.4, 0.6, 0.8, and 1.0 ml/min, respectively.

This stage was proceeded for 10 min or more to collect the solid particles sufficient to perform the analysis of the precipitate. This experiment finished when the solution delivery was ended. However, supercritical CO_2 was continuously delivered for 120 min to wash the residual content of liquid solvent in the precipitation chamber and particles dissolved into the supercritical antisolvent. If this washing process is not done, particles is not formed and

residual solvent is still remained in the precipitation chamber even after washing. When the washing process was completed, the CO_2 flow was stopped and the chamber was depressurized down to atmospheric pressure.
A schematic representation of the SAS apparatus in this study is shown in Fig. 1.

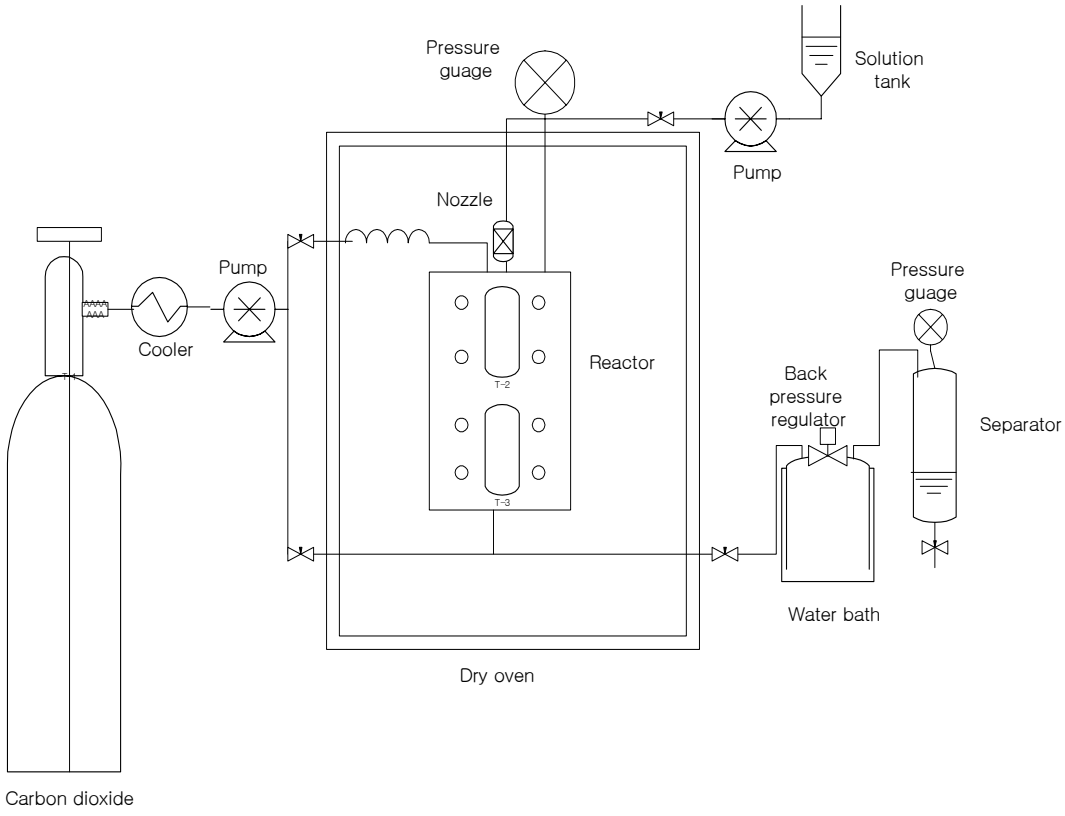

Fig. 1. Schematic diagram of SAS system.

Results and Discussion

The process of SAS micronization process depends, as in liquid antisolvent precipitation, on the solubility of the liquid solvent in the supercritical antisolvent and the fact that the solute is not soluble in the antisolvent. However, it also depends on the fast solubilization of the liquid due to the gas-like diffusion characteristic of supercritical fluids. This last characteristic is fundamental in assuring that very small particles are obtained.

According to prior authors[1-5], it is known that particle types and sizes depend on the experimental variables such as concentration of solution, temperature, pressure, feed flowrate of solution, and types of solvent etc. The experimental conditions were also applied to this work. The experiments were performed under the condition 150 bar, 50 ℃ and 20 % solution, and the range of experimental flowrate of solution was from 0.2 ml/min to 1.0 ml/min.
As the feed flowrate of solution is increased, the particle size is gradually decreased. And when the flowrate is 1 ml/min, the particle size is smallest. These results were shown in Fig. 2. and Fig. 3.

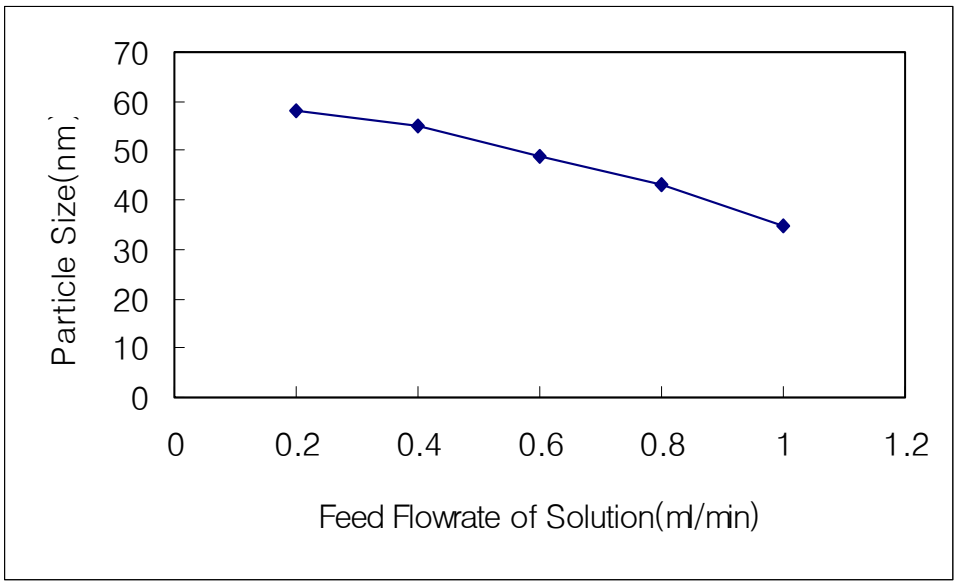

Fig. 2. ITO particle size determined by XRD analyses to the feed flowrates of solution

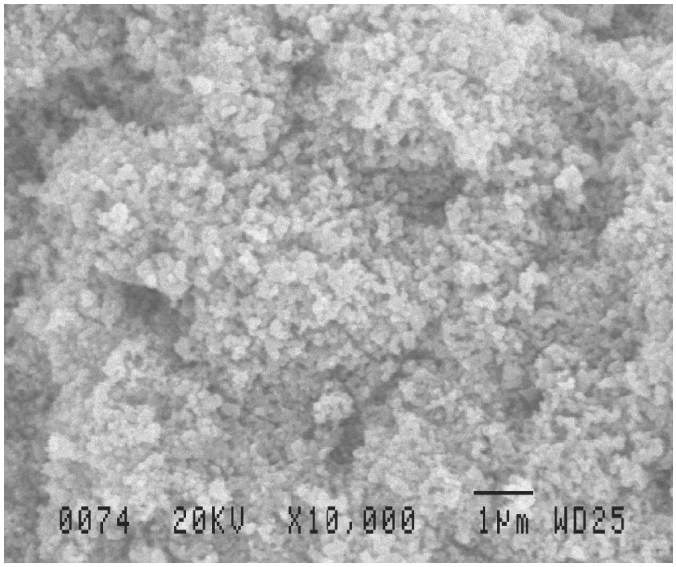

Fig. 3. SEM image of ITO synthesized by SAS process from ethanol operation at 150 bar, 50 ℃, 20 % solution, and 1.0 ml/min

Conclusion

To study the influence of the solution flowrates on the particle size and the morphology in manufaturing ITO nanoparticle using SAS process, the experiments varing the range of the solution feed flowrates from 0.2 ml/min to 1.0 ml/min at 150 bar, 50 ℃, 20 % solution were proceeded.

The consequences were the faster the feed flowrate of solution , the smaller the particle size and the particle size is smallest when the solution flowrate is 1.0 ml/min. But more studies are needed to optimize SAS process with the solution feed flowrates.

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