



# Annealing effects for calcination of tin oxide powder prepared via homogeneous precipitation

Hyang Ho Son, Won Gyu Lee\*

Department of Chemical Engineering, Kangwon National University, KNU Ave 1, Chuncheon, Kangwon 200-701, Republic of Korea

## ARTICLE INFO

### Article history:

Received 27 December 2010

Accepted 6 April 2011

Available online 7 November 2011

### Keywords:

Tin oxide powder

Rapid thermal annealing

Calcination

Homogeneous precipitation

## ABSTRACT

Tin oxide powders were prepared from a homogeneous precipitation using the aqueous solution of  $\text{SnCl}_4$  with urea as a precipitator at  $90^\circ\text{C}$  and followed by a calcination process. The calcination was performed using two different methods; conventional furnace annealing (CFA) and rapid thermal annealing (RTA). The crystallization of the tin oxide finished at  $600^\circ\text{C}$  regardless of the calcination method used. The crystallite size increased with as the calcination temperature increased due to the crystal growth and agglomeration. The tin oxide calcined using RTA has a relative smaller crystallite size than CFA at the same temperature. The tin oxide powders calcined with RTA showed higher specific surface areas than those that used CFA over a wide range of temperatures.

© 2011 The Korean Society of Industrial and Engineering Chemistry. Published by Elsevier B.V. All rights reserved.

## 1. Introduction

Tin oxide is a versatile material that can be applied in a wide range of areas such as heat mirrors [1,2], transparent electrodes for solar cells [3,4], optoelectronic devices [5], catalysts [6], and gas sensors [7–9]. The sensing properties of tin oxide gas sensors, such as sensitivity, selectivity, and reproducibility, depend primarily on the crystallite size and specific surface area [10]. The microstructural control of tin oxide has been widely studied through temperature treatments, dopants, and the preparation method [11,12].

Various methods have been developed to prepare tin oxide powders such as homogeneous precipitation [12], hydrothermal [13], sol–gel [14], gel combustion [15], spray pyrolysis [16], and so on. Among the currently available methods, homogeneous precipitation is suitable for synthesizing monodispersed spherical powders of a variety of oxide ceramics. In the homogeneous precipitation method with urea as a precipitator, the gradual and uniform rise in pH levels results in the nucleation and growth of uniformly sized and shaped tin oxide precursor particles [11,12,17]. Usually, tin oxide powders fabricated by a homogeneous precipitation method have higher surface areas than those fabricated from the direct precipitated powders. Crystalline tin oxide powders with large specific surface area and a small particle size are required to obtain the best performance in the specific gas sensor application. Therefore, controlling the crystalline grain size and specific surface area of tin oxide is critical.

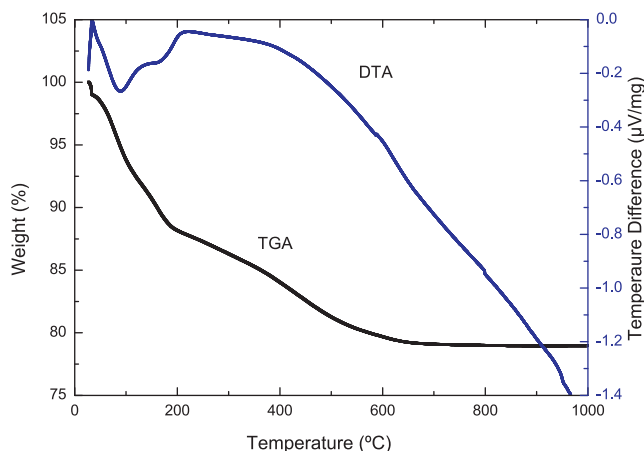
In this study, tin oxide powders prepared using a homogeneous precipitation method have been calcined with conventional furnace annealing (CFA) and rapid thermal annealing (RTA). The influence of the crystalline grain sizes and specific surface areas of the different calcinations methods were compared and analyzed. The physical and chemical properties of the tin oxide powders were analyzed using X-ray diffraction (XRD), thermal analysis, Fourier-transform infrared spectroscopy (FTIR), and a scanning electron microscope (SEM). The specific surface areas of the tin oxide powders were analyzed using a BET surface analyzer.

## 2. Experimental procedure

All chemical reagents were analytically pure and used without further purification. The tin oxide powders were prepared using a homogeneous precipitation method modified in terms of the data in the literatures [12,18]. In this study, 0.25 M aqueous solutions of tin tetrachloride ( $\text{SnCl}_4$ , Tokyo Chemical Industry) and urea ( $(\text{NH}_2)_2\text{CO}$ , Tokyo Chemical Industry) were used as starting materials. The feedstock solutions for synthesis were prepared by mixing the urea and tin tetrachloride solutions at a ratio of 2:1. The feedstock solution was heated to and maintained at  $90^\circ\text{C}$  for 90 min to complete the synthesis process; during this time, the solution was constantly stirred. After synthesis, the solutions were washed with distilled water five times in a centrifuge to remove ions. Then, the precipitates were dried at  $90^\circ\text{C}$  for 24 h in a drying oven. The calcinations for crystallization were undertaken using the conventional furnace annealing (CFA) and rapid thermal annealing (RTA) methods under atmospheric conditions in ambient air. The RTA apparatus, using 10 infrared lamps, is electronically controlled by a computer program. Various cycling

\* Corresponding author. Tel.: +82 33 2506337; fax: +82 33 2513658.

E-mail address: [wglee@kangwon.ac.kr](mailto:wglee@kangwon.ac.kr) (W.G. Lee).



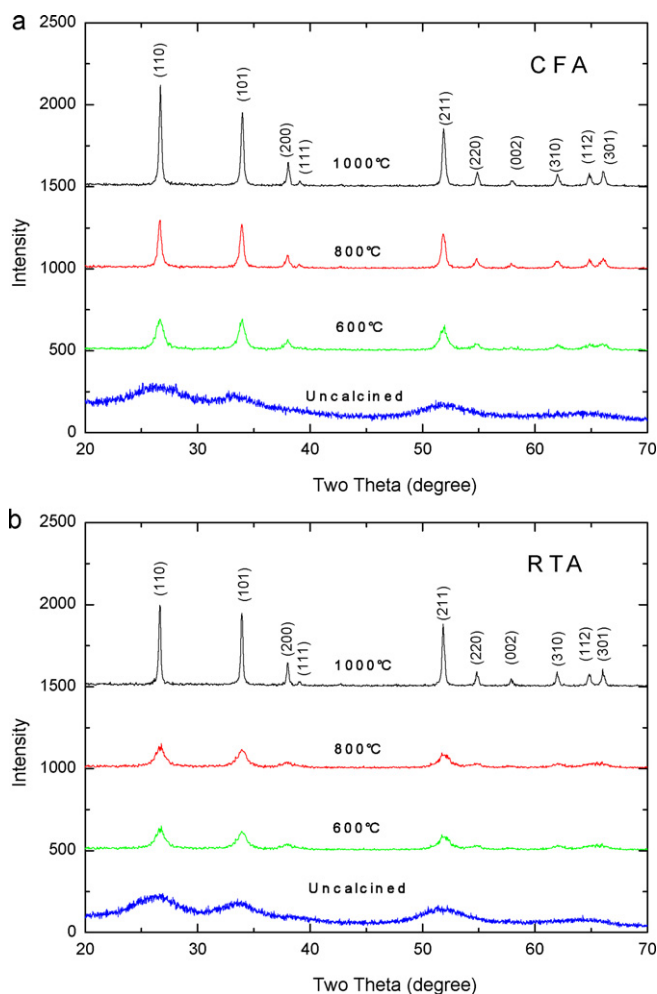
**Fig. 1.** TGA–DTA curve of the precipitated tin oxide powder from the homogeneous precipitation.

schemes can be defined, and the heating rate on the substrate can be controlled over a wide range of temperatures. Table 1 summarizes the calcination conditions for this study.

The thermochemical behavior of the precipitated phase was characterized using a thermogravimetric and differential thermal analyzer (TGA/DTA, DSC Q2000/SDT Q600). The phase development during the calcination process was monitored by Fourier-transform infrared spectroscopy (FTIR, FT-3000 MX Excaliber) and X-ray diffraction (XRD, X'Pert PRO MPD). The morphological properties of the prepared powders were observed via a field emission scanning electron microscope (FESEM, S-4300 Hitachi). The specific surface areas of the powders were measured using the BET analyzer (Micromeritics ASAP 2020). All samples were degassed at 100 °C for 24 h prior to the BET analyses.

### 3. Results and discussion

Measurement of the thermal behavior of the stannic acid as the precipitated phase was performed using TGA and DTA to determine and choose an optimum calcination temperature, as shown in Fig. 1 [18]. The TGA curve shows a marked weight loss with increase in the temperature. The weight loss is due to the elimination of ammonia, physically adsorbed water and chemically bonded water [11,12]. At temperatures above 600 °C, there was no further weight loss in the TGA curve, indicating the completion of the reactions involving a weight change. In the DTA curve, two endothermic peaks were observed at approximately 90 °C and 180 °C which correspond to the evaporation of the physically adsorbed water and to the removal of the chemically adsorbed water, respectively [11]. The results of the thermal analyses indicate that when the stannic acids in the precipitated phases are heated at temperatures as low as 600 °C, further heating does not cause any thermal and/or weight changes due to the completion of the reactions. The total weight loss after treating at



**Fig. 2.** XRD patterns of the precipitated tin oxide powders calcined at various temperatures using the CFA and RTA methods.

600 °C was approximately 22 wt%, which was a higher value than previous studies [11,12,19].

Fig. 2 shows the XRD patterns of the tin oxide as a function of the calcination temperatures. The peak positions fit with the previously reported values of tin oxide phases (JCPDS 41-1445). In the calcination with CFA and RTA, the patterns show that the samples exhibit tendencies of high degrees of crystallinity as the temperature increases. It is believed that the crystal growth continues with an increasing calcination temperature. Using the Scherrer equation, the crystalline sizes of tin oxide can be calculated using an X-ray line broadening measurement [19]. The crystallite sizes of the samples, calcined with CFA (800 °C, 2 h) and RTP (800 °C, 2 min), are 26.4 nm and 18.7 nm, respectively. Thus, the calcination results not only in crystal growth but also in the agglomeration of homogeneously precipitated tin oxide powders. The SEM micrographs in Fig. 3 show the agglomerated

**Table 1**  
Calcination conditions using CFA and RTA methods.

Conventional furnace annealing (CFA)				Rapid thermal annealing (RTA)			
Condition	Ramp-up time <sup>a</sup> (min)	Calcination temp (°C)	Calcination time (h)	Condition	Ramp-up time <sup>a</sup> (min)	Calcination temp (°C)	Calcination time (min)
1	30	600	2	1	2	600	2
2	30	800	2	2	2	800	2
3	30	1000	2	3	2	1000	2

<sup>a</sup> Time taken to heat from room temperature to calcination temperature.

Download English Version:

<https://daneshyari.com/en/article/228177>

Download Persian Version:

<https://daneshyari.com/article/228177>

[Daneshyari.com](https://daneshyari.com)