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Lead Oxide Nanorods Obtained by Thermal Decomposition of Lead Hydroxide Nanorods and Its Melting Point

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Abstract. A thermal gravity analysis on the white precipitates obtained by solution reaction of lead nitrate and alkali with adding NaCl was presented. Two thermal absorption peaks on the thermal absorption curve correspond to decomposition of lead hydroxide nanorods and melting lead oxide nanorods. And the melted temperature of the lead oxide nanorods is about 700 ºC, it is obviously lower than that of body lead oxide. A serial of controlled thermal treatment experiments confirm these results. This gives a simple and effective strategy to obtain lead oxide nanorods by thermal treatment in the temperature range from 400ºC to 600ºC.

1. Introduction

Lead oxides have two types of α- and β- phase. Both of them are n-type semiconductors and have wide applications, such as in lead-acid batteries, glass industry and pigment [1-4].

Lead oxide nanostructures have varies of morphologies, such as nanorods [5-7], nanobelts [8] and nanoparticles [9], and attract much attention under a background of research upsurge of nanoscience and nanotechnology. And different methods were developed for synthesizing lead oxide nanostructure, including hydrothermal method [5, 7], solid-state reaction [6], thermal evaporation [8] and electrochemical deposition [10]. However, these methods suffer from some drawbacks, such as complex setup, or complex preparation procedure, or low productivity, or bad reproducibility.

Lead oxide could be prepared by solution reaction of lead salt and alkali; however, the morphologies of the lead oxide precipitates are plate-like [11, 12]. In our previous work, we developed this work to obtain white precipitates of nanorods by adding NaCl into the reaction solution [13, 14]. The white precipitates are lead hydroxide nanorods. It needs a thermal treatment to change lead hydroxide nanorods to lead oxide nanorods.

In this paper, we present a detailed thermal treatment experiments on the white precipitates in order to understand the effects of the thermal treatment on the morphologies and crystal structure of the lead hydroxide nanorods. And our results will give a properly thermal treatment condition for the transition from lead hydroxide nanorods to lead oxide nanorods.

So, a simple and effective strategy to obtain lead oxide nanorods is obvious. The strategy has two key steps: (1) to obtain white precipitates by lead salt and alkali in solution with adding chloride ions; (2) thermal treatment on the white precipitates to produce yellow lead oxide nanorods. Obviously, this strategy has excellent reproducibility and productivity, and it can provide sufficient lead oxide nanorods for fundamental and applied researches.
2. Experiments
The experimental detail on the preparation of the white precipitates has been reported in previous reports [13, 14]. The obtained white precipitates are mainly lead hydroxide nanorods. The thermal treatment on the white precipitates was conducted in a horizontal quartz tube furnace. Only for higher temperature at 400ºC and 750ºC, nitrogen gas was employed for protection. The treatment time was 30 min.

A thermogravity analysis has been performed for the white precipitates at the temperature from room temperature to 900ºC under nitrogen gas protection.

We have characterized the deposits by employing Hitachi S-4300 scanning electron microscopy (SEM), Bruker D8 Focus X-ray diffraction (XRD).

3. Results and discussion
In our previous report [14], we mentioned that our original goal was to obtain lead oxide nanorods directly by solution reaction of lead nitrate and alkali with adding NaCl. We did get nanorods, but it is mainly lead hydroxide. We also mentioned the lead hydroxide nanorods could change to lead oxide by thermal treatment; however, only one experimental result was presented to prove the as-heat-treated products to be lead oxide nanorods.

To have a detailed analysis on the thermal treatment of the white precipitates, we conducted a thermogravity analysis on the precipitates. Figure 1 shows the thermogravity curves at the temperature from 25ºC to 900ºC. The red line (the upper one) in figure 1 is the thermal absorption curve. There are two absorption peaks on the thermal absorption curve at about 200ºC (marked by number 1) and 700ºC (marked by number 2). The black line (the lower one) in figure 1 is the weight loss curve. It gives the weight loss of the tested sample at every temperature stage.

Figure 1. Thermogravity curves of the white precipitates.

For the first absorption peak, it has a wide heat absorption range from about 100ºC to 600ºC. This implies that the heat absorption rate is slow. In this temperature range, there is an obvious step on the weight loss curve. And the height of the step, which is the weight loss, is about 5.93%. According to our previous reports [13, 14], we know the white precipitates are the lead hydroxide nanorods. So the weight loss at the first absorption peak is obviously due to decomposition of the lead hydroxides. The chemical formula is given below:

\[ \text{Pb(OH)}_2 \text{ (solid)} \xrightarrow{\text{by heat}} \text{PbO (solid)} + \text{H}_2\text{O (gas)} \]
According to this formula, the weight loss due to decomposition is 7.46% for absolute pure lead hydroxide. To compare our testing value of weight loss (5.93%) with calculated value for pure lead hydroxide, we can estimate the content of the lead hydroxide of the white precipitates is about 80%. This value is in good agreement with the XRD results in previous report [14].

For the second absorption peak, it is very sharp. And there is no any weight loss step on the weight loss curve at around 700°C. Obviously, a phase transition occurs and the transition temperature is about 700°C. According to weight loss curve, we can confirm the sample is lead oxide at 600°C. So it is a phase transition of lead oxide. We know lead oxides have $\alpha$- and $\beta$- phase. The transition temperature of $\alpha$-PbO to $\beta$-PbO is about 540°C [15]. However, there is no obvious heat absorption peak on absorption curve at round 540°C. So we predict that lead hydroxides are decomposed to $\beta$-PbO directly, and we exclude the phase transition at around 700°C belongs to the transition of $\alpha$-PbO to $\beta$-PbO, and we suggest that the transition at 700°C is from solid to liquid of $\beta$-PbO.

![XRD curves and SEM images](image)

Figure 2. XRD curves and SEM images of the white precipitates under different thermal treatment temperature. (a, b) room temperature, (c, d) 100°C, (e, f) 200°C, (g, h) 400°C.

To prove our suggestion, we conduct a serial of controlled experiment of the white precipitates at thermal treatment temperature of room temperature, 100°C, 200°C, 400°C and 750°C. And the morphologies and crystal structure of the as-heat-treated sample has been characterized. Figure 2 shows the SEM morphologies and XRD curves of the sample at the thermal treatment temperature of room temperature, 100°C, 200°C, 400°C. In the XRD curves, Fe-Cr diffraction peaks are from stainless steel substrates. From these XRD curves, we can find that the precipitates have changed from lead hydroxide to lead oxide with increasing thermal treatment temperature. And the sample is almost $\beta$-
PbO after thermal treatment at 400°C. No any diffraction peak of α-PbO appears in these XRD curves. This confirms that lead hydroxides are actually decomposed to β-PbO directly.

From figure 2(b), 2(d), 2(f) and 2(h), we can see all samples have nanorod morphologies. This reveals that the morphologies of the white precipitates have no dramatic change during decomposition of lead hydroxides with the increasing thermal treatment temperature. However, when the temperature rises to above 700°C, nanorods disappear.

Figure 3(a) shows the SEM image of the sample at the thermal treatment temperature of 750°C. From figure 3(a), we see a huge ball instead of nanorods. To determine what the ball is, we have gotten EDS spectrum of the ball. Figure 3(b) shows its EDS spectrum. In the spectrum, there are C, O, Pb, Cr peaks. In these peaks, C originates from the carbon contamination of the SEM, Cr originates from the stainless steel substrates, Pb and O come from the ball. These results indicate that the ball is lead oxide.

How did the ball form? Obviously, the ball came from the nanorods. To have a detailed observation on figure 3(a), it is easy to find a sunken surface on the top of the lead oxide ball. This is the typical solidification characteristics. This confirms that the sample is liquid when the temperature is 750°C, that is to say, the lead oxide nanorods are melted. According to figure 1, we confirm that the melted temperature of the lead oxide nanorod is about 700°C. It is very interesting that the melted temperature of the lead oxide nanorods is lower than that of body lead oxides (about 888°C) [16]. We infer that the lower melted temperature could be due to the small size effects of nanomaterials.

Figure 3. SEM image (a) and EDS spectrum (b) of the sample at the thermal treatment temperature of 750°C.

So according to the experimental results, we can get the properly thermal treatment temperature in the range of 400°C to 600°C for transition of lead hydroxide nanorods to lead oxide nanorods.

4. Conclusion
The decomposition of lead hydroxide nanorods occurs in the temperature of about 100°C-400°C. During decomposition, the morphologies remain nanorods. At 400°C, the lead hydroxide nanorods almost decompose to lead oxide nanorods. And the melted temperature of the lead oxide nanorods is about 700°C. So to obtain lead oxide nanorods, the properly thermal treatment temperature is about from 400°C to 600°C.

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References
[1] Veluchamy P, Sharon M, Shimizu M and Minoura H 1994 *J. Electroanal. Chem.* 365 179.
[2] Yang D, Liu J, Wang Q, Yuan X, Zhu X, Li L, Zhang W, Hu Y, Sun Xiaojia, Kumar R V and Yang J 2014 *J. Power Sources* 257 27-36.
[3] Karami H, Karimi M A and Haghdar S 2008 *Materials Ressearch Bulletin* 43 3054-3065.
[4] Taunka P B, Dasm R, Bisenc D P and Tamrakarb R 2016 *Optik* 127 (2016) 6028–6035.
[5] Cao M H, Hu C W, Peng G, Qi Y J and Wang E B 2003 *J. Am. Chem. Soc.* 125 4982-4983.
[6] Cao Y L, Liu L, Jia D Z and Xiao D Q 2005 *Material Science Forum* 475-479 3579-3582.
[7] Jia B P and Gao L 2006 *Mater. Chem. Phys.* 100 351-354.
[8] Pan Z W, Dai Z R and Wang Z L 2001 *Appl. Phys. Lett.* 80 309.
[9] Farda M J S, Hayatib P, Naraghas H S and Tabeiea S A, 2017 *Ultrasonics - Sonochemistry* 39 129-136.
[10] Ren P F, Zou X P, Cheng J, Zhang H D, Li F, Wang M F and Zhu G, 2009 *J. Nanosci. Nanotechnol.* 9 1487-1490.
[11] Samberg J P, Kajbafvala A and Koolivand A 2014 *Materials Research Bulletin* 51 356-360.
[12] Veluchamy P and Minoura H 1996 *J. Mater. Sci. Lett.* 15 1705-1707.
[13] Cheng J, Zou X P, Song W L, Cao M S, Su Y, Gang G Q, Lv X M and Zhang F X 2010 *Chin. Phys. Lett.* 27 057302
[14] Cheng J, Zou X P, Song W L, Meng X M, Su Y, Gang G Q, Lv X M, Zhang F X and Cao M S 2010 *CrystEngComm* 12 1790-1794.
[15] Giefers H and Porsch F 2007 *Physica B*, 400 53-58.
[16] From Wikipedia, the free encyclopedia. http://en.wikipedia.org/wiki/Lead(II)_oxide.