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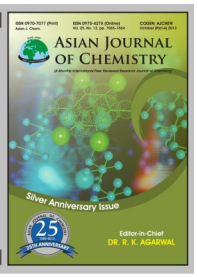

# Preparation and Characterization on Nano-Powder $Al_2O_3$-$ZrO_2$ by Ultrasound Field Co-precipitation

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In this study, $Al_2(SO_4)_3 \cdot 18H_2O$ and $ZrOCl_2 \cdot 8H_2O$ were used as raw materials to synthesize $Al_2O_3$-$ZrO_2$ nano-powder with an average diameter of 70 nm was prepared by co-precipitation method in the ultrasound field. The synthetic process and properties of nano-powder $Al_2O_3$-$ZrO_2$ were studied by X-ray diffraction, differential thermal analysis, scanning electron microscopy and laser particle size analyzer. The results show that because of the activation ultrasonic radiation can not only make the precursor smaller and reduce it agglomeration but also slow down the speed of change to gel. Ultrasonic radiation can refine $Al_2O_3$-$ZrO_2$ nano-powder effectively. The product was sphere-shaped particle with good dispersity.

**Key Words: Ultrasound field, Co-precipitation, $Al_2O_3$-$ZrO_2$ nano-powder.**

## INTRODUCTION

The ultrasonic wave which is composed of a series of density and longitudinal wave, is a special kind of energy and wave forms and through the liquid medium to spread around and with a range of field radiation. When the ultrasonic energy is high enough, it can bring the ultrasonic cavitation phenomenon. It was exploded to releasing huge amounts of energy and to generating a speed of about 110 m/s and a strong impact of the micro jet and the collision density as high as 1.5 kg/cm$^2$. The cavitation bubbles can produce about 4000 K local high temperature and l00 MPa high pressure environment at the instant of the explosion[1]. The ultrasonic cavitation can greatly improve heterogeneous reaction speed and to accelerate the spread of reactant and product and to promote the formation of new solid phase[2,3].

Preparation of alumina and zirconia nano-powder compound is an important raw material of ceramic good performance. The materials as engineering structure materials have been paid much attention. At present, the co-precipitation method[4] is commonly used to synthesize compound ultrafine powder zirconia and alumina. The advantage is simple equipment and is easy to control process[5].

The preparation process in ultrasonic field yielded, the product as spherical nanoparticle and particle size distribution uniformity and good dispersion[6,7].

## EXPERIMENTAL

Firstly, 3 % poly(vinyl alcohol) solution was prepared by dissolving it into deionized water and heated in the muffle furnace to 90 °C until the solution became clear and colourless.

**Preparation of $Al_2(SO_4)_3$ solution:** A certain amount of $Al_2(SO_4)_3 \cdot 18H_2O$ dissolved in deionized water and heated on a magnetic stirrer until the solution became colorless. A certain amount of $ZrOCl_2 \cdot 8H_2O$ was weighed and dissolved in water. The two solutions were mixed and the concentrations of $Al^{3+}$ and $Zr^{4+}$ in the solution were 0.15 and 0.03 mol/L, respectively. Then poly(vinyl alcohol) solution was selected as a dispersant, according to the molar ratio of 15:1 of urea and metal ion, in which the urea was precipitating agent. Then the solution was placed in the ultrasonic field, was controlled at 60 °C and pH value to 7, till no precipitation existed in the solution.

The solution was centrifuged and washed until there was no turbidity in the supernatant. After centrifugation, the precipitate was placed in a vacuum oven and dried. The dried precipitate was removed by grinding for differential thermal analysis. According to the results of DTA, the calcining temperature of nano-powder was determined.

The nano-powder crystal form was analyzed by means of X-ray diffraction (XRD). The particle size and shape was characterized by scanning electron microscopy (SEM). The

particle size and distribution was studied by laser particle size analyzer (PSA).

## RESULTS AND DISCUSSION

**DTA analysis:** Fig. 1 represents the DTA results of synthesized $Al_2O_3$-$ZrO_2$ nano-powder in the range of room temperature to 1200 °C. It can be seen from the TG curve that the curve continuous declined from room temperature to 1200 °C, suggesting that the loss of sample was a continuous process. The weight loss reached over 37.94 % before 650 °C, indicating the $Al_2O_3$-$ZrO_2$ nano-powder mainly converted to oxides at 650 °C precipitate and the mass loss was mainly caused by the decomposition of the evaporation of the adsorbed water in the $Al(OH)_3$ and $Zr(OH)_4$.

In the DTA curve, there were three endothermic peaks and two exothermic peaks. The exothermic peak appeared at about 147.4 °C was due to the loss of adsorbed water and crystallization water in the precursor. The endothermic peaks at 730 or 948.1 °C were attributed to the crystalline of $Al_2O_3$-$ZrO_2$ phase. The endothermic peaks of (528.4 °C or 927.6 °C) illustrated the $Al_2O_3$-$ZrO_2$ phase began to form.

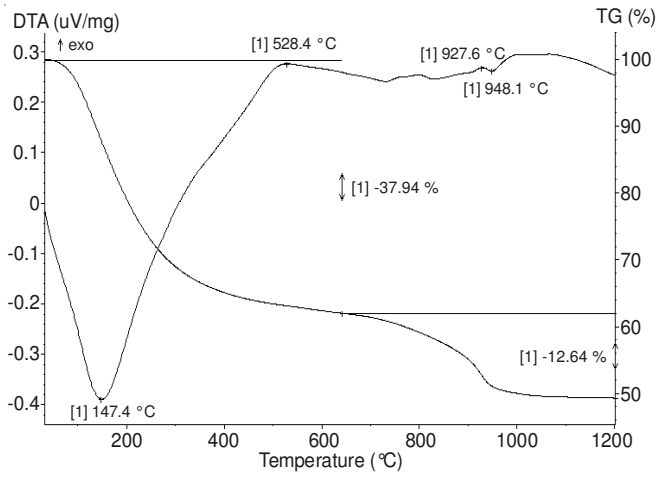


Fig. 1. DTA curve and TG curve of synthesized $Al_2O_3$-$ZrO_2$ nano-powder

**XRD analysis:** Fig. 2 shows the XRD pattern of synthesized nano-powder after calcination. It can be seen that there is no other peaks other than $Al_2O_3$-$ZrO_2$ phase, which is agreeable with the standard diffraction chart of $Al_2O_3$-$ZrO_2$ in JCPDS-270997 indicating the $Al_2O_3$-$ZrO_2$ phase has good crystallinity and there is no impurities. The crystal size was estimated from the broadening of the peaks by using the Scherrer formula:

$$D_{hkl} = \frac{k\lambda}{\beta(2\theta)\cos\theta}$$

Where, $\beta(2\theta)$ is the width of the pure diffraction profile in radians, k is 0.89, $\lambda$ is the wavelength of the X-rays and $\lambda$ is 0.1541 nm, $\theta$ is the diffraction angle and  is the age diameter of the crystallite. By fitting various peaks to this formula and taking into account the instrumental broadening, is could be found that the particles was about 10 nm.

**Microstructure of $Al_2O_3$-$ZrO_2$ nano-powder:** We used the scanning electron microscopy (SEM) to measure the $Al_2O_3$-$ZrO_2$ nano-powder sample. Fig. 3 shows the microstructure of

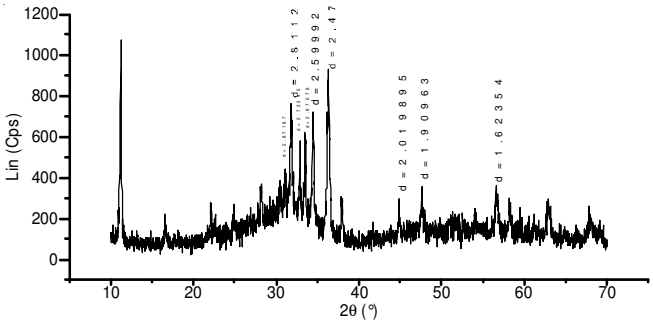


Fig. 2. XRD pattern of synthesized $Al_2O_3$-$ZrO_2$ nano-powder after calcination

synthesized $Al_2O_3$-$ZrO_2$ nano-powder. It can be seen that the most of the particles of calcined $Al_2O_3$-$ZrO_2$ powder was relatively small and distributed well. The morphology of particle was regular and nearly spherical. Only a small part of particles agglomerated, just as shown in Fig. 3. The average diameter of particle was about 70 nm.

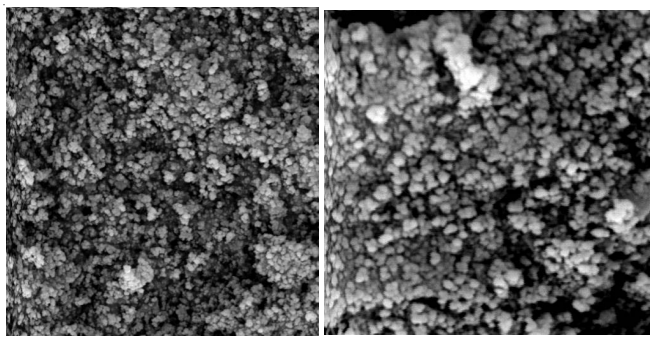

Fig. 3. Microstructure of synthesized $Al_2O_3$-$ZrO_2$ nano-powder

Fig. 4. shows the grading analysis result of synthesized $Al_2O_3$-$ZrO_2$ nano-powder. It can be seen that the particle size of the $Al_2O_3$-$ZrO_2$ samples agreed with normal distribution. The minimum particle diameter was less 40 nm and the average particle diameter was about 70 nm. The median diameter of particles was approximately 73 nm.

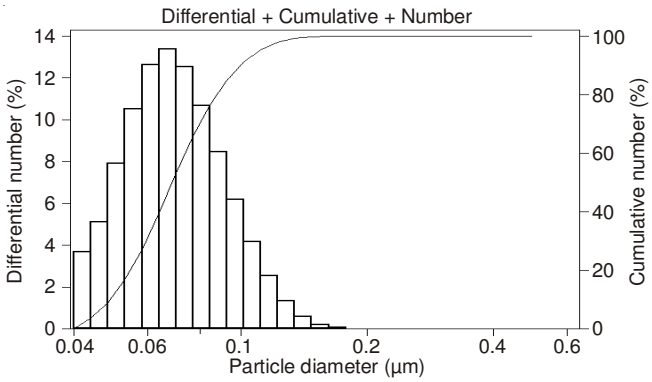


Fig. 4. Grading analysis result of synthesized $Al_2O_3$-$ZrO_2$ nano-powder

Table-1 listed the size distribution of $Al_2O_3$-$ZrO_2$ nano-powder. It can be seen that the particles less than 43 nm, 41 nm and 70 nm were nearly 10, 25 and 50 % in the YAG nano-powder, respectively. The particle less than 86 nm was nearly 75 %, indicating the size distribution of particle was well and the synthesized powder was nano size.

TABLE-1
THE GRADING ANALYSIS TABLE OF SYNTHESIZED $Al_2O_3$-$ZrO_2$ NANO-POWDER

| < 10 % | < 25 % | < 50 % | < 75 % | < 90 % |
|---|---|---|---|---|
| 0.043 μm | 0.051 μm | 0.070 μm | 0.086 μm | 0.127 μm |

## Conclusion

$Al_2O_3$-$ZrO_2$ nano-powder was synthesized by co-precipitation method in the ultrasound field. The minimum size of particle was less than 40 nm and the average diameter of particle of was about 70 nm. The median diameter of spherical particles was approximately 73 nm.

The advantage of co-precipitation method in the ultrasound field was simple instrument and equipment and convenient operation and low synthesis temperature. It is a kind of industrial application prospect of nano-powder preparation of new technology.

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