

## The Preparation of Perovskite Nano Powder by Sol-gel from DTPA and the Mechanism of Thermal Decomposition

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**Abstract:** Dy<sub>0.8</sub>Sr<sub>0.2</sub>FeO<sub>3</sub> nano powder, a synthetic oxide, is made by sol-gel method from metal nitrate and diethylenetriaminepentaacetic acid (DTPA), and the processing parameters are optimized. The process of the preparation, thermal decomposition and the property of the powder are studied by TG-DTA, IR, TEM, and XRD. The diameter of the average grain is about 70 nm. This new technique can be used in the preparation and the studying of nano materials in the complex oxide system.

**Keywords:** Nano powder, sol-gel, perovskite, diethylenetriaminepentaacetic acid.

Synthetic perovskite-type oxide (ABO<sub>3</sub>) is an important inorganic material. It is of thermal stability and good catalysis<sup>1</sup>. After A and B are replaced by other metal atoms resulting in such compounds as A<sub>x</sub>A<sub>1-x</sub>BO<sub>3</sub> or AB<sub>x</sub>B<sub>1-x</sub>O<sub>3</sub>, the crystal structure still remains unchanged, showing many good properties. Thus there is a promising prospect in the fields of catalysis, photoelectricity, magnetic medium and sensors<sup>2,3</sup>. Citric acid is one of the most commonly used gels for preparing ABO<sub>3</sub> and so is DTPA. This paper reported the preparation of Dy<sub>0.8</sub>Sr<sub>0.2</sub>FeO<sub>3</sub> synthetic nano oxide powder and analysis the process of its preparation and the mechanism of thermal decomposition.

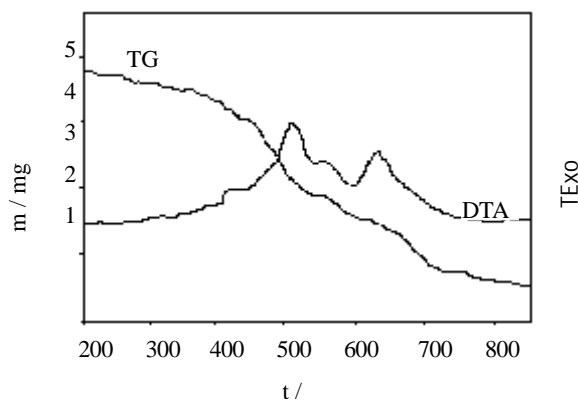
### Experimental

#### *Analysis of thermal decomposition process*

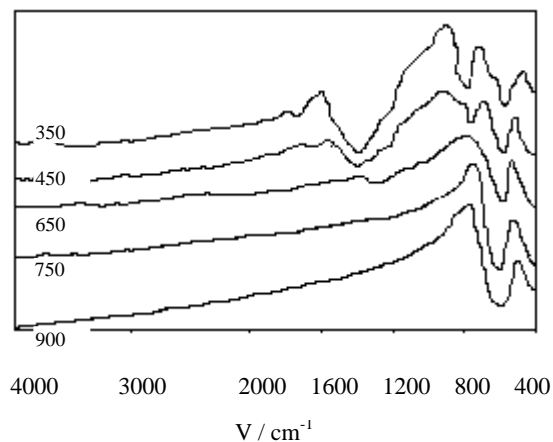
TG-DTA curve (**Figure 1**) of gel is shown, the loss of the weight in the samples occurs at 240~650°C and tends to be in balance over 700°C. At 280~550°C DTA, there are two kurtosis of heat discharge. The kurtosis between 280~400°C is for the decomposition and heat discharge of NH<sub>4</sub>NO<sub>3</sub> DTPA in free state. The kurtosis between 400~550°C suggests decomposition of DTPA metal gel, resulting in a lot of organic combustion. Over 700°C the weight remains more or less stable, indicating a complete decomposition.

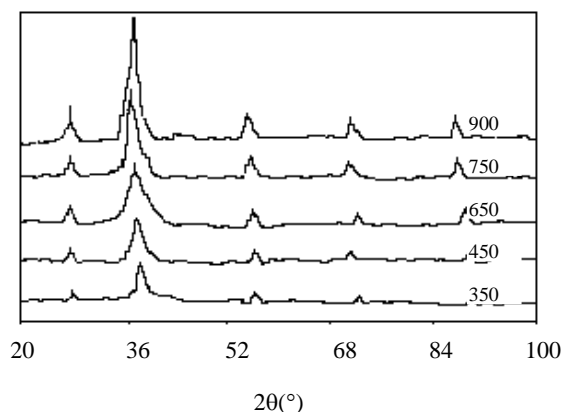
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**Figure 1** TG-DTA curve of samples*IR analysis*

**Figure 2** shows IR absorption curves of gel after calcinations at different temperatures. It can be seen that after calcinations at 350°C the vibration kurtosis of C-H, C-C framework in the sample disappears, showing that the decomposition of ganic matter is completed. At 1450  $\text{cm}^{-1}$  there is a wide kurtosis, which is the stretching vibration kurtosis of carbonate hydroxy, indicating the formation of carbonate. At 1338  $\text{cm}^{-1}$  and 853  $\text{cm}^{-1}$  there is a sharp absorption kurtosis, which is the vibration kurtosis showing the characteristics of nitric radical in free state. The kurtosis is getting lower with the rise of temperature and disappears at 750°C. Only at 570  $\text{cm}^{-1}$  a moderate kurtosis appears. It is for the stretching vibration of M-O in perovskite compound  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$ , indicating the complete formation of perovskite phase. Over 900°C, IR profile remains stable. Judging by IR result it is clear that the changing from nitrate to carbonate is a necessary stage in roasting.

**Figure 2** IR curve of samples after calcinations at different temperatures for 2 hours

**Figure 3** XRD profiles of samples calcinated at different temperatures for 2 hours

#### *XRD and the analysis of relevant substance phases*

XRD profiles (**Figure 3**) of samples roasted at different temperatures for 2 hours  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$  is formed at 350°C, because organic matters emitted a lot of heat in decomposition as well as in oxidation, thus the temperature of the gel was risen. Nitric ion provides an oxidizing environment around DTPA gel, which makes the gel to be easier to decompose and get oxidized, emitting much of heat. Thus  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$  forms only at 350°C and a small amount of  $\text{SrCO}_3$  also appears at the same time. With the increase of the temperature, the diffraction kurtosis of  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$  is rising while that of  $\text{SrCO}_3$  was dropped and disappeared at 750°C.

This is agreeable with the IR result. Judging by IR, XRD experiments it is clear that the loss of weight between 600-800°C results from the decomposition and removal of  $\text{SrCO}_3$  in the sample. At 900°C, the kurtosis becomes sharper and no new one emerges. This shows that with rise of the temperature the size of grains grows while crystal phase remains unchanged. It can be concluded that roasting temperature is very important during the composition of  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$ . No matter what method is applied (samples roasted at 750°C for 4 hours or at 650°C for 2 hours and then cooled before re-roasted, or keep 650°C for 2 hours). Therefore, roast method has little effect on crystallization.

#### *Studies of the properties of $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$ powder*

**Figure 4** shows the TEM picture of  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$  samples after calcinations at 450°C for two hours. Clearly the grain is of granularity with good dispersity, and about 30~50 nm in size. The powder is of softening agglomeration, which can be broken by supersonic wave.

#### **Conclusion**

Nano composite oxide powder of  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$  is synthesized from complex DTPA solution. Dry gel calcinated at 750°C for two hours will get pure phase. This method

has many advantages, such as even mixture, available material, simple operation, easy-to-control process and chemical measurement. And fine powder of good dispersity can be obtained at a low temperature. This method is useful for preparation of  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$  nano powder.

**Figure 4** The TEM picture of  $\text{Dy}_{0.8}\text{Sr}_{0.2}\text{FeO}_3$  samples



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