

Research Article

Influence of Eu^{3+} -Doped on Phase Transition Kinetics of Pseudoboehmite

Fuliang Zhu and Yanshuang Meng

School of Material Science and Engineering, Lanzhou University of Technology, Lanzhou 730050, China

Correspondence should be addressed to Fuliang Zhu; chzfl@126.com

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The influence of Eu^{3+} -doped on phase transition kinetics of pseudoboehmite has not been reported in the literature. Through dropping $\text{Eu}(\text{NO}_3)_3$ into pseudoboehmite colloidal solution, pseudoboehmite xerogel was produced using spray pyrolysis. The influence of Eu^{3+} -doped on the mechanism of pseudoboehmite phase transition kinetics has been calculated and analyzed by TG/DSC, XRD, and Kissinger equation. Part of Eu^{3+} ion formed compound $\text{EuAl}_{12}\text{O}_{19}$, which existed between $\alpha\text{-Al}_2\text{O}_3$ grains. Bulk diffusion of Al^{3+} was prevented from compound $\text{EuAl}_{12}\text{O}_{19}$. Therefore, phase transition kinetics rate of $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ was slowed down, causing an increase of phase transition activation energy and elevation of phase transition temperature.

1. Introduction

Pseudoboehmite ($\gamma\text{-AlOOH}$) is a crystal imperfection boehmite. Pseudoboehmite experienced a variety of intermediate phases in its phase change process, and ultimately formed a stable $\alpha\text{-Al}_2\text{O}_3$ [1–4]. Phase transitions of Alumina were studied by many researchers [5–16], and the results were shown in Figure 1. When pseudoboehmite was sintered in the range of 673 K to 973 K, product $\gamma\text{-Al}_2\text{O}_3$ was widely applied as a catalyst, catalyst carrier, and adsorbent et al. Nano $\alpha\text{-Al}_2\text{O}_3$, which is produced by sintering pseudoboehmite at 1473 K, was proverbially utilized as a paint additive, top-grade ceramic, petrochemical efficient catalyst, submicron/nanoabrasive and polishing materials, cosmetic filling materials, and inorganic membrane materials. Investigation of pseudoboehmite phase transition kinetics on its appliance had a vital significance, therefore it has gotten wide attentions [17, 18].

According to reports in the literature [19–21], phase transition temperatures of $\gamma\text{-Al}_2\text{O}_3 \rightarrow \theta\text{-Al}_2\text{O}_3$ and $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ can be changed by adding the metal ion salts or metal oxide. Adjunctions of La_2O_3 , B_2O_3 , CaO , and Y_2O_3 and salts of Ba, Sr, and Ca can increase $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ phase transition temperature. $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ phase transition temperature can be decreased by adding a

certain amount of $\text{CuO}/\text{Cu}_2\text{O}$, MgAl_2O_4 , Fe_2O_3 , V_2O_5 , TiO_2 , $\gamma\text{-Al}_2\text{O}_3$, and $\alpha\text{-Al}_2\text{O}_3$.

The effect of Eu^{3+} -doped on phase transition kinetics of the pseudoboehmite phase transition process has not been reported in the literature. Firstly, $\text{Eu}(\text{NO}_3)_3$ was dissolved in the pseudoboehmite colloidal solution, where Eu^{3+} ions are uniformly dispersed. And then, pseudoboehmite sol is dried to obtain the Eu^{3+} ion doped pseudoboehmite xerogel as a precursor. The effect of Eu^{3+} ion on phase, phase transition temperature, and phase transition kinetics in the pseudoboehmite phase change process was investigated.

2. Experimental

Pseudoboehmite powder was provided by the Aluminum Corporation of China Limited. All other reagents were analytical grade. Pseudoboehmite powder and redistilled water were mixed in a definite mass ratio, stirring to produce a suspension of solid content of 5%. The suspension was continuously stirred and concentration of 5 mol/L nitric acid solutions was added simultaneously. Pseudoboehmite colloid was obtained under the conditions of $\text{pH} = 2$ and stirring time = 3 h. Pseudoboehmite xerogel drying by SD-06 spray dryer was marked as Sp. $\text{Eu}(\text{NO}_3)_3$ with a molar ratio of $\text{Eu}(\text{NO}_3)_3 : \text{AlOOH} = 0.02 : 1$ was joined to pseudoboehmite

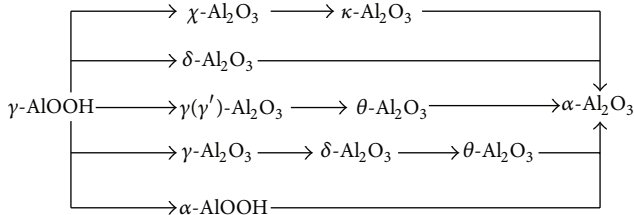


FIGURE 1: Phase transition process of pseudoboehmite may have happened.

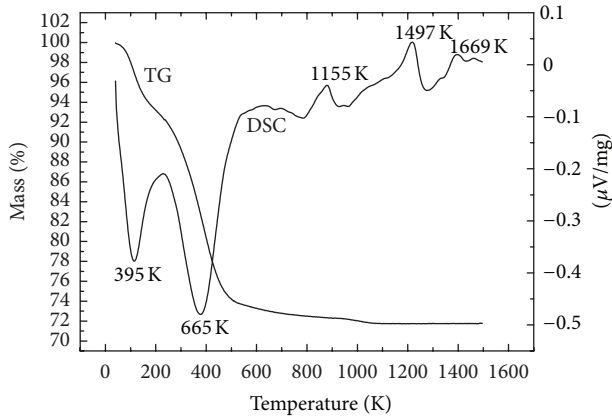


FIGURE 2: TG and DSC curves of Sp.

sol. $\text{Eu}(\text{NO}_3)_3$ was fully dissolved in pseudoboehmite sol for stirring time of 1 h. Eu^{3+} doped pseudoboehmite xerogel ($\text{AlOOH}:\text{Eu}$ xerosol) sample drying by spray dryer was denoted as Sp-E. Spray drying conditions were determined as follows: the colloid solution flow of 15 mL/min, the samples outlet temperature of 335 K, and hot air temperature of 423 K. Samples of Sp and Sp-E were calcined at different temperature in GSL1600X type tube furnace under air atmosphere with the heating rate of 10 K/min and holding time of 3 h.

XRD result of the sample was carried out on a D/max-2500/PC type XRD diffractometry (Rigaku, Japan). TG/DSC analysis of the sample was performed on a STA 449C type thermal analysis (Netsch, Germany). Sample weighed about 15 mg into the platinum crucible under N_2 atmosphere with nitrogen flow of 15 mL/min, heating rate of 10 K/min, the detection temperature in the range of 313 K to 1773 K, and the temperature error of ± 0.1 K.

3. Results and Discussions

Thermal decomposition curves of Eu^{3+} doped (Sp-E) and undoped (Sp) pseudoboehmite xerogel samples under optimum conditions from the ambient temperature to 1773 K in nitrogen atmosphere are shown in Figures 2 and 3, respectively. In Figure 2, there was an endothermic peak appeared at 395 K accompanied with 4% weight loss. The reason was that the pseudoboehmite contained part of the interlayer water. When temperature was 395 K, pseudoboehmite xerogel samples (Sp) lost interlayer water by changing of $(\text{AlOOH}) \cdot n\text{H}_2\text{O}$

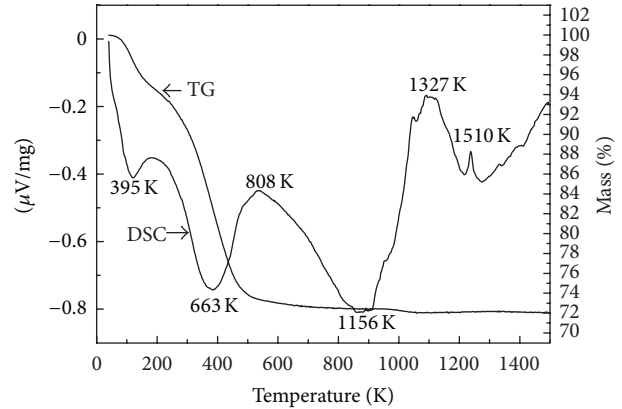


FIGURE 3: TG and DSC curves of Sp-E.

($n = 0.080 \sim 0.602$) into AlOOH . As can be seen from Figure 2, endothermic effect occurred from 493 K, endothermic peak was existed at 665 K, and endothermic effect was finished at 723 K. Combined with TG curve in Figure 2, 23% weight loss between 493~723 K was considered for decomposition of $\text{AlOOH} \cdot n\text{H}_2\text{O}$, and phase transition of $\text{AlOOH} \rightarrow \gamma\text{-Al}_2\text{O}_3$ was finished. Phase transition temperature of $\text{AlOOH} \rightarrow \gamma\text{-Al}_2\text{O}_3$ was about 773 K. Two exothermic peaks at 1155 K and 1497 K were thought to be phase transition temperatures of $\gamma\text{-Al}_2\text{O}_3 \rightarrow \theta\text{-Al}_2\text{O}_3$ and $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$, respectively. Similar results were obtained in the literature [22]. The exothermic peak formed at 1669 K, which owing to the reduction of surface energy, was considered to be the crystal growth of $\alpha\text{-Al}_2\text{O}_3$.

TGA curve of Eu^{3+} -doped pseudoboehmite Sp-E specimen was shown in Figure 3. Endothermic peak occurred at 395 K was the removal of physically adsorbed water from the sample Sp-E. Endothermic peak at 663 K was the loss of crystal water in Sp-E sample. Eu^{3+} ion in the form of substitution doping replaced Al^{3+} ion into the lattices of $\gamma\text{-Al}_2\text{O}_3$ or $\theta\text{-Al}_2\text{O}_3$. Substitution doping of Eu^{3+} ion was an endothermic reaction. There is no apparent phase transition endothermic peak of $\text{AlOOH} \rightarrow \gamma\text{-Al}_2\text{O}_3$ in the temperature range of 773 K to 1173 K. Therefore, $\text{AlOOH} \rightarrow \gamma\text{-Al}_2\text{O}_3$ phase transition temperature of Sp-E sample cannot be confirmed according to the DSC curve in Figure 3. Exothermic peaks appeared at 1327 K and 1510 K were correspondingly considered for the $\gamma\text{-Al}_2\text{O}_3 \rightarrow \theta\text{-Al}_2\text{O}_3$ and $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ phase transition temperatures, respectively.

XRD results of Pseudoboehmite xerogel Sp samples sintered at different temperature were shown in Figure 4. Figure 4 shows the main phase was cubic structure of $\gamma\text{-Al}_2\text{O}_3$ (PDF#10-0425) at 873 K. $\gamma\text{-Al}_2\text{O}_3$ with a small amount of monoclinic structure of $\theta\text{-Al}_2\text{O}_3$ (PDF#04-0877) was obtained after calcinations at 1173 K. $\theta\text{-Al}_2\text{O}_3$ with a small amount of rhombohedral structure of $\alpha\text{-Al}_2\text{O}_3$ (PDF#10-0173) was gained at 1373 K. After calcination at 1573 K, sample completely converted to $\alpha\text{-Al}_2\text{O}_3$ phase. Simultaneously, Figure 4 further indicated that phase transition temperatures of $\gamma\text{-Al}_2\text{O}_3 \rightarrow \theta\text{-Al}_2\text{O}_3$ and $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ were existed at 1155 K and 1497 K, respectively. Figure 4 also displayed that

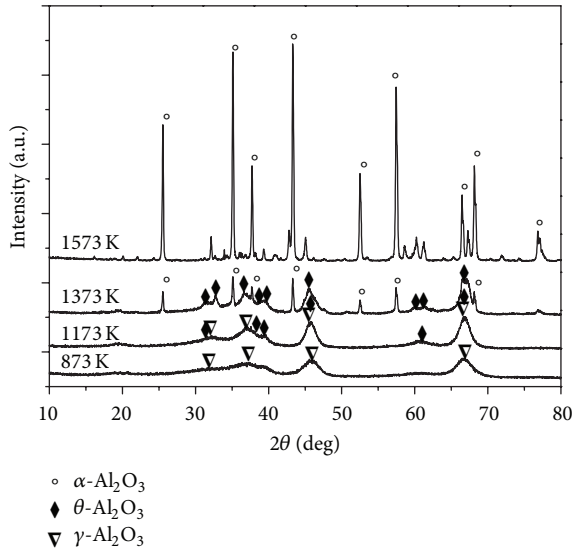


FIGURE 4: XRD patterns of Sp which were heated under different temperature.

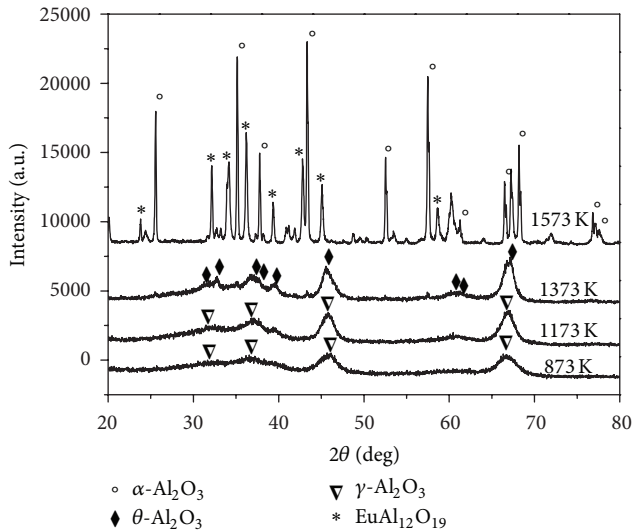


FIGURE 5: XRD patterns of Sp-E which were heated under different temperature.

diffraction peaks of γ - Al_2O_3 and θ - Al_2O_3 significantly broadened and diffraction peak of α - Al_2O_3 sharpened, indicating that both γ - Al_2O_3 and θ - Al_2O_3 were nanograins with a lower crystallinity degree and α - Al_2O_3 has a higher crystallinity degree.

XRD results of Eu^{3+} -doped pseudoboehmite Sp-E samples were shown in Figure 5. It indicated that the main phase were cubic structure of γ - Al_2O_3 (PDF#10-0425) when Sp-E samples were sintered at 873 K and 1173 K. Therefore, γ - Al_2O_3 and θ - Al_2O_3 (PDF#04-0877) were obtained when Sp-E was calcined at 1173 K and 1373 K, respectively. It further explained that 1327 K in Figure 3 was γ - $\text{Al}_2\text{O}_3 \rightarrow \theta$ - Al_2O_3 phase transition temperature. The principal phase was rhombohedral structure of α - Al_2O_3 (PDF#10-0173) with a small

amount of $\text{EuAl}_{12}\text{O}_{19}$ compound. In the literature [10, 15, 20], formation of six aluminate ($\text{BaO} \cdot 6\text{Al}_2\text{O}_3$) by doping of BaO can prevent the Al^{3+} bulk diffusion and make the transition state structure of γ - Al_2O_3 more stable. Introductions of Ca and Sr can slow down the sintering rate of γ - Al_2O_3 and phase transformation kinetic rate α - Al_2O_3 , improving the thermal stability of γ - Al_2O_3 . This paper argues that phase transition temperature of θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 was increased by the introduction of Eu^{3+} ion for two reasons. On one hand, ion diffusion was prevented from formation of $\text{EuAl}_{12}\text{O}_{19}$ compound existed between α - Al_2O_3 grains, resulting in a higher phase transition temperature of θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 . On the other hand, Eu^{3+} ions partly substitute Al^{3+} ion into the θ - Al_2O_3 lattice. This may hinder the ion migration rate in the process of θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 phase transition, elevating the Phase-change resistance. Mohanty and Ram also got the similar results [23].

Mechanism of Eu^{3+} -doped on θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 phase transition of pseudoboehmite was similar as the literature [10, 15, 20], where Ba^{2+} , Ca^{2+} , and Sr^{2+} ions can increase θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 phase transition temperature of pseudoboehmite. Eu^{3+} -doped can raise θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 phase transition temperature of pseudoboehmite to 1510 K. γ - $\text{Al}_2\text{O}_3 \rightarrow \theta$ - Al_2O_3 and θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 phase transition temperatures of pseudoboehmite through Eu^{3+} doping was elevated by 172 K and 13 K, respectively.

Phase transition kinetics parameters can be calculated through many equations [16]. Owing to the simplicity and accuracy of Kissinger equation, it is widely used. Kissinger equation describes the following:

$$\ln\left(\frac{\Phi}{T_p^2}\right) = \ln A - \ln\left(\frac{E_a}{R}\right) - \frac{E_a}{RT_p}, \quad (1)$$

where Φ denotes heating rate (K/min), T_p is peak temperature (K), R is ideal gas constant [8.314 J/(mol·K)], E_a is phase transition activation energy (kJ/mol), and A is preexponential factor (min^{-1}).

DSC curves of samples at different heating rates Φ were carried out. Peak temperature T_p (K) was acquired. The relation between $\ln(\Phi/T_p^2)$ and $1/T_p$ was plotted. Putting the slope and intercept of the straight line into (1), reaction activation energy E_a and the nucleation rate ν can be solved. DSC curves of Sp and Sp-E samples at different heating rates ($\Phi = 5 \text{ K/min}, 10 \text{ K/min}, 15 \text{ K/min}, 20 \text{ K/min}$) were measured. According to (1), phase transition activation energy E_a and preexponential factor A of γ - $\text{Al}_2\text{O}_3 \rightarrow \theta$ - Al_2O_3 and θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 phase transition can be calculated.

DSC curves of θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 phase transition process of pseudoboehmite xerogel Sp samples were shown in Figure 6. Peak temperatures T_p (K) were listed in Table 1. When $-\ln(\Phi/T_p^2)$ was plotted against $1/T_p$ as shown in Figure 7. Fitting experimental data in Figure 7 to a straight line, $-\ln(\Phi/T_p^2) = -53.3153 + 98874.8(1/T_p)$ was gotten. The linear coefficient of $r = 0.9998$ was included in Table 1. According to $E_a/R = 98874.8$ and $\ln(E_a/R) - \ln A = -53.3153$, phase transition activation energy E_a and preexponential factor A of θ - $\text{Al}_2\text{O}_3 \rightarrow \alpha$ - Al_2O_3 were calculated

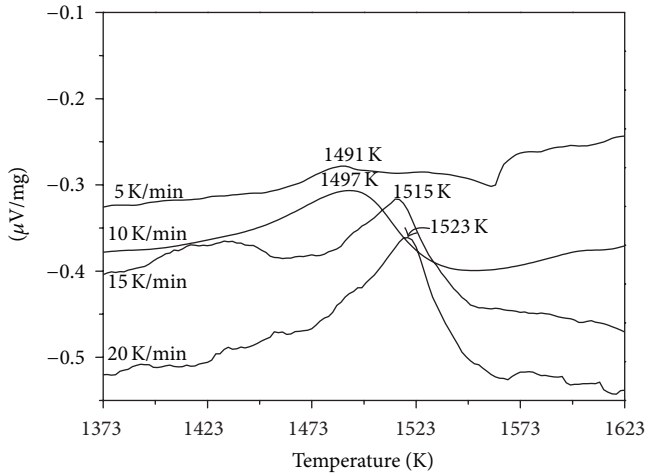


FIGURE 6: DSC curves for the pseudoboehmite xerogel Sp samples at different heating rates.

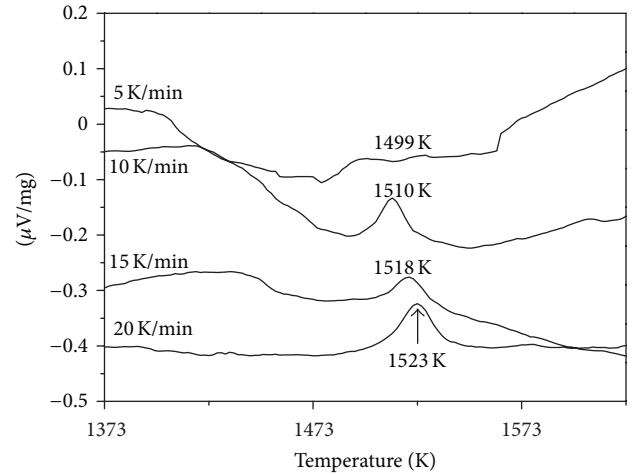


FIGURE 8: DSC curves for the Eu^{3+} doped Pseudoboehmite xerogel Sp-E samples at different heating rates.

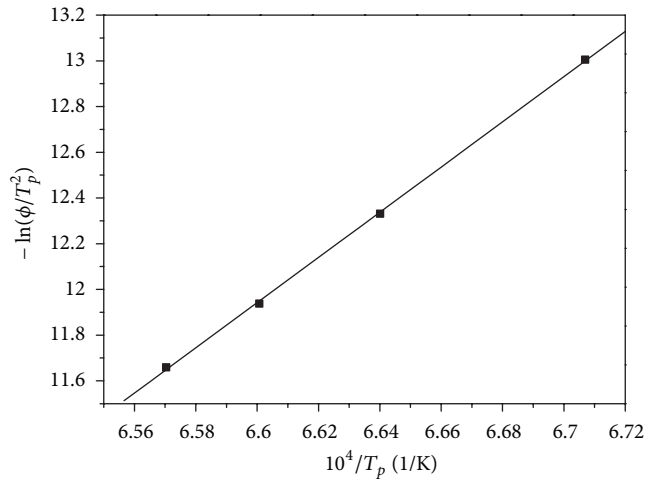


FIGURE 7: Kissinger plot for pseudoboehmite xerogel Sp samples at heating rates of 5, 10, 15, and 20 K/min.

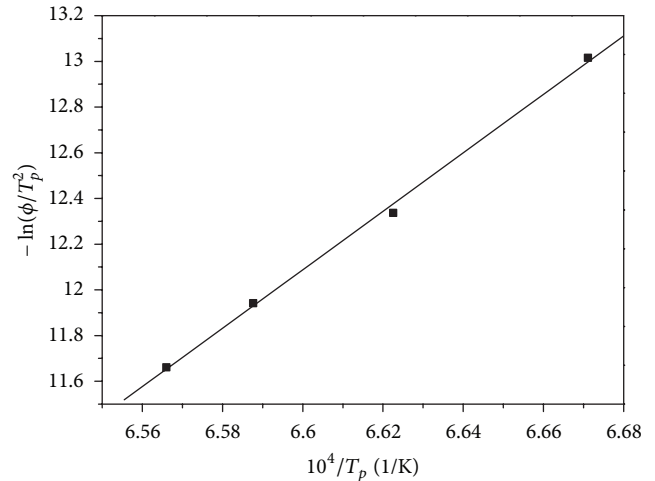


FIGURE 9: Kissinger plot for Eu^{3+} doped pseudoboehmite xerogel Sp samples at heating rates of 5, 10, 15, and 20 K/min.

for $E_a = 822.0 \text{ kJ/mol}$ and $A = 1.14 \times 10^{28}$, respectively. The consequences were also listed in Table 1.

DSC curves of $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ phase transition of Eu^{3+} doped pseudoboehmite xerogel Sp-E samples were described in Figure 8. The relations between $-\ln(\Phi/T_p^2)$ and $1/T_p$ were plotted as showed in Figure 9. The data in Figure 9 have done a linear fitting, and $-\ln(\Phi/T_p^2) = -72.3094 + 127874.8(1/T_p)$ equation was obtained. Linear coefficient r was 0.9990. Phase transition activation energy E_a and pre-exponential factor A of $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ were calculated by equations $E_a/R = 127874.8$ and $\ln(E_a/R) - \ln A = -72.3094$ for $E_a = 1063.1 \text{ kJ/mol}$ and $A = 3.24 \times 10^{36}$. All results were listed in Table 1.

Using pseudoboehmite xerogel as a precursor in this paper, phase transition activation energy of $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ is 822.0 kJ/mol , which is higher than those of reports in the literature ($557\text{--}850 \text{ kJ/mol}$) using $\gamma\text{-Al}_2\text{O}_3$ as a precursor

and (522 kJ/mol) using boehmite as a precursor. It indicated that the precursor type had an apparent effect on phase transition activation energy of $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$. $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ phase transition activation energy of Eu^{3+} -doped pseudoboehmite xerogel was 1063.1 kJ/mol , which increased 241.1 kJ/mol than that of Eu^{3+} undoped pseudoboehmite xerogel (822.0 kJ/mol). Phase transition kinetics rate of $\theta\text{-Al}_2\text{O}_3 \rightarrow \alpha\text{-Al}_2\text{O}_3$ was slowed down, resulting in an increase of phase transition activation energy and elevation of phase transition temperature.

4. Conclusions

Utilizing pseudoboehmite sol as a precursor, Eu^{3+} ion in the form of $\text{Eu}(\text{NO}_3)_3$ was dropped into sol. Eu^{3+} -doped (Sp-E) and undoped (Sp) pseudoboehmite xerogel were prepared by spray drying method. Impact of Eu^{3+} -doped on pseudoboehmite phase transition kinetics and microstructure

TABLE 1: Kinetics parameters of the θ -Al₂O₃ to α -Al₂O₃ phase transition.

Sample	Slope, E_a/R	Intercept, $\ln(E_a/R) - \ln A$	Linear coefficient, r	E_a , kJ/mol	A , min ⁻¹
Sp	98874.8	-53.3153	0.9998	822.0	1.14×10^{28}
Sp-E	127874.8	-72.3094	0.9990	1063.1	3.24×10^{36}

has been investigated utilizing TG-DSC and XRD. Kissinger equation was proposed to calculate phase transition kinetics parameters of Sp-E and Sp samples. The main conclusions are as follows.

- (1) Pseudoboehmite xerogel (Sp) experienced γ -Al₂O₃ \rightarrow θ -Al₂O₃ and θ -Al₂O₃ \rightarrow α -Al₂O₃ phase transition at 1155 K and 1497 K, respectively.
- (2) After doping with Eu³⁺ ion, phase temperatures of γ -Al₂O₃ \rightarrow θ -Al₂O₃ and θ -Al₂O₃ \rightarrow α -Al₂O₃ of Sp-E sample were 1327 K and 1510 K, respectively. Comparing with the results of Sp samples, phase transition temperatures of γ -Al₂O₃ \rightarrow θ -Al₂O₃ and θ -Al₂O₃ \rightarrow α -Al₂O₃ were increased by 172 K and 13 K, respectively.
- (3) Phase transition activation energy of θ -Al₂O₃ \rightarrow α -Al₂O₃ for Sp-E and Sp samples was 1063.1 kJ/mol and 822.0 kJ/mol, respectively. Phase transition activation energy of θ -Al₂O₃ \rightarrow α -Al₂O₃ was added by 241.1 kJ/mol as Eu³⁺ was doped to pseudoboehmite. EuAl₁₂O₁₉ compound occurred between α -Al₂O₃ grains can hinder diffusion of Al³⁺ ion. Therefore, θ -Al₂O₃ \rightarrow α -Al₂O₃ phase transition rate was reduced, resulting in an increase of θ -Al₂O₃ \rightarrow α -Al₂O₃ phase transition activation energy and elevation of phase transition temperature.

Acknowledgments

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