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Sintering densification of CaO-UO2-Gd2O3 nuclear fuel pellets

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ABSTRACT

CaO-doped UO₂-10 wt% Gd₂O₃ burnable poison fuel was prepared by co-precipitation reaction method. It was found that 0.3 wt% CaO-doping significantly improved the sintered density, grain sizes and crushing strength of UO₂--Gd₂O₃ fuel pellets at the sintering temperature of 1650 °C in the sintering atmosphere of hydrogen for 3.5 h. In addition, homogeneous solid solution without precipitation of free phases of CaO and Gd₂O₃ was successfully achieved. CaO doping in UO₂--Gd₂O₃ fuel pellet system accelerated the thermally activated material transport, so the onset temperature of densification as well as the temperature of the maximum densification rate shifted to a lower temperature region.

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1. Introduction

Developing nuclear power is an inevitable stage to solve the contradiction between supply and demand of energy, maintain sustainable economic development and protect ecological environment. In order to improve the efficiency of nuclear power, alleviate the pressure of the treatment and disposal of spent nuclear fuel, nuclear fuel elements with high burn-up and long life are the long-term goals for nuclear power plant.

The goal for a new generation and the future of nuclear power is in pursuit of inherent safety performance as well as greater economic benefits. $UO_2-Gd_2O_3$ burnable poison fuel is widely used in nuclear power reactors in the world, especially reactor cores of pressurized water reactor (PWR), to control the initial reactivity of reactors, spread the distribution of core power, increase fuel burnup and prolong refueling cycle, which thus contribute to the reduction of nuclear power operation cost and the improvement of operation safety and reliability [1–6]. Currently the preparation method of $UO_2-Gd_2O_3$ fuel pellets in nuclear power plants is similar to that of commercially used UO_2 pellets, but it is far more difficult to prepare $UO_2-Gd_2O_3$ fuel pellets than UO_2 pellets. In the

* Corresponding author. *E-mail address:* yinchanggeng5525@163.com (H. Wang). same condition, the additive gadolinium oxide reduces not only the grain sizes and the density of UO_2 pellets, but also the thermal conductivity [1,7–10], resulting in more fission gas release, irradiation swelling and other issues during nuclear reactor operation, which reduce nuclear reactor safety and economy.

In order to reduce fission gas release, one often attempted to produce large grain-sized fuel, which could be obtained by suitable doping. The results of research on CaO-doped UO₂ showed that CaO doping significantly improved the sintered density, grain sizes of UO₂ and the crushing strength of UO₂, which enhanced the security performance of the reactor fuel elements [11,12].

The concentration of gadolinium oxide in $UO_2-Gd_2O_3$ fuel pellets is in the range of 4–8 wt%. However, with the increase of the fuel cycle length, gadolinium oxide with higher concentration is needed for $UO_2-Gd_2O_3$ fuel pellets. Therefore, rising gadolinium oxide concentration would further reduce the sintered density, grain sizes and thermal conductivity of $UO_2-Gd_2O_3$ fuel pellets. For the purpose of increasing fuel burn-up and prolonging fuel cycle length, it is of great significance to research and develop UO_2-10 wt % Gd₂O₃ fuel pellet with high concentration of gadolinium oxide, high density, large grain sizes and good microstructure.

Although it is more economical to prepare CaO-doped UO₂-10 wt% Gd_2O_3 fuel pellets by traditional powder metallurgy method, $UO_2-Gd_2O_3$ fuel pellets prepared in this way reveal shortcomings such as poor uniformity of gadolinium oxide,





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difficulty in sintering densification, more internal pores and flaws and a lot of dust, which bring about environmental pollution and damage to people's physical health.

In view of a lot of advantages by the co-precipitation method, such as the uniform material chemical composition, good purity and good crystal structures and low sintering temperatures, it becomes the research and development focus of the preparation of ceramic nuclear fuel and material in the world [11]. As an advanced technology to produce ceramic materials, however, there are rare research reports on the preparation of CaO-doped UO₂-10 wt% Gd₂O₃ fuel pellets by co-precipitation reaction method.

UO₂(NO₃)₂, Gd(NO₃)₃ and Ca(OH)₂ were adopted as the initial experimental materials in this paper. UO₂-10 wt% Gd₂O₃ fuel with different contents of CaO (the mass fraction of 0, 0.3%, 0.5% and 0.6%, respectively) were prepared by co-precipitation reaction method and sintered in different sintering process conditions. The paper studied the effects of sintering temperatures and CaO contents on the sintered density, densification dynamics, grain sizes and crushing strength. In addition, its microstructure and phase structure were analyzed. The research results provided technological base for the preparation and application of CaO-doped UO₂-10 wt% Gd₂O₃ burnable poison fuel with high burn-up and inherent safety for advanced nuclear power use.

2. Experimental method

2.1. Fuel preparation

(1) The preparation of mixed solution of $UO_2(NO_3)_2$, $Gd(NO_3)_3$ and $Ca(NO_3)_2$

First, in accordance with the traditional approach to the preparation of nuclear fuel for pressurized water reactors, UO₂ pellets with the ration of O/U = 2.10 are prepared with the raw material of ammonium diuranate (ADU, 235 U content = 0.2 at%). The UO₂ pellets are broken into UO₂ powder with the particle size \leq 1–2 µm.The UO₂ powder was dried at 350 °C for 2.5 h and 99.9% Gd_2O_3 powder with the particle size $\leq 1 \mu m$ (Ganzhou holmium xin new materials co.,LTD) was dried at 550 °C for 2 h. Based on the mass fractions of Gd₂O₃ in UO₂-10 wt% Gd₂O₃ fuel pellets, a certain amount of dry powder (90 g UO₂ powder, 10 g Gd₂O₃ powder) was put into the dissolving tank together with a certain amount (450-500 ml) of deionized water. By rapid stirring, moderate (95-105 ml) concentrated nitric acid was added into the dissolving tank. After complete dissolution, insolubles were filtered out and the remaining clear filtrate was mixed solution of UO₂(NO₃)₂ and $Gd(NO_3)_3$.

The mass fractions of CaO in doped UO₂-10 wt% Gd₂O₃ fuel pellets were 0, 0.3%, 0.5% and 0.6%, respectively calcium hydroxide was weighed according to the proportion of CaO and stirred with 5 ml water. 1–2 ml concentrated nitric acid was added and dissolved to get stable Ca(NO₃)₂ solution. By rapid stirring, Ca(NO₃)₂ was slowly dropped into the mixed solution of UO₂(NO₃)₂ and Gd(NO₃)₃. In the end, uniform mixed solution of UO₂(NO₃)₂, Gd(NO₃)₃ and Ca(NO₃)₂ was prepared.

(2) The preparation of saturated (NH₄)₂CO₃ solution

22 g analytically pure solid ammonium carbonate was put into a beaker together with 100 ml deionized water. In the condition of rapid stirring, saturated (NH₄)₂CO₃ solution was prepared.

(3) The preparation of fuel pellets

The mixed solution of UO₂(NO₃)₂, Gd(NO₃)₃ and Ca(NO₃)₂ was

stirred and then heated to the reaction temperature in the precipitation tank. Then saturated (NH₄)₂CO₃ solution was added into the tank at a certain flow rate (5 ml/min). When the feeding finished, a certain volume (60-80 ml) of anhydrous ethanol was added. After a period of time (0.5-1 h) the precipitation reaction of the mixed solution finished and the solution lost liquidity. After sediment filtering, the filter cake was washed three times with anhydrous ethanol and dried at 65 °C for 15 h to get precursor. The precursor was kept at 600 °C for 2.5 h, cooled and ground to produce UO_2 -Gd₂O₃-CaO powder, which was pressed by 4 ton cm⁻² pressure into cylindrical powder bulks with the diameter of about 8 mm and the height of about 10 mm. The pressed powder bulks were sintered in the reducing atmosphere into CaO-doped UO₂-10 wt% Gd₂O₃ ceramic fuel pellets. The sintering atmosphere was H₂, the sintering temperature was 1650 °C, and the hold time was 3.5 h.

In order to study the effect of CaO doping on UO₂ and Gd₂O₃, respectively, UO₂-1 wt% CaO and Gd₂O₃-1 wt% CaO samples were prepared by the above-mentioned method, which were sintered at 1100, 1200, 1300, 1400, 1500 and 1600 °C in the sintering atmosphere of H₂ with the hold time of 3 h.

2.2. Characterization and measurement

After the cylindrical powder bulks was heated up to 1650 °C in H_2 , its shrinkage was measured by a lever dilatometer. The densities of fuel pellets are measured with water immersion method. The Optical microscope (OM) is used to observe the metallurgical structures of fuel pellets. Line intercept method is adopted to test the average grain sizes of pellets. The phase structures of fuel pellets are tested with D/max22400X X-ray diffractometer (XRD).

Electronic universal testing machine was used to measure the crushing strength of cylinders with height-diameter ratio of 1:1. The specific measurements were as follows: (1). The pressure surfaces sizes of tested samples were measured and the areas were calculated. The samples for each group are not fewer than 3; (2). The samples were put in the center of the pressure plate of the tester. Load was added at a rate of 2×10^2 N/s until the sample was destroyed. The maximum load was recorded during that moment; (3). The test results were put in the following formula to calculate the crushing strength. $R_C = P/S$, where R_C is the crushing strength, MPa, P is failure load, N, and S is stressed area.

3. Results and discussion

3.1. Phase analysis of CaO-doped UO₂-Gd₂O₃ fuel pellets

Fig. 1 is XRD diffraction pattern of UO₂–Gd₂O₃ fuel pellets with different CaO contents after sintering at 1600 °C in H₂ with the hold time of 3.5 h at the heating rate of 4 K min⁻¹. There were few differences among the XRD diffraction patterns of UO₂-Gd₂O₃ fuel pellets with different CaO contents as shown in Fig. 1. Almost all were UO₂ phase diffraction peaks, demonstrating that substitutional solid solution formed in UO2-Gd2O3 fuel pellets with different contents of 10 wt% Gd₂O₃. Fig. 1 shows that when 0.3 wt% CaO was added into UO_2 -Gd₂O₃ fuel pellets, there were not other peaks in the XRD diffraction pattern, indicating that CaO solved into the fuel pellets and uniform solid solution formed. And when 0.5% CaO was added into UO₂-Gd₂O₃ fuel pellets, the XRD pattern showed weak CaO diffraction peaks. When CaO content increased to 0.6%, the precipitated CaO from UO₂-Gd₂O₃ fuel pellets also increased. The XRD pattern showed the increase of CaO diffraction peaks.

From Fig. 1, the diffraction peaks in XRD diffraction pattern showed tiny changes with the increase of CaO content. XRD



Fig. 1. XRD diffraction pattern for UO₂-Gd₂O₃ pellets with different CaO contents.

diffraction peaks of UO_2 -Gd₂O₃ fuel pellets decreased. According to Bragg equation and interplanar spacing formula, the formula for the diffraction direction of tetragonal crystal system is:

$$\sin^2 \theta = \frac{\lambda^2}{4\alpha^2} \left(h^2 + k^2 + l^2 \right) \tag{1}$$

From Formula (1), when the diffraction angle θ remains the same, the smaller α is, the smaller the diffraction peak λ is. Because the ionic radius of Ca²⁺ is smaller than that of U⁴⁺, more CaO content in UO₂–Gd₂O₃ fuel pellets leads to smaller α of solid solution and smaller diffraction peak. Fig. 1 provides the direct data.

3.2. The effect of doped CaO contents on the sintered density and microstructure of UO_2 -Gd₂O₃ fuel pellets

Fig. 2 shows the sintered density curve for $UO_2-Gd_2O_3$ fuel pellets with different doped CaO contents. The sintering temperature was 1650 °C, the sintering atmosphere was H₂, and the hold time was 3.5 h. It can be seen from Fig. 2 that 0.1 wt% doped CaO had a great effect on the sintered density of $UO_2-Gd_2O_3$ fuel pellets, which significantly improved the sintering. When the doped CaO content was more than 0.3 wt%, the dopant had little impact on the sintered density of fuel pellets was slightly lower instead.

A small amount of CaO could increase the sintered density of UO_2 bulks. A tiny amount of Ca element could dissolve in UO_2 and might affect its dislocation structure, thereby enhancing the



Fig. 2. Sintered density of UO₂-Gd₂O₃ pellets with various CaO contents.

diffusion rate of uranium atoms and the sintered density. According to the analysis of Fig. 1, however, after the doped CaO content was more than 0.5 wt%, there was a small amount of CaO free phase precipitation from fuel pellets sintered at high temperatures. The precipitated free dispersed particles, perhaps enriching in intracrystalline place or at the grain boundary, had a "pinning" effect on the uranium atoms and inhibited the diffusion rate. Thus, the sintered density rarely changed or even slightly decreased.

Fig. 3 shows the microstructure of UO₂–Gd₂O₃ fuel pellets with different doped CaO contents. The sintering temperature was 1650 °C, the sintering atmosphere was H₂, and the hold time was 3.5 h. It can be seen from Fig. 3 that the grain size of the undoped UO₂-Gd₂O₃ pellet was about 4 µm. Doped CaO had a distinct effect on the grain sizes of UO₂–Gd₂O₃ fuel pellets. When the doped CaO content was more than 0.3 wt%, the grain size of UO₂–Gd₂O₃ pellet was 10 µm. But when the doped CaO contents were 0.5 wt% and 0.6 wt%, the grain sizes were similar to those of 0.3 wt% CaO-doped UO₂-Gd₂O₃, which also suggested that a small amount of doped CaO could significantly increase the grain sizes of fuel pellets. However, a further increase in the doped amount did not bring about obvious increase in the grain sizes. Some clusters of small grains could be seen in Fig. 3(a–c). Kim et al. [13] reported that this cluster was the Gd deficient region. The area fraction of the smallgrained cluster decreased with an increasing CaO content.

Based on the above analysis, a small amount of doped CaO could significantly improve the densification and increase the grain sizes of fuel pellets. It is wrong that more doping content will be better. According to the study in this paper, 0.3 wt% doped CaO played an obvious role.

Although small amounts of CaO has a significant effect on the sintered density of UO₂-Gd₂O₃ pellets, in order to clarify the influence of CaO in the sintered density of UO2 and Gd2O3 respectively, it is required to investigate the sintering temperature of 1100, 1200, 1300, 1400, 1500 and 1600 °C, hold time 3 h according to our experiment. In this experiment, the sintering temperatures are low, the hold time is short. Therefore, more amount of CaO doping is needed in order to obtain a satisfactory effect relationship data. 1wt% CaO doping is chosen for pure UO₂ or Gd₂O₃. Fig. 4 shows the effect of different sintering temperatures on the sintered density of UO2-1 wt% CaO and Gd2O3-1 wt% CaO. The sintering atmosphere was H₂, the holding time was 3 h and the heating rate was 4 K/min. A small amount of doped CaO significantly improved the densification and sintered density of UO₂, which was in accordance with the data in Fig. 4. Besides, it can be seen that a small amount of doped CaO significantly improved the sintered density of Gd₂O₃. These samples were almost fully densified at 1300 °C, even at a very low temperature with short sintering time. It is also seen that the dedensification occurred in both cases at 1600 °C.

3.3. The effect of sintering temperatures and CaO contents on the crushing strength of $UO_2-Gd_2O_3$ fuel pellets

According to Fig. 5, at the sintering temperatures of 1350, 1450, 1550 and 1650 °C, the variation trends of the crushing strength of $UO_2-Gd_2O_3$ fuel pellets with different CaO contents were almost the same. The strength of fuel pellets greatly increased when CaO content in $UO_2-Gd_2O_3$ fuel pellets increased from 0 to 0.3 wt%. When the CaO content further increased from 0.3 wt% to 0.6 wt%, the strength of fuel pellets insignificantly changed with a slight downward trend.

From the analysis of Figs. 1 and 2, when CaO content in $UO_2-Gd_2O_3$ fuel pellets was 0.3 wt%, the sintered density of the sample significantly increased and the sintering pellet showed a good microstructure with no free phase. So $UO_2-Gd_2O_3$ fuel pellets



Fig. 3. Microstructures of the undoped and doped UO2-Gd2O3 pellets sintered at 1650 °C for 3.5 h; (a) undoped; (b) 0.3% CaO; (c) 0.5% CaO; (d) 0.6% CaO.



Fig. 4. Sintered density of the UO_2-1 wt% CaO and Gd_2O_3-1 wt% CaO pellets after sintering at different temperatures.



Fig. 5. The effect of sintering temperatures and CaO content on the crushing strength of UO_2 -Gd₂O₃ fuel pellets.

had higher crushing strength. 0.3 wt% CaO-doped UO₂--Gd₂O₃ fuel pellet sintered at 1650 °C showed the highest crushing strength (25.8 MPa). The further increasing content of CaO had little effect on the strength of fuel pellets, which was consistent with the influence of CaO content on the sintered density.

3.4. The effect of CaO content on the sintering shrinkage rate of $UO_2-Gd_2O_3$ fuel pellets

Fig. 6 shows the sintering shrinkage curves for $UO_2-Gd_2O_3$ fuel pellets with different CaO contents. Fig. 7 shows their shrinkage rate curves. It can be seen from Figs. 6 and 7 that the curves of the sintering shrinkage and the maximum sintering shrinkage rate for $UO_2-Gd_2O_3$ fuel pellets shifted to lower temperatures. The temperature for the maximum shrinkage rate shifted from 1510 °C for the undoped $UO_2-Gd_2O_3$ pellet to 1240 °C for the 0.6 wt% CaOdoped $UO_2-Gd_2O_3$ pellet. The shifts in the shrinkage curves and the temperature for the maximum shrinkage rate increased with



Fig. 6. Sintering shrinkage curves for $\rm UO_2-Gd_2O_3$ fuel pellets with different doped CaO contents.



Fig. 7. Sintering shrinkage rate curves for $\rm UO_2-Gd_2O_3$ fuel pellets with different doped CaO contents.

the content of CaO. These results suggested that the addition of CaO considerably affected the densification in the initial or the intermediate stage.

Young [14] developed an equation for an initial-stage sintering in the condition of a constant heating rate:

$$\frac{(\Delta L/L_0)}{T} = A \exp\left[-\frac{Q}{(m+1)RT}\right]$$
(2)

where, $\Delta L/L_0$ was the relative length change. *T* was the temperature. A was a constant depending on the materials parameters and the sintering mechanisms. The exponent, *m*, had a value of 0 for viscous flow, 1 for volume diffusion and 2 for grain boundary diffusion mechanism.

Eq. (2) was treated with mathematical derivation with natural logarithms on both sides. The results were as follows:

$$\ln\frac{(\Delta L/L_0)}{T} = \ln A - \frac{Q}{(m+1)R} \cdot \frac{1}{T}$$
(3)

Based on Eq. (3), $\ln(\Delta L/L_0)/T$ versus 1/T at the heating rate of 4 K min⁻¹ was plotted as shown in Fig. 8. If the value of *m* was known, the apparent activation energy, Q, could be estimated from the slope of the plots in Fig. 8.

In the case of the undoped UO₂–Gd₂O₃, there were two linear



Fig. 8. Sintering activation energy curves for $UO_2-Gd_2O_3$ fuel pellets with different doped CaO contents.

parts of different slopes in the vicinity of 1365 °C. The lower slope at the temperatures between 1240 °C and 1365 °C was attributed to a delay of the densification of the undoped $UO_2-Gd_2O_3$ pellet. A delay of the densification was associated with the formation of the (U,Gd)O₂ solid solution [15]. CaO-doped $UO_2-Gd_2O_3$ compacts appeared to have one linear part for each content of CaO and they had a different initial-stage sintering mechanism from the undoped $UO_2-Gd_2O_3$ compact. The slopes of the CaO-doped $UO_2-Gd_2O_3$ compacts seemed to decrease with CaO contents.

Assuming that all the initial-stage sintering mechanisms of CaOdoped UO₂–Gd₂O₃ pellets were the same, the apparent activation energy decreased with increasing CaO contents. For the case of m = 1 (volume diffusion), the activation energies were estimated to 32,185, 28,640 and 27,475 Cal mol⁻¹ for CaO-doped UO₂–Gd₂O₃ pellets with CaO contents from 0.3, 0.5 to 0.6 wt%, respectively. Those values were lower than the activation energy of the undoped UO₂–Gd₂O₃ pellet, 48,000 Cal mol⁻¹. Further studies are needed to establish the mechanisms of the enhanced densification in early stage and the promoted grain growth in CaO-doped UO₂–Gd₂O₃ pellets.

4. Conclusions

- (1) 0–0.3 wt% CaO-doped UO₂–Gd₂O₃ fuel pellets, prepared by co-precipitation reaction method at 1650 °C in sintering atmosphere of hydrogen with the hold time of 3.5 h, had bigger grain sizes with uniform distribution and good densification. In addition, homogeneous solid solution formed without precipitation of free phases of CaO and Gd₂O₃.
- (2) 0.3 wt% CaO-doping significantly improved the sintered density (Increased from 10.42 to 10.52 g/cm³), grain size (Increased from 4 to 10 μ m) and crushing strength (Increased from 17.9 to 25.8 MPa) of UO₂–Gd₂O₃ fuel pellets. However, the further increase of doped CaO content had little effect on the sintered density, the grain size growth and the improvement of the crushing strength.
- (3) CaO doping in UO₂–Gd₂O₃ fuel pellets accelerated the thermally activated material transport, so the onset temperature of densification as well as the temperature of the maximum densification rate shifted to the lower temperature region. The temperature of the maximum shrinkage rate shifts from 1510 °C for the undoped UO₂–Gd₂O₃ pellet to 1240 °C for the 0.6% CaO-doped UO₂–Gd₂O₃ pellet.

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