Fabrication Methods of Dy_xZr_yO_z Pellets with Homogeneous Microstructures

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1. Introduction

In nuclear power generation, the used neutron absorber materials should have suitable characteristics in order to control the power of reactors and to compensate for an excess reactivity of fuels. Currently, Dy, Eu, Sm and so forth for absorber material have been examined with the purpose to replace (n,α) -absorbers as a control rod [1]. Several dysprosium compounds are known as an attractive control rod material for the thermal neutron reactors [2,3]. If $Dy_xZr_yO_z$ were to be fabricated by a powder process using Dy_2O_3 and ZrO_2 oxides, it must be more attractive than dysprosium alumina or dysprosium titanate as a control rod material and have suitable in-pile properties and a desirable control efficiency.

In this study, ZrO_2 , as a diluent additive, was mixed with Dy_2O_3 , and $Dy_xZr_yO_z$ pellets were fabricated by a series of mixing, wet milling, compacting and sintering processes. Sintered density and pore structures were controlled using a pore former. Sintered density, phase structure and micro-structural homogeneity of the $Dy_xZr_yO_z$ pellets were evaluated.

2. Experimental

A fine, high purity Dy₂O₃ (ALDRICH, 99.9%) and ZrO₂ (ALDRICH, 99.9%) powder were used as raw materials. The mixing ratio of Dy₂O₃ and ZrO₂ was calculated according to the Dy density and sintered density of the $Dy_x Zr_y O_z$ pellet. The weighed amount of both Dy₂O₃ and ZrO₂ was blended in a Turbula mixer for 30min. The milling was performed in a planetary mill for 3 hours at 300rpm using a zirconia jar containing both 8mm and 20mm balls together. In the wet milling process, CCl₄ was used as a liquid milling media. After the wet milling, the powder was separated from the balls and liquid media and then dried in an oven. Acrowax powder having the characteristics of a lubricant and a pore former with contents of 0.5, 1, 1.5 and 2.5 wt% was added to the milled powder, and mixed in a Turbula mixer for 30min. The powders were pre-compacted with a pressure of 25Mpa, then crushed the compacts and sieved with a 63um sieve to prepare powder granules. 0.5wt% of Acrowax was added to the powder granules as a pore former. The powder granules was pressed into green pellets with 10.0mm diameter and sintered in a box furnace at 1943K for 4 hours in air. The density of the pellets was measured by the immersion method, and the phases of each pellet were analyzed by XRD. The XRD analysis was performed by using a Ni filtered CuKa radiation. Microstructures of

the pellets with different compositions were analyzed by a ceramography.

3. Results

The variation of the green density and sintered density of the $Dy_xZr_yO_z$ pellets due to the pore former addition methods are shown in Fig. 1. Green density and sintered density were regularly decreased with an increasing pore former amount, but the decrease gradient of the sintered densities was higher than those of the green densities.

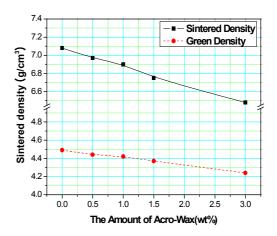


Fig. 1 The change in green density and sintered density of $Dy_x Zr_y O_z$ pellets.

The XRD patterns for each pellet specimen sintered at 1943K shows the pattern of a single cubic phase.



Fig. 2 Microstructures of $Dy_x Zr_y O_z$ pellet sintered by using previous process.

The microstructure of the $Dy_xZr_yO_z$ pellet manufactured by using a previous process in the identical sintering condition is shown in Fig. 2. This microstructure of the pellet manufactured by adding a pore former after a granulation revealed very irregular pores and large granules as a grain. It is due to the distribution of the pore former around the outer surface of the granules.

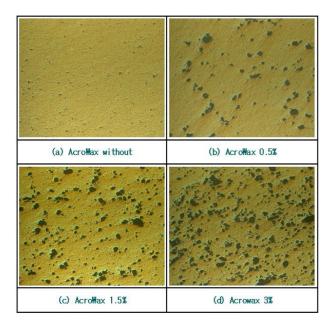


Fig. 3 Microstructures of Dy_xZr_vO_z pellets sintered at 1943K

The microstructures of $Dy_x Zr_y O_z$ are shown in Fig. 3. The pore fractions were increased with the amount of pore former. Pores have more or less a round shape and their distribution was homogeneous. This homogeneous microstructure is due to the powder treatment using a wet milling process and adding the pore former before a granulation.

4. Conclusion

The fabrication process of the $Dy_xZr_yO_z$ pellets which have homogeneous microstructures was investigated by using a new powder treatment process. Green density and sintered density were linearly decreased with an increasing pore former amount. The microstructures and pore distribution were homogeneous by using a wet milling process and adding the pore former before a granulation.

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