Phase study on the Dy$_x$Ti$_y$O$_z$ pellets.

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1. Introduction

Currently, More than 60 ceramic absorber materials based on Dy, Eu, Sm, Dy, Hf, Cd, pure Hf and Hf alloys have been examined with the purpose to replace (n,α)-ab-sorbers. Dysprosium titanate is an attractive control rod material for the thermal neutron reactors. Its main advantages are: insignificant swelling, no out-gassing under neutron irradiation, rather high neutron efficiency, a high melting point (~1870°C), non-interaction with the cladding at temperatures above 1000°C, simple fabrication and easily reprocessed non-radioactive waste.

Dy$_x$Ti$_y$O$_z$ is a solid solution formed by sintering of Dy$_2$O$_3$-TiO$_2$ compact. This solid solution has Dy density of 3.6 and 4.9 g/cm$^3$ as a absorber material.[1][2]

The sinterability of Dy$_2$O$_3$+TiO$_2$ mixed oxides was tested for various mixing ratios and sintering parameters.

Sintered density and XRD phases of Dy$_x$Ti$_y$O$_z$ pellets were evaluated in this study.

2. Experimental

The mixing ratio of Dy$_2$O$_3$/TiO$_2$ was calculated according to the Dy density and sintered density of Dy$_x$Ti$_y$O$_z$ pellet. The weighed amount of both Dy$_2$O$_3$ and TiO$_2$ was blended in a Turbula mixer for 1h, then milled by using Planetary mill with a zirconia jar containing 10mm zirconia balls at a rotation speed of 300rpm for 1h. The milled powder was pressed into cylindrical compacts using a double-acting hydraulic press under 300MPa. The compacts were sintered at 1650°C, 1500°C and 1350°C in air atmosphere for 4hr. Density of sintered pellet was measured by water immersion method. Ceramography of the pellets was done and pore structure was analyzed by using image analysis system on the polished sections. XRD on the pellet was analyzed by using Cu target(Kα 1.54056 Å) with sampling width of 0.02 degree and scanning speed of 4.00 deg/min.

3. Results

3.1 X-ray diffraction studies

The XRD patterns of Dy$_x$Ti$_y$O$_z$ with different sintering temperatures are shown in Fig. 1. It was observed that Dy$_2$TiO$_5$ crystallize in an orthorhombic structure at 1350°C and hexagonal and cubic at 1500°C and 1650°C. It was observed that Dy$_2$TiO$_5$(hexagonal) and Dy$_2$Ti$_2$O$_7$ phases in the 4.00g Dy g/cm$^3$ of Dy$_x$Ti$_y$O$_z$ sintered pellets(Fig.2).

![Fig. 1. XRD patterns of 4.88 Dy g/cm$^3$ sintered pellets;](image-url)
Fig. 2. XRD patterns of Dy$_x$Ti$_y$O$_z$ pellets sintered at 1650°C with Dy density of 4.00 Dy g/cm$^3$.

Fig. 3. Photos of microstructure; 4.88 Dy g/cm$^3$ of Dy$_x$Ti$_y$O$_z$ at (a) 1350°C, (b) 1500°C, (c) 1650°C and (d) 4.00 Dy g/cm$^3$ at 1650°C.

4. Conclusion

1. The white spots on the Dy$_x$Ti$_y$O$_z$ micrograph were cubic crystal of Dy$_2$TiO$_5$ phase.
2. The spots increases with sintering temperature.
3. Phase transformation of Dy$_x$Ti$_y$O$_z$ is irreversible during sintering process.

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Reference