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Phase study on the $Dy_xTi_yO_z$ 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℃), non-interaction with the cladding at temperatures above 1000℃, simple fabrication and easily reprocessed non-radioactive waste.

$Dy_xTi_yO_z$ is a solid solution formed by sintering of Dy_2O_3-TiO_2 compact. This solid solution has Dy dens-ity of 3.6 and 4.9 g/cm^3 as a absorber material. [1][2]

The sinterability of Dy_2O_3+TiO_2 mixed oxides was tested for various mixing ratios and sintering parameters.

Sintered density and XRD phases of $Dy_xTi_yO_z$ pellets were evaluated in this study.

2. Experimental

The mixing ratio of Dy_2O_3/TiO_2 was calculated according to the Dy density and sintered density of $Dy_xTi_yO_z$ pellet. The weighed amount of both Dy_2O_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 campacts were sintered at 1650℃, 1500℃ and 1350℃ 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 1.54056Å) with sampling width of 0.02degree and scanning speed of 4.00 deg/min.

3. Results

3.1 X-ray diffraction studies

The XRD patterns of $Dy_xTi_yO_z$ with different sintering temperatures are shown in Fig. 1. It was obser -ved that Dy_2TiO_5 crystallize in an orthorhombic stru-cture at 1350℃ and hexagonal and cubic at 1500℃ and 1650℃. It was observed that Dy_2TiO_5(hexagonal) and $Dy_2Ti_2O_7$ phases in the 4.00g Dy g/cm^3 of $Dy_xTi_yO_z$ sintered pellets(Fig.2).

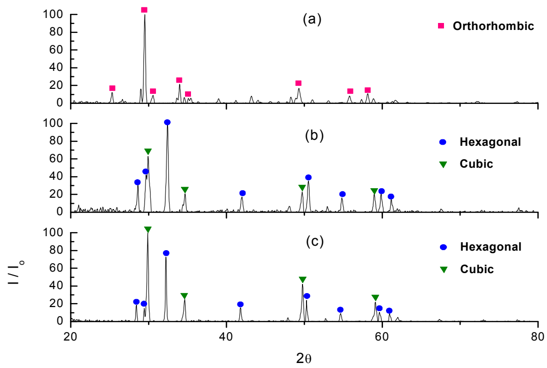


Fig. 1. XRD patterns of 4.88 Dy g/cm^3 sintered pellets;

(a) 1350℃, (b) 1500℃ , (c) 1650℃

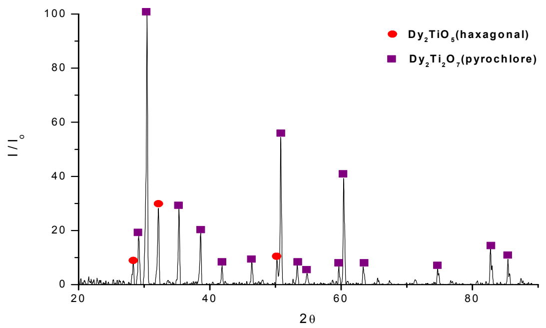
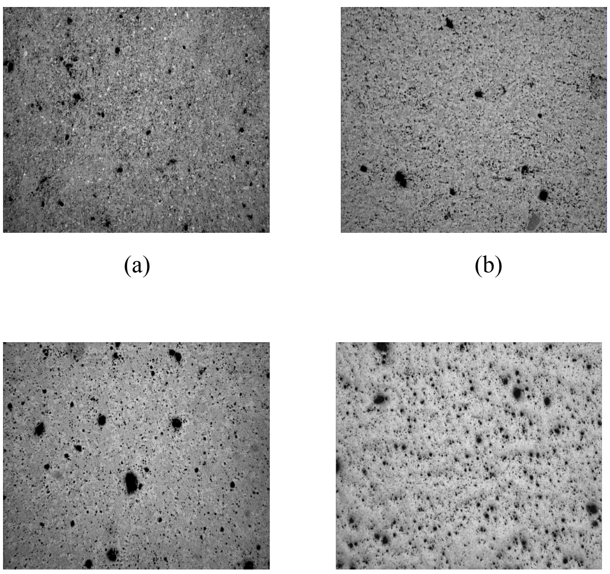


Fig. 2. XRD patterns of $Dy_xTi_yO_z$ pellet sintered at 1650℃ with Dy density of 4.00 Dy g/cm^3.

3.2 Sinterability

Sintered density of 4.00g Dy g/cm^3 and 4.88g Dy g/cm^{3}of $Dy_xTi_yO_z$ sintered pellets were 6.64 and 7.07 g/cm^3 at 1650℃.

Fig. 3 shows micro-structures of the $Dy_xTi_yO_z$ pellets sintered at different. There are a lot of white spots on the (b) and (c) micrographs. The amount of spots increases with sintering temperature. The XRD pattern of the $Dy_xTi_yO_z$ shows the spots are Dy_2TiO_5 phase which has cubic crystal structure.



(a) (b)

(c) (d)

Fig. 3. Photos of microstructure ; 4.88 Dy g/cm^3 of $Dy_xTi_yO_z$ at (a) 1350℃, (b) 1500℃, (c) 1650℃ and (d) 4.00 Dy g/cm^3 at 1650℃

4. Conclusion

1. The white spots on the $Dy_xTi_yO_z$ micrograph were cubic crystal of Dy_2TiO_5 phase.
2.. The spots increases with sintering temperature.
3. Phase transformation of $Dy_xTi_yO_z$ is irreversible during sintering process..

Acknowledgment

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Reference

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[2] G. Panneerselvam, R. Venkata Krishnan, M.P. Antonty, K. Nagarajan, T. Vasudevan, P.R. Vasudeva Rao, Thermophysical measurements on dysprosium and gadolinium titanates, Journal of nuclear materials, 327 p.220~225, 2004