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Synthesis and Characterization of a Novel Compound $SnDy_2O_4$

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Abstract: A new phase, the rare earth complex oxide $SnDy_2O_4$ was synthesized by the thermal decomposition of its oxalate precursor that was prepared by rheological phase reaction method. TG, IR, XRD and EPS were used to prove the formation of the compound $SnDy_2O_4$. The structure of $SnDy_2O_4$ was refined by Rietveld analysis. $SnDy_2O_4$ is cubic, a = 7.40366 Å, V = 405.82 $Å^3$, Z = 4.

Keywords: Rare earth complex oxide, $SnDy_2O_4$, rheological phase reaction, thermal analysis.

In recent years, the research on rare earth became a focus, and a lot of rare earth complex compounds with AB_2O_4 stoichiometry were prepared[1-3]. The compound $SnDy_2O_4$ is expected to spinel-type phase while it exhibits a new structure type.

$SnDy_2O_4$ was prepared by the thermal decomposition of the oxalate precursor that was prepared by rheological phase reaction method. SnO(AR), Dy_2O_3(99.9%) and $H_2C_2O_4 \cdot 2H_2O$ are ground in molar ratio 1:1:4.1, and placed into the reaction apparatus. Then nonionic water is added until rheological phase appears[4]. Precursor will be obtained after 10 hours at 100°C. Grind the precursor and remove off superfluous $H_2C_2O_4$, and then place the precursor with a reaction boat, raise the temperature to the decomposition point in nitrogen atmosphere and keep this temperature for 24 hours. The white powder will be obtained. It was kept into a desiccator for future use.

The TG curve of precursor was obtained by using SHIMADZU DT-40 thermal analysis instrument in nitrogen atmosphere and raising the temperature at a steady rate of 20°C/min. **Figure 1** is the TG curve of the precursor. From the TG curve we know that at the decomposition temperature the precursor completely decomposed into oxide, and that the decomposition of the precursor is processed in 3 steps. In the first step, the crystal water is lost with the weight lost ratio of 9.098; Then $SnDy_2O_3CO_3$ is formed, finally $SnDy_2O_3CO_3$ decomposed to the complex oxide. The weight lost ratio in every step matches with that of calculation. The number of water in the precursor is four that is calculated by the weight lost ratio of the first step in the TG plot.

The IR spectrum is obtained in Nicoletsxb IR instrument within 400~4000 cm^{-1}. In the IR spectrum, the stretching vibrations of $SnDy_2O_4$, Dy-O and Sn-O are corresponding to 634, 565 and 507cm^{-1} respectively[5].

The powder specimen obtained was evaluated by X-rays diffraction(D/MAX-RA)

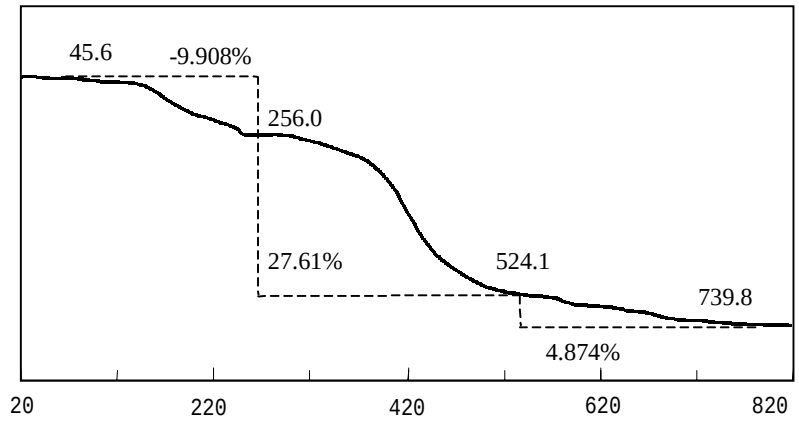
with Cu$K\alpha_1$ radiation. Intensity data were collected in the range $20^o<2\theta<90^o$ using a step width of 0.02^o. The XRD data are listed in **Table 1**.

Table 1 The index data of the X-rays diffraction

hkl	d_{OBS} (Å)	d_{CAL}(Å)	I/I_o	hkl	d_{OBS} (Å)	d_{CAL}(Å)	I/I_o
111	4.28537	4.27450	3	440	1.30922	1.30879	4
211	3.02743	3.02253	100	532	1.20084	1.20103	5
220	2.61816	2.61759	28	611		1.20103	
400	1.85109	1.85091	30	620	1.17023	1.17062	5
332	1.57939	1.57846	24	444	1.06805	1.06863	5
422	1.51230	1.51126	6				

In order to decide the valence of Sn in $SnDy_2O_4$, the EPS spectra is measured in XSAM800 EPS instrument. In XPS figure, the B. E. of Sn(3d) in the specimen is 486.2 eV which matches with the standard Sn(II) 3d electron energy of 486.0 eV.

Figure 1 The TG plot of the precursor



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References and Notes

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* The crystallographic parameters have been deposited in the editorial office of CCL

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