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Thermophysical measurements on dysprosium and gadolinium titanates

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Abstract

Dysprosium and gadolinium titanates were prepared by the solid state route and by the wet chemical route. The compounds were characterized using inductively coupled plasma mass spectrometry (ICPMS) and X-ray diffraction (XRD) techniques. Their thermal expansion coefficients were measured using high temperature X-ray diffraction technique. The quasi-isotropic mean linear thermal expansion coefficient $\bar{\alpha}_m$ of Dy_2TiO_5 and Gd_2TiO_5 at room temperature are 6.55×10^{-6} and 6.56×10^{-6} K^{-1}, respectively, and in the temperature range 298–1573 K are 1.006×10^{-5} and 1.023×10^{-5} K^{-1}, respectively. Heat capacity measurements were carried out in the temperature range 320–820 K using differential scanning calorimetry. From the polynomial fit the $C_{p,298}$ of Dy_2TiO_5 and Gd_2TiO_5 have been computed to be 178.26 and 168.61 $J\,K^{-1}\,mol^{-1}$, respectively.

1. Introduction

Elements with large absorption cross-sections for thermal neutrons such as cadmium (2450 b), boron (755 b), hafnium (105 b), iridium (440 b) and their compounds are generally used as control rod materials in nuclear reactors [1]. Boron in the form of boron carbide, B_4C, is the absorber material generally used in fast breeder reactors. The major advantages of boron carbide are the relatively high neutron absorption cross-section of boron in the fast spectrum, high thermal stability and commercial availability. However, during neutron irradiation, the $B(n,\alpha)Li$ reaction induces helium formation. The helium bubbles nucleate cracks and thereby reduce the lifetime of control rods.

Lanthanides or their oxides as alloying constituents or as dispersions in stainless steel, aluminium and titanium can also be used as control rod materials. Among the rare earth elements, dysprosium and gadolinium have relatively large thermal neutron absorption cross-sections, 930 and 4900 b, respectively, and hence compounds of dysprosium and gadolinium are considered potential candidates for use in nuclear reactors as control rod materials. Dysprosium titanate (Dy_2TiO_5) is an attractive control rod material for thermal neutron reactors [2]. Its main advantages are the high neutron absorption cross-section, relatively low swelling, no outgassing under neutron irradiation, high melting point (2143 K), non-interactive nature with the cladding at high temperatures, ease of fabrication and final product being non-radioactive. Moreover, titanates of Dy and Gd have good mechanical properties and thermal stability. Therefore, an accurate knowledge of the thermophysical properties of these materials and their behaviour during irradiation is necessary for the design of control rods and for modeling their performance. There is not much information available in the open literature on the heat capacity and thermal expansion of gadolinium titanate. For dysprosium titanate, one set of

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data from the work of Risovany et al. [2] is currently available. These researchers have used a calorimeter built in-house for heat capacity measurements. However, the accuracy of these measurements was not reported and their data also display a huge amount of scatter [3]. Hence there is a general need for generating reliable thermal expansion and heat capacity data for dysprosium and gadolinium titanates. The present study attempts to fill this gap.

2. Experimental details

The oxides of dysprosium, gadolinium and titanium (99.9%) used in this study were obtained from M/s. Indian Rare Earths, India. Dysprosium titanate (Dy_2TiO_5) and gadolinium titanate (Gd_2TiO_5) were prepared by standard solid-state synthesis (ceramic method) route as well as by wet chemical methods.

2.1. Solid-state synthesis

Stoichiometric amounts of dysprosium and titanium oxide powders were mixed for about 1 h in an agate mortar. The homogeneous powder mix was compacted at a pressure of 500 MPa, using a uniaxial hydraulic press to get 10 mm × 2 mm pellets. The green pellets were initially sintered at 1673 K for 24 h. The pellets were subsequently ground to get fine powders that were compacted again and heated at 1673 K for another 24 h to get the final sintered pellets. A similar procedure was adopted for the preparation of gadolinium titanate.

2.2. Wet chemical synthesis

Stoichiometric amounts of dysprosium oxide and titanium sponge were dissolved in concentrated HNO_3. The solution was then evaporated to obtain dry powder. The powder was calcined at 1673 K for 24 h. The sample was then ground, compacted and sintered at 1673 K for 24 h. This procedure was repeated for gadolinium titanate as well.

For compositional characterisation, a small portion of the sample was dissolved in concentrated HNO_3 and an elemental analysis for dysprosium, gadolinium and titanium was done using inductively coupled mass spectrometry (ICPMS).

2.3. X-ray diffraction studies

For characterization by X-ray powder diffraction, the sintered pellet was powdered to obtain ≈100 μm sized particles. The XRD experiment was performed using Ni filtered Cu Kα radiation ($\lambda = 154.098$ pm), in a Philips X'pert MPD system that is equipped with a graphite monochromator and a scintillation detector. The X-ray diffraction pattern was recorded in the two-theta range, $10° < 2\theta < 70°$. Peak positions and the relative intensities were estimated using a peak-fit program of the Philips X'pert Plus software. The calibration of the diffractometer was carried out using silicon and α-alumina standards obtained from the National Institute of Standards and Technology (NIST), USA.

2.4. Thermal expansion studies

The thermal expansion behaviour of polycrystalline dysprosium titanate and gadolinium titanate was studied from room temperature to 1573 K at a pressure of 10^{-5} Pa. High-temperature XRD measurements were carried out using a high-temperature attachment supplied by M/s. Johanna Otto, Germany. The detailed description of the instrument has been given elsewhere [4]. The temperature of the sample was controlled within ±1 K. Before each run, the vacuum chamber was first evacuated to a pressure of about 10^{-3} Pa and then filled with high pure argon gas. The evacuation and argon filling cycle was repeated 4–5 times, before the X-ray patterns were recorded.

Lattice parameters at different temperatures were calculated using the values of d-spacings derived from high angle reflections. The approximate lattice parameters calculated from the Miller indices and d-spacings were refined by the method of least squares using the computer program AIDS 83 [5]. The estimated error in the lattice parameter is of the order of ±0.5 pm.

For an orthorhombic lattice, the lattice parameters a, b and c are related to the d-spacing by the following relation

$$h^2/a^2 + k^2/b^2 + l^2/c^2 = 1/d_{hkl}^2. \quad (1)$$

Hence, $d_{(hkl)}$-values corresponding to all major reflections between 20° and 60° were calculated at each temperature. Three linear equations were set up by substituting the $d_{(hkl)}$-values and corresponding h, k, l values in the abovementioned equation. The lattice parameters a, b and c were computed by solving the three linear equations simultaneously. The estimated lattice parameters of Dy_2TiO_5 and Gd_2TiO_5 as a function of temperature are given in Tables 1 and 2, respectively.

2.5. Calorimetric measurements

2.5.1. Equipment

A heat flux type differential scanning calorimeter, model DSC 821e/700 of M/s. Mettler Toledo GmbH, Switzerland, was used in this study.

2.5.2. Measurements

The pellet samples for the DSC measurements were prepared so as to fit into the 40 μl Al-pans and were

Table 1
Lattice parameters (*a*, *b* and *c*), instantaneous ($\alpha_i^a, \alpha_i^b, \alpha_i^c$), relative ($\alpha_r^a, \alpha_r^b, \alpha_r^c$), mean ($\alpha_m^a, \alpha_m^b, \alpha_m^c$) and average linear thermal expansivities ($\bar{\alpha}_i, \bar{\alpha}_r, \bar{\alpha}_m$) of Dy_2TiO_5

Temperature (K)	*a*	*b*	*c*	α_i^a	α_i^b	α_i^c	α_r^a	α_r^b	α_r^c	α_m^a	α_m^b	α_m^c	$\bar{\alpha}_i$	$\bar{\alpha}_r$	$\bar{\alpha}_m$
	(pm)			(10^{-6} K^{-1})											
298	1049.2	1126.4	369.5	6.42	6.80	6.34	6.43	6.81	6.36	6.45	6.83	6.37	6.52	6.53	6.55
450	1050.3	1127.6	369.9	6.87	7.28	6.69	6.89	7.30	6.71	6.90	7.30	6.71	6.94	6.96	6.97
570	1051.2	1128.6	370.2	7.22	7.65	6.96	7.25	7.68	6.98	7.25	7.69	6.99	7.28	7.31	7.31
680	1052.0	1129.6	370.5	7.55	8.00	7.20	7.58	8.04	7.24	7.58	8.03	7.24	7.58	7.62	7.62
795	1053.0	1130.7	370.8	7.89	8.36	7.46	7.93	8.40	7.50	7.93	8.40	7.50	7.90	7.95	7.94
970	1054.5	1132.4	371.3	8.40	8.90	7.85	8.46	8.97	7.91	8.45	8.95	7.89	8.39	8.44	8.43
1185	1056.4	1134.6	371.9	9.03	9.57	8.33	9.11	9.66	8.40	9.09	9.63	8.38	8.98	9.06	9.03
1373	1058.3	1136.7	372.5	9.58	10.15	8.75	9.68	10.26	8.83	9.65	10.23	8.81	9.49	9.59	9.56
1573	1060.4	1139.1	373.2	10.15	10.76	9.19	10.28	10.90	9.30	10.24	10.68	9.26	10.03	10.16	10.06

Table 2
Lattice parameters (*a*, *b* and *c*), instantaneous ($\alpha_i^a, \alpha_i^b, \alpha_i^c$), relative ($\alpha_r^a, \alpha_r^b, \alpha_r^c$), mean ($\alpha_m^a, \alpha_m^b, \alpha_m^c$) and average linear thermal expansivities ($\bar{\alpha}_i, \bar{\alpha}_r, \bar{\alpha}_m$) of Gd_2TiO_5

Temperature (K)	*a*	*b*	*c*	α_i^a	α_i^b	α_i^c	α_r^a	α_r^b	α_r^c	α_m^a	α_m^b	α_m^c	$\bar{\alpha}_i$	$\bar{\alpha}_r$	$\bar{\alpha}_m$
	(pm)			(10^{-6} K^{-1})											
298	1048.3	1131.6	375.6	6.39	6.69	6.38	6.40	6.70	6.39	6.52	6.72	6.41	6.49	6.56	6.55
450	1049.3	1132.8	376.0	6.87	7.18	6.73	6.89	7.21	6.75	6.98	7.22	6.76	6.93	6.95	6.99
570	1050.2	1133.8	376.3	7.24	7.58	7.01	7.27	7.60	7.03	7.35	7.61	7.04	7.28	7.30	7.33
680	1051.1	1134.8	376.6	7.59	7.93	7.26	7.62	7.97	7.29	7.69	7.97	7.34	7.59	7.63	7.67
795	1052.0	1135.8	376.9	7.95	8.30	7.53	7.99	8.35	7.57	8.04	8.35	7.56	7.93	7.97	7.98
970	1053.6	1137.5	377.4	8.49	8.87	7.93	8.55	8.93	7.98	8.58	8.92	7.97	8.43	8.48	8.49
1185	1055.6	1139.8	378.1	9.15	9.56	8.41	9.23	9.65	8.48	9.24	9.63	8.47	9.04	9.12	9.11
1373	1057.4	1141.9	378.7	9.73	10.16	8.84	9.83	10.27	8.93	9.82	10.24	8.90	9.56	9.68	9.65
1573	1059.6	1144.3	379.4	10.34	10.79	9.29	10.47	10.93	9.40	10.43	10.90	9.36	10.14	10.27	10.23

hermetically sealed. High purity argon was used as the purge gas in these measurements. DSC measurements on the samples were carried out in the temperature range 320–820 K at a heating rate of 10 K min^{-1} with a purge gas flow rate of 50 ml min^{-1}. A disc of sapphire was used as the heat capacity standard. A three- segment heating program was used in these measurements. The first segment lasting for 5 min was an isothermal one at the initial temperature; the second segment was a dynamic one with a heating rate of 10 K min^{-1} and the final segment lasting for 5 min was another isothermal one at the final temperature.

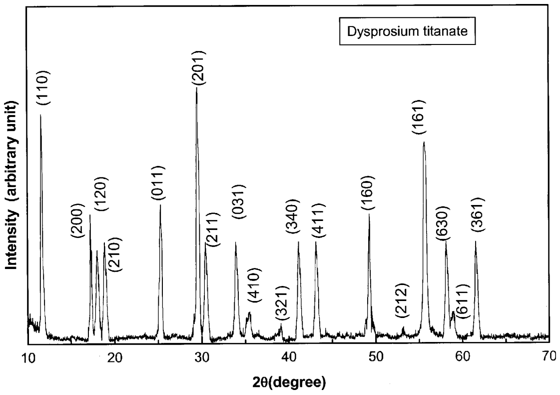

Fig. 1. Room temperature XRD pattern of Dy_2TiO_5.

3. Results and discussion

3.1. X-ray diffraction studies

The XRD patterns of Dy_2TiO_5 and Gd_2TiO_5 are shown in Figs. 1 and 2, respectively. It was observed from the XRD patterns that both Dy_2TiO_5 and Gd_2TiO_5 crystallize in an orthorhombic structure. The room temperature lattice parameters of the low temperature modification of Dy_2TiO_5 are $a = 1049.2$ pm, $b = 1126.4$ pm and $c = 369.5$ pm and those of Gd_2TiO_5 are $a = 1048.2$ pm, $b = 1131.6$ pm and $c = 375.6$ pm. The diffraction patterns and the lattice parameter values are in good agreement with the literature [6] data.

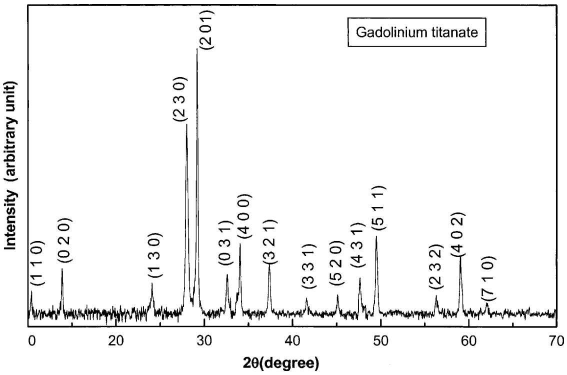

Fig. 2. Room temperature XRD pattern of Gd_2TiO_5.

The elemental compositions of Dy_2TiO_5 and Gd_2TiO_5 were estimated using ICPMS. The amount of Dy and Ti in Dy_2TiO_5 were determined to be 70.9% and 10.49%, respectively, which are in good agreement with the computed values, 71.76% and 10.59%, respectively. Similarly, the amount of Gd and Ti in Gd_2TiO_5 determined by analysis are 70.1% and 10.74%, respectively, which are in good agreement with the computed values, 71.08% and 10.82%, respectively.

3.2. Thermal expansion studies

Since the crystal structure of Dy_2TiO_5 and Gd_2TiO_5 are non-cubic, their thermal expansion characteristics are expected to be anisotropic. In the present study, we define the following measures of linear thermal expansion coefficients along the three principal crystallographic direction:

$$\alpha_i^a = 1/a_T \cdot (\partial a/\partial T)_p, \tag{2}$$

$$\alpha_r^a = 1/a_{298} \cdot (\partial a/\partial T)_p, \tag{3}$$

$$\alpha_m^a = 1/a_{298} \cdot [(a_T - a_{298})/(T - 298)], \tag{4}$$

where a_T is the lattice parameter at temperature T and a_{298} is the lattice parameter at the reference temperature 298 K. α_i^a, α_r^a, α_m^a are the instantaneous, relative, and mean linear thermal expansion coefficients along the direction a. In a similar fashion, one can define related quantities of the other directions b and c. They are designated as $(\alpha_i^b, \alpha_r^b, \alpha_m^b)$ and $(\alpha_i^c, \alpha_r^c, \alpha_m^c)$. In addition, we also define a quasi-isotropic mean linear thermal expansion coefficient pertaining to instantaneous, relative and mean linear thermal expansion coefficients. They are given by the following relations:

$$\bar{\alpha}_i = 1/3(\alpha_i^a + \alpha_i^b + \alpha_i^c), \tag{5}$$

$$\bar{\alpha}_r = 1/3(\alpha_r^a + \alpha_r^b + \alpha_r^c), \tag{6}$$

$$\bar{\alpha}_m = 1/3(\alpha_m^a + \alpha_m^b + \alpha_m^c). \tag{7}$$

In Tables 1 and 2, the values of $\bar{\alpha}_i$, $\bar{\alpha}_r$, $\bar{\alpha}_m$ are listed for Dy_2TiO_5 and Gd_2TiO_5, respectively. These data are also portrayed in Figs. 3 and 4, respectively. It is evident from Figs. 3 and 4 that the $\bar{\alpha}_i$ values are less than $\bar{\alpha}_r$ and $\bar{\alpha}_m$ values. This is in accordance with the respective definitions of these quantities. The mean linear thermal expansion coefficients for the a, b and c axes of Dy_2TiO_5 between 298 and 1573 K were found to be 1.025×10^{-5}, 1.068×10^{-5} and 9.26×10^{-6} K^{-1}, respectively. Similarly, the mean linear thermal expansion coefficients for a, b and c axes of Gd_2TiO_5 between 298 and 1573 K were found to be $1.043\ \times10^{-5}$, 1.089×10^{-5} and 9.36×10^{-6} K^{-1}, respectively.

The linear thermal expansion coefficient of Dy_2O_3–TiO_2 (Dy_2O_3 – 78.1%, TiO_2 – 9.7%, Mo – 1.8%) has been measured by Risovany et al. [2] up to 750 K using dilatometry [3]. The quasi-isotropic mean linear thermal expansion coefficient obtained in the present study in the same temperature range is 7.9×10^{-6} K^{-1} which is

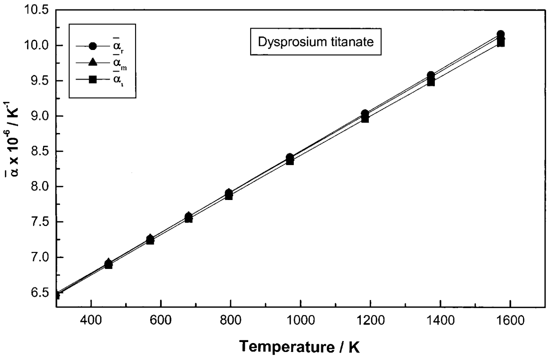

Fig. 3. Quasi-isotropic values of instantaneous, mean and relative thermal expansion coefficients of Dy_2TiO_5 in the temperature range 298–1600 K.

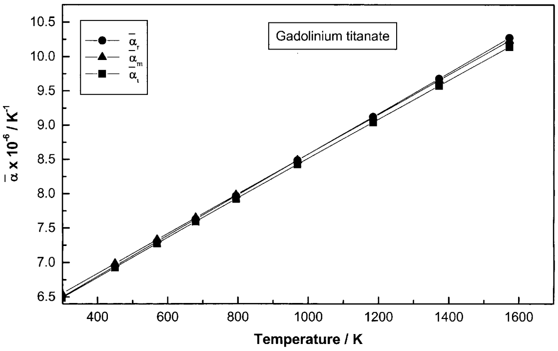

Fig. 4. Quasi-isotropic values of instantaneous, mean and relative thermal expansion coefficients of Gd_2TiO_5 in the temperature range 298–1600 K.

slightly lower than the value reported by Risovany et al. [2] is 8.55×10^{-6} K^{-1}. The slight difference may be due to the fact that the Risovany et al. have used dilatometry whereas we have used high temperature XRD. The linear thermal expansion coefficients of Gd_2TiO_5 are reported for the first time. Gd_2TiO_5 is isostructural with Dy_2TiO_5. The lattice parameters and the linear thermal expansion coefficients of Gd_2TiO_5 are also similar to those of Dy_2TiO_5. The values of quasi-isotropic mean linear thermal expansion coefficient, $\bar{\alpha}_m$ of Dy_2TiO_5 and Gd_2TiO_5 at room temperature are 6.55×10^{-6} and 6.56×10^{-6} K^{-1}, respectively, and in the temperature range 298–1573 K are 1.006×10^{-5} and 1.023×10^{-5} K^{-1}, respectively.

3.3. Calorimetric studies

The heat capacity data of sapphire given by National Institute of Standards and Technology (NIST), USA, were used for computing the heat capacities of the samples.

The heat capacity data of Dy_2TiO_5 measured in the present work given in Table 3 are the mean values of five or six measurements and the relative standard deviations are in the range 1–2%. The measured heat capacity values of Dy_2TiO_5 were fitted to the following polynomial in temperature using the least-squares method:

$$C_p\ (\mathrm{J\,K^{-1}\,mol^{-1}}) = 177.5428 + 4.877 \times 10^{-2}T - 1.2289 \times 10^{6}T^{-2}\ (320\text{–}820\ \mathrm{K}). \tag{8}$$

The standard error of the fit is 1.2 J K^{-1} mol^{-1}. The measured data along with the fit values and the data of Risovany et al. [3] are given in Fig. 5. As can be seen, there is a large amount of scatter in the data of Risovany et al. [3]. Also, the accuracy of their measurements has not been reported. Hence the present data are more reliable. The heat capacity data of Dy_2TiO_5 computed from the heat capacity data of Dy_2O_3 [6] and TiO_2 (rutile) [6] using Neumann–Kopp's law are also shown in Fig. 5. As can be seen, the present data are in agreement with the computed values within 4%. From the heat capacity data, other thermodynamic functions have been computed such as enthalpy, entropy and Gibbs energy functions which are also given in Table 3. The S^0_{298} value of Dy_2TiO_5 needed for the computation was estimated from those of Dy_2O_3 [7] and TiO_2 (Rutile) [7] from literature by assuming Neumann–Kopp's rule to be valid.

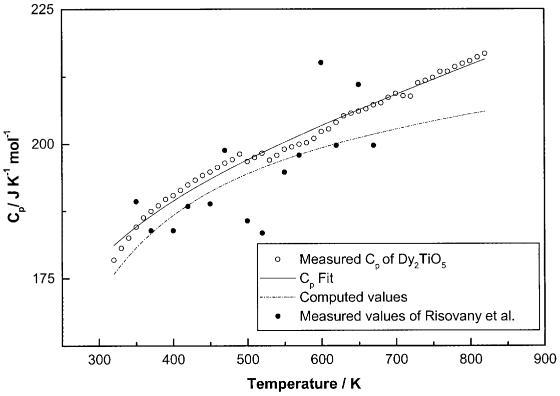

Fig. 5. Heat capacity of Dy_2TiO_5 in the temperature range 320–820 K.

The measured heat capacity data of Gd_2TiO_5 given in Table 4 are the mean values of five or six measurements and the relative standard deviations are in the range 1–2%. The measured heat capacity values of Gd_2TiO_5 were fitted to the following polynomial in temperature using the least-squares method:

Table 3
Thermodynamic functions of Dy_2TiO_5

Temperature (K)	C_p (J K^{-1} mol^{-1})				$H^0_T - H^0_{298}$	S^0_T	$(G^0_T - H^0_{298})/T$
	Measured	Fit	Literature [3]	Estimated by Neumann–Kopp's rule	(J mol^{-1})	(J K^{-1} mol^{-1})	
298.15		178.26		171.36	0	200.41	−200.41
300.00		178.52		171.77	330	201.52	−200.42
350.00	184.54	184.58	189.29	180.67	9414	229.51	−202.61
400.00	190.37	189.37	183.86	186.70	18 767	254.48	−207.56
450.00	194.76	193.42	188.84	191.07	28 339	277.03	−214.05
500.00	196.71	197.01	185.67	194.40	38 102	297.59	−221.39
550.00	199.00	200.30	194.73	197.05	48 036	316.53	−229.19
600.00	202.31	203.39	215.11	199.25	58 129	334.09	−237.21
650.00	206.08	206.33	211.03	201.12	68 372	350.49	−245.30
700.00	209.36	209.17		202.75	78 760	365.88	−253.37
750.00	212.30	211.94		204.21	89 288	380.41	−261.36
800.00	215.38	214.64		205.54	99 953	394.17	−269.23

Table 4
Thermodynamic functions of Gd_2TiO_5

Temperature (K)	C_p ($J K^{-1} mol^{-1}$)			$H_T^0 - H_{298}^0$	S_T^0	$(G_T^0 - H_{298}^0)/T$
	Measured	Fit	Estimated by Neumann–Kopp's rule	($J mol^{-1}$)	($J K^{-1} mol^{-1}$)	
298.15		168.61	161.71	0	207.53	−207.53
300.00		168.91	162.13	312	208.57	−207.53
350.00	175.79	175.64	171.22	8935	235.14	−209.61
400.00	182.57	180.75	177.43	17 850	258.94	−214.32
450.00	186.73	184.90	181.96	26 994	280.48	−220.49
500.00	188.82	188.46	185.45	36 330	300.15	−227.49
550.00	189.84	191.64	188.26	45 834	318.26	−234.93
600.00	194.69	194.55	190.61	55 489	335.06	−242.58
650.00	194.77	197.28	192.63	65 286	350.74	−250.30
700.00	198.07	199.87	194.42	75 215	365.46	−258.01
750.00	203.46	202.37	196.03	85 271	379.33	−265.64
800.00	206.90	204.79	197.50	95 451	392.47	−273.16

$$C_p(\mathrm{J\ K^{-1}\ mol^{-1}}) = 173.985 + 4.159 \times 10^{-2}\ \mathrm{T} - 1.5799 \times 10^{5}\ \mathrm{T}^{-2}\ (320\text{–}820\ \mathrm{K}). \tag{9}$$

The standard error of the fit is 1.8 $J K^{-1} mol^{-1}$. The measured data along with the fit values as well as the heat capacity data of Gd_2TiO_5 computed from the heat capacity data of Gd_2O_3 [7] and TiO_2(rutile) [7] using Neumann–Kopp's law are given in Fig. 6. As can be seen, the present data are in agreement with the Neumann–Kopp law values within 5%. From the heat capacity data, other thermodynamic functions have been computed such as enthalpy, entropy and Gibbs energy functions and these values are also given in Table 4. The S_{298}^0 value of Gd_2TiO_5 required for the computation was estimated in a similar way, as was done for Dy_2TiO_5. From the polynomial fit the $C_{p,298}$ of Dy_2TiO_5 and Gd_2TiO_5 have been computed to be 178.26 and 168.61 $J K^{-1} mol^{-1}$, respectively.

As the measured values of heat capacity of Dy_2TiO_5 and Gd_2TiO_5 deviate from the value estimated by Neumann–Kopp's rule by 4–5%, the estimated S_{298}^0 values are also expected to have an inaccuracy of 4–5% and therefore the computed values such as entropy and Gibbs energy functions will also have similar errors.

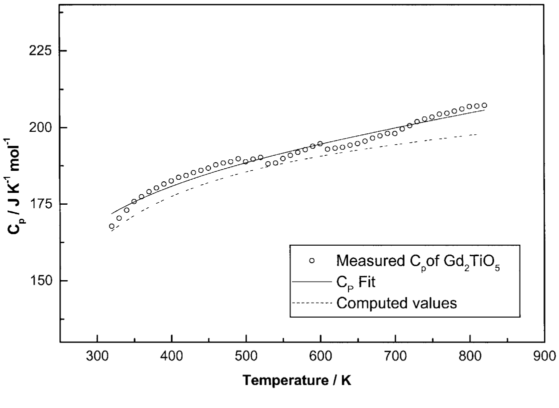

Fig. 6. Heat capacity of Gd_2TiO_5 in the temperature range 320–820 K.

4. Conclusion

Dysprosium and gadolinium titanates were prepared by the ceramic route and by the wet chemical method. The compounds were characterized using X-ray diffraction. It is generally known that both the compounds crystallize in the orthorhombic structure. Thermal expansion coefficients were determined using high-temperature XRD. The thermal expansion and heat capacity data of gadolinium titanate have been measured for the first time. The study provides more reliable heat capacity data for dysprosium titanate than that of Risovany et al. [3].

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