Standard enthalpies of formation of some carbides, silicides, germanides, stannides and borides of terbium determined by high temperature direct synthesis calorimetry

S.V. Meschel, O.J. Kleppa

The James Franck Institute, The University of Chicago, 5640 South Ellis Avenue, Chicago, IL 60637, USA

Abstract

The standard enthalpies of formation for some terbium alloys in the binary systems Tb–X (where X = C, Si, Ge, Sn, B) have been determined by direct synthesis calorimetry at 1473 ± 2 K. The following values of ΔH_f^0 (kJ (mol atoms)$^{-1}$) are reported: TbC_2, -24.6 ± 1.0; TbSi, -80.9 ± 1.8; $TbSi_2$, -61.5 ± 2.4; Tb_5Si_3, -61.0 ± 2.5; Tb_5Ge_3, -81.7 ± 2.7; Tb_5Sn_3, -73.1 ± 3.1; TbB_2, -34.2 ± 1.8.

The results are compared with reported calorimetric data for the corresponding compounds of some other lanthanide elements and with predicted values from the semiempirical model of Miedema and coworkers. Systematic comparisons of the enthalpies of formation are presented for the compounds of Tb with elements from groups IIIB and IVB in the periodic table.

Possible correlations among the systematic changes in the reduced temperature, the relative molar volume ratios, and the enthalpies of formation as suggested by Gschneidner are also examined.

Keywords: Enthalpies of formation; High temperature direct synthesis calorimetry; Terbium alloys

1. Introduction

During recent years we have pursued systematic studies of the enthalpies of formation of transition metal and rare earth carbides, silicides, germanides and borides by means of high temperature reaction calorimetry [1]. These investigations began with the work of Topor and Kleppa on LaB_6 [2], and were then continued by the same researchers on some characteristic silicides of group III metals [3,4] and later by Jung and Kleppa on the corresponding germanides [5]. During the last year this study has been extended by the present authors to the enthalpies of formation of group III carbides [6] and more recently to the carbides, silicides, germanides and borides of the lanthanide elements La, Ce, Pr, Nd, Gd and Lu [7–10]. In the present investigation we report new thermochemical data for some compounds in the binary systems Tb–C, Tb–Si, Tb–Ge, Tb–Sn and Tb–B. The information in the literature regarding the phase diagrams of these systems is less extensive than for the corresponding alloys of the early lanthanide elements. For example, there are no established phase diagrams

for the Tb–C and Tb–Si systems. For this reason the melting points were not available for four of the compounds which we studied [11,12]. Information regarding the structural properties is more generally available; however, the X-ray diffraction patterns for two of the considered compounds were not included in the ASTM powder diffraction file [13–20].

The published literature offers a value for the heat of formation of TbB_6, derived from e.m.f. measurements and mass spectrometric data [21–23]. However, we found no experimental values for the enthalpies of formation of any of the other alloys which we studied. We will compare our results for the Tb compounds with the enthalpies of formation of the corresponding alloys formed by the earlier lanthanide elements. We will also compare our results with predictions based on the semiempirical model of Miedema and coworkers [24].

Our new thermochemical data allow us to test further the systematic correlation of the heats of formation of the compounds of the lanthanide elements with elements in columns IIIB and IVB in the periodic table [7–10].

Our new data also enable us to test the possible correlations between the enthalpies of formation, the reduced temperatures and the relative molar volumes of the lanthanide compounds as suggested by Gschneidner [25,26].

2. Experimental and materials

The experiments were carried out at 1473 ± 2 K in a single unit differential microcalorimeter which has been described in an earlier communication from this laboratory [27]. All the experiments were performed under a protective atmosphere of argon gas which was purified by passing it over titanium chips at about 900 °C. A BN (boron nitride) crucible was used to contain the samples.

All the materials except boron were purchased from Johnson Matthey–Aesar, Ward Hill, MA; the Tb metal was in ingot form. Boron was obtained from Alfa Products–Ventron. The purities of the samples ranged from 99.0% for carbon to 99.999% for germanium. The particle sizes of the powders used in the experiments were about -80 mesh for Tb, -100 mesh for Sn, -150 mesh for Ge, -300 mesh for C and -325 mesh for Si. In order to avoid as much as possible any oxidation of the samples by the atmosphere, the Tb powders were hand filed from the ingot immediately prior to the preparation of the sample pellets. Carbon was in the crystalline graphite modification. As purchased, the Ge sample had an average particle size of -10 mesh. We ground this sample in an agate mortar and sifted it through a 150 mesh sieve. The boron sample consisted of the crystalline material in the rhombohedral form. To obtain a particle size suitable for the calorimetric experiments, this sample was also ground in an agate mortar and sifted through a 150 mesh sieve.

The two components were carefully mixed in the appropriate molar ratio, pressed into 4 mm pellets and dropped into the calorimeter from room temperature. In a subsequent set of experiments the reaction products were dropped into the calorimeter from room temperature to measure their heat contents. Between the two sets of experiments the samples were kept in a vacuum desiccator to prevent reaction with oxygen or moisture.

Calibration of the calorimeter was achieved by dropping weighed segments of 2 mm diameter high purity copper wire from room temperature into the calorimeter at 1473 ± 2 K. The enthalpy of pure copper at this temperature, $46\,465$ J (mol atoms)$^{-1}$, was obtained from Hultgren et al. [28]. The calibrations were reproducible to within $\pm 1.2\%$.

The reacted samples were examined by X-ray diffraction to assess their structures and to ascertain the absence of unreacted metals. Some of the samples were also subjected to scanning electron microscopy and X-ray microprobe analyses. As we noted in an earlier communication [6], the rare earth carbides, since they decompose on mounting, cannot be tested by these methods.

There is no established phase diagram for the Tb–C system. The eutectic temperature of TbC_2–C has been measured as 2275 ± 20 °C [18]. However, in an earlier paper the melting point of TbC_2 was reported to be 2100 ± 60 °C [13]; TbC_2 undergoes a structural transformation from tetragonal to cubic at 1288 ± 4 °C [18]. We prepared TbC_2 using the procedure which we described in detail for other rare earth carbides in our earlier communication [6]. The diffraction pattern matched well that of the low temperature tetragonal modification listed in the ASTM powder diffraction file. There was no evidence for the presence of any unreacted metal. However, we observed about 5%–7% of Tb_2C_3. In our sample we also found a small amount of the high temperature, cubic modification of TbC_2. The X-ray diffraction pattern of this modification is not listed in the ASTM powder diffraction file, and its atomic coordinates are not available [20]. However, we generated a pattern from its unit cell parameter and the atomic coordinates of its structural prototype, CaF_2 [20].

The phase diagram of the Tb–Si system is not known; however, the existence of Tb_5Si_3, $TbSi_2$ and TbSi has been established [11]. There is no information about the melting points of these compounds, but their structures are known [14–16]. We prepared all three compounds in the calorimeter. The X-ray diffraction patterns of $TbSi_2$ and Tb_5Si_3 matched well the patterns in the ASTM powder diffraction file. The pattern for TbSi is not listed in this file, and its atomic coordinates are not available [20]. However, the X-ray diffraction pattern of our sample agreed well with the pattern generated from its unit cell parameters and the atomic coordinates of its structural prototype FeB. In the pattern of $TbSi_2$ we observed about 1% oxide, and in Tb_5Si_3 one minor unidentified peak estimated at about 3%. The X-ray diffraction pattern of TbSi was tested immediately after the experiment was completed and the sample was found to be essentially single phase. However, when the test was repeated one week later, we found about 5% of $TbSi_2$.

The phase diagram of the Tb–Ge system exhibits one congruently melting compound, Tb_5Ge_3, which melts at 1900 °C [11]. The X-ray diffraction pattern of our sample matched well the pattern in the ASTM powder diffraction file. There was no evidence of unreacted metal or even of oxide.

The phase diagram of the Tb–Sn system displays one congruently melting compound, Tb_5Sn_3, which melts at 1850 °C [12]. However, this diagram is not

included in Massalski et al.'s compilation [11]. Its X-ray diffraction pattern was not included in the ASTM powder diffraction file, and the atomic coordinates were not available [20]. The X-ray diffraction pattern of our sample agreed well with the pattern generated from the unit cell parameters and the atomic coordinates of its structural prototype. Mn_5Si_3 [20]. There was no evidence for the presence of unreacted metal or other alloy phases; however, we found 1%–2% of oxide. Since this sample was very sensitive to oxidation by air during the X-ray diffraction analysis, we followed the same practice as with the carbide samples, and prepared the Tb_5Sn_3 powder for X-ray diffraction under a layer of vaseline [6].

The phase diagram of the Tb–B system exhibits two congruently melting phases, TbB_4, which melts at 2600 °C, and TbB_2, which melts at 2100 °C [11]. We prepared both compounds in the calorimeter. However, the X-ray diffraction pattern of our sample of TbB_4 indicated a mixed phase. Apart from the major component, we found nearly 40% of TbB_2 and 8%–10% of TbB_6. On the contrary, the X-ray diffraction pattern of our sample of TbB_2 (AlB_2 type) matched well the pattern in the ASTM powder diffraction file. There was no evidence for the presence of unreacted metal, of oxide or of TbB_6. However, we found about 5% of TbB_4.

3. Results and discussion

The standard enthalpies of formation of the rare earth alloys determined in this study were obtained as the difference between the results of two sets of measurements. In the first set the following reaction takes place in the calorimeter:
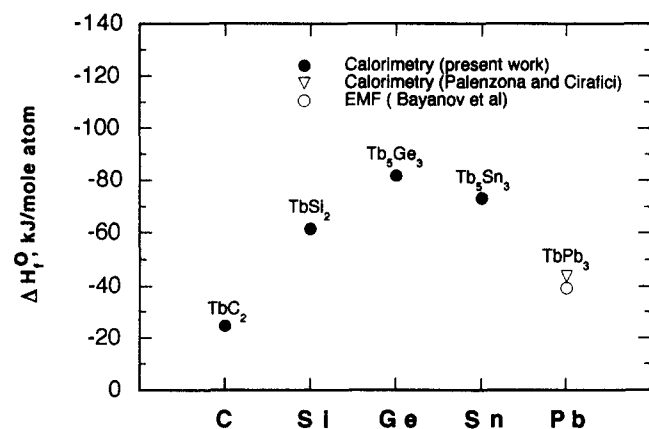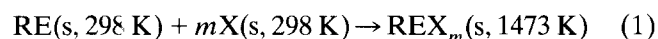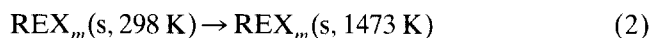
$$RE(s, 298\ K) + mX(s, 298\ K) \rightarrow REX_m(s, 1473\ K) \qquad (1)$$

Here m represents the molar ratio X:RE and RE is the considered rare earth metal, while X represents C, Si, Ge, Sn, or B and s denotes solid. The reacted pellets were reused in a subsequent set of measurements to determine their heat contents:

$$REX_m(s, 298\ K) \rightarrow REX_m(s, 1473\ K) \qquad (2)$$

The standard enthalpy of formation is given by

$$\Delta H_f^0 = \Delta H(1) - \Delta H(2) \qquad (3)$$

where $\Delta H(1)$ and $\Delta H(2)$ are the enthalpy changes per mole of atoms of the compound associated with reactions (1) and (2).

The experimental results are summarized in Table 1. The heat effects associated with reactions (1) and (2) are given in kilojoules per mole of atoms as the average of 5–7 consecutive measurements with the appropriate standard deviations. The last column shows the standard enthalpy of formation of the considered phases. The standard deviations given in the last column also reflect small contributions from the uncertainties in the calibrations.

Table 2 compares the measured standard enthalpies of formation reported in the present work with some experimental values from the published literature and with predicted values from the semiempirical model of Miedema and coworkers [24]. Note that the only published enthalpies of formation we found for the considered binary systems were for the compound TbB_6. The reported values for this compound were derived from e.m.f. measurements by Timofeev and Timofeeva and from mass spectrometric data by Gordienko et al. and by Krikorian [21–23]. As Table 2 shows, there is considerable scatter in the derived values.

Our values for the enthalpies of formation of TbC_2, $TbSi_2$ and $TbSi$ compare well with the values for the corresponding compounds of the early lanthanide elements reported by the present authors [6–10]. The new value for Tb_5Si_3 (-61.0 ± 2.5 kJ (mole atoms)$^{-1}$) is slightly less exothermic than our earlier value for

Fig. 1. Standard enthalpies of formation for characteristic compounds of Tb with group IVB elements, C, Si, Ge, Sn and Pb.

Table 1
Summary of the measured standard enthalpies of formation for some Tb compounds (data in kilojoules per mole of atoms)

Compound	$\Delta H(1)$	$\Delta H(1)$	ΔH_f^0
TbC_2	$+1.2 \pm 0.2\ (7)$	$25.8 \pm 0.8\ (6)$	-24.6 ± 1.0
$TbSi$	$-49.7 \pm 1.0\ (5)$	$31.2 \pm 1.5\ (5)$	-80.9 ± 1.8
$TbSi_2$	$-27.7 \pm 1.6\ (6)$	$33.8 \pm 1.7\ (6)$	-61.5 ± 2.4
Tb_5Si_3	$-31.3 \pm 2.2\ (6)$	$29.7 \pm 0.8\ (6)$	-61.0 ± 2.5
Tb_5Ge_3	$-51.9 \pm 2.3\ (6)$	$29.8 \pm 1.0\ (6)$	-81.7 ± 2.7
Tb_5Sn_3	$-38.1 \pm 2.6\ (5)$	$35.0 \pm 1.2\ (5)$	-73.1 ± 3.1
TbB_2	$-2.8 \pm 1.1\ (6)$	$31.4 \pm 1.3\ (6)$	-34.2 ± 1.8

The numbers in parentheses indicate the numbers of experiments averaged.

Table 2
Comparison of the measured standard enthalpies of formation with some experimental data in the literature and with predicted values from the semiempirical model of Miedema and coworkers [24] (data in kilojoules per mole of atoms)

Compound	(°C)	ΔH_f^0 (experimental), this study	ΔH_f^0 (experimental), literature	Method	ΔH_f^0 (predicted)
TbC$_2$	2100 ± 60	−24.6 ± 1.0	–	–	−66
TbSi		−80.9 ± 1.8	–	–	−79
TbSi$_2$		−61.5 ± 2.4	–	–	−68
Tb$_5$Si$_3$		−61.0 ± 2.4	–	–	−65
Tb$_5$Ge$_3$	1900	−81.7 ± 2.7	–	–	−74
Tb$_5$Sn$_3$	1850	−73.1 ± 3.1	–	–	−81
TbB$_2$	2100	−34.2 ± 1.8	–	–	−67
TbB$_6$	2340	–	−70.4	E.m.f. [21]	∼20
			−23.7 ± 6.6	Mass spectrometry [22]	
			−43	assmt.	

Gd$_5$Si$_3$ (−68.0 ± 2.3 kJ (mol atoms)$^{-1}$ [8]) and the value for Lu$_5$Si$_3$ (−67.8 ± 3.3 kJ (mol atoms)$^{-1}$) measured by Topor and Kleppa [4]. Since the melting points of Lu$_5$Si$_3$ and Tb$_5$Si$_3$ are not known, we cannot make a comparison.

Our new value for the enthalpy of formation of Tb$_5$Ge$_3$ (−81.7 ± 2.7 kJ (mol atoms)$^{-1}$; melting point, 1900 °C) is nearly identical to that of Gd$_5$Ge$_3$ (−82.0 ± 2.6 kJ (mol atoms)$^{-1}$; melting point, 1790 °C [8]). The other germanides which we have studied so far, all have considerably lower melting points (1500–1600 °C). They also have somewhat lower exothermic heats of formation [7,8].

The enthalpy of formation of Tb$_5$Sn$_3$ (−73.1 ± 3.1 kJ (mol atoms)$^{-1}$) is nearly identical with our earlier value for Lu$_5$Sn$_3$ (−73.6 ± 1.4 kJ (mol atoms)$^{-1}$ [10]). It is is also quite comparable with the corresponding values for La$_5$Sn$_3$ and Ce$_5$Sn$_3$ (−77.4 ± 2.1 kJ (mol atoms)$^{-1}$ and −73.2 ± 4.2 kJ (mol atoms)$^{-1}$ respectively) reported by Borzone et al. [29,30] and (−73.2 ± 4.2, −75.3 ± 4.2 kJ (mol atoms)$^{-1}$) by Borsese et al. [31].

Our new value for the hat of formation of TbB$_2$ (−34.2 ± 1.8 kJ (mol atoms)$^{-1}$) compares well with the corresponding values for other rare earth diborides, e.g. LuB$_2$ (−29.8 ± 0.9 kJ (mol atoms)$^{-1}$) and YB$_2$ (−35.7 ± 2.6 kJ (mol atoms)$^{-1}$), both measured by the present authors [10,32]. Note that the enthalpies of formation of rare earth diborides typically are considerably less exothermic than the values for the tetraborides and hexaborides of the earlier lanthanide metals [9]. This reflects the difference in structure, and also correlates with the significantly lower melting points of the diborides compared with the tetra- and hexaborides.

As Table 2 shows, the predicted enthalpies of formation are in reasonable agreement with our new measurements for TbSi$_2$, Tb$_5$Si$_3$, TbSi, TbS$_5$Ge$_3$ and

Tb$_5$Sn$_3$. We noted already in our earlier communications that the predicted values for the rare earth carbides and borides are considerably more exothermic than our experimental values [6–10].

In Fig. 1 we compare our new enthalpies of formation for the Tb carbides, silicides, germanides and stannides with earlier values for alloys of Tb with Pb. One of the plotted values for the heat of formation of TbPb$_3$ was measured by calorimetry by Palenzona and Cirafici [33]. A very similar value was reported in the e.m.f. study by Bayanov et al. [34]. We noted in our earlier communications that the enthalpies of formation of the compounds of early lanthanide elements, and Lu with the IVB elements in the periodic table, show a roughly parabolic correlation [7,8,10]. Fig. 1 shows that a very similar correlation exists also for the Tb compounds. Again, the germanide has the most exothermic heat of formation.

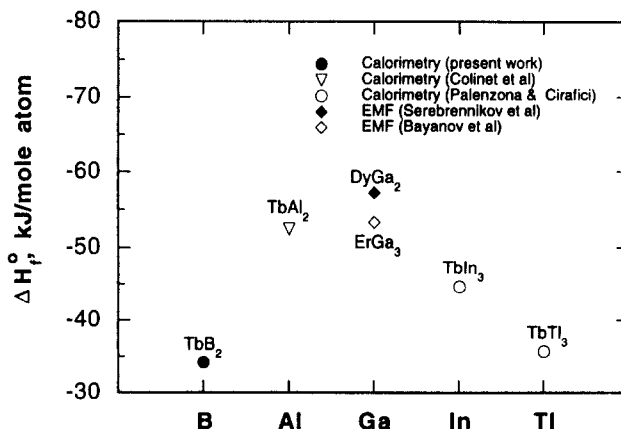In Fig. 2 we compare our enthalpy of formation for

Fig. 2. Standard enthalpies of formation for characteristic compounds of Tb with group IIIB elements, B, Al, Ga, In and Tl. The value for DyGa$_2$ represents ΔG_f^0 obtained by Serebrennikov et al. [38].

TbB$_2$ with earlier calorimetric values for the compounds of Tb with the other group IIIB elements Al, In and Tl. The value for the heat of formation of TbAl$_2$ was measured by Colinet et al. by high temperature solution calorimetry [35]. The corresponding values for TbIn$_3$ and TbTl$_3$ were determined by Palenzona and Cirafici by direct synthesis calorimetry [36]. We found no experimental heat of formation value for any alloy in the Tb–Ga system. The compound TbGa$_2$ is listed in *Chemical Abstracts*; however, no thermochemical information is available. However, the heats and free energies of formation of the corresponding Dy and Er gallides have been derived from e.m.f. data by Bayanov et al. and by Serebrennikov et al. respectively [37,38]. As an approximate value, we have plotted ΔG_f^0 for DyGa$_2$ and ΔH_f^0 for ErGa$_3$. In analogy to the corresponding compounds of the earlier lanthanide elements the enthalpies of formation of the considered compounds also exhibit a roughly parabolic correlation when plotted against the IIIB elements [9].

It was suggested by Gschneidner that the enthalpies of formation of rare earth alloys, their reduced melting temperatures and their relative molar volumes should be related and all reflect the lanthanide contraction in the compounds compared with the contraction in the pure metals [25,26]. For example, if the lanthanide contractions in the compounds from La to Lu become greater than in the corresponding metals, then the enthalpies of formation are predicted to become more exothermic in the same sequence. In this scheme the reduced temperature (i.e. the ratio of the melting point of the compound to the melting point of the rare earth element), and the ratio of the molar volume of the compound to the atomic volume of the element, normalized with respect to the ratio for the first member of the series (i.e., La), are used as indicators to predict the systematic behavior of the stability of the alloys.

In Fig. 3 we compare the systematic changes in the reduced melting point ratios, in the relative molar volume ratios and in the experimental standard enthalpies of formation for the REC$_2$ systems for which data are now available. This figure shows that the reduced melting point ratio systematically decreases when plotted against the atomic number. However, we do not find a comparable decrease in the corresponding plot of the enthalpies of formation. On the contrary, the relative molar volume ratios, with the exceptions of the values for EuC$_2$ and YbC$_2$, are nearly constant, or may decline slightly, when plotted against the atomic number. Our experimental enthalpy of formation values suggest that ΔH_f^0 for REC$_2$ also is very nearly constant from La to Tb and for Lu. Hence, a prediction based on the relative molar volume ratios
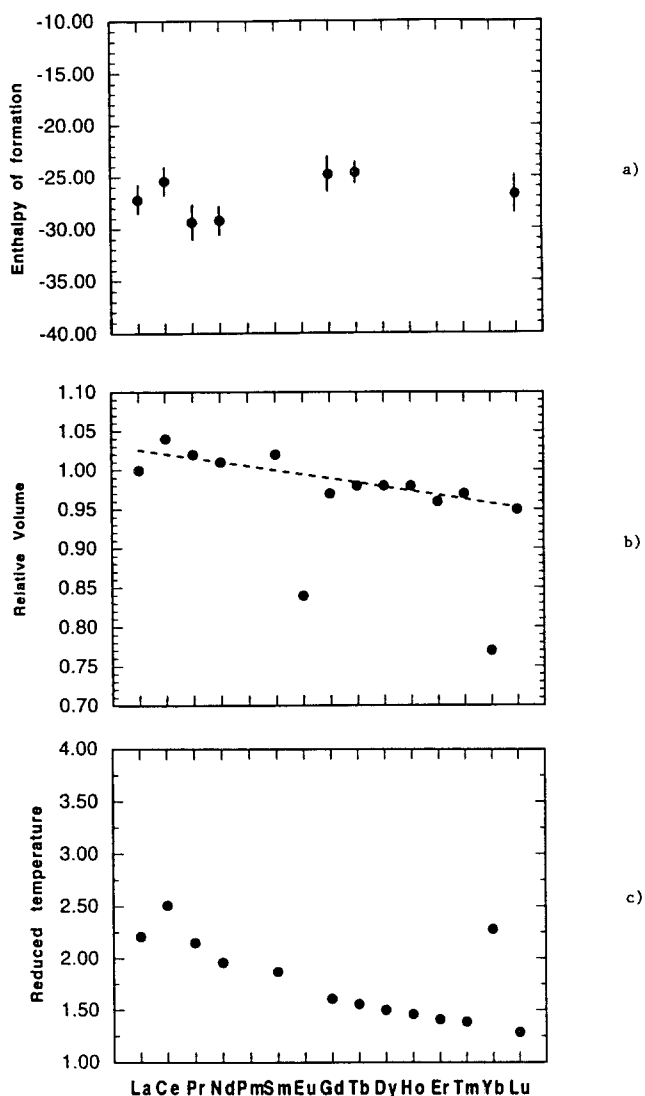

Fig. 3. Some selected properties of REC$_2$ compounds plotted against the atomic number of RE; (a) standard enthalpy ΔH_f^0 (kJ (mol atoms)$^{-1}$) of formation; (b) relative molar volume V_m = $[V(REC_2)/V(RE)]/[V(LaC_2)/V(La)]$; (c) reduced temperature T_r = $T_m(REC_2)/T_m(RE)$.

agrees reasonably well with our experimental data for the considered REC$_2$ system. We found roughly comparable agreement for the so far studied RESi$_2$, RESi, and RE$_5$Sn$_3$ systems.

Acknowledgements

the X-ray diffraction patterns of TbSi and Tb_5Sn_3 from the reported unit cell parameters and the appropriate atomic coordinates.

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