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J. Chem. Thermodynamics **1986**, 18, 727–734

Thermodynamic stabilities of TeO_2 and Sb_2Te_3 by a solid-oxide electrolyte e.m.f. technique

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(Received 30 September 1985; in revised form 9 December 1985)

The thermodynamic stabilities of $TeO_2(s)$ and $Sb_2Te_3(s)$ were determined by the solid-electrolyte technique using the following galvanic cells in the temperature ranges 588 to 722 K and 726 to 898 K for $TeO_2(s)$ and 645 to 712 K for $Sb_2Te_3(s)$:

$$\mathrm{Pt|C|Te(s)|TeO_2(s)|YSZ|air}\{p(\mathrm{O_2}) = 0.21p^\circ\}\mathrm{|Pt}, \qquad \text{(I)}$$

$$\mathrm{Pt|C|Te(l)|TeO_2(s)|YSZ|air}\{p(\mathrm{O_2}) = 0.21p^\circ\}\mathrm{|Pt}, \qquad \text{(II)}$$

$$\mathrm{Pt|C|Sb_2Te_3(s)|Te(s)|Sb_2O_3(s)|YSZ|air}\{p(\mathrm{O_2}) = 0.21p^\circ\}\mathrm{|Pt}, \qquad \text{(III)}$$

where $p^\circ = 101325$ Pa and YSZ denotes 15 mass per cent of Y_2O_3 stabilizing ZrO_2. The standard molar Gibbs energies of formation calculated from the above cells are:

$$\Delta_f G_m^\circ(\mathrm{TeO_2, s})/(\mathrm{kJ \cdot mol^{-1}}) = -(323.28 - 0.17600T/\mathrm{K}) \pm 1.21, \quad (588 \text{ to } 722\ \mathrm{K});$$

$$\Delta_f G_m^\circ(\mathrm{TeO_2, s})/(\mathrm{kJ \cdot mol^{-1}}) = -(339.31 - 0.19861T/\mathrm{K}) \pm 0.60, \quad (726 \text{ to } 898\ \mathrm{K});$$

$$\Delta_f G_m^\circ(\mathrm{Sb_2Te_3, s})/(\mathrm{kJ \cdot mol^{-1}}) = -(91.57 - 0.04339T/\mathrm{K}) \pm 1.20, \quad (645 \text{ to } 712\ \mathrm{K}).$$

From these results, values of $-(321.11 \pm 1.29)\ \mathrm{kJ \cdot mol^{-1}}$, $71.51\ \mathrm{J \cdot K^{-1} \cdot mol^{-1}}$, and $16.0\ \mathrm{kJ \cdot mol^{-1}}$ were computed for $\Delta_f H_m^\circ(\mathrm{TeO_2, s, 298.15\ K})$, $S_m^\circ(\mathrm{TeO_2, s, 298.15\ K})$, and $\Delta_{fus} H_m^\circ(\mathrm{Te, s})$, respectively, and were compared with literature values.

1. Introduction

The interaction of tellurium, an important fission product, with other fission products and also with the cladding of the fuel pins is of interest to designers of fast nuclear reactors.(1) Besides this, Te(s) is also a daughter product of decay of ^{124}Sb formed in the (antimony sesquioxide + beryllium) assembly which is used as a neutron start-up facility for fast reactors.(2) Hence the thermodynamic stabilities of $TeO_2(s)$ and $Sb_2Te_3(s)$ are of interest to fast-reactor designers especially in the temperature range 600 to 900 K. In addition, $Sb_2Te_3(s)$ is a narrow-energy-gap semiconductor(3) of interest to materials scientists. The standard molar Gibbs energy of formation $\Delta_f G_m^\circ$ of $TeO_2(s)$ was determined using a solid-oxide electrolyte e.m.f. method with air as reference electrode by Chatterji and Smith(4) and also by Otsuka and Kozuka(5) over the temperature ranges 793 to 993 K and 733 to 873 K, respectively. Both these measurements cover a temperature range which is above the melting temperature of Te(s). There has been no reliable measurement of $\Delta_f G_m^\circ$ of

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TeO_2(s) in the temperature range 600 to 723 K, the melting temperature of Te(s). However, values of $\Delta_f G^\circ_m(TeO_2, s, 298.15\ K)$ had been reported from adiabatic calorimetry by Rabinovich *et al.*(6) and Pashinkin *et al.*(7) and from aqueous electrolyte e.m.f. studies by Schuhmann.(8) In the present work, $\Delta_f G^\circ_m(TeO_2, s)$ coexisting with Te(s) from 588 to 722 K and with Te(l) from 726 to 898 K is reported from oxide-electrolyte e.m.f. measurements with air as the reference electrode material. For Sb_2Te_3(s), standard molar Gibbs energies are rather scanty. For instance, Voronin and Degtyarev(9) had to compute $\Delta_f G^\circ_m(Sb_2Te_3, s)$ from calorimetric results and phase diagrams. They had also compared their results with those from e.m.f. measurements by Semenkovich and Melekh(10) at 540 K and by Gerasimov and Nikol'skaya(11) at 673 K. Other studies reported on Sb_2Te_3(s) were mainly calorimetric.(12–16) In the present investigation $\Delta_f G^\circ_m$ of Sb_2Te_3(s) relative to Te(s) and Sb(s) was determined over the temperature range 645 to 712 K.

2. Experimental

TeO_2(s) was prepared by heating Te granules (purity greater than 99.99 mass per cent, supplied by B.D.H., U.K.) in air at 673 K for 3 to 5 h. The intermetallic Sb_2Te_3(s) was synthesized by mixing Sb powder (purity greater than 99.999 mass per cent, supplied by Johnson Matthey Chemicals, U.K.) with Te granules in the required stoichiometric amounts. This mixture was then compacted into pellets of dimensions 12.7 mm in diameter and 5 mm thick at a pressure of 100 MPa, followed by sintering at 573 K for 120 h and 623 K for 10 h in evacuated and sealed silica ampoules. Sb_2Te_3(s) and TeO_2(s) were shown to be pure within the limits of detection by X-ray diffractometry.

Mixtures of $(0.833Te+0.167TeO_2)$ and $(0.330Sb_2Te_3+0.404Te+0.266Sb_2O_3)$ were compacted into pellets of dimensions 6 mm in diameter and 3 to 4 mm thick at a pressure of 100 MPa. Graphite cups were used to contain the electrode pellets so as to prevent chemical attack of the Pt lead wire by the constituents of the electrode. Both the Pt leads on either side of the cell were located in the isothermal zone of the furnace; the absence of asymmetric potentials had been verified earlier in similar cell configurations.(17)

The following galvanic cells were used in the present study:

$$\mathrm{Pt|C|Te(s)|TeO_2(s)|YSZ|air}\{p(\mathrm{O_2}) = 0.21p^\circ\}\mathrm{|Pt}, \tag{I}$$

$$\mathrm{Pt|C|Te(l)|TeO_2(s)|YSZ|air}\{p(\mathrm{O_2}) = 0.21p^\circ\}\mathrm{|Pt}, \tag{II}$$

$$\mathrm{Pt|C|Sb_2Te_3(s)|Te(s)|Sb_2O_3(s)|YSZ|air}\{p(\mathrm{O_2}) = 0.21p^\circ\}\mathrm{|Pt}, \tag{III}$$

where $p^\circ = 101325$ Pa and YSZ denotes 15 mass per cent of Y_2O_3 stabilizing ZrO_2. The solid-electrolyte tube (Corning, U.S.A.) with dimensions 9.5 mm i.d., 12.7 mm o.d., and 305 mm long was used to separate the gaseous environments of the test electrode and air-reference electrode compartments. Ar(g) purified as described elsewhere(18) was used as a cover gas for the test electrode. The temperature of the cell was measured with a Pt-to-(Pt+10 mass per cent of Rh) thermocouple which had been calibrated at the freezing temperatures of Sn, Zn, Sb, and Ag. The e.m.f.

readings were taken after equilibrating the galvanic cell at 700 K for at least 24 h when the drift in the cell potential was found to be $<3\times10^{-8}$ $V\cdot s^{-1}$ and this drift was only a random one caused by fluctuations in the temperature. Cell III generally required longer periods of initial equilibration. The e.m.f. readings were found to be reproducible only during the heating cycles. After the initial equilibration at 700 K, the cell was cooled to the lowest temperature of the e.m.f. measurement and then was heated in steps of 10 to 20 K for each isothermal reading of cell potential. At least three independent runs with fresh electrode pellets were carried out for each cell.

3. Results

The e.m.f. values for cells I to III are shown in figure 1. The least-squares expressions derived for cells I, II, and III are

$$E(\mathrm{I})/\mathrm{mV} = (837.63-0.48972T/\mathrm{K})\pm3.14, \quad (588 \text{ to } 722 \text{ K}); \quad (1)$$

$$E(\mathrm{II})/\mathrm{mV} = (879.17-0.54830T/\mathrm{K})\pm1.44, \quad (726 \text{ to } 898 \text{ K}); \quad (2)$$

$$E(\mathrm{III})/\mathrm{mV} = (1027.69-0.38396T/\mathrm{K})\pm0.80, \quad (645 \text{ to } 712 \text{ K}). \quad (3)$$

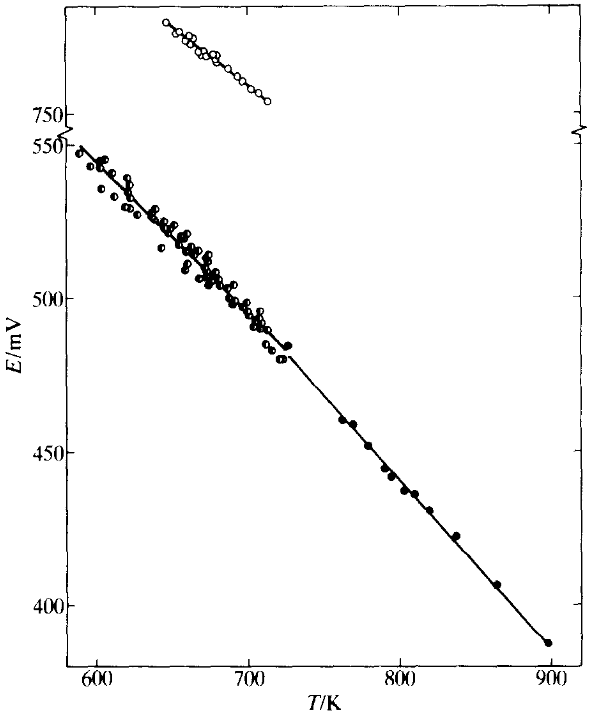

FIGURE 1. Experimental e.m.f. E results for cells I, II, and III:

◐, Cell I: Pt|C|Te(s)|TeO_2(s)|YSZ|air{$p(O_2) = 0.21p°$}|Pt;

●, Cell II: Pt|C|Te(l)|TeO_2(s)|YSZ|air{$p(O_2) = 0.21p°$}|Pt;

○, Cell III: Pt|C|Sb_2Te_3(s)|Te(s)|Sb_2O_3(s)|YSZ|air{$p(O_2) = 0.21p°$}|Pt;

where $p° = 101325$ Pa and YSZ denotes 15 mass per cent of Y_2O_3 stabilizing ZrO_2.

4. Discussion

The reaction in cell I for the passage of four electrons is

$$\mathrm{Te(s)+O_2(g) = TeO_2(s)}. \tag{4}$$

The e.m.f. given by reaction (4) was corrected for the standard state of oxygen in the air reference electrode, yielding for the standard molar Gibbs energy of formation of TeO_2 (588 to 722 K):

$$\Delta_f G^\circ_m(\mathrm{TeO_2, s})/(\mathrm{kJ \cdot mol^{-1}}) = -(323.28-0.17600T/\mathrm{K}) \pm 1.21. \tag{5}$$

To assess the temperature-dependent errors in the above results and to check the consistency with the calorimetric results, a third-law treatment was used to compute $\Delta_f H^\circ_m$ at 298.15 K. For this purpose, values of $-(G^\circ_m - H^\circ_m)/T$ for $TeO_2(s)$ were constructed from the molar heat capacity $C^\circ_{p,m}$ and the standard entropy S°_m(298.15 K) given by Barin and Knacke.(19) Likewise, values from Hultgren *et al.*(20) were used for Te(s) and $O_2(g)$. The values for $-(G^\circ_m - H^\circ_m)/T$ at a few interpolated temperatures along with the change in $-(G^\circ_m - H^\circ_m)/T$ for reaction (4) are listed in table 1. The values of $\Delta_f H^\circ_m(\mathrm{TeO_2}$, s, 298.15 K) computed from the results on cell I are plotted against temperature in figure 2. This third-law plot confirmed the absence of significant temperature-dependent errors in the e.m.f. values. An average of $-(321.11 \pm 1.29)$ $\mathrm{kJ \cdot mol^{-1}}$ was calculated for $\Delta_f H^\circ_m(\mathrm{TeO_2}$, s) at 298.15 K. This value is in good agreement with those given in thermochemical tables(19, 21, 22) and with some of the recent experimental measurements(4, 6–8, 23–26) as shown in table 2. Equation (5) was combined with the relevant $C^\circ_{p,m}$ values, integrated from 298.15 to 655 K (the mean temperature of the present galvanic-cell measurements) so as to yield the change in the standard molar entropy for the formation reaction (4). A value of -183.02 $\mathrm{J \cdot K^{-1} \cdot mol^{-1}}$ was thus obtained for $\Delta_f S^\circ_m$(298.15 K) of $TeO_2(s)$. Combining that value with the values of 49.50 and 205.04 $\mathrm{J \cdot K^{-1} \cdot mol^{-1}}$ for S°_m of Te(s) and $O_2(g)$, respectively, at 298.15 K from Barin and Knacke,(19) a value of 71.51 $\mathrm{J \cdot K^{-1} \cdot mol^{-1}}$ was computed for S°_m(298.15 K) of $TeO_2(s)$, which is compared with the literature values(4, 6, 19, 21, 22, 27) in table 2.

TABLE 1. Values of $-(G^\circ_m - H^\circ_m)/T$ used for the computation of $\Delta_f H^\circ_m(\mathrm{TeO_2}$, s, 298.15 K)

T/K	$-(G^\circ_m - H^\circ_m)/T$ / $\mathrm{J \cdot K^{-1} \cdot mol^{-1}}$			$\Delta\{(G^\circ_m - H^\circ_m)/T\}$ / $\mathrm{J \cdot K^{-1} \cdot mol^{-1}}$
	Te(s)[a]	$O_2(g)$[a]	$TeO_2(s)$[b]	
550	54.33	209.94	89.82	174.45
600	54.94	210.94	92.70	173.18
700	57.40	213.50	98.48	172.42
723	57.95	214.79	99.80	172.94

[a] Values taken from Hultgren *et al.*(20)

[b] Computed from S°_m(298.15 K) and $C^\circ_{p,m}$ given by Barin and Knacke;(19) for reaction (4) the least-squares expression for the change in $-(G^\circ_m - H^\circ_m)/T$ is

$$\Delta\{(G^\circ_m - H^\circ_m)/T\}/(\mathrm{J \cdot K^{-1} \cdot mol^{-1}}) = (181.12-0.01260T/\mathrm{K}), \quad (550 \text{ to } 723 \text{ K}).$$

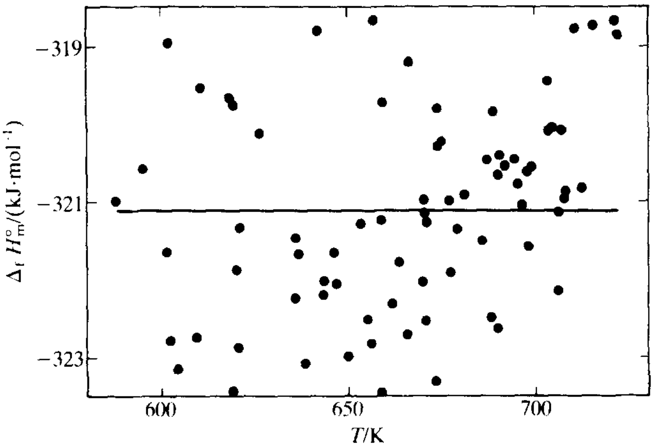

FIGURE 2. Third-law plot of the enthalpy of formation $\Delta_f H^\circ_m(TeO_2, s, 298.15\ K)$: the horizontal line is the mean result.

Above the melting temperature of Te, 723 K,(19) $\Delta_f G^\circ_m(TeO_2, s)$ could be calculated as follows from the expression for the e.m.f. of cell II (after correcting for the standard state of oxygen in the air reference electrode):

$$\Delta_f G^\circ_m(TeO_2, s)/(kJ \cdot mol^{-1}) = -(339.31 - 0.19861\,T/K) \pm 0.60, \quad (726 \text{ to } 898\ K). \quad (6)$$

The consistency of equation (6) with equation (5) can be checked by calculating the molar enthalpy of fusion $\Delta_{fus} H^\circ_m$ of Te(s) which was found to be 16.0 $kJ \cdot mol^{-1}$. This value is in good agreement with 17.5 $kJ \cdot mol^{-1}$ from thermochemical tables.(19, 20) The e.m.f. lines for cells I and II in figure 1 intersect at 710 K which is somewhat lower than the accepted melting temperature of Te(s). The partial phase diagram of (tellurium + oxygen)(28) showed that the melting temperature of Te(s) was unaffected by the presence of TeO_2(s). Since an error of about $\pm 1.0\ J \cdot K^{-1} \cdot mol^{-1}$ in the slope of equation (6) could affect the point of intersection by more than 10 K, the internal consistency between the results from

TABLE 2. The third-law standard molar enthalpy of formation and standard molar entropy of TeO_2(s) at 298.15 K

$\frac{-\Delta_f H^\circ_m}{kJ \cdot mol^{-1}}$	Reference	$\frac{-\Delta_f H^\circ_m}{kJ \cdot mol^{-1}}$	Reference	$\frac{S^\circ_m}{J \cdot K^{-1} \cdot mol^{-1}}$	Reference
321.11 ± 1.29	This work	321.75 ± 5.02	23	71.51	This work
322.59	19	379.07 ± 1.26	24	79.50	19
323.42 ± 4.18	21	351.90 ± 11.80	25	74.06	21
325.06	22	316.40 ± 1.10	26	71.09	22
301.25 ± 2.09	4			97.91	4
321.75 ± 5.02	6			70.13	6
325.10	8			58.58	27

TABLE 3. Comparison of standard molar Gibbs energy of formation of $TeO_2(s)$ coexisting with Te(l) from e.m.f. measurements: $\Delta_f G^\circ_m/(kJ \cdot mol^{-1}) = A + B(T/K)$

$\frac{T}{K}$	A	B	$\frac{\Delta_f G^\circ_m(800\ K)}{kJ \cdot mol^{-1}}$	Reference
588 to 722	−323.28	0.17600		This work
722 to 898	−339.31	0.19861	−180.42	This work
793 to 993	−314.30	0.17230	−176.46	4
733 to 873	−334.40	0.19460	−178.72	5

cells I and II is considered reasonable. The $\Delta_f G^\circ_m$ values from the present study are compared with those from Chatterji and Smith(4) and Otsuka and Kozuka(5) in table 3. The agreement is quite good.

To determine the activity of Sb in a mixture of (antimony sesquitelluride + tellurium), it would be desirable to measure the e.m.f. of the cell:

$$\mathrm{Pt|C|Sb(s)|Sb_2O_3(s)|YSZ|Sb_2O_3(s)|Sb_2Te_3(s)|Te(s)|C|Pt}. \qquad \text{(IV)}$$

Using the same experimental set-up Azad *et al.*(18) measured the e.m.f. of the cell:

$$\mathrm{Pt|C|Sb(s)|Sb_2O_3(s)|YSZ|air}\{p(O_2) = 0.21p^\circ\}\mathrm{|Pt}, \qquad \text{(V)}$$

and reported

$$E(V)/mV = (1185.86 - 0.45891T/K) \pm 1.2, \quad (662 \text{ to } 890 \text{ K}). \qquad (7)$$

Combining equations (3) and (7), $\Delta_f G^\circ_m(Sb_2Te_3, s)$ may be calculated for the reaction:

$$2Sb(s) + 3Te(s) = Sb_2Te_3(s), \qquad (8)$$

for the passage of six electrons and is given by

$$\Delta_f G^\circ_m(Sb_2Te_3, s)/(kJ \cdot mol^{-1}) = -(91.57 - 0.04339T/K) \pm 1.20, \quad (645 \text{ to } 712 \text{ K}). \qquad (9)$$

A value of 1.2 $kJ \cdot mol^{-1}$ is computed as the uncertainty in $\Delta_f G^\circ_m(Sb_2Te_3, s)$ by adding the uncertainties: 1.2 and 0.8 mV in the potentials of cells III and V respectively, and multiplying by $6F$. Such a small uncertainty limit could be achieved because of the absolute nature of cells III and V which use air as the reference electrode material. Possible systematic errors were eliminated by using the same experimental set-up. Further, absence of asymmetric and thermoelectric potentials in cell V was verified as reported earlier.(18) In view of these an overall precision of ±1.2 $kJ \cdot mol^{-1}$ could be achieved for $\Delta_f G^\circ_m(Sb_2Te_3, s)$ in this study as compared with that of 4.2 $kJ \cdot mol^{-1}$ for $Sb_2O_3(s)$ compiled by Kubaschewski and Alcock.(21) Equation (9) is valid only over a narrow range of temperature and is of limited application in extrapolating over very wide temperature ranges. Hence no third-law treatment was attempted. However, equation (9) yields values of −68.1 and −62.3 $kJ \cdot mol^{-1}$ for $\Delta_f G^\circ_m(Sb_2Te_3, s)$ at 540 and 673 K respectively which compare well with the value of −64.5 $kJ \cdot mol^{-1}$ at 540 K by Semenkovich and

Melekh⁽¹⁰⁾ and $-61.7\ kJ \cdot mol^{-1}$ at 673 K by Gerasimov and Nikol'skaya.⁽¹¹⁾ Equation (9) also shows reasonable agreement (within $\pm 4\ kJ \cdot mol^{-1}$) with the standard molar Gibbs energy calculated by Voronin and Degtyarev⁽⁹⁾ over the temperature range of the present investigation. The lower limit of temperature for this investigation was restricted both by the high impedance of the solid-electrolyte cells and by the kinetics of the electrochemical equilibrium. The upper bound of temperature is dictated by the eutectic reaction between Sb_2Te_3(s) and Te(s) reported to take place at 697 K.⁽²⁹⁾ However, cell III did not show any evidence for a eutectic reaction up to 712 K.

The authors are grateful to Shri J. B. Gnanamoorthy, Head, Chemical Metallurgy Section, Materials Development Laboratory and to Dr P. Rodriguez, Head, Metallurgy Programme, Reactor Research Centre, Kalpakkam, for their interest and constant encouragement throughout the course of this work. They are also thankful to Shri P. Kuppusamy, Materials Development Laboratory, for the X-ray diffraction work and to Shri M. Arumugam, Materials Development Laboratory, for technical assistance. This paper forms a part of the work to be reported in the thesis of C. Mallika for the Ph.D. degree in the University of Madras.

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