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Reanalysis of Solidus Points and Te Chemical Potential for Te-Rich CdTe(s)

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The author's previously published partial pressures of tellurium over tellurium-rich CdTe are reanalyzed to yield new solidus points as well as temperatures and partial pressures for compositions within the homogeneity range. Although the results are qualitatively the same as before, they are more satisfactory in that some thermodynamic inconsistencies have been removed. The composition range of the CdTe phase extends slightly beyond the equal atom fraction, $X_{Te} = 0.5$, to a Te-rich composition of $(X_{Te} - 0.5) = (1 \text{ to } 7) \times 10^{-5}$. The data are in fair agreement with that obtained from total pressure measurements at generally higher compositions. Since both types of measurement are near the limits of attainable accuracy, their agreement would seem to confirm the essential correctness of both.

Keywords chemical potential, experimental phase equilibria, materials characterization

1. Introduction

Generally, information on the homogeneity range and thermodynamic properties of compound semiconductors is limited to (a) electrical measurements, often only on quenched samples and not at temperature; (b) molar properties independent of exact composition such as heat capacity, enthalpy or Gibbs energy of formation; and (c) partial pressure or chemical potential along the solidus curve. The partial pressures have most often been determined by measuring the optical density of the vapor for the high-temperature solid-liquid-vapor equilibrium in which case the composition of a two-component solid is fixed. The defect structure of the semiconductor compound is then chosen as the simplest possible consistent with the electrical measurements. Tin telluride[1] and bismuth telluride[2] are two exceptions because their homogeneity ranges are a few tenths to 1 at.% wide and samples of known composition can be made for measurement. Cadmium telluride is also a notable exception in that solidus points and tellurium chemical potential data have been obtained from total pressure measurements[3,4] and from partial pressures determined by optical density measurements.[5] These indicate that the homogeneity range extends on the Te-rich side to about $(X_{Te} - 0.5) = 10^{-4}$. High-temperature Hall measurements[6,7] indicate the homogeneity range is about ten times smaller. However, the valence band hole concentrations needed for this conclusion are somewhat uncertain since the higher-mobility electrons dominate the Hall coefficient, and extrapolated values for the hole mobility must be used. If one accepts the low hole concentrations, the simple defect model of Cd vacancy acceptors for Te-rich CdTe must be extended. Most often, Te place exchange donors are assumed, although self-diffusion measurements[8] favor neutral Te interstitials. A homogeneity range for CdTe that is much larger than inferred from electrical measurements is further supported by total pressure measurements[9] on $Cd_{1-x}Zn_xTe$ for x between 0.05 and 0.15. These show the solidus maximum increasing with X_{Zn} and becoming as large as $(X_{Te} - 0.5) = 7 \times 10^{-4}$ at 1073 K. One might expect this trend since the homogeneity range of CdTe includes both Cd- and Te-rich compositions, whereas that for ZnTe lies entirely at X_{Te} greater than 0.5 as determined[10] by high-temperature conductivity measurements under controlled Zn pressure. However, optical density measurements[11] for $X_{Zn} = 0.20$ in the Cd-Zn-Te system give smaller values of only $(X_{Te} - 0.5) = 10^{-4}$, although a maximum may not have been attained.

Currently, CdTe is the subject of much effort to obtain high-resistivity material suitable for x-ray and gamma ray detectors. Information on the basic properties of CdTe is obviously useful to this end. The author believes this is particularly true for the temperature, partial pressure, and composition data, which imply a more complicated defect structure than the simplest one of Cd vacancies for Te-rich material. Because of this and because of unsuccessful efforts on the author's part to simultaneously fit previous temperature-composition data and the hole concentrations for Te-saturated material,[7] the previous analysis has been rexamined. A better way has been chosen to smooth the measured partial pressure to which the calculated composition is extremely sensitive. Although the new results are not grossly different from the previous ones, they seem more firmly based. Except for a set of results at the highest tellurium pressure, the C-shape of the previous T-composition data and some thermodynamic inconsistency is removed. The new results are complementary to results from total pressure measurements[3] in that they cover a composition range closer to 50 at.%, but overlap slightly. Consequently, the basis for greater confidence in the essential correctness of both sets of results for Te-rich CdTe is established.

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Table 1 Cell 92D

Optical cell				CdTe reservoir					
Temperature, K	100 P_2 (obs)	100 P_2 (sm)	(e+4) × P_1	Temperature (T_R), K	100 P_2	P_2, kPa	(e+4) × P_1	P(Cd)	Comp
1267.7	3.020	3.031	6.56	1005.7	3.085	3.126	0.247	4.57e−5	0.60
1269.2	3.050	3.037	0.398	1036.8	3.095	3.136	0.398	1.28e−4	0.70
1269.2	3.000	2.999	6.62	1067.2	3.057	3.097	0.614	3.33e−4	0.91
1265.6	2.970	2.986	6.37	1097.5	3.039	3.079	0.926	8.15e−4	1.08
1273.0	3.070	2.996	6.87	1108.2	3.050	3.090	1.07	1.10e−3	1.13
1272.8	3.050	3.045	6.92	1128.3	3.10	3.141	1.39	1.91e−3	1.16
1271.5	3.000	3.090	6.89	1139.3	3.14	3.182	1.60	2.54e−3	1.16
1274.0	3.250	3.143	7.11	1149.0	3.19	3.232	1.82	3.26e−3	1.15
1270.6	3.080	3.215	6.95	1159.8	3.26	3.303	2.10	4.27e−3	1.13
1270.7	3.280	3.390	7.25	1179.7	3.43	3.475	2.72	6.88e−3	1.07
1260.5	3.700	3.561	6.61	1194.8	3.594	3.642	3.30	9.74e−3	1.02
1269.6	4.100	4.011	7.69	1225.4	4.039	4.092	4.89	1.89e−2	1.13
1269.5	4.500	4.583	8.21	1254.9	4.594	4.655	7.10	3.44e−2	1.64
1269.9	4.600	4.582	8.24	1254.9	4.593	4.654	7.09	3.43e−2	1.64
1269.6	…	2.760	6.38	960.0	2.799	2.837	0.110	9.36e−6	1.09

Note: Partial pressures in the optical cell proper and in the CdTe reservoir. Last column titled Comp is the composition of CdTe(s) in units of (e+5)(X_{Te} − 0.5). In this and Tables 2 to 5, pressure is in atm except for the second P_2 column in the CdTe reservoir part of table, which is in kPa. In the exponential notation 4.57e−5 stands for 4.57 times ten to the minus fifth power. The column heading (e+4) × P_1 means the values in that column are ten thousand times the value of P_1.

2. Analysis

The experimental technique with important experimental details such as the weight of the elements, path lengths of the optical cells, methodology of measurement, and so forth have been reported earlier.[5] Here the author gives only the overall scheme and concentrates on the analysis. The temperature of an optical cell was held fixed, while that of Cd-Te contained in an attached sidearm reservoir was varied over a number of lower temperatures. At each reservoir temperature, the optical density of the vapor in the optical cell was measured over a range of wavelengths and a partial pressure of the diatomic Te_2 species calculated for a number of wavelengths. The calculated values were then averaged. These average values are shown as the second entry, labeled P_2(obs), in Tables 1 through 4 for the different optical cells where P_2 represents the partial pressure of the diatomic species in the overall vapor pressure. Pressures in this paper are in atmospheres (1 atm = 0.10132 MPa) except for P_2 in the reservoir section, which is given in both atm and kPa. The first entry in each table is the optical cell temperature, while the first entry in the CdTe reservoir section is the temperature of the CdTe reservoir T_R. Now the fractional error in values of $10^5(X_{Te} - 0.5)$, calculated as indicated subsequently, is at least 8 to 10 times larger than that in the starting value of P_2. In some instances, values calculated starting with P_2(obs) for closely adjacent values of T_R vary from a smooth curve by a significant fraction of their values. A further smoothing of P_2(obs) is therefore required and justified by the reasonable expectation that the variance of composition with temperature be a smooth one. Here the values of P_2(obs) falling within the CdTe three-phase curve were fit to a cubic in T_R and are listed as the third entry in Tables 1 through 4 and labeled P_2(sm). In the earlier report,[5] this second smoothing was achieved by making a least squares fit of one or two line segments to the logarithm of P_2(obs) as a function of $1/T_R$. However, P_2(obs), rather than its logarithm, is the quantity measured, and it is the quantity on which the calculated composition depends.

In order to calculate the composition of the CdTe in the reservoir, the partial pressures must be known throughout the cell, the connecting stem, and the reservoir. It was assumed earlier[5] and also here that local thermodynamic equilibrium holds, that Te, Te_2, and Te_5 are present and their partial pressures governed by the equilibrium constants given by Mills,[12] and that the partial pressures of Cd and Te_2 in the reservoir where CdTe is present are related by:

$$P_{Cd} P_2^{1/2} = \exp(\Delta_f G^\circ / RT) \quad \text{(Eq 1)}$$

Here $\Delta_f G^\circ$ is the Gibbs energy of formation of CdTe(s) from Cd(g, 1 atm) and Te_2(g, 1 atm) and is given[13] by:

$$\Delta_f G^\circ \text{ (J/mol)} = -287{,}190 + 188.03T \quad \text{(Eq 2)}$$

Finally, it is assumed that, at a given reservoir temperature, the sum of the partial pressures of all Te species is constant throughout the cell and the partial pressure of Cd is also constant. Two alternative calculations were also made, both of which gave values for $10^5(X_{Te} - 0.5)$ within 5% of each other and those from the calculation described previously. In one type of calculation, the sum of the partial pressures of all the species including Cd was assumed constant, and the temperature dependence of the partial pressure of Cd given by $\exp(\mu^\circ_{Cd}/RT)$, where μ°_{Cd} is the standard

Table 2 Cell 92C

Optical Cell				CdTe Reservoir					
Temperature, K	100 P_2 (obs)	100 P_2 (sm)	(e+4) × P_1	Temperature (T_R), K	100 P_2	P_2, kPa	(e+4) × P_1	P(Cd)	Comp
1272.2	5.460	5.329	9.09	1013.0	5.386	5.457	0.365	4.43e−5	1.90
1272.2	5.550	5.427	9.18	1022.7	5.490	5.563	0.428	6.07e−5	1.74
1269.0	5.260	5.576	9.01	1043.0	5.645	5.720	0.589	1.16e−4	1.51
1272.1	5.600	5.668	9.37	1063.5	5.743	5.819	0.798	2.17e−4	1.44
1271.4	5.600	5.715	9.34	1082.0	5.791	5.868	1.04	3.76e−4	1.45
1271.9	5.800	5.745	9.41	1103.0	5.821	5.898	1.38	6.89e−4	1.53
1268.2	5.810	5.773	9.10	1123.8	5.843	5.920	1.80	1.23e−3	1.62
1272.1	6.010	5.812	9.49	1143.0	5.882	5.960	2.30	2.05e−3	1.74
1272.3	5.910	5.890	9.57	1163.9	5.955	6.034	2.98	3.51e−3	1.83
1271.7	6.280	6.006	9.61	1182.4	6.064	6.144	3.73	5.53e−3	1.88
1272.2	6.140	6.011	9.65	1183.0	6.069	6.149	3.75	5.61e−3	1.88
1272.1	6.130	6.097	9.72	1192.9	6.151	6.232	4.23	7.10e−3	1.90
1272.4	6.030	6.126	9.77	1195.8	6.179	6.261	4.38	7.60e−3	1.90
1272.4	6.050	6.211	9.83	1203.5	6.261	6.344	4.80	9.09e−3	1.92
1272.3	6.360	6.371	9.95	1215.6	6.414	6.499	5.55	1.19e−2	1.93
1272.2	6.150	6.495	10.0	1223.5	6.534	6.620	6.10	1.42e−2	1.94
1273.0	6.760	6.714	10.3	1235.5	6.747	6.836	7.03	1.84e−2	1.97
1272.1	6.910	6.907	10.3	1244.5	6.931	7.023	7.83	2.22e−2	2.01
1271.9	6.980	7.132	10.5	1253.8	7.149	7.244	8.76	2.69e−2	2.06
1273.2	7.260	7.147	10.6	1254.4	7.165	7.260	8.82	2.72e−2	2.07
1271.8	7.160	7.176	10.5	1255.5	7.192	7.287	8.93	2.78e−2	2.07
1272.2	7.120	7.366	10.7	1262.4	7.376	7.474	9.71	3.19e−2	2.14
1272.4	7.410	7.386	10.7	1263.1	7.395	7.493	9.78	3.24e−2	2.14
1293.5	8.030	7.690	13.4	1273.0	7.714	7.816	11.0	3.92e−2	2.35

Note: Partial pressures in the optical cell and in the CdTe reservoir. Last column labeled Comp is composition of CdTe(s).

Table 3 Cell 92A

Optical cell				CdTe reservoir					
Temperature, K	10 P_2 (obs)	10 P_2 (sm)	1000 P_1	Temperature (T_R), K	10 P_2	P_2, kPa	(e+4) × P_1	P(Cd)	Comp
1275.0	…	1.020	1.29	1075.0	1.028	10.42	1.25	2.29e−4	2.38
1275.9	1.040	1.043	1.32	1113.3	1.052	10.66	2.12	6.85e−4	2.30
1276.2	1.060	1.055	1.33	1137.5	1.064	10.78	2.89	1.32e−3	2.31
1276.3	1.070	1.068	1.34	1162.7	1.077	10.91	3.95	2.53e−3	2.37
1277.2	1.070	1.081	1.36	1185.2	1.089	11.03	5.16	4.42e−3	2.48
1277.3	1.110	1.099	1.37	1210.8	1.105	11.20	6.91	8.13e−3	2.68
1277.3	1.110	1.114	1.38	1228.8	1.119	11.34	8.45	1.23e−2	2.93
1277.5	1.130	1.141	1.41	1255.1	1.144	11.59	11.2	2.19e−2	3.53
1325.8	1.170	1.183	2.26	1286.4	1.190	12.06	15.6	4.19e−2	5.21

Note: Partial pressures in the optical cell and in the CdTe reservoir. Last column titled Comp is composition of CdTe(s) in units of (e+5)(X_{Te} − 0.50).

chemical potential of Cd(g, 1 atm). One arrives at this result for Cd by writing the diffusion flux for each species and a heat flux following the procedure of irreversible thermodynamics.[15] The diffusion fluxes then depend on all the concentration gradients as well as the temperature gradient. The result quoted previously follows if one neglects all of the cross diffusion coefficients and the thermal diffusion coefficients. In another type of calculation, the total pressure of all species was assumed constant and ratio of the dominant ones, Cd and Te_2, given by the thermal diffusion equation. A typical coefficient of −0.3 was used so that tellurium concentrates in the hotter region. Calculated values for the partial pressures in the optical cell and in the reservoir are given as the remaining entries in the Tables 1–4 along with the calculated composition, $10^5(X_{\mathrm{Te}} - 0.5)$, of the CdTe(s). Diatomic tellurium constitutes 98% or more of the vapor for all of the sample points. In general, the partial pressure of the five-atom molecule is greater than that of the monatomic species in the reservoir, while the reverse is true in the optical cell.

Table 4 Cell 93E

Optical cell				CdTe reservoir					
Temperature, K	10 P_2 (obs)	10 P_2 (sm)	(e+4) × P_1	Temperature (T_R), K	10 P_2	P_2, kPa	(e+4) × P_1	P(Cd)	Comp
1272.7	1.31	1.307	14.3	1111.3	1.316	13.33	2.30	5.74e–4	5.40
1273.8	1.30	1.323	14.6	1122.3	1.332	13.50	2.66	7.74e–4	5.20
1274.7	1.36	1.335	14.8	1131.9	1.345	13.63	3.03	1.01e–3	5.09
1273.5	1.51	1.335	14.6	1131.9	1.345	13.63	3.04	1.01e–3	5.07
1274.5	1.36	1.345	14.7	1142.4	1.355	13.73	3.45	1.32e–3	5.00
1273.6	1.51	1.352	14.7	1151.6	1.361	13.79	3.91	1.71e–3	5.00
1275.0	1.38	1.353	14.9	1152.0	1.363	13.81	3.91	1.71e–3	4.99
1273.9	1.45	1.359	14.8	1161.5	1.368	13.86	4.41	2.21e–3	5.01
1272.9	1.51	1.364	14.7	1171.0	1.373	13.91	4.91	2.77e–3	5.05
1275.0	1.38	1.365	15.0	1171.9	1.374	13.92	4.97	2.84e–3	5.07
1274.1	1.59	1.369	14.8	1181.6	1.377	13.95	5.59	3.63e–3	5.17
1273.8	1.35	1.373	15.0	1191.1	1.381	13.99	6.20	4.53e–3	5.29
1275.1	1.53	1.374	14.9	1191.5	1.382	14.00	6.27	4.64e–3	5.28
1274.0	1.63	1.378	14.9	1201.2	1.385	14.03	6.94	5.75e–3	5.41
1273.2	1.63	1.383	14.8	1210.8	1.390	14.08	7.77	7.29e–3	5.56
1275.2	1.29	1.383	15.0	1211.4	1.389	14.07	7.77	7.28e–3	5.59
1274.3	1.57	1.389	14.9	1220.6	1.395	14.13	8.68	9.19e–3	5.76
1275.3	1.32	1.396	15.1	1231.1	1.401	14.19	9.67	1.15e–2	5.98
1273.5	1.60	1.404	15.0	1240.4	1.408	14.27	10.6	1.41e–2	6.16
1274.4	1.61	1.405	15.0	1240.7	1.409	14.28	10.8	1.44e–2	6.19
1274.2	1.27	1.416	15.1	1251.1	1.419	14.38	12.0	1.79e–2	6.43
1286.0	1.70	1.428	17.0	1260.2	1.432	14.51	13.2	2.18e–2	6.81

Note: Partial pressures in the optical cell and in the CdTe reservoir. The last column titled Comp is the composition of CdTe(s) in units of (e+5)(X_{Te} – 0.50).

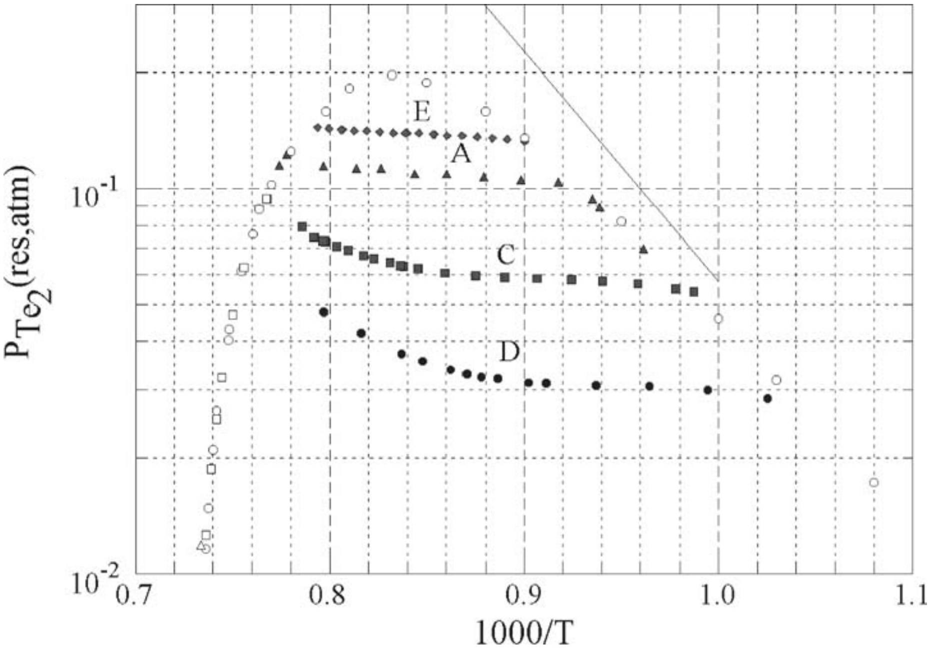

Fig. 1 The partial pressure of diatomic tellurium over CdTe(s) as a function of its reciprocal temperature for four optical cells and the three-phase curve for CdTe(s). The line segment at upper right shows the vapor pressure of tellurium. The parabola-like curve defined by the open circles and squares is the Te-rich portion of the three-phase curve for CdTe(s). From the lowest pressure upward, the solid symbols are for optical cells 92D, 92C, 92A, and 93E.

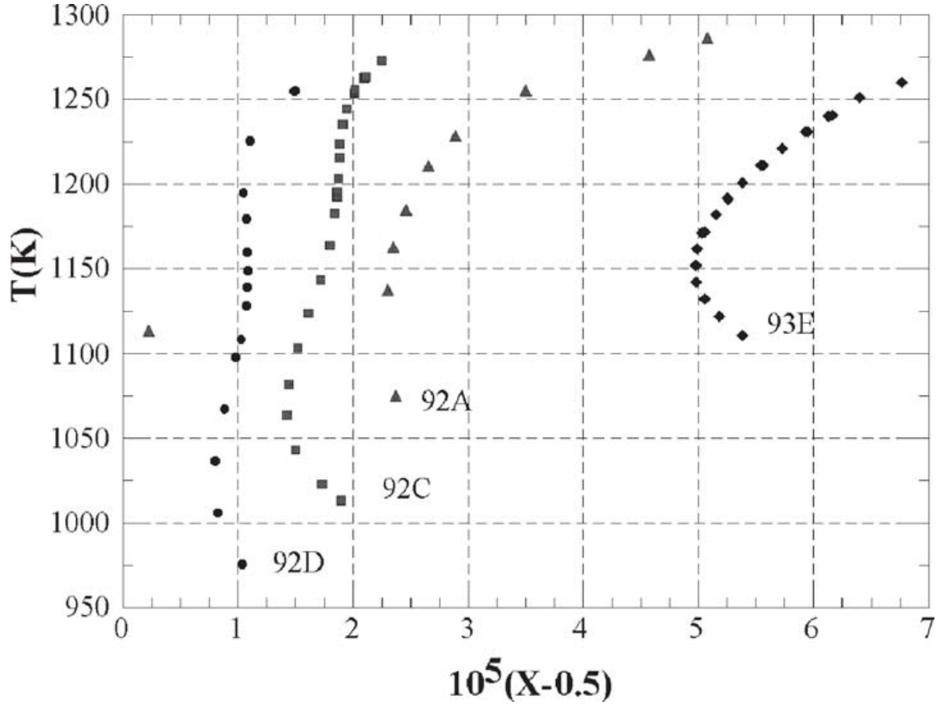

Fig. 2 For the points in Fig. 1 and on the horizontal axis, the composition of CdTe(s) expressed as 10^5 times the difference between the atom fraction of tellurium, and one-half is plotted as a function of the temperature on the vertical axis. Different symbol denote results from different optical cells, and from left to right are for cells 92D, 92C, 92A, and 93E.

The quantities characterizing the CdTe(s) are then T, P_2, where this is the partial pressure of Te_2 in the reservoir, and the composition. These are shown in the next two figures. Figure 1 shows the partial pressure of $Te_2(g)$ over the CdTe on a log scale as a function of the reciprocal temperature for four optical cells. The points falling along the uppermost parabola-like curve define the Te-rich leg of the CdTe three phase curve.[14] The line segment at the upper right shows the vapor pressure of tellurium where it just deviates from the three-phase curve. Figure 2 shows the composition of the CdTe on the horizontal axis as a function of its temperature. High-temperature points for sample 92D were omitted in the previous paper[5] because they overlapped those for sample 92C. Here, the overlap has been removed by the new smoothing step, and these points are included. The range of compositions covered is about the same as in the earlier paper,[5] but the C-shape of the temperature-composition data is now much less pronounced except for 93E. A part of the data for 93E is inconsistent with the thermodynamic criterion for a stable phase in that partial pressure-temperature curves constructed from Fig. 1 and 2 show the partial pressure for $10^5(X_{Te} - 0.5) = 5.4$ to lie slightly below that for 5.2.

3. Discussion

The value of P_2 and T at a solidus point can be seen in Fig. 1 where the value of P_2 for a given optical cell intersects the three-phase curve. The corresponding composition can be seen in Fig. 2 and in Tables 1 through 4. The solidus points are tabulated in Table 5. They are only slightly different from the author's previous values. The solidus points obtained here are shown with those from total pressure measurements[3] in Fig. 3. At the highest temperatures the author's two points fall close to the curve defined by those from the total pressure measurements. Between 960 and 1000 K the lowest temperature points seem to be leading into those from the total pressure measurements, but then rise above them between 1000 and 1100 K.

Table 5 Solidus points for Te-rich CdTe

Optical cell	Temperature, K	P_2, atm	P_2, kPa	Comp
92D	960	0.028	2.84	1.08
92C	1013	0.0539	5.46	1.90
92A	1075	0.103	10.44	2.37
92A	1286	0.123	12.46	5.08
93E	1111	0.132	13.37	5.38
93E	1260	0.144	14.59	6.77

Note: Temperature, partial pressure of diatomic tellurium, and composition obtained with various optical cells. Composition expressed as 10^5 times the difference between the atom fraction of tellurium and one-half.

The data for compositions within the three-phase curve are compared with that from total pressure measurements in Fig. 4. There, P_2 is plotted against composition for three temperatures, 1111, 1150, and 1204.8 K. The open symbols are from this work. The filled symbols are from total pressure measurements[3] and are calculated from their equations, which show compositions 5 through 7 with identical

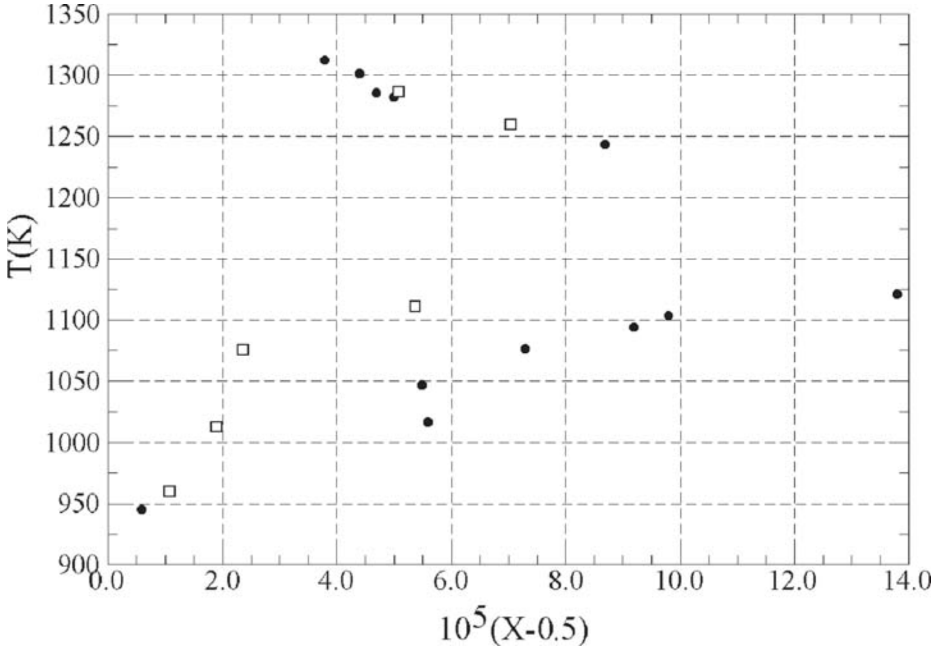

Fig. 3 Solidus points for Te-rich CdTe(s). Open squares, this work; filled circles (Ref 3)

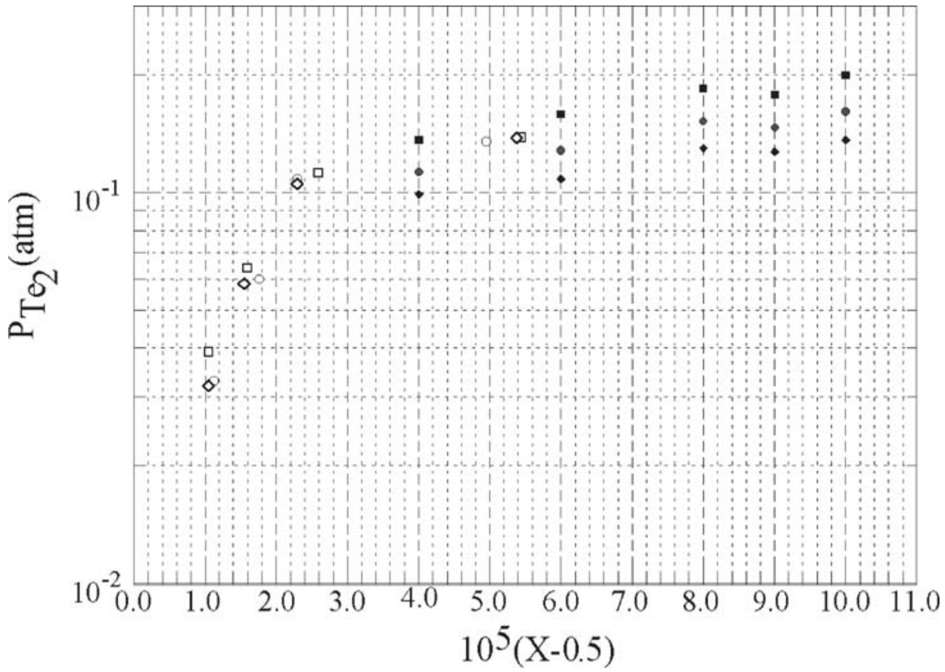

Fig. 4 Partial pressure of diatomic tellurium over CdTe(s) as a function of composition for 1111 K shown as diamonds, 1150 K shown as circles, and 1204.8 K shown as squares. Filled symbols are from Ref 3. Open symbols are from this work. Composition is expressed as 10^5 times the difference between the atom fraction of tellurium and one-half.

P_2. These are plotted as 6. On a log-log plot the author's compositions below 3 depend approximately on the three-fourths power of P_2. The compositions above 5 depend approximately on the sixth power of P_2. The near-zero power dependence of P_2 on composition, $10^5(X_{Te} - 0.5)$, for compositions above 5 is interesting for two reasons. First of all, in order that the CdTe(s) phase be stable relative to internal decomposition, the isothermal chemical potential of tellurium and hence P_2 must increase montonically with atom fraction of tellurium. The measurements at high composition then show a close approach to spinodal decomposition. Second, for two of the simplest defect models for

Te-rich CdTe the power dependence is much higher. For doubly ionized Cd vacancy acceptors in the case that the material is extrinsic—that is, the concentration of valence band holes is twice that of Cd vacancies—P_2 depends on the sixth power of $10^5(X_{Te} - 0.5)$. In the case of neutral tellurium interstitials, it depends on the second power.

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