The Pb-Se (Lead-Selenium) System

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Equilibrium Diagram

The assessed Pb-Se phase diagram, Fig. 1, is based on the present evaluators' modeling and calculations. An intermediate phase, PbSe, with a narrow composition range of stability, forms at 50 at.% Se. In the Pb-PbSe region, a eutectic reaction occurs near pure Pb. In the PbSe-Se region, a liquid miscibility gap, a monotectic reaction, and a eutectic reaction exist. Figure 2 gives the Pb-rich portion of the liquidus, and Fig. 3 shows the homogeneity range of the PbSe phase.

Liquidus

The melting points of Pb and Se are 327.502 and 221 °C, respectively. The liquidus in the Pb-Se system was determined by [07Pel], [08Fri], [55Noz], [56Pel], [61Sei], [66Sei], [66Mil], and [74Sch2]. The calculated liquidus shown in Fig. 1 and 2 is in fairly good agreement with the experimental data.

[55Noz] suggested a monotectic point at ~20.5 at.% Se and 860 °C and a liquid miscibility gap in the Pb-PbSe region of the phase diagram. However, [61Sei] and [66Mil] did not show any miscibility gap in the Pb-PbSe region. Table 1 lists the liquidus data for the Pb-Se system.

Invariant Equilibria

The eutectic in the Pb-PbSe region is very close to pure Pb. [39Gre] determined this eutectic point at 0.013 at.% Se and 0.2 °C below the melting point of Pb. The monotectic point in the PbSe-Se region was determined by [55Noz], [66Sei], and [74Sch2]. The eutectic in the PbSe-Se region is very close to pure Se and has not been experimentally determined. Table 2 gives the experimental data for different invariant equilibria in the Pb-Se system from the literature along with the assessed values based on the present calculations.

Solid Solubilities

The mutual solid solubilities of Pb and Se are very limited. [39Gre] determined the solubility of Se in (Pb) to be 0.004 at.% Se at 300 °C. There are no reported measurements on the solubility of Pb in (Se).

PbSe

The homogeneity range of PbSe is very narrow, and there are no direct measurements of the stability range of PbSe in the literature. [74Leu] estimated the homogeneity range of PbSe from transport number measurements. PbSe is a semiconductor compound. A number of researchers have reported measurements on the sign and concentration of charge carriers along the stability limit of PbSe as a function of temperature [62Bre, 68Abr, 69Cho, 74Sch1, 74Sch3, 75Leu]. These data have been used to compute the stability limit of PbSe by assuming that nonstoichiometry is caused by the presence of fully ionized vacancies on the Pb or Se sublattices. (See also "Thermodynamics.") Results are shown in Fig. 3 along with the suggested line based on the present calculations. Table 3 gives the congruent melting point of PbSe.

High-Pressure Phase

PbSe, which has a cubic (NaCl-type) structure at atmospheric pressure, transforms to an orthorhombic (GeS-type) structure at high pressures. The transformation is of first order and occurs at ~43 kbar [64Tak].

Phase Equilibrium Calculations

The Pb-Se phase diagram was calculated assuming that the PbSe phase is a line compound. The thermodynamic data used

Table 1 Liquidus Data

Composition, at.% Se	Temperature, °C
0.0	327.5
0.002	327
2.5	682
5.0	752
7.5	797
10.0	832
12.5	859
15.0	881
17.5	901
20.0	918
22.5	933
25.0	947
27.5	959
30.0	971
32.5	983
35.0	995
37.5	1005
40.0	1013
42.5	1024
45.0	1040
47.5	1070
50.0	1079
52.5	1065
55.0	1020
57.5	980
60.0	950
62.5	908
65.0	855
67.5	775
70.0	735
72.5	705
75.0	685
98.8	678
99.992	220
100	221

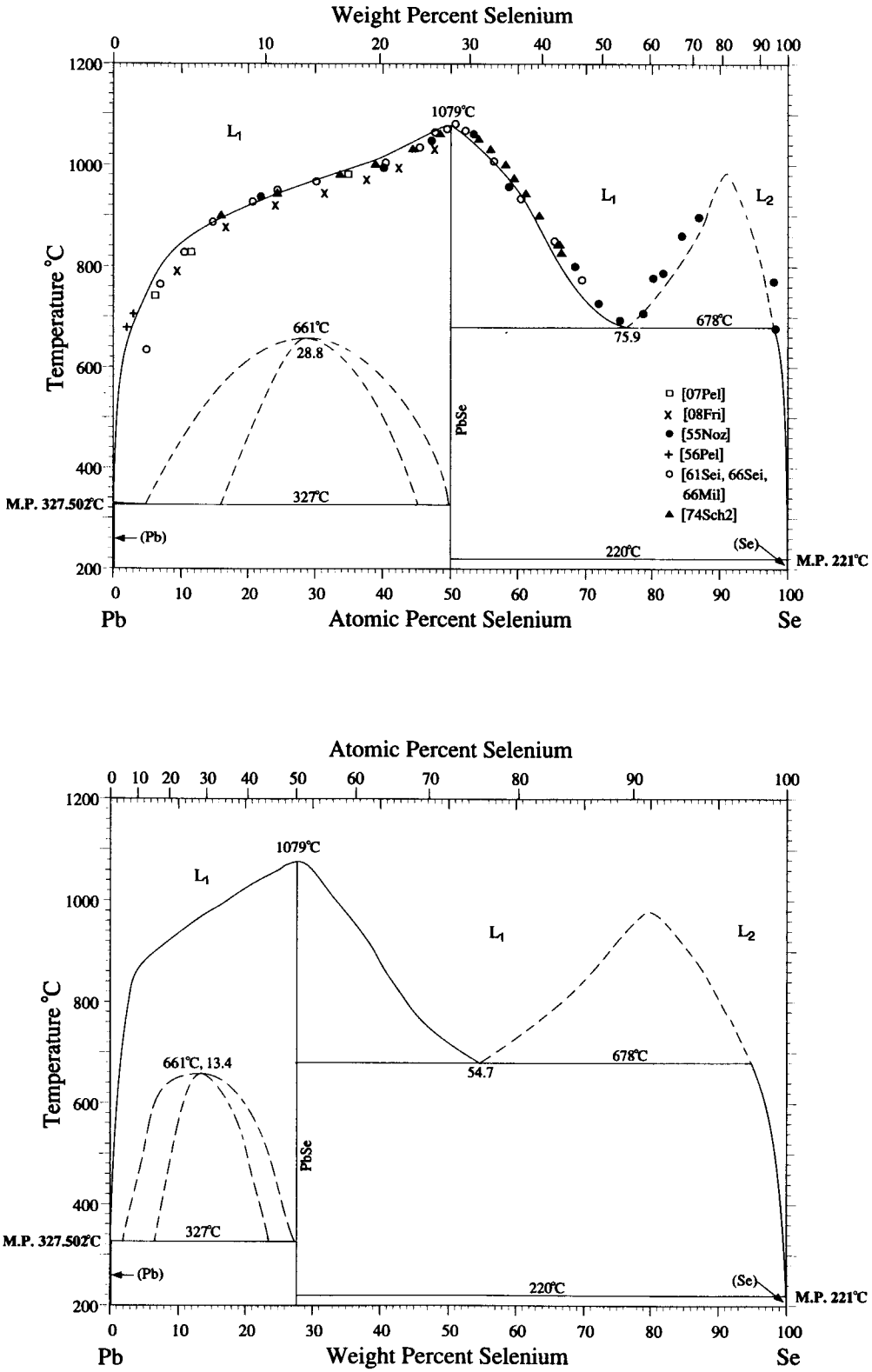

Fig. 1 Assessed Pb-Se phase diagram

for the liquid phase and PbSe are discussed in later sections. Agreement between the calculated and experimental phase diagram is good. The assessed phase diagram (Fig. 1 and 2) is based on the calculated phase equilibrium.

Metastable Phases

The liquidus curve in the Pb-PbSe region has an inflection point (Fig. 1) that indicates a metastable liquid miscibility gap at lower temperatures. This miscibility gap was calculated based on the thermodynamic model described in the next section and is shown as a dashed line in Fig. 1. It has not been confirmed experimentally.

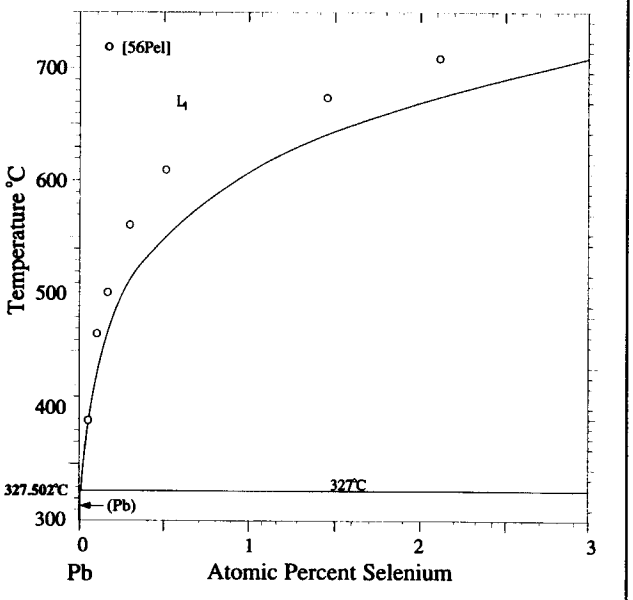

Fig. 2 Pb-rich region of the Pb-Se phase diagram.

Crystal Structures and Lattice Parameters

Crystal structure and lattice parameter data for the Pb-Se system are given in Table 4. No measurements have been reported for the lattice parameters of the high-pressure, orthorhombic, PbSe phase.

Thermodynamics

Liquid Phase

[74Sch2] measured the activity of Pb in the Pb-Se liquid phase as a function of temperature and composition and along the

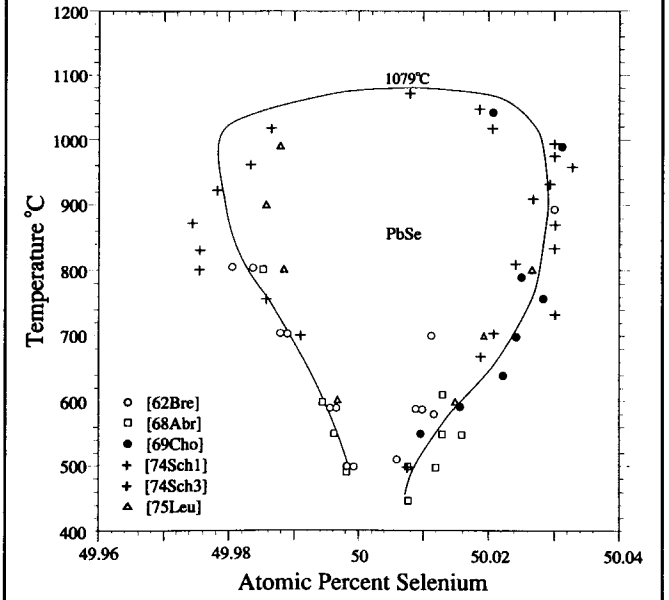

Fig. 3 Stability range of PbSe.

Table 2 Special Points of the Assessed Pb-Se Phase Diagram

Reaction		Composition, at.% Se		Temperature, °C	Reaction type	Reference
L ↔ Pb		~0		327.502	Melting	[Massalski2]
L_1 ↔ (Pb) + PbSe		~0		327.3	Eutectic	[39Gre]
L_1 ↔ PbSe + L_2	0.002	...	50	327	Eutectic	Calculated
	76	...	98.8	681.0	Monotectic	[55Noz]
	76.1	...	...	678.3	Monotectic	[66Sei]
	75.9	50	98.8	678	Monotectic	this study
L_2 ↔ PbSe + (Se)	99.992	50	~100	220	Eutectic	Calculated
L ↔ Se		100		221	Melting	[Massalski2]

liquidus. [84Kot] determined the enthalpy of mixing of liquid Pb-Se alloys up to 50 at.% Se in the temperature range from 610 to 1092 °C. The present evaluators used the associated solution model to describe the thermodynamic properties of the liquid phase [85Sch]. The liquid Pb-Se phase is assumed to consist of "Pb," "Se," and "PbSe" species in the liquid, governed by the following equilibrium:

"Pb"(L) + "Se"(L) = "PbSe"(L) (Eq 1)

with an equilibrium constant given by:

$$K = \frac{f_3 y_3}{(f_1 y_1)(f_2 y_2)} \quad \text{(Eq 2)}$$

where f_1, f_2 and f_3 are the activity coefficients and y_1, y_2, and y_3 are the mole fractions of "Pb," "Se," and "PbSe," respectively, in the liquid. The mole fractions, y_i, are related to the actual mole fractions, X_{Pb} and X_{Se} of Pb and Se, respectively, by the following mass balance equations:

$$y_1 = X_{Pb} - X_{Se} y_3 \quad \text{(Eq 3)}$$

and

$$y_2 = X_{Se} - X_{Pb} y_3 \quad \text{(Eq 4)}$$

The activity coefficients, f_i, are described by the three-suffix Margules equations [76Kel] as:

$$\ln f_i = \frac{1}{2}\sum_{j=1}^{n}(w_{ij} + w_{ji})y_j - \frac{1}{2}\sum_{j=1}^{n}\sum_{p=1}^{n} w_{jp} y_j y_p + \sum_{j=1}^{n}(w_{ij} - w_{ji})y_j\left(\frac{y_j}{2} - y_i\right) + \sum_{j=1}^{n}\sum_{p=1}^{n}(w_{jp} - w_{pj})\, y_j^2 y_p \quad \text{(Eq 5)}$$

where i, j, and p refer to "Pb," "Se," and "PbSe," respectively. w_{ij}, w_{ji}, w_{jp}, and w_{pj} are the interaction parameters of the solution. y_i, y_j, and y_p are the corresponding mole fractions, and $w_{ii} = w_{jj} = w_{pp} = 0$. The interaction parameters are represented as:

$$w_{ij} = \frac{A}{T} + B \quad \text{(Eq 6)}$$

where A and B are constants whose values must be obtained from experimental data. The activities of "Pb" and "Se" species are:

$$a_1 = f_1 y_1 \quad \text{(Eq 7)}$$

and

$$a_2 = f_2 y_2 \quad \text{(Eq 8)}$$

with Pb(L) and Se(L) as the standard states. Because the activities of the "Pb" and "Se" species are the same as those of the Pb and Se components [65Pri], the following equations are generated:

$$a_1 = f_1 y_1 = a_{Pb} = \gamma_{Pb} X_{Pb} \quad \text{(Eq 9)}$$

and

$$a_2 = f_2 y_2 = a_{Se} = \gamma_{Se} X_{Se} \quad \text{(Eq 10)}$$

where γ_{Pb} and γ_{Se} are the activity coefficients of Pb and Se that differ from f_1 and f_2. The values for the different parameters are obtained using the available thermodynamic and phase equilibria information to give a self-consistent set of parameters. These parameters are given in Table 5.

Table 3 PbSe Congruent Melting Point

Temperature, °C	Composition, at.% Se	Reference
1088	...	[08Fri]
1076	...	[55Noz]
1080.7	...	[61Sei]
1080.7	...	[66Mil]
1079	50.012	Assessed

Table 4 Crystal Structure and Lattice Paremeter Data

Phase	Composition, at.% Se	Pearson symbol	Space group	Strukturberich designation	Prototype	Lattice parameters, nm a	c
(Pb)	~0	*cF*4	$Fm\bar{3}m$	*A*1	Cu	0.49502	...
PbSe	50	*cF*8	$Fm\bar{3}m$	*B*1	NaCl	0.6122	...
PbSe(a)	50	*oP*8	*Pnma*	*B*16	GeS	...	...
(Se)	~100	*hP*3	$P3_121$	*A*8	γSe	0.43659	0.49537

Note: From [Massalski2]. **(a)** High pressure.

Table 5 Thermodynamic Data for Pb-Se Liquid Alloys

"Pb"(L) + "Se"(L) = "PbSe"(L)

$$K = \frac{(f_3 y_3)}{(f_1 y_1)(f_2 y_2)}$$

$$\ln K = 0.6503 + \frac{7358}{T}$$

$$w_{13} = w_{31} = -0.600 + \frac{2051.6}{T}$$

$$w_{23} = 9.44 - \frac{12\,732.2}{T}$$

$$w_{32} = -21.75 + \frac{23\,197.4}{T}$$

$$w_{12} = w_{21} = 0.0$$

Note: Subscript 1 ≡ the "Pb" species; subscript 2 ≡ the "Se" species; subscript 3 ≡ the "PbSe" species. The other parameters are defined in Eq 1 to 10.

The various thermodynamic properties of the liquid can be obtained by standard thermodynamic procedures [84Sch]. Figure 4 compares the calculated integral enthalpy of mixing, ΔH, and the partial molar enthalpy of mixing for Se, ΔH_{Se}, at 1087 °C with the experimental data of [84Kot]. The calculated values are generally less negative than those of [84Kot]. Figure 5 compares the activity of Pb along the liquidus with the experimental data of [74Sch2], and the agreement is satisfactory. The calculated thermodynamic properties for the liquid Pb-Se phase are suggested here because they are consistent with the phase diagram data. Table 6 lists the partial enthalpy and entropy of mixing for Pb and Se derived from the above parameters at 1087 °C.

[79Cha] and [80Cha] determined the viscosity of the Pb-Se melts over a range of composition and temperature. Results were analyzed in terms of a hard sphere model and the associated solution thermodynamics.

The PbSe Phase

[60Fin] and [81Sha] determined the Gibbs energy of formation of PbSe by electromotive force in the temperature range 200 to 300 °C and 325 to 585 °C, respectively. [70Haj] determined the enthalpy of formation of PbSe. [74Mil] assessed the data prior to 1974 and suggested $\Delta H^0(298)$ for the reaction Pb(c) (crystalline solid) + Se(c) ↔ PbSe(c) to be 100.0 kJ/mol. The present evaluators used $\Delta H^0(298)$ given by [74Mil] along with the $(G^0(T) - H^0(298))/T$ values from [74Mil] for the PbSe phase and from [Hultgren,E] for Pb and Se to arrive at the following expression for the Gibbs energy of formation of PbSe:

$$\Delta G^0 = -118\,000.0 + 36.0\,T \text{ J/mol} \qquad \text{(Eq 11)}$$

for the reaction Pb(L) + Se(L) ↔ PbSe(c).

The enthalpy and entropy of melting of the PbSe, determined by [81Sha], are:

$$\Delta_{fus}H(\text{PbSe}) = 44\,800 \text{ J/mol} \qquad \text{(Eq 12)}$$

and

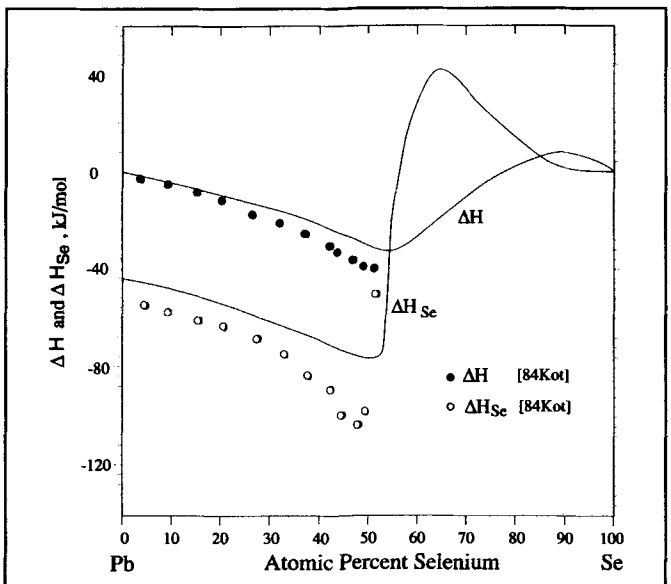

Fig. 4 Calculated and experimental ΔH and ΔH_{Se} for the Pb-Se liquidus at 1087 °C (1360 K).

$$\Delta_{fus}S(\text{PbSe}) = 33.06 \text{ J/K·mol} \qquad \text{(Eq 13)}$$

Pressure

Vapor Pressure

[59Zlo], [69Han], and [69Sok] determined the partial pressure of PbSe(g) in equilibrium with PbSe(c). These data were evaluated by [74Mil]. Table 7 gives $p_{PbSe(g)}$ in equilibrium with PbSe(c) from different investigators, along with the suggested values. The suggested data are based on the thermodynamic properties selected by [74Mil].

P-T-X Diagram for PbSe

[84Nga] developed a thermodynamic model to describe the *P-T-X* diagrams of Pb and Sn chalcogenides. This model is similar to the one developed by [77Bre]. The present evaluators analyzed the *P-T-X* diagram for the PbSe phase according to the model of [84Nga], which shows the dominant point defects in the PbSe phase as double-ionized Pb vacancies on the Pb sublattice and as double-ionized Se vacancies on the Se sublattice. The nonstoichiometry is caused by the presence of excess Se vacancies on the Pb-rich side and the excess Pb vacancies on the Se-rich side. The partial pressure of Se_2, p_{Se_2}, in equilibrium with the PbSe phase can then be expressed as:

$$\ln p_{Se_2} = \ln p^0_{Se_2} + 2Z\,\text{Sinh}^{-1}\left[\frac{p-n}{2n_i}\right] + 2\,\text{Sinh}^{-1}\left[\frac{p-n}{2ZK_s^{1/2}}\right] \qquad \text{(Eq 14)}$$

where $p^0_{Se_2}$ is the partial pressure of Se_2 in equilibrium with the stoichiometric PbSe. p and n are the concentrations (cc^{-1}) of holes in the valence band and electrons in the conduction band of the semiconductor compound PbSe, respectively. n_i is the intrinsic carrier concentration (cc^{-1}) of PbSe; $n_i^2 = p{\cdot}n$. Z is the degree of ionization of the vacancies ($Z = 2$ in this case), and K_s is the equilibrium constant, given by:

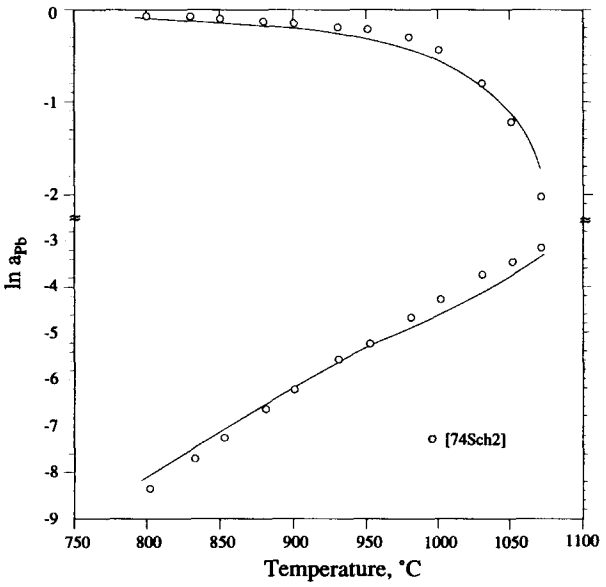

Fig. 5 Calculated and experimental activity of Pb along the liquidus.

$$K_s = \left[\begin{smallmatrix}-Z\\ Pb\end{smallmatrix}\right]\left[\begin{smallmatrix}+Z\\ Se\end{smallmatrix}\right] \quad \text{(Eq 15)}$$

where $\left[\begin{smallmatrix}-Z\\ Pb\end{smallmatrix}\right]$ and $\left[\begin{smallmatrix}+Z\\ Se\end{smallmatrix}\right]$ are the concentrations (cc^{-3}) of vacancies on the Pb and Se sublattices, respectively. For intrinsic (stoichiometric) PbSe, $p = n$ and $\left[\begin{smallmatrix}-Z\\ Pb\end{smallmatrix}\right] = \left[\begin{smallmatrix}+Z\\ Se\end{smallmatrix}\right]$. Also:

$$(p - n) = Z\left\{\left[\begin{smallmatrix}-Z\\ Pb\end{smallmatrix}\right] - \left[\begin{smallmatrix}+Z\\ Se\end{smallmatrix}\right]\right\} \quad \text{(Eq 16)}$$

and

$$(X_{Se} - 0.5) = \frac{(p-n)M_{PbSe}}{2Z\rho N} = \frac{(p-n)}{6.858 \times 10^{22}} \quad \text{(Eq 17)}$$

where M_{PbSe} is the molecular weight of PbSe, ρ is its density, and N is Avogadro's number.

The experimental data on the carrier concentration along the stability limit of PbSe [62Bre, 68Abr, 69Cho, 74Sch1, 74Sch3, 74Zlo, 75Leu] and within the stable PbSe phase [64Oha] have to be used to obtain the optimum model parameters given below in addition to the calculated activity of Se along the liquidus.

$$\log n_i = -\frac{352.4}{T} + 14.92 + 1.5\log T \quad \text{(Eq 18)}$$

and

Table 6 Thermodynamic Properties of Liquid PbSe

Composition, at.% Se	Partial enthalpy (ΔH_{Pb}), J/mol	Partial entropy (ΔS_{Pb}), J/mol·K	Partial enthalpy (ΔH_{Se}), J/mol	Partial entropy (ΔS_{Se}), J/mol·K
0	0	0	–43 854	∞
5	44.7	0.461	–45 560	34.22
10	200	1.032	–47 456	26.98
15	595	1.917	–50 072	21.01
20	1 037	2.896	–52 081	17.54
25	1 882	3.909	–54 974	13.31
30	3 186	5.60	–58 397	8.86
35	5 153	8.03	–62 462	3.85
40	8 054	11.69	–67 277	–2.28
45	12 356	18.42	–73 071	–10.82
50	14 426	31.49	–75 916	–25.75
55	–73 016	–25.47	1 922	24.6
60	–116 289	–53.39	32 495	44.2
65	–132 003	–58.88	41 294	47.03
70	–123 924	–44.81	37 693	40.48
75	–97 774	–15.94	28 646	30.19
80	–60 329	22.26	18 877	19.92
85	–16 243	66.19	10 590	11.40
90	31 587	113.9	4 609	5.22
95	81 314	165.3	1 116	1.47
100	131 734	∞	0	0

Note: At 1087 °C (1360 K).

$$\log K_S^{1/2} = -\frac{5851.7}{T} + 23.01 \qquad \text{(Eq 19)}$$

and

$$\log p^0_{Se_2} = -\frac{11\,120.4}{T} + 6.016 \qquad \text{(Eq 20)}$$

Figure 6 gives the calculated *P-T-X* diagram for the PbSe phase. The stability range of PbSe, given in Fig. 3, is also derived from the above model. [67Nov], [73Sea], and [81Nov] also calculated the *P-T-X* diagram for the PbSe phase from point defect models.

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Table 7 Partial Pressure of PbSe(g) in Equilibrium with PbSe(c)

Reference	Method	log (*p*, bar) = -(*A*/*T*) + *B* *A*	*B*
[59Zlo]	Knudsen effusion	11 032	7.203
[69Han]	Knudsen effusion	12 260	8.163
[69Sok]	Knudsen effusion	11 650	7.781
Assessed(a)	...	11 433	7.528

(a) 700 K <*T* <1100 K.

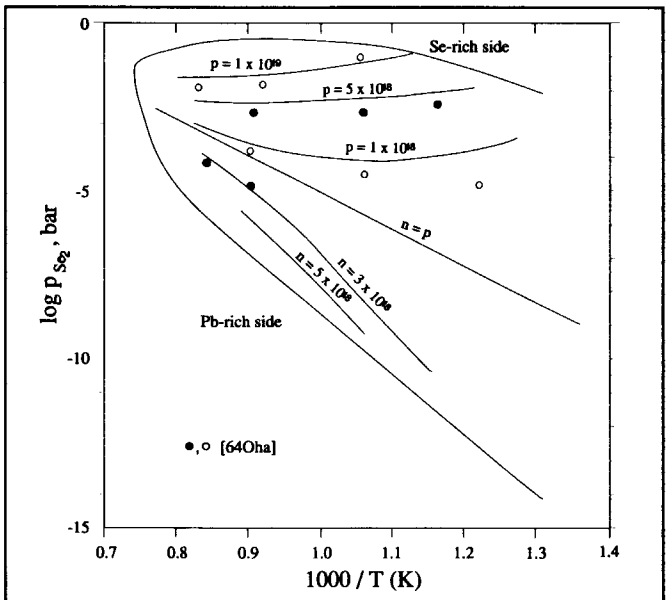

Fig. 6 *P-T-X* diagram for PbSe.

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*Indicates key paper.

#Indicates the presence of a phase diagram.

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