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Thermodynamic assessment of the Ru-Si and Os-Si systems

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Abstract

The thermodynamic properties and phase diagrams of the Ru–Si and Os–Si systems are assessed. The calculated enthalpies of fusion and entropies of fusion of ruthenium and osmium silicides are compared with the reported values of different transition metal silicides. Both the thermodynamic properties and the phase diagrams of Ru–Si and Os–Si systems are in good agreement with the available experimental data. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

There has been an increasing interest in the transition metal silicides. Their stability and oxidation resistance make these alloys excellent candidates as materials for high temperature applications. Their relatively low electrical resistance has been utilized in microelectronics for the development of integrated circuit technology. Some transition metal silicides exhibit superconducting properties. The ruthenium silicides have been of considerable interest for their possible application as luminescent material in silicon based light emitters (LEDs) and as a promising new material for thermoelectrical applications [1,2]. The osmium silicides are reported to be semiconducting [3]. Despite the great interest in the ruthenium and osmium silicides for electrical and electronic devices, the thermodynamic properties of the Ru-Si and Os-Si systems are still incomplete. Reliable thermodynamic data are essential for assessing and predicting the behavior of these silicides. In this paper, the phase relations and thermodynamic properties of the Ru-Si and Os-Si systems are assessed using the CALPHAD technique.

2. Experimental information

2.1. The Ru-Si system

An early phase diagram of the Ru–Si system was presented by Obrowski [4]. On the basis of metallographic analysis of various alloys, three intermediate phases were observed and were assigned as Ru_2Si_3 , RuSi and Ru_2Si . Another phase, Ru_5Si_3 , was observed in as-melted samples, but not in annealed ones, by Engström [5]. Weitzer et al. [6] investigated the phase diagram of the Ru–Si system between 1273 and 1603 K using X-ray diffraction and reported five intermetallic compounds: Ru_2Si_3 , RuSi, Ru_4Si_3 , Ru_5Si_3 and Ru_2Si .

Recently, Perring et al. [7] revised the phase diagram of the Ru–Si system using differential thermal analysis, Xray diffraction, and electron microprobe investigations. The occurrence of Ru_5Si_3 in Refs. [5,6] was not confirmed. Three eutectic and two peritectic transformations were identified,

E1: liquid= Ru_2Si_3 +(Si) at 1573 K, instead of 1643 K in Ref. [6].

E2: liquid= Ru_2Si_3 +RuSi at 1953 K, similar to 1963 K in Ref. [6].

E3: liquid=(Ru)+ Ru_2Si at 1813 K, instead of 1778 K in Ref. [6].

P1: liquid+ $Ru_4Si_3 = Ru_2Si$ at 1817 K.

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P2: liquid+RuSi= Ru_4Si_3 at 1833 K instead of 1968 K in Ref. [6].

It has been reported that the RuSi exists in two crystal structures: a CsCl-type and a FeSi-type structure. However, the composition range and the relationship between the two forms of RuSi are still in debate. Finnie [8] proposed that the CsCl-type may be stable over only part of the temperature range between 1613 K and room temperature and is slightly more metal-rich than the FeSitype. Buschinger et al. [9] found a phase transition in stoichiometric RuSi from the high temperature CsCl-structure to the low temperature FeSi-type structure at about 1578 K, with the transition temperature decreasing rapidly with increasing Ru-excess. Perring et al. [10] reported that the stoichiometric RuSi had the CsCl-type instead of the FeSi-type structure at 1515 K. Recently, Perring et al. [7] reported that the two structures coexisted as distinct phases near the equi-atomic composition. The composition ranges of CsCl-type and FeSi-type RuSi measured by electron probe microanalysis are about $47.1-48.2(\pm 0.1)$ and 49.1(± 0.1) at.% Si, respectively. No transformation of the two forms of RuSi was reported at the temperature range 1273 K to 1773 K [7], this is not consistent with the results of [9,10].

In the Ru–Si system, the phase transformation between the two crystal structures of the Ru_2Si_3 (orthorhombic and tetragonal) was reported by Poutcharovsky et al. [11], but the transformation temperature was not well defined. However, the tetragonal Ru_2Si_3 was not detected by [6,7]. In this paper, we only take into account the thermodynamic properties of the FeSi-type RuSi and the orthorhombic Ru_2Si_3 .

The thermodynamic properties of the Ru-Si system have been investigated by several researchers. Kuntz et al. [12] determined the molar heat capacity of RuSi and Ru₂Si₃ by differential scanning calorimetry in the temperature range from 310 K to 1080 K. Table 1 summarizes the reported enthalpies of formation of the ruthenium silicides. Meschel and Kleppa [13] measured the enthalpy of formation of RuSi and Ru₂Si₃ using high temperature direct synthesis calorimetry. The enthalpy of formation of RuSi given by Meschel and Kleppa [13] agrees well with the previously determined values by Perring et al. [10] and Topor and Kleppa [14]. The enthalpy of formation of Ru₂Si₃ given by Meschel and Kleppa [13] agrees reasonably with the result of [10]. Perring et al. [10] reported the enthalpy of formation of Ru₄Si₃ at 1505 K using high temperature calorimetry.

Because of the importance of the thermodynamic prop-

Table 1

Comparison of the assessed enthalpies of formation ΔH_t of silicides in the Ru–Si system with some experimental and predicted data reported in the literature

Silicide	$\Delta H_{\rm f}$, kJ/(mole-atoms)	Method	Ref.
RuSi	-58.3 ± 2.1	High temperature direct	[13]
		synthesis calorimetry	
	-57.7±1.4 (1505 K)	High temperature calorimetry	[10]
	-56.5	_	[12]
	-58.1 ± 3.7	Solute-solvent drop calorimetry	[14]
	-32	Prediction	[15]
	-43.9	Prediction	[16]
	-33.2	Estimation	[17]
	-39.2	Prediction	[18]
	-33.4	_	[19]
	-42	Assessment	[20]
	-33.4	Assessment	[21]
	-56.454	Assessment	This work
Ru ₂ Si ₃	-60.7 ± 1.7	High temperature direct	[13]
		synthesis calorimetry	
	-50.3±1.0 (1704 K)	High temperature calorimetry	[10]
	-49.2	_	[12]
	-26.8	_	[22]
	-26	Prediction	[15]
	-26.8	Estimation	[17]
	-49.060	Assessment	This work
Ru ₄ Si ₃	-45.9±0.6 (1505 K)	High temperature calorimetry	[10]
	-57.561	Assessment	This work
Ru ₂ Si	-29	Prediction	[15]
	-37.6	Prediction	[16]
	-22.2	Assessment	[21]
	-38.700	Assessment	This work

erties of the transition metal silicides, some models or semi-empirical methods were proposed to predict their enthalpies of formation. Table 1 also shows the enthalpies of formation predicted using the semi-empirical model of Miedema and coworkers [15]. It can be seen that the predicted values are considerably less exothermic than the experimental results of [10,13,14].

Pasturel et al. [16] proposed a model to predict the enthalpies of formation of transition metal silicides and germanides. The authors proposed that the enthalpies of formation of silicides and germanides had two contributions. The first is the energy necessary to convert Si and Ge from the non-metallic into the metallic state and the second is the result of the filling of the d band of the transition metal by the free valence electrons of the metallic Si and Ge. It can be seen that the predicted values of [16] is less exothermic than the experimental results of [10,13,14]. Chart [20] assessed the thermochemical data for transition metal silicides, the results are also shown in Table 1. Until now, no thermodynamic properties of the liquid phase have been reported.

2.2. The Os-Si system

An early phase diagram of the Os–Si system was proposed by Finnie [8]. The eutectic temperature on the silicon-rich side of the Os–Si system was found to be at 1633 ± 15 K [8]. Mason and Muller-Vogt [23] studied the preparation, crystal growth and the physical properties of OsSi₂. It was found that the mixture of 15 at.% Os and 85 at.% Si was completely liquid at 1730 K. On slow cooling, OsSi₂ crystallized until the eutectic temperature was reached at 1630 K. The eutectic composition was slightly above 90 at.% Si.

The complete phase diagram of the Os–Si system has been investigated by Schellenberg et al. [3] using X-ray powder diffraction, differential thermal analysis, metallography, microprobe analysis and electrical resistivity measurements. Three intermetallic phases existed in this system, OsSi, Os_2Si_3 and $OsSi_2$. Two eutectic and two peritectic transformations were identified:

E1: liquid=(Si)+OsSi₂ at 1633 K, consistent with the results of [8,23]. E2: liquid=(Os)+OsSi at 1993 K. P1: liquid+Os₂Si₃=OsSi at 2003 K. P2: liquid+Os₂Si₃=OsSi₂ at 1913 K.

Table 2 summarizes the reported enthalpies of formation of the osmium silicides. The only experimentally determined thermodynamic data of the Os–Si system is the enthalpies of formation of Os_2Si_3 measured by Meschel and Kleppa [24]. The enthalpy of formation of OsSi given by Topor and Kleppa [14] is estimation only, due to the incomplete dissolution of the compound and components in the solvent used in the solute–solvent drop methods.

Table 2

Comparison of the assessed enthalpies of formation $\Delta H_{\rm f}$ of silicides in the Os–Si system with some experimental and predicted data reported in the literature

Silicide	$\Delta H_{\rm f}$, kJ/ (mole-atoms)	Method	Ref.
Os ₂ Si ₃	-30.5 ± 2.1	High temperature direct synthesis calorimetry	[24]
	-23	Prediction	[15]
	-41.4	Estimation	[17]
	-30.450	Assessment	This work
OsSi	$-30 \sim -50$	Solute-solvent drop calorimetry	[14]
	-29	Prediction	[15]
	-16.7	Prediction	[16]
	-32.6	_	[19]
	-42	Assessment	[20]
	-32.6	Assessment	[21]
	-25.415	Assessment	This work
OsSi ₂	-16	Prediction	[15]
	-20.9	Prediction	[16]
	-34.2	Estimation	[17]
	-36.8	Assessment	[21]
	-28.745	Assessment	This work

The predicted values using the semi-empirical model of Miedema and coworkers [15] and the model proposed by Pasturel et al. [16] are also shown in Table 2. No thermodynamic data of the liquid phase has been reported.

3. Thermodynamic model

3.1. Elements

The Gibbs energy of a pure element is taken from Ref. [25], which is referred to as ${}^{0}H_{i}^{SER}$, the enthalpy for its stable state at 298.15 K.

3.2. Stoichiometric phases

Since the homogeneity range of the ruthenium silicides and osmium silicides are not well defined, all intermetallic compounds in the Ru–Si and Os–Si systems are treated as stoichiometric phases. The Gibbs energy of a stoichiometric compound ${}^{0}G_{M_{a}Si_{a}}$ is expressed as follows:

$${}^{0}G_{M_{p}Si_{q}} = p \, {}^{0}G_{M} + q \, {}^{0}G_{Si} + a + bT + cT \ln T + dT^{2} + e/T$$
(1)

where, M refers to Ru or Os. ${}^{0}G_{M}$ and ${}^{0}G_{Si}$ are the Gibbs energy of the pure elements M and Si, respectively. *a*, *b*, *c*, *d* and *e* are parameters to be determined.

3.3. Solution phases

For a substitutional solution phase ϕ , such as the liquid and hcp–Ru phases, the molar Gibbs energy is equal to

$$G_m^{\phi} = G_m^{ref} + G_m^{id} + {}^{ex}G_m \tag{2}$$

with

$$G_{m}^{ref} = x_{\rm M}^{0} G_{\rm M} + x_{\rm Si}^{0} G_{\rm Si}$$
(3)

$$G_m^{id} = RT[x_M \ln x_M + x_{Si} \ln x_{Si}]$$
(4)

$$^{ex}G_m = x_{\rm M} x_{\rm Si} L \tag{5}$$

where the interaction term L can be composition- and temperature-dependent as follows:

$$L = \sum_{i=0}^{n} (a_i + b_i T)(x_{\rm M} - x_{\rm Si})^i$$
(6)

where, a_i and b_i are parameters to be determined.

4. Evaluation of the thermodynamic parameters

The model parameters are evaluated using the Parrot module in the Thermo-Calc program package [26]. This program is able to take various kinds of experimental data in the operation. It works by minimizing an error sum with each kind of the selected data values, given a certain weight. The weight is chosen and adjusted based on the data uncertainties given in the original publications, until most of the selected experimental information is reproduced within the expected uncertainty limits. All thermodynamic calculations are carried out using the Thermo-Calc program package.

4.1. The Ru–Si system

The enthalpies of formation of different ruthenium silicides are used as a guide for selecting the initial values of interaction parameters. At first, the heat capacity data of Kuntz et al. [12] are used to optimize the parameters of RuSi and Ru_2Si_3 phases. The phase diagram data summarized by [6,7] are used to optimize the parameters of other phases.

4.2. The Os-Si system

The enthalpy of formation of Os_2Si_3 given by Meschel and Kleppa [24] and the predicted enthalpies of formation of OsSi and OsSi₂ from the semiempirical model of Miedema and coworkers [15] are used as a guide for selecting the initial values of interaction parameters. The phase diagram data given by Schellenberg et al. [3] are used to optimize the parameters.

5. Results and discussion

By means of the computerized optimization, a complete and self-consistent thermodynamic description for the Ru– Si and Os–Si systems is obtained and listed in Appendix A.

5.1. The Ru-Si system

The calculated molar heat capacity of RuSi and Ru_2Si_3 at different temperatures are shown in Fig. 1(a) and (b), respectively. It can be seen that the calculation results agree well with the experimental values [12].

The calculated enthalpies of formation of ruthenium silicides at 298.15 K are listed in Table 1. All available literature values (experimental, assessed and predicted) are listed in the table. It can be seen that the enthalpy of formation of RuSi calculated in the present work agrees well with the values given by Refs. [10,12–14]. The



Fig. 1. Calculated heat capacity of RuSi (a) and Ru_2Si_3 (b) at different temperatures, together with experimental data. The heat capacities correspond to one mole of atoms. \times , [12].

calculated enthalpy of formation of Ru_2Si_3 agrees well with the values given by [10,12].

Murarka [27] proposed that the magnitude of the enthalpies of formation of transition metal silicides should increase with increasing silicon content in the alloys, provided that the values are normalized with respect to the number of moles of metal atoms. The calculation results of this work generally agree with this prediction.

Chart [20] pointed out that the entropy of fusion of transition metal silicides versus their melting points obeys the similar trends to those shown by the pure elements [28], that is, there is a correlation between the entropy of fusion and melting point within a group of elements that have a particular crystal structure. The enthalpies of fusion and the melting points reflect the cohesion energy. The relationships between enthalpies of fusion and entropy of fusion of TMSi (TM represents transition metal) vs. melting points are shown in Fig. 2(a) and (b), respectively. The relationships between the enthalpies of fusion and

entropy of fusion of TM_2Si vs. melting points are shown in Fig. 3(a) and (b), respectively. The calculated enthalpies of fusion and entropies of fusion of RuSi and Ru₂Si agree reasonably with the general trends.

The calculated phase diagram of the Ru–Si system is compared with various experimental phase boundary data in Fig. 4. Table 3 presents the comparison of the calculated invariant equilibria with the literature values. It can be seen that the calculation agrees well with the experimental data.

It should be pointed out that the reported melting points of RuSi differ greatly, from 2143 K [29], 2023 K [22] to 2003 K [6,30]. Our results show that the values given by [6,22,30] were more reasonable.

5.2. The Os-Si system

The calculated enthalpies of formation of osmium silicides at 298.15 K are listed in Table 2. All available literature values (experimental, assessed and predicted) are listed in the table. It can be seen that the values scatter



Fig. 2. Correlation of the enthalpy of fusion (a), and the entropy of fusion (b) to the melting point of TMSi, (TM represents transition metal). \triangle CoSi, [20,31]; \square CrSi, [34], \diamond FeSi, [32]; \blacksquare MnSi, [20]; + NiSi, [20]; \triangledown TiSi, [35]; *ZrSi, [33]; \times RuSi, this work; \diamond OsSi, this work.



Fig. 3. Correlation of the enthalpy of fusion (a), and the entropy of fusion (b) to the melting point of TM_2Si , (TM represents transition metal). Δ Co₂Si, [20,31]; \square Fe₂Si, [32]; \Diamond Zr₂Si, [33]; Ξ Ru₂Si, this work.



Fig. 4. The calculated phase diagram of the Ru–Si system compared with experimental data. \square , [6]; \triangle , [7]; \aleph , [22]; \heartsuit , [29]; \diamondsuit , [30].

greatly. The calculated enthalpy of formation of Os_2Si_3 agrees well with the recently determined value of [24].

Similar to the ruthenium silicides, the calculated en-

thalpies of formation of the osmium silicides increase with increasing silicon content when the data are normalized with respect to the number of moles of osmium atoms. This is consistent with the predictions of Murarka [27].

The calculated enthalpies of fusion and entropy of fusion of OsSi are shown in Fig. 2(a) and (b), respectively. The relationships between enthalpy of fusion and entropy of fusion of $TMSi_2$ vs. melting points are shown in Fig. 5(a) and (b), respectively. The calculated enthalpies of fusion and entropies of fusion of OsSi and OsSi₂ are consistent with the general trends.

The calculated phase diagram of the Os–Si system is compared with various experimental phase boundary data in Fig. 6. Table 4 presents the comparison of the calculated invariant equilibria with the literature values. The calculation is in good agreement with the experimental data.

6. Summary

The thermodynamic properties and phase diagrams of the Ru–Si and Os–Si systems are assessed. The calculated enthalpies of fusion and entropies of fusion of ruthenium and osmium silicides are compared with the reported values of different transition metal silicides. Both the

Table 3 Comparison between the calculated invariant equilibria and literature values in the Ru–Si system

Phase reaction	Composition of respective phases, x_{Si}			T (K)	Ref.
liquid=RuSi +Ru ₂ Si ₃	0.55-0.58 0.56 0.5 0.6 0.568 0.5 0.6		0.6 0.6	1953±10 1963 1968	[7] [6,29] This work
$liquid = Ru_2Si_3 + Diamond$	0.83-0.84 0.83 0.860	0.6 0.6	1.0 0.97 1.0	1573±5 1643 1571	[7] [6,29] This work
liquid + RuSi = Ru ₄ Si ₃	0.343	0.5	0.429	1833±5 1968 1836	[7] [6,29] This work
liquid $+Ru_4Si_3=Ru_2Si$	0.329	0.429	0.333	1817±5 1821	[7] This work
liquid = $(Ru) + Ru_2Si$	0.30–0.31 0.3	0.0452	0 333	1813±5 1778 1763 1809	[7] [6] [29] This work
$Ru_2Si = (Ru) + Ru_4Si_3$	0.333	0.0432	0.429	1518±5 1498 1518	[7] [6] This work
liquid=RuSi				2003 2023 2003 2143 2004	[6] [22] ^a [30] [29] This work
$liquid = Ru_2Si_3$				1983 1976	[6,29,30] This work

^a High-temperature phase.



Fig. 5. Correlation of the enthalpy of fusion (a), and the entropy of fusion (b) to the melting point of $TMSi_2$, (TM represents transition metal). \triangle CoSi₂, [20,31]; \square CrSi₂, [20]; \Diamond FeSi₂, [32]; \bigstar MoSi₂, [36]; \bigtriangledown NbSi₂, [35]; + TiSi₂, [35]; *VSi₂, [20]; \times ZrSi, [33]; \diamondsuit OsSi₂, this work.



Fig. 6. The calculated phase diagram of the Os–Si system compared with experimental data. Δ , [3]; \Diamond , [8]; \square , [23].

thermodynamic properties and the phase diagrams of the Ru–Si and Os–Si systems are in good agreement with the available experimental data.

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Appendix A

Summary of the optimized thermodynamic parameters for the Ru–Si and Os–Si systems. Values are given in SI units and correspond to one mole of atoms.

The Ru–Si system:

Phase liquid
Description: (Ru, Si)

$$L_{Ru,Si}^{liquid} = -127858 - 27.620T + (59628 - 23.850T)$$

 $(x_{Ru} - x_{Si}) + 50985(x_{Ru} - x_{Si})^2$

Phase hcp

Description: (Ru, Si) $L_{Ru,Si}^{hcp} = -121500 + 15.002T$

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Phase Ru<sub>2</sub>Si<sub>3</sub>
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$$\begin{array}{l} Description: (Ru)_{0.4}(Si)_{0.6} \\ {}^{0}G_{\mathrm{Ru}_{2}\mathrm{Si}_{3}} - 0.4 \, {}^{0}G_{\mathrm{Ru}}^{\mathrm{hep}} - 0.6 \, {}^{0}G_{\mathrm{Si}}^{\mathrm{diamond}} \\ = -47969 - 24.002T + 1.529T \, \mathrm{ln}T \\ + 1.026 \times 10^{-3}T^{2} - 81094/T; \, 298.15 < \mathrm{T} \leq 620 \\ - 45010 - 101.091T + 13.745T \, \mathrm{ln}T \\ - 4.440 \times 10^{-3}T^{2} - 1161172/T; \, 620 < \mathrm{T} < 3000 \end{array}$$

Phase RuSi

$$Description: (Ru)_{0.5}(Si)_{0.5}$$

 ${}^{0}G_{RuSi} - 0.5 \; {}^{0}G_{Ru}^{hcp} - 0.5 \; {}^{0}G_{Si}^{diamond}$
 $= -55276 - 17.892T + 2.205T \ln T$
 $- 6.122 \times 10^{-5}T^{2} - 78350/T$

Phase Ru_4Si_3 Description: $(Ru)_{0.572}(Si)_{0.428}$

$${}^{6}G_{\mathrm{Ru}_{4}\mathrm{Si}_{3}} = 0.572 {}^{6}G_{\mathrm{Ru}}^{\mathrm{m}} = 0.428 {}^{6}G_{\mathrm{Si}}^{\mathrm{dramond}}$$

= $-57561 + 3.639T$

Phase Ru_2Si Description: $(Ru)_{0.667}(Si)_{0.333}$ ${}^{0}G_{Ru_2Si} - 0.667 \; {}^{0}G_{Ru}^{hcp} - 0.333^{0}G_{Si}^{diamond}$ = -38700 - 1.178T

The Os-Si system: Phase liquid

Description: (Os, Si)

Table 4 Comparison between the calculated invariant equilibria and literature values in the Os–Si system

Phase reaction	Composition of respective phases, x_{s_i}			<i>T</i> (K)	Ref.
liquid=Os ₂ Si ₃				2113±20 2102	[3] This work
$liquid + Os_2Si_3 = OsSi_2$	0.72 ± 0.02 0.78	0.6 0.6	0.667 0.667	1913±20 1921	[3] This work
$liquid = OsSi_2 + Diamond$	0.88 ± 0.01 ~0.90	0.667	1.0	1633 ± 10 1630 1633 \pm 15	[3] [23] [8]
	0.90	0.667	1.0	1615	This work
$liquid + Os_2Si_3 = OsSi$	0.46	0.6 0.6	0.5 0.5	2003±10 2011	[3] This work
liquid=OsSi+(Os)	0.45 ± 0.01 0.42	0.5 0.5	0 0	1993±10 1988	[3] This work

$$\begin{split} L_{\rm Os,Si}^{\rm liquid} &= -125865 + 31.560T \\ &+ (27569 - 11.068T)(x_{\rm Os} - x_{\rm Si}) \end{split}$$

Phase hcp

Description: (Os, Si) $L_{Os,Si}^{hcp} = 464047$

Phase OsSi

Description: $(Os)_{0.5}(Si)_{0.5}$ ${}^{0}G_{OsSi} - 0.5 \; {}^{0}G_{Os}^{hcp} - 0.5 \; {}^{0}G_{Si}^{diamond}$ = -25415 + 1.242T

Phase Os₂Si₃

Description:
$$(Os)_{0.4}(Si)_{0.6}$$

 ${}^{0}G_{Os_{2}Si_{3}} - 0.4^{0}G_{Os}^{hcp} - 0.6^{-0}G_{Si}^{diamond}$
= $-30450 + 1.805T$

Phase OsSi₂

Description:
$$(Os)_{0.333}(Si)_{0.667}$$

 ${}^{0}G_{OsSi_{2}} - 0.333 {}^{0}G_{Os}^{hcp} - 0.667 {}^{0}G_{Si}^{diamond}$
 $= -28745 + 2.001T$

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