

Phase Diagram of the Iridium-Rhenium System

A useful model of the available experimental data is presented

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The experimental data on the binary metallic iridium-rhenium system available to date are inconsistent. The present work summarises recent experimental data on the Ir-Re system with the aim of allowing the correct solidus parts of the equilibrium phase diagram to be constructed. The examination of data obtained by different synthetic methods and thermodynamic calculations has revealed that the maximum solid-phase solubilities at 2000°C of Re in Ir and Ir in Re are 20 at% and 68 at%, respectively. The binary phase diagram proposed in this paper serves as a reliable model for representing the available experimental data.

Polymetallic compositions containing platinum group metals (pgms) and rhenium have high hardness and high thermal stability, and are useful to industry in constructional, high-temperature and high-hardness materials and as components of superalloys (1). Iridium-rhenium solid solutions are used as the working elements of high-temperature thermocouples (2) and in high-temperature and chemically resistant materials for crucibles (3), while Ir/Re/Al₂O₃ and Ir/ReO_x/SiO₂ composites have been proposed as active heterogeneous catalysts (4,5). It is well known that the properties of metallic solid solutions are affected by their composition and preparatory conditions, as well as the presence of impurities. The composition and temperature dependence of alloy properties is widely studied in order to analyse and predict the behaviour of metallic phases. However, difficulties in preparing solid solutions have led to poor and inconsistent information about the Ir-Re system and the corresponding physical properties of its metallic phases.

To investigate the chemical, physical and material properties of Ir-Re solid solutions, the construction of the correct equilibrium phase diagram is required. Prediction of ternary and quaternary phase diagrams is impossible without profound knowledge of two-component systems, so accurate data on the latter are essential. These systems are characterised by the extremely high melting points of their metal

components, making the collection of such data difficult. To solve this problem, different research groups have used thermodynamic modelling as a reliable tool for understanding and predicting phase behaviour. Thermodynamic calculation of equilibrium phase diagrams gives information about possible solubility limits and peritectic temperature, and can be used for materials design and industrial process optimisation. For example, thermodynamic calculations using the CALPHAD method have been performed for different binary phase diagrams including platinum-ruthenium (6–8), rhenium-tantalum (9), rhenium-tungsten (9) and rhenium-rhodium (10), where the regular solution model was applied and showed a good agreement with available experimental data.

The present paper aims to critically analyse recent experimental data and thermodynamic calculations with the application of mixing parameters for solid

and liquid phases in the Ir-Re system and to construct a reliable model for further investigations of the phase behaviour in the system described.

The Iridium-Rhenium System: Experimental Data

Experimental data on the Ir-Re system were obtained during the 1950s and 1960s using high-temperature melting and annealing of fine metallic powders in vacuum (11–13). A phase diagram was constructed based on the analyses of bimetallic solid solutions prepared at wide concentration intervals (14). As obtained, the peritectic phase diagram had three regions in the solid state: face-centred cubic (fcc) solid solutions in the Ir part, hexagonal close packed (hcp) solid solutions in the Re part and a broad two-phase region between the single-phase sections (Figure 1). The maximum solubilities of Re in Ir and Ir in Re at 1000°C were estimated as 28 at% and

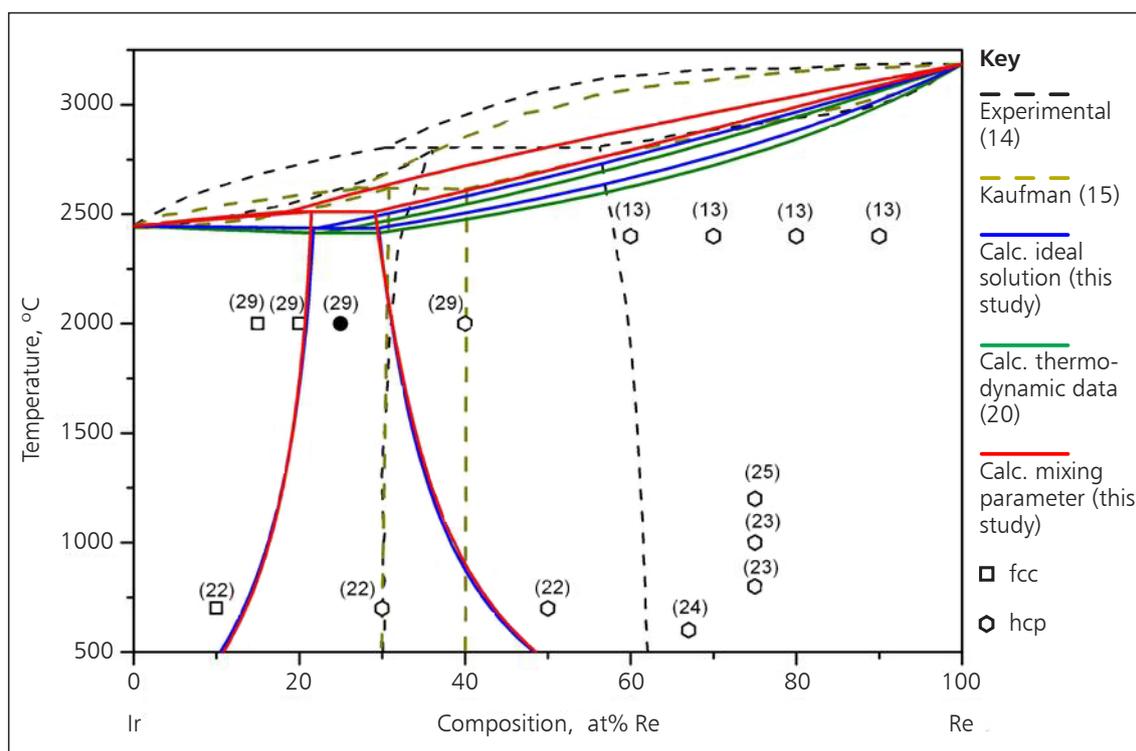


Fig. 1. Phase diagram for the iridium-rhenium system: experimental (14); proposed by Kaufman (15); calculated using the ideal solution model for all phases; calculated using thermodynamic data for solid solutions ${}^0L_{Ir,Re}^{hcp} = -652.2 + 0.83T$ and ${}^0L_{Ir,Re}^{fcc} = -1070.3 + 1.03T$ (20); and calculated using also a liquid mixing parameter of ${}^0L_{Ir,Re}^{liq} = 8000$. Squares and hexagons correspond to fcc and hcp solid solutions with the respective references. A two-phase sample with 25 at% Re is shown as a black circle. Compositions and preparatory temperatures are given according to Table I

39 at%, respectively, and the peritectic temperature was determined as 2800°C. The corresponding tie-lines for the liquidus part of the diagram were not examined experimentally and have been drawn schematically between the invariant reaction line and the melting points of the pure metals. The purities of the initial Ir and Re powders used for the preparation of the solid solutions were not reported (11–13). As single-phase samples showed unreliable atomic volumes (12) some experimental data are likely to be incorrect.

Two-component phase diagrams for Re with all pgms were later calculated by Kaufman using the ideal solution model (15). The correctness of the model was confirmed by further measurements of mixing volumes and formation enthalpy. The calculated and experimentally obtained phase diagrams were adequate for the majority of metallic systems with the exception of Re-Rh and Ir-Re. The disagreements between theoretical calculations and earlier and more recent experimental data can be explained in terms of the incorrectness of the corresponding experimental diagram as it occurs for the Re-Rh system (10, 16). Therefore high precision experimental data are needed to help understand the equilibrium behaviour of the Re-Rh and Ir-Re systems.

Thermodynamic data (integral enthalpy, entropy and Gibbs free energy), which are available for pure Ir (17, 18) and Re (19) and also for a number of Ir-Re solid solutions (20), might be taken into account to precisely describe the phase equilibria. Thirteen solid solutions across the full range of concentrations were prepared and used to determine Gibbs formation energies as a function of temperature and concentration by the electromotive force method with a solid electrolyte (rhenium oxides and fluorides) (20). Data interpretation was partly based on a previously published phase diagram (14). The experimental data obtained in the ranges 0 at% to 20 at% Re (fcc region) and 60 at% to 100 at% Re (hcp region) could be used to estimate the mixing energies for Ir-Re solid solutions. The following mixing parameters for hcp and fcc solid solutions can be extracted from the data and used for further phase diagram calculation:

$${}^0L_{Ir,Re}^{hcp} = -652.2 + 0.83T \quad (i)$$

$${}^0L_{Ir,Re}^{fcc} = -1070.3 + 1.03T \quad (ii)$$

Single source precursors are widely used to prepare different oxide and metallic phases (21). This approach has also been applied to the preparation of Ir-Re solid solutions under mild conditions using bimetallic

double complex salts as precursors (22–25). For example, an Ir_{0.50}Re_{0.50} solid solution can be obtained by thermal decomposition of [Ir(NH₃)₅Cl][ReCl₆] in a hydrogen or argon stream at 600°C. A variety of other precursors have been used to prepare a range of other solid solutions of differing compositions. As well as thermal decomposition of the individual double complex salts, Ir-Re metallic phases have also been prepared by thermolysis of their solid solutions (22). The isoformular compounds [Ir(NH₃)₅Cl]-[ReCl₆] and [Ir(NH₃)₅Cl][IrCl₆] are isostructural and their co-crystallisation gives the single-phase solid solution [Ir(NH₃)₅Cl][IrCl₆]_x[ReCl₆]_{1-x}. Thermal decomposition of the latter results in a metallic phase with the composition Ir_{0.5+0.5x}Re_{0.5-0.5x}. In the same manner, Ir_xRe_{1-x} solid solutions can be prepared by thermal decomposition of the easily synthesised compound (NH₄)₂[IrCl₆]_x[ReCl₆]_{1-x}. This method allows a broad range of metallic compositions to be prepared and can also be applied to other systems such as Ir-Os (27) and Ir-Ru (28).

Recently, several Ir-Re solid solutions were prepared by high-pressure high-temperature annealing of metallic powders (29). To improve the solidus part of the diagram, the composition of the Ir-Re mixtures was selected from the two-phase region. High-pressure high-temperature annealing was chosen as a quick and reproducible method for preparation of metallic phases with extremely high melting points. Special boron nitride reactors with graphite covering were used to avoid the formation of oxides and nitrides.

All of the metallic phases described in the literature have been characterised using powder X-ray diffraction, elemental analysis and scanning electron microscopy, with lattice parameters and atomic volumes measured. The data for all single phases known in the literature are summarised in **Table I**. According to Zen's law for hcp-fcc bimetallic systems, the atomic volumes V/Z (where V is the volume of the elemental cell and Z corresponds to the number of atoms in the elemental cell, with $Z = 6$ for hcp and $Z = 4$ for fcc) should display nearly linear dependence on composition with a relatively small positive or negative deviation (30, 31). For systems with phase separation, V/Z dependences need to be determined separately for each single-phase part of the phase diagram. Solid solutions in the Ir-Re system show a small negative deflection from linearity (<2%), which roughly reflects the idealness of the system (**Figure 2**). The corresponding V/Z dependence is described by the following second order polynomial functions:

Table I
Crystallographic Data on Known Metallic Phases in the Iridium-Rhenium System

Composition	Cell parameters, a, Å c, Å	Cell parameters ratio, c/a	Space group	Density, D_x , g cm ⁻³	Atomic volume, V/Z , Å ³	Preparation conditions	Reference
Ir	3.83998	–	$Fm\bar{3}m$	22.56	14.15	Melting point 2446°C	(26, No. 46-1044)
Ir _{0.90} Re _{0.10}	3.842(2)	–	$Fm\bar{3}m$	22.44	14.18(2)	Thermal decomposition in H ₂ of [Ir(NH ₃) ₅ Cl][IrCl ₆] _{0.8} [ReCl ₆] _{0.2} (700°C, 1 h)	(22)
Ir _{0.85} Re _{0.15}	3.843(2)	–	$Fm\bar{3}m$	22.39	14.19(2)	High-pressure high-temperature annealing (2000°C, 4 GPa, 15 min)	(29)
Ir _{0.80} Re _{0.20}	3.845(2)	–	$Fm\bar{3}m$	22.33	14.21(2)	High-pressure high-temperature annealing (2000°C, 4 GPa, 5 min)	(29)
Ir _{0.70} Re _{0.30}	2.736(2) 4.390(3)	1.605	$P6_3/mmc$	22.22	14.23(4)	Thermal decomposition in H ₂ of [Ir(NH ₃) ₅ Cl][IrCl ₆] _{0.4} [ReCl ₆] _{0.6} (700°C, 1 h)	(22)
Ir _{0.60} Re _{0.40}	2.752(1) 4.371(2)	1.588	$P6_3/mmc$	22.00	14.33(4)	High-pressure high-temperature annealing (2000°C, 4 GPa, 5 min)	(29)
Ir _{0.50} Re _{0.50}	2.749(2) 4.371(3)	1.590	$P6_3/mmc$	21.97	14.30(4)	Thermal decomposition in H ₂ of [Ir(NH ₃) ₅ Cl][ReCl ₆] (700°C, 1 h)	(22)
Ir _{0.50} Re _{0.50}	2.743(2) 4.380(3)	1.597	$P6_3/mmc$	22.02	14.27(4)	Thermal decomposition in H ₂ of [Ir(NH ₃) ₅ Cl][ReBr ₆] (700°C, 1 h)	(22)
Ir _{0.40} Re _{0.60}	2.7545(7) 4.3739(12)	1.588	$P6_3/mmc$	21.79	14.37(8)	Annealing in vacuum (2400°C, 1 h)	(13)
Ir _{0.33} Re _{0.67}	2.765(5) 4.383(6)	1.585	$P6_3/mmc$	21.59	14.48(8)	Thermal decomposition in H ₂ of [Ir(NH ₃) ₅ Cl][ReO ₄] ₂ (600°C, 1 h)	(24)

(Continued)

Table I (Continued)

Composition	Cell parameters, a, Å c, Å	Cell parameters ratio, c/a	Space group	Density, D_x , g cm ⁻³	Atomic volume, V/Z , Å ³	Preparation conditions	Reference
Ir _{0.30} Re _{0.70}	2.7578(7) 4.3865(12)	1.591	<i>P6₃/mmc</i>	21.61	14.45(8)	Annealing in vacuum (2400°C, 1 h)	(13)
Ir _{0.25} Re _{0.75}	2.758(2) 4.393(3)	1.593	<i>P6₃/mmc</i>	21.55	14.47(4)	Thermal decomposition in He of [Ir(NH ₃) ₅ Cl] ₂ [Re ₆ S ₈ (CN) ₆]:3H ₂ O (1200°C, 1 h)	(25)
Ir _{0.25} Re _{0.75}	2.758(2) 4.394(3)	1.593	<i>P6₃/mmc</i>	21.54	14.47(4)	Thermal decomposition in H ₂ of (NH ₄) ₂ [IrCl ₆] _{0.25} [ReCl ₆] _{0.75} (1000°C, 1 h)	(23)
Ir _{0.25} Re _{0.75}	2.758(2) 4.392(3)	1.592	<i>P6₃/mmc</i>	21.55	14.47(4)	Annealing in vacuum of Ir _{0.25} Re _{0.75} (800°C, 48 h)	(23)
Ir _{0.20} Re _{0.80}	2.7608(7) 4.4052(12)	1.596	<i>P6₃/mmc</i>	21.40	14.54(8)	Annealing in vacuum (2400°C, 1 h)	(13)
Ir _{0.10} Re _{0.90}	2.7610(7) 4.4314(12)	1.605	<i>P6₃/mmc</i>	21.21	14.63(8)	Annealing in vacuum (2400°C, 1 h)	(13)
Re	2.760 4.458	1.615	<i>P6₃/mmc</i>	21.03	14.71	Melting point 3180°C	(26, No. 5-702)

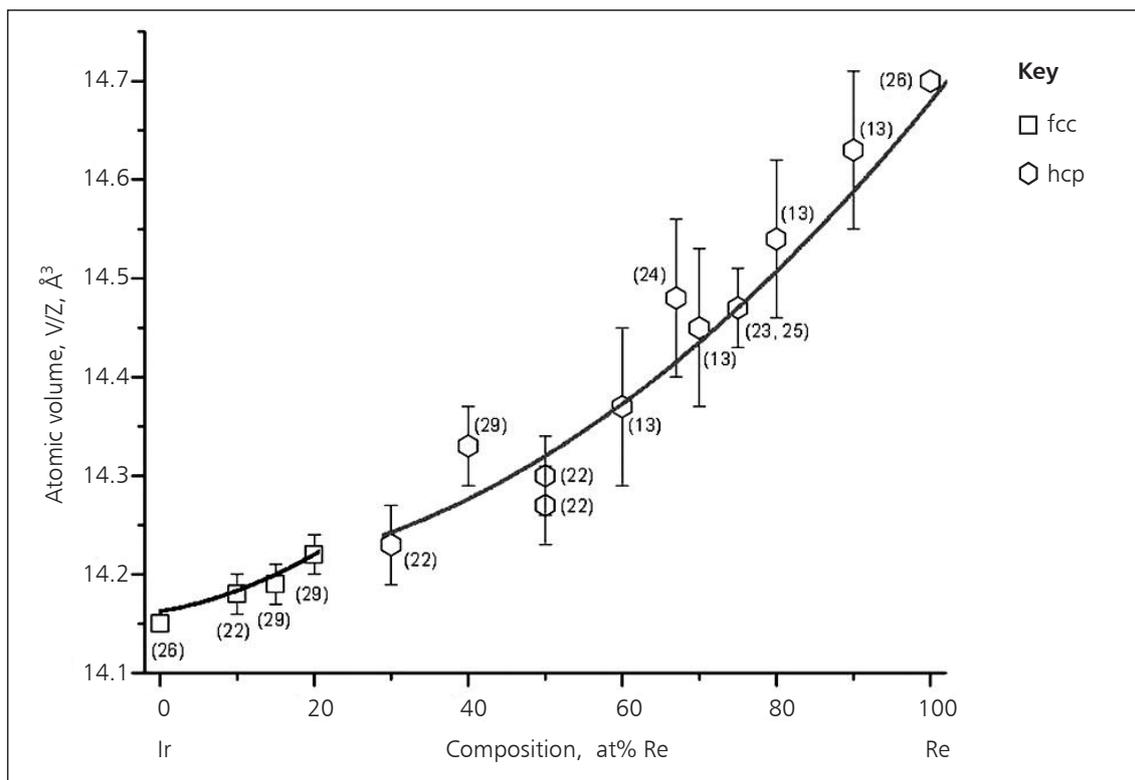


Fig. 2. Atomic volumes for known fcc and hcp iridium-rhenium solid solutions with the corresponding references. Compositions and V/Z volumes are given according to Table I

$$(V/Z)_{\text{hcp}} = 14.20 - 0.16 \times X_{\text{Re}} + 0.53 \times X_{\text{Re}}^2 \quad (\text{iii})$$

$$(V/Z)_{\text{fcc}} = 14.15 + 0.17 \times X_{\text{Re}} + 0.82 \times X_{\text{Re}}^2 \quad (\text{iv})$$

where X_{Re} is the proportion of Re (at%) in the binary solid solution. The given functions can be used to estimate the composition of Ir-Re solid solutions with known lattice parameters.

The reported lattice parameters for $\text{Ir}_{0.72}\text{Re}_{0.28}$ ($a = 3.65 \text{ \AA}$, $V/Z = 12.16 \text{ \AA}^3$) and $\text{Ir}_{0.90}\text{Re}_{0.10}$ ($a = 3.76 \text{ \AA}$, $V/Z = 13.29 \text{ \AA}^3$) fcc solid solutions prepared by annealing of the pure metals at 2500°C (12) seem to be incorrect, since the atomic volumes are less than the corresponding value for pure Ir. The reduced lattice parameters can be attributed to the formation of individual or mixed compounds of Ir and/or Re with light elements (primarily carbon, nitrogen and oxygen) as well as metallic impurities (mainly other light and heavy metals) in the initial Ir and Re powders. Therefore, these solid solutions are not taken into consideration in the present work.

Overall, it was found that phases prepared using different methods correspond to the same curves on the experimental phase diagram, showing that solid solutions obtained by thermal decomposition of single-source precursors or by high-temperature and high-pressure annealing are nearly equivalent and therefore both methods can be applied to the preparation of Ir-Re solid solutions. Heating the metallic solid solutions up to their melting points does not change the phase composition or lattice parameters, indicating that the phases are thermodynamically stable. The squares and hexagons in Figure 1 schematically show composition and preparatory temperatures for known single-phase Ir-Re solid solutions. Single-phase fcc solid solutions can be obtained up to 13 at% Re at 1200°C , while the maximum concentration for hcp solid solutions was 25 at% Re (16, 32). The maximum solid phase solubility of Ir in Re is 68 at%, and that of Re in Ir is 20 at% at 2000°C (29) which is comparable with the data obtained by Filatov (16, 32). This experimental

data for solid solutions is inconsistent with the previously known phase diagram.

Theoretical Calculations

The phase diagram for the Ir-Re system was calculated using the PANDAT 8 software (33) and the Scientific Group Thermodata Europe (SGTE) v. 4.4 thermodynamic database (34) in order to understand the inconsistency between the known phase diagram and the experimental data, and to help classify the available experimental data. Mixing energies used for the regular solution model calculated from the thermodynamic data obtained in (20) were compared with those obtained by calculation based on the ideal solution model.

Three calculated phase diagrams with different mixing parameters are shown in **Figure 1**. It can be seen that the calculated solid solubility limits are not in accordance with the existing experimental phase diagram (14). Nevertheless, there is good agreement between the solidus part of the theoretical phase diagrams and recently obtained experimental data for all single-phase Ir-Re solid solutions. The comparison clearly shows that the previously known experimental phase diagram for the Ir-Re system should be reconsidered and corrected for both the Ir (fcc solid solutions) and Re (hcp solid solutions) parts.

Calculations performed with or without the experimental thermodynamic data for Ir-Re solid solutions produce nearly identical results, which indicates that the mixing energies have little influence on the solid equilibria. Direct thermodynamic and equilibrium data for the liquidus part of the diagram are absent which makes it impossible to analyse the high-temperature part of the diagram. Diagrams calculated without mixing parameters or with mixing parameters only for the solid phases appear to be eutectic. The corresponding eutectic temperatures are 2436°C (calculation using the ideal solution model for solid and liquid phases) and 2414°C (calculation using mixing parameters for solid solutions only), which is close to the melting point of pure Ir (2446°C) and shows a need for precise direct observation of the invariant reaction temperature. Optimisation of the ${}^0L_{Ir,Re}^{liq}$ parameter for the liquid phase results in a change in the invariant temperature without significant change in the two-phase field fcc+hcp. A positive value of ${}^0L_{Ir,Re}^{liq} = 4000$ results in a eutectic temperature of 2465°C. The peritectic temperature of 2512°C can be obtained with ${}^0L_{Ir,Re}^{liq} = 8000$. Mixing parameters ${}^0L_{Ir,Re}$ for hcp and fcc solid solutions and

liquid phases are negative and relatively small which indicates nearly ideal behaviour of the Ir-Re system in both the solid and liquid parts.

Recommendations for Further Work

Despite the nearly ideal behaviour indicated above, further high precision thermodynamic and equilibrium data need to be included in the CALPHAD-based phase diagram optimisation to obtain better agreement between experimental and calculated data. *Ab initio* calculations would also improve our knowledge of the system discussed. The calculated fcc+hcp two-phase field below 1000°C is broad and would also benefit from further improvement, since an hcp solid solution with a composition $Ir_{0.7}Re_{0.3}$ (22) was obtained in the two-phase region of the calculated phase diagram.

A critical analysis of all available experimental data on solid solutions of platinum and rhenium would enable a consistent database of thermodynamic data and phase diagrams for these systems to be constructed. This would also facilitate the prediction of the relevant ternary and quaternary phase diagrams. In addition, precise investigation of the physical properties of Ir-Re solid solutions such as hardness, electrical and thermal conductivity as well as thermoelectric characteristics and catalytic activity will improve the practical importance of the system discussed. Since not only Ir-Re but also oxide-containing compositions are of interest, the study of temperature, pressure and oxidation stability of metallic single- and poly-phase particles would also be beneficial.

The ordering in the Ir-Re system is still not understood and experimental investigation into the stability and properties of the intermetallic compounds will help reveal this. Nevertheless, four ordered phases: Ir_8Re (Pt₈Ti structure), Ir_2Re (ZrSi₂), $IrRe$ (B19 structure designation) and $IrRe_3$ (D0₁₉) – stable at zero temperature have been predicted using high-throughput *ab initio* calculation (35). Further modelling of the ordered phases at high temperature as well as calculation of temperature dependent thermodynamic functions will allow ordered phases to be included in the phase diagram calculation.

Conclusions

In conclusion, the phase diagram proposed in this paper for the Ir-Re system serves as a reliable model for representing the experimental data available to date. Additional thermodynamic investigations especially on the melting of high purity metals will

further improve the liquid part of the phase diagram. The author believes that the data presented in this article will inspire further investigations on binary systems based on rhenium and the pgms, especially direct calorimetric and electrochemical examination of thermodynamic functions such as activity and mixing parameters for Ir-Re metallic solution in the solid and liquid states, which will in turn allow the practical usefulness of these alloys to be extended.

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