The Nb-Pt (Niobium-Platinum) System

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Equilibrium Diagram

The Nb-Pt phase diagram was studied by [60Kna], [61Kim], and [85Wat]. The assessed phase diagram of the Nb-Pt system is shown in Fig. 1. The phase boundaries and the reaction temperatures are based primarily on the work of [85Wat], who investigated the system over the entire composition range in the temperature interval of 1000 to 2400 °C by metallography, electron microprobe, XRD, and thermal analysis of 35 arc-melted alloy samples prepared from 99.6% pure Nb and 99.9% pure Pt. The Nb-rich alloys up to 20 at.% Pt were given homogenization anneals in a vacuum furnace at 1900 °C for 3 hours, and the other compositions were annealed at 1600 to 1700 °C for 6 to 12 hours and quenched at rates of 30 to 100 °C per second. The compositions and the temperatures in Fig. 1 have an accuracy of ±1 at.% and ±10 °C, respectively.

The present diagram differs significantly from the one published by [61Kim] and the previous reviews of [Elliott], [Metals], [Hultgren, B], and [Massalski1].

Six nonstoichiometric intermetallic compounds exist: Nb_3Pt, "~Nb_2Pt," $Nb_{1-x}Pt_{1+x}$, α′Pt, $NbPt_2$, and $NbPt_3$. "Nb_2Pt" is usually referred to as σ phase. The two Nb-rich phases, Nb_3Pt and σ, are known as Frank-Kasper phases.

The equilibrium phases of the Nb-Pt system are: (1) the liquid, L; (2) the bcc solid solution of Pt in αNb phase with a maximum solubility of Pt up to ~12 at.%, at 2040 °C; (3) the cubic Nb_3Pt crystallizing peritectically at 2040 °C; (4) the tetragonal σ phase forming peritectically at 1800 °C; (5) the orthorhombic $Nb_{1-x}Pt_{1+x}$ phase crystallizing peritectically at 1750 °C; (6) the high temperature α′ Pt phase of unknown structure, forming peritectically at 1780 °C; (7) the orthorhombic $NbPt_2$ forming by peritectic reaction at 1990 °C; the $NbPt_3$ phase solidifying congruently at 2040 °C; and (9) the fcc solid solution of Nb in αPt with solubility up to ~20 at.% Nb at 2000 °C.

Liquidus and Solidus

The melting point work of [60Kna] showed that the liquidus on the Pt-rich side of the phase diagram has a maximum near 75 at.% Pt. It was later confirmed by [85Wat], who made an approximate determination of the liquidus by microscopic examination of the melting point samples with visual estimates of the relative amounts of solid and liquid phases present in each sample. Therefore, the liquidus in Fig. 1 is shown by broken lines.

The solidus boundaries were determined by [85Wat] employing metallography, XRD, and thermal methods for phase analysis of the samples quenched after homogenization anneals. With solidus temperatures measured to an accuracy of ±10 °C and compositions within ±1 at.%, the solidus lines are known with a high degree of confidence and are, hence, represented by solid lines. Nine invariant reactions including eight three-phase equilibria were identified in addition to the normal melting of the pure end-members. The compositions of the phases involved in the invariant reactions and the temperatures of equilibria are presented in Table 1.

Terminal Solid Solutions

Both terminal solid solution phases exhibit considerable solid solubility. [60Kna] observed larger solid solubilities in the terminal phases of the Nb-Pt system as compared to those of Nb alloys with Rh, Ir, Ru, and Os. This was confirmed by [85Wat]. The heat treated and quenched alloys were examined for determination of composition by electron microprobe, XRD, and metallography. The terminal solid solution boundary of bcc Nb was established by analysis of equilibrated and quenched alloys in the composition range 10 to 18 at.% Pt. The solubility

Table 1 Special Points of the Assessed Nb-Pt Phase Diagram

Reaction	Compositions of the respective phases, at.% Pt			Temperature (a), °C	Reaction type
L ↔ Nb		0		2469	Melting
L + (αNb) ↔ Nb_3Pt	~26	~12	~19	2040	Peritectic
L + Nb_3Pt ↔ σ	36.4	28	31	1800	Peritectic
L ↔ $Nb_{1-x}Pt_{1+x}$ + σ	43	51.2	38	1700	Eutectic
L + α′Pt ↔ $Nb_{1-x}Pt_{1+x}$	47	57	52	1750	Peritectic
L + $NbPt_2$ ↔ α′Pt	49	65	57	1780	Peritectic
α′Pt ↔ $Nb_{1-x}Pt_{1+x}$ + $NbPt_2$	57.5	54	66	1670	Eutectoid
L + $NbPt_3$ ↔ $NbPt_2$	66	74	67	1990	Peritectic
L ↔ $NbPt_3$		75		2040	Congruent
L + $NbPt_3$ ↔ (αPt)	82.5	76	80	2000	Peritectic
L ↔ αPt		100		1769	Melting

(a) Temperature is within ±10 °C.

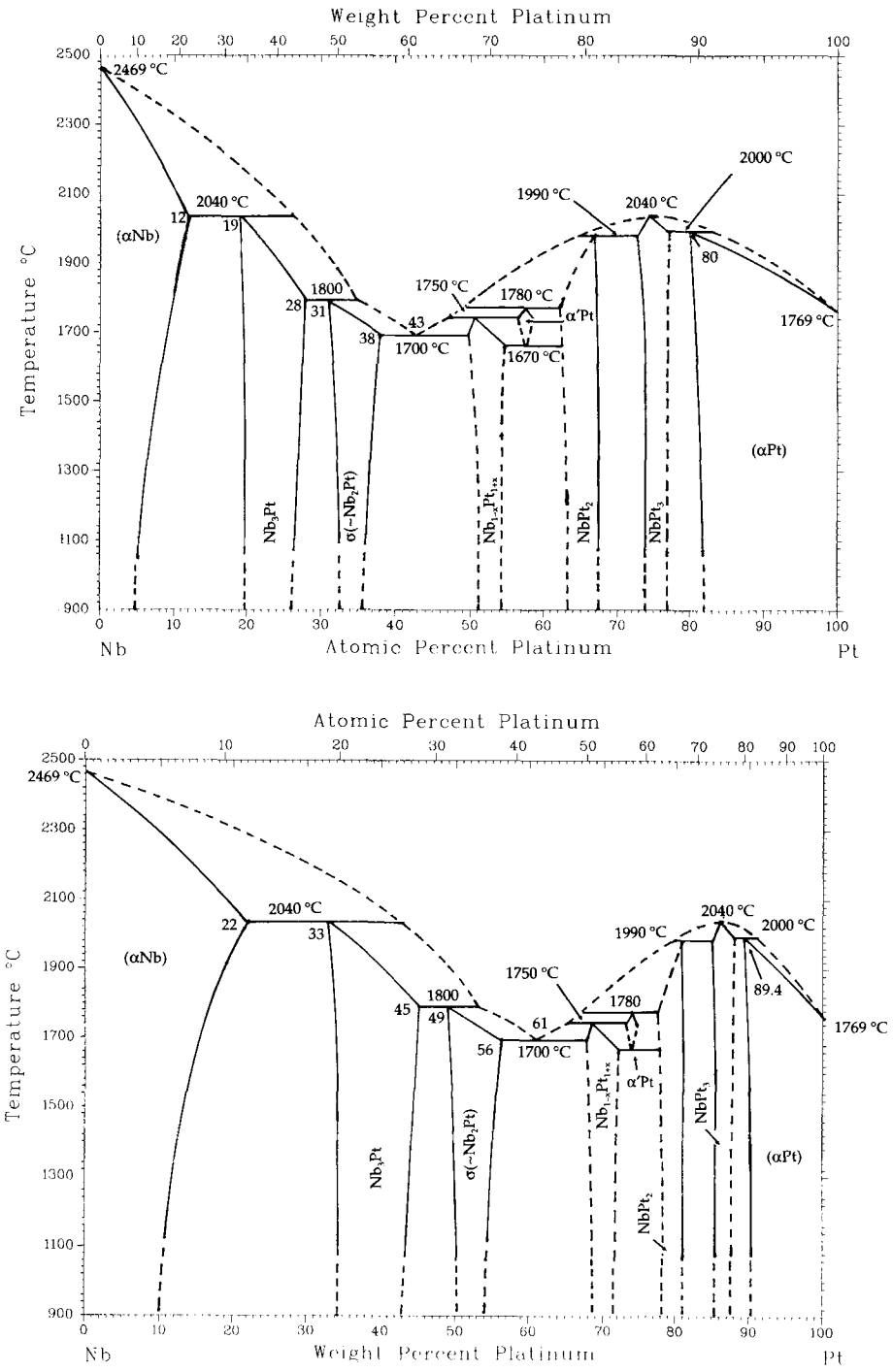

Fig. 1 Assessed Nb-Pt phase diagram.

of Pt in αNb solid solution reaches ~12 at.% at 2040 °C, but it decreases at lower temperatures and remains only 5 at.% at 1150 °C.

The fcc αPt solid solution dissolves ~20 at.% Nb at 2000 °C and ~18 at.% Nb at lower temperatures. The metastable coherent structure [85Wat] of the 80 at.% Pt alloy annealed at 1500 °C suggests that the solvus boundary of the αPt phase may be metastable. It is quite likely that the true equilibrium boundary is located at lower Nb concentrations.

Crystal Structures and Lattice Parameters

The invariant reactions of Fig. 1 are presented in Table 1. Crystal structures for the equilibrium phases are presented in Table

Table 2 Nb-Pt Crystal Structure Data

Phase	Composition, at.% Pt	Pearson symbol	Space group	Strukturbericht designation	Prototype	References
(αNb)	0	*cI*2	$Im\bar{3}m$	*A*2	W	[Massalski2]
Nb_3Pt	19 to 28	*cP*8	$Pm\bar{3}n$	*A*15	Cr_3Si, βW	[56Gre], [55Gel]
σ(Nb_2Pt)	31 to 38	*tP*30	$P4_2/mnm$	$D8_b$	βU	[56Gre]
$Nb_{1-x}Pt_{1+x}$	51 to 53	*oP*4	*Pmma*	*B*19	AuCd	[64Gie1], [64Mal]
α′Pt	57	...	...	Not determined	...	[64Gie2]
$NbPt_2$	~67	*oI*6	*Immm*	...	$MoPt_2$	[64Gie1], [65Gie], [64Mal]
α$NbPt_3$	~75	*oP*8	*Pmnm*	$D0_a$	β$TiCu_3$	[64Gie1], [64Mal]
β$NbPt_3$	~76	*mP*48	$P2_1/m$	...	β$NbPt_3$	[64Gie1], [65Gie]
(αPt)	80 to 100	*cF*4	$Fm\bar{3}m$	*A*1	Cu	[Massalski2]

Table 3 Nb-Pt Lattice Parameter Data

Phase	Composition, at.% Pt	Lattice parameters, nm			Comment	References
		a	*b*	*c*		
αNb	0	0.33004	...	...	...	[Massalski2]
	5	0.3277	...	...	...	[85Wat]
	10	0.3260	...	...	...	[85Wat]
Nb_3Pt	...	0.511	...	...	...	[56Gre]
	...	0.5153	...	...	...	[55Gel]
	20	0.5182	...	...	...	[85Wat]
	25	0.5152	...	...	...	[68Reu]
	28	0.5139	...	...	...	[85Wat]
	19 to 28	0.5147 to 0.5166	...	...	...	[61Kim]
σ	32	0.9940	...	0.5145	...	[83Wat], [85Wat]
	38	0.9902	...	0.5132	...	[83Wat], [85Wat]
	...	0.991	...	0.512	...	[61Kim]
$(Nb_{1-x}Pt_{1+x})$?	50	0.2780	0.4983	0.4611	...	[83Wat], [64Gie1]
	50	0.278	0.498	0.462	...	[65Dwi]
α$NbPt_3$	75	0.5534	0.4873	0.4564	...	[64Gie1]
β$NbPt_3$	75	0.5537	0.4870	0.2733	α = 90° 32′	[64Gie1]
αPt	82	0.3940	...	...	...	[85Wat]
	85	0.3935	...	...	...	[85Wat]
	90	0.3927	...	...	...	[85Wat]
	95	0.3924	...	...	...	[85Wat]
Pt	100	0.39236	...	...	...	[Massalski2]

2. The lattice parameters of a number of alloys with different compositions for four intermediate phases and two terminal solid solutions are given in Table 3.

Intermediate Alloy Phases

Six intermediate phases exist: Cr_3Si-type, cubic Nb_3Pt; βU-type, tetragonal close-packed σ phase, ~Nb_2Pt; AuCd-type, orthorhombic $Nb_{1-x}Pt_{1+x}$; α′ Pt phase of unknown structure; $MoPt_2$-type, orthorhombic $NbPt_2$; β$TiCu_3$-type, orthorhombic low-temperature form, α$NbPt_3$; and monoclinic high-temperature modification, β$NbPt_3$.

Nb_3Pt Phase

First discovered by [55Gel], Nb_3Pt was identified by [56Gre] as having a Cr_3Si-type (βW, *A*15) structure, as a result of their studies of 7 alloys in the composition range 25 to 57 at.% Pt, annealed and quenched at 1000 °C. Nb_3Pt was further confirmed by [61Kim], [64Gie1], [65Gie], [83Wat], and [85Wat]. [85Wat] found a homogeneity range in Nb_3Pt with a composition span of 19 to 28 at.% Pt at 1800 ± 10 °C; this span narrows at lower temperatures. The solvus and the solidus boundary of the phase are known to good precision. Nb_3Pt forms as an ordered structure from the disordered bcc αNb at 2040 ± 10 °C by peritectic reaction: L + αNb ↔ Nb_3Pt.

Nb_3Pt is a highly ordered (order parameter, $S = 0.93$) phase [55Gel]. The ideal composition is A_3B, but the phase is also stable at significantly deviating compositions implying that the crystallographic sites in the lattice are not exclusively occupied by only one chemical element. The remarkable stability of Nb_3Pt and other *A*15-type phases [68Reu] in alloys of the transition metals is attributed to the intimate involvement of the d-electrons in the bonding and the interdependence of the electronic structure and the ability of the atoms to undergo deformation to conform to the geometrical packing requirements.

Table 4 C_p data for σ phase at 38 at.% Pt

Temperature, K	C_p, J/g-atom K
5	0.2339
10	0.0950
13	0.1904

Table 5 Gibbs Energy Limits of Solid Alloys at 1727 °C

Phase	Composition, at.% Pt	$\Delta_f G$, kJ/g-atom
Nb_3Pt	25	< −10
Nb_2Pt	38	< −16
$Nb_{1-x}Pt_{1+x}$	52	< −20
$NbPt_2$	67	< −29
$NbPt_3$	75	< −33

In a recent investigation by [88Rob] on Nb-Pt alloys with compositions ranging between 16 and 32 at.%, undercooling by as much as 585 K was reported for liquid alloys. The Nb-Pt alloys of 16 to 18 at.% Pt yielded, on undercooling, a single Nb_3Pt phase in contrast to the αNb and Nb_3Pt phases expected on the basis of Fig. 1.

σ Phase

The σ phase, ~Nb_2Pt, also first reported by [56Gre] together with Nb_3Pt, was first identified with a tetragonal close-packed crystal structure that was later confirmed by [61Kim], [64Gie1], [64Mal], and [85Wat]. Like Nb_3Pt, the σ phase is also nonstoichiometric and highly stable. It has a homogeneity range of 31 to 38 at.% Pt at 1700 °C narrowing at lower temperatures. The lattice parameters of the optimum compositions are listed in Table 3. The σ phase crystallizes from a mixture of Nb_3Pt + melt at 1800 ± 10 °C by the peritectic reaction, Nb_3Pt + L ↔ σ phase.

$Nb_{1-x}Pt_{1+x}$ Phase

[61Kim] found evidence for an equiatomic composition alloy which was identified by [64Gie2] and [65Gie] to possess the AuCd-type (*B*19) orthorhombic structure. The phase is reported to be hyperstoichiometric [85Wat] and is represented as $Nb_{1-x}Pt_{1+x}$. It forms at 1700 ± 10 °C and ~43 at.% Pt by a eutectic reaction, L ↔ σ + $Nb_{1-x}Pt_{1+x}$, and there is agreement [61Kim, 85Wat] as to the temperature and nature of the reaction. There is no structural data on 50 and 51 at.% Pt alloys at 1700 °C, so it is not justifiable to anchor the Nb-rich phase boundary of the single-phase field of $Nb_{1-x}Pt_{1+x}$ at 1700 °C at ~51.2 at.% Pt, as seen in Fig. 1 of [85Wat] and [Massalski2]. Microprobe analysis [85Wat] demonstrated the existence of a phase boundary at 1150 °C at 52 at.% Pt and at 1500 °C at 51 at.% Pt. The existence of a two-phase field at 49 at.% Pt in the temperature range 1500 to 1700 °C is consistent with the likely farming out of the single-phase field from 52 at.% Pt at 1150 °C to ~50 at.% Pt at 1700 °C via 51 at.% Pt at 1500 °C. Hence the Nb-rich phase boundary at 1700 °C is shifted to ~50 at.% Pt. XRD of 53 at.% Pt alloy showed the single phase down to 1600 °C, but two phases at 1500 °C and below, indicating a decomposition that is in contradiction with the microprobe results. In view of insufficient information in the range 1500 to 1700 °C and conflicting observations at and below 1500 °C, the Nb-rich phase boundary all along, and the Pt-rich one below 1600 °C are considered tentative and are shown by dashed lines in the assessed phase diagram. Hence the extent of the phase field of $Nb_{1-x}Pt_{1+x}$ in the assessed diagram is different from those of [85Wat] and [Massalski2]. A 50 at.% alloy is a two-phase mixture according to [85Wat], hence the lattice parameters determined for this composition by [64Gie1], [65Dwi], and [83Wat] are not representative of $Nb_{1-x}Pt_{1+x}$.

α′ Pt Phase

[64Gie1], [64Gie2], and [65Gie] reported a phase with a composition close to $Nb_{1-x}Pt_{1+x}$. [64Gie1] found the phase to be stable only at high temperatures, *i.e.*, 1670 to 1780 °C. [75Flu] established the existence of α′Pt on the basis of their solidus measurements by DTA and metallography. The thermal arrests observed by [75Flu] in 57 and 62 at.% Pt alloys agree completely with the temperatures of the peritectic and eutectoid reactions of α′Pt [85Wat]. The temperature of the eutectoid decomposition of α′Pt is precisely known from the metallography of 55 and 57.5 at.% Pt alloys quenched from above and below 1670 °C.

The crystal structure of α′Pt remains undetermined. By analogy to V-Pt and Nb-Pd, [85Wat] speculated that this phase was an extension of fcc αPt solid solution. XRD at high temperatures is expected to reveal the structure conclusively.

This phase forms from $NbPt_2$ by a peritectic reaction at 1780 ± 10 °C and undergoes another peritectic reaction at 1750 ± 10 °C: α′Pt + L ↔ $Nb_{1-x}Pt_{1+x}$. On cooling to 1670 ± 10 °C, it decomposes eutectoidally to a mixture of $Nb_{1-x}Pt_{1+x}$ and $NbPt_2$.

$NbPt_2$ Phase

$NbPt_2$ is essentially a nonstoichiometric compound having a narrow homogeneity range spanning over 2 to 3 at.% at 1780 °C [85Wat]. It forms by the peritectic reaction, $NbPt_3$ + L ↔ $NbPt_2$, at 1990 ±10 °C as observed in a solidus study of a 70 at.% Pt alloy. It crystallizes with orthorhombic $MoPt_2$-type structure [64Gie, 64 Mal, 65 Gie].

[61Kim] suggested that the $NbPt_2$ phase is an extension of αPt solid solution, but [65Gie], [64Mal, and [85Wat] assigned $NbPt_2$ a distinct crystal structure.

The Nb-rich boundary of the $NbPt_2$ phase [85Wat] is tentative because there is no information as to whether the alloys in the composition range 62.5 to 67 at.% Pt are monophasic or biphasic. Hence it is presumptuous to draw the Nb-rich phase boundary as shown in [85Wat]. A judicious choice would be a tentative phase field extending up to 62.5 at.% Pt below 1780 °C and to higher compositions in the range 1780 to 1990 °C. Hence the Nb-rich phase boundary of $NbPt_2$ in the assessed phase diagram is tentatively shifted towards the left as compared to those of [85Wat] and [Massalski2].

$NbPt_3$ Phase

The 75 at.% Pt melt solidifies congruently to $NbPt_3$ on cooling to 2040 ± 10 °C. Two polymorphs of $NbPt_3$ were identified and characterized. The low-temperature phase designated as α$NbPt_3$ [64Gie1, 64Mal] possesses an orthorhombic Cu_3Ti-

$D0_a$ type structure with two-layer stacking sequence. The high-temperature polymorph is $\beta NbPt_3$, identified as a monoclinic structure, confirmed by [85Wat] with twelve-layer stacking sequence and the same layer type as $\alpha NbPt_3$. Earlier [61Dwi] reported a tetragonal structure ($D0_{22}$) for $NbPt_3$, which was isotypic with Al_3Ti. It is isomorphous with $\beta TaPt_3$. [64Gie1] suggested that the $\alpha NbPt_3$ phase maybe be stabilized with respect to $\beta NbPt_3$ by the trace amount of interstitial impurities. [64Mal] indicated that $\alpha NbPt_3$ is stable below 1000 °C and $\beta NbPt_3$ has a Pt content higher than the stoichiometric composition. The regions of thermal stability of the two forms of $NbPt_3$ are not yet established. Additional work by XRD, including high-temperature measurements, is required to clarify the stability ranges of the α and β forms. $NbPt_3$ undergoes peritectic reactions at 1990 ± 10 °C, $NbPt_3 + L \leftrightarrow NbPt_2$, and at 2010 ± 10 °C, $NbPt_3 + L \leftrightarrow \alpha Pt$.

The Nb-rich boundary of the homogeneity region of $NbPt_3$ is substantiated by microprobe results from a 74 at.% alloy [85Wat] whereas on the Pt-rich end no structural information is available about intermediate alloys between 75 and 77.5 at.% Pt, as to whether they are monophasic or biphasic. Hence the Pt-rich phase boundary of $NbPt_3$ is considered tentative and is drawn accordingly by a dashed line in Fig. 1.

Thermodynamics

C_p measurements were reported by [64Buc] for a 38 at.% Pt alloy in the temperature range 4 to 13 K. Selected values are listed in Table 4.

The only experimental data on Nb activity from 50 ± 4 at.% Pt alloy was reported by [84Gib], and these are given by $G_{Nb}/R = -4.3 \pm 2.0$ kK at 2594 K. [73Bre] calculated the limits of Gibbs energies of the solid phases of the Nb-Pt system from the fact that the reaction $NbC + 3Pt \rightarrow NbPt_3 + C$ occurs spontaneously at 1727 °C, i.e., $\Delta G_R \leq 0$. The data on limits of ΔG are presented in Table 5.

The σ phase exhibits a superconducting transition. [61Bla], and [64Buc] reported T_c values of 3.73, 2.4, and 4.21 K for compositions of 37.5, 37.5, and 38 at.% Pt, respectively. Having largest mean atomic volumes, V, among the various phases of the Nb-Pt system [85Wat], the Nb_3Pt and σ phases are expected to have the highest T_c values in accordance with the direct proportionality relationship between V and T_c [57Mat]. As a result, it is highly likely that Nb_3Pt has T_c very close to that of σ phase (~ 4 K) whereas $Nb_{1-x}Pt_{1+x}$, $NbPt_2$, and $NbPt_3$ are likely to have progressively decreasing T_c parallel to their respective V. $Nb_{1-x}Pt_{1+x}$ and $NbPt_2$ exhibit normal conductivity [66Sad] down to 1.39 and 1.46 K respectively, but their T_c are not yet known. No conductivity data exist for $NbPt_3$.

Suggestions for Future Experimental Work

Despite an in-depth study of the Nb-Pt system by [85Wat] and others, discrepancies exist, and some areas in the phase diagram remain inconclusive. The features deserving careful reinvestigation are:

- The αPt solvus needs to be reexamined carefully in samples equilibrated for longer periods.
- The low temperature domain of stability of $Nb_{1-x}Pt_{1+x}$ below 1500 °C is yet to be established.
- The crystal structure of the high-temperature α'Pt phase needs to be determined by high-temperature XRD.
- The Nb-rich end of $NbPt_2$ (up to ~63 at.% Pt) and the Pt-rich boundary of $NbPt_3$ (up to ~77 at.% Pt) need to be redetermined for authentic compositional information.
- Nb_3Pt, $Nb_{1-x}Pt_{1+x}$, $NbPt_2$, and $NbPt_3$ deserve close scrutiny for their superconducting behavior, the first one around 4 K and the remaining three below 1.4 K.

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# Indicates presence of a phase diagram.

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