

The Ta-V (Tantalum-Vanadium) System

180.9479

50.9415

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Phase Relationships

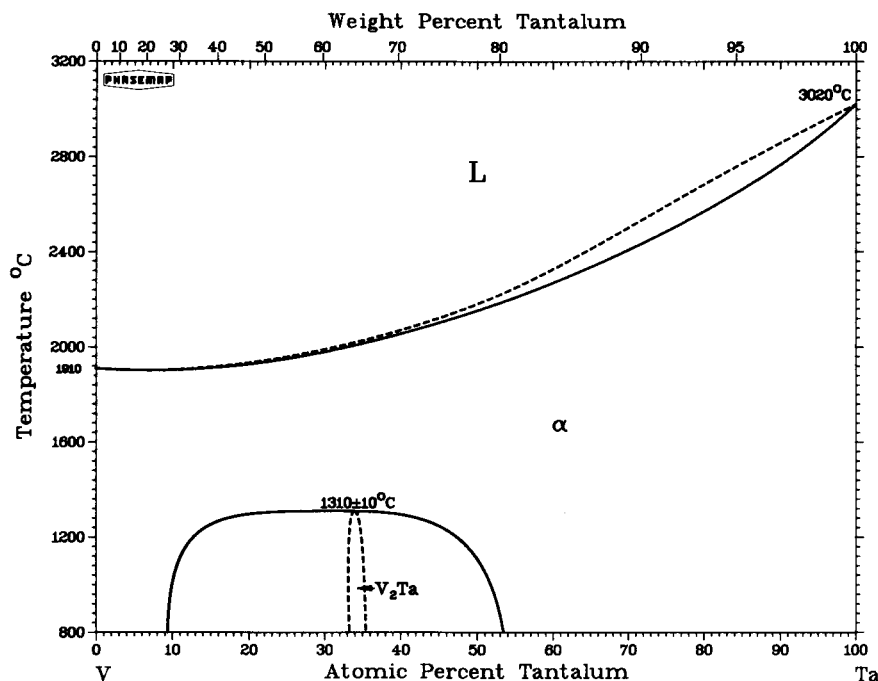
General Features. There is general agreement that Ta and V alloy to form a continuous bcc solid solution at elevated temperatures [54Ros, 59Car1, 60Ere, 69Rud] and that an intermediate phase forms from this solid solution at lower temperatures [54Ros, 59Car1, 62Ere, 69Rud, 72Sav, 75Wen]. The V-Ta diagram in Fig. 1 is a composite of the currently available data. There is still some uncertainty as to the exact nature of the V-rich solidus. Rudy [69Rud] and Eremenko *et al.* [60Ere] reported a continuously rising solidus from the melting point of V to that of Ta. Carlson *et al.* [59Car1], on the other hand, found an azeotropic melting minimum at 1820 °C and ~11 at.% Ta. Russian diagrams were published [64Nef, 72Sav] that also show this minimum, but these diagrams appear to be an acceptance of the Carlson report without additional corroborating measurements. All three sets of melting data are shown in Fig. 2.

The V in Carlson's alloys had a purity of about 99.7 wt.%, with a melting point of 1885 ± 15 °C, and the Ta had a purity of 99.9 wt.%, with a melting point of 2996 ± 25 °C. The V in Rudy's alloys had a melting point of 1926 ± 3 °C and the Ta a melting point of 3014 ± 10 °C, but no purities were specified. Rudy's uncertainties may be overly optimistic, but the reasonable accord between his melting points and the currently accepted values of 1910 °C for V

[81Smi] and 3020 °C for Ta [81BAP] suggests relatively high purity for Rudy's base metals. The V in Eremenko's alloys was indicated to contain 0.5% Al and 0.22% Fe with a considerable, but not quantitatively specified, amount of oxygen, and the Ta was specified as 99.8% pure. It is not clear whether Eremenko's percentages are meant to be wt.% or at.%. The low melting points of 1810 °C for V and 2950 °C for Ta indicate that Eremenko's base metals were the least pure among those of the three solidus investigations.

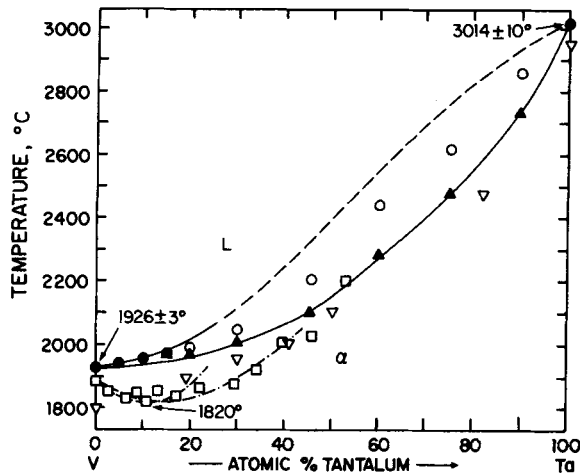
For the present review, an attempt was made to determine whether or not an azeotropic melting minimum occurs in the pure binary system. An alloy was prepared with 11 at.% Ta, which is the composition associated by Carlson with the melting minimum. Electrolytically refined V of 99.99+ wt.% purity and electron-beam melted Ta of 99.95+ wt.% purity were arc melted together, and the resulting ingot was fabricated into a rod suitable for a melting point determination. The alloy specimen was heated by internal resistance under an argon atmosphere until melting was first observed in a small black-body hole in the specimen. Temperature was measured by a calibrated optical pyrometer and corrected for sight glass absorption. Six independent measurements yielded a melting point of 1880 °C, with a mean deviation of ± 5 °C. The melting point of the pure, base V metal was determined in identical manner and was found to be 1888 ± 5 °C. Although

Fig. 1 V-Ta Assessed Phase Diagram



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Fig. 2 Melting Data for V-Ta Alloys



As compiled by Rudy [69Rud]. The symbols ▲, ○, ● are Rudy's experimental points based, respectively, on incipient melting, specimen collapse, and isothermal melting. The upper dashed and solid lines that extend from 1926 to 3014 °C are Rudy's selections for liquidus and solidus. Melting data from Carlson *et al.* [59Car1] are represented by the symbol □ and from Eremenko *et al.* [60Ere] by the symbol ▽. The dash-dot lines show that the Carlson data imply a melting minimum.

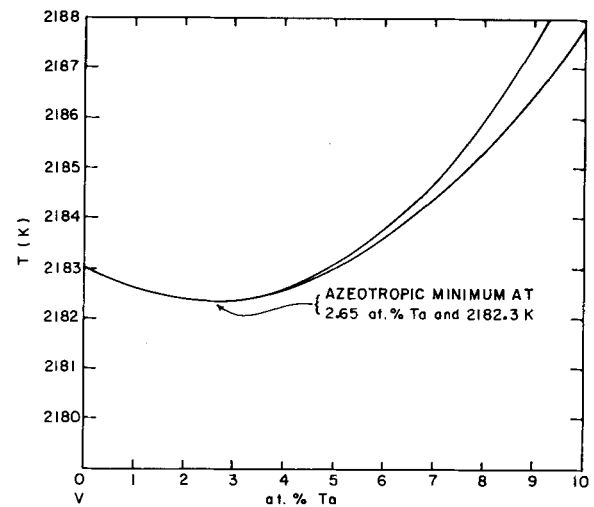
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these melting points also indicate the existence of an azeotropic melting minimum in the system, the uncertainties are sufficiently large to leave the evidence equivocal.

This equivocal status is not alleviated by a simple modeling calculation. The calculation was done with a regular solution approximation [70Kau]. Input data consisted of theoretically generated interaction parameters of 13 673 J/mol for the bcc solid solution and 2418 J/mol for the liquid solution, a heat of fusion of 21 500 J/mol with a melting point of 2183 K for V, and a heat of fusion of 31 600 J/mol with a melting point of 3293 K for Ta. The calculation indicated a very weak azeotropic minimum with liquidus and solidus lines touching tangentially between 2 and 3 at.% Ta at 2182 K. This is shown in Fig. 3. The separation between liquidus and solidus on the V-rich side of the minimum does not exceed ~0.02 at.%, so the separation is not resolvable on the scale of the graph. The composition and depth of the minimum are, of course, sensitive to the input melting points, heats of fusion, and interaction parameters, all of which have some degree of uncertainty. However, on the basis of the present experimental measurements on pure materials in combination with the calculated results, a weak azeotropic minimum has been included in Fig. 1. Nonetheless, the reader is cautioned that utilizations or predictions based on the existence of this minimum should be treated with some reservation.

The Intermediate Phase. In the following section, "Crystallography", data concerning the structure of the intermediate phase are discussed, and most reports [59Car2, 59Eil, 72Sav, 74Sch, 75Wen] agreed that an intermediate phase, V_2Ta , exists, with the cubic Cu_2Mg -type Laves structure. Three investigations [54Ros, 62Ere, 64Nef] indexed X-ray diffraction patterns from the

Fig. 3 Liquidus and Solidus Lines for the V-Rich Region



As calculated with a regular solution model.

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intermediate phase as tetragonal, but in two of these studies [54Ros, 62Ere], there is definite evidence (see "Crystallography" section) that the intermediate phase was cubic V_2Ta . The third report gave too little information to justify the tetragonal indexing. It should also be noted that, although Savitskii *et al.* [72Sav] agreed that V_2Ta has the cubic Cu_2Mg -type structure at lower temperatures, they reported that the structure changes to the $MgZn_2$ type at higher temperatures.

The first report of an intermediate phase was that of Rostoker and Yamamoto [54Ros]. In their investigation, microstructures of as-cast alloys showed only the bcc solid solution, but alloys that were annealed at 900 °C for 170 h showed a second phase at compositions ranging from 9 to 53 at.% Ta. With their restricted number of samples, the maximum amount of intermediate phase was observed at 22 at.% Ta. Carlson *et al.* [59Car1] found the intermediate phase to form from the bcc solid solution by congruent transformation at 1320 °C with a composition of 33 at.% Ta, in accord with a stoichiometry of V_2Ta . At 900 °C, their microstructures showed evidence of the phase through the range 10 to 60 at.% Ta. They inferred that the phase had a limited to negligible composition range. Eremenko *et al.* [62Ere] also found the intermediate phase to form by congruent transformation at a composition near 33 at.% Ta, but at 1420 °C, which is 100 °C higher than found by Carlson. At 1000 °C, Eremenko found the phase to be detectable through the range 5 to 65 at.% Ta with a composition range between 30 and 43 at.% Ta. Nefedov *et al.* [64Nef] used microscopic evidence, hardness values, thermal analyses, and resistivity measurements to study the occurrence of the intermediate phase. In agreement with Carlson *et al.*, Nefedov *et al.* found the phase to form congruently near 33 at.% Ta at 1300 ± 10 °C. In alloys annealed at 1250 °C, the phase was detectable through the composition range 15 to 45 at.% Ta, and, in alloys annealed at 900 °C, the phase appeared at compositions 9 through 52 at.% Ta. The width of the homogeneous phase field was found to increase from zero at the congruent transformation to ~3 at.% at 900 °C.

The diagram proposed by Savitskii *et al.* [72Sav] indicated the intermediate phase to be dimorphic, and in this sense the Savitskii diagram differs significantly from other proposed diagrams. Savitskii *et al.* [72Sav], like Eremenko *et al.* [62Ere], found a congruent transformation to occur at 33 at.% Ta and 1420 °C, but Savitskii reported the transformation as being from the bcc solid solution to a hexagonal Laves structure of MgZn₂ type. A two-phase field between the MgZn₂- and Cu₂Mg-type phases was found on the Ta-rich side of the hexagonal phase through the temperature range 1125 to 1280 °C, with the lower temperature being associated with eutectoidal decomposition of the hexagonal phase at ~29 at.% Ta. The most Ta-rich composition for the hexagonal phase was given as 36 at.% Ta at 1280 °C, at which temperature the hexagonal phase was indicated to react in peritectoidal equilibrium with the bcc solid solution to form the cubic Cu₂Mg-type Laves phase with a composition near 37 at.% Ta. At 1400 °C, the hexagonal phase was reported to be detectable in alloys from 20 to 45 at.% Ta, and at 1125 °C from 9 to 31 at.% Ta. At 1125 °C, the cubic phase was found to be homogeneous from 31.5 to 38 at.% Ta, and at 800 °C, from 32 to 39.5 at.% Ta. The cubic phase was reported to exist in alloys from 8 to 72 at.% at 800 °C; this V-rich composition at 800 °C for the terminus of the two-phase field is quite comparable to the boundaries found in the other investigations, but the Ta-rich composition is much greater than any other reported value.

Savitskii *et al.* [72Sav] gave no information in their report on metal purity or on details of their experimental techniques. The decision not to include their reported dimorphic transformation in Fig. 1 was made on the basis of information from other V alloy studies. Savitskii *et al.* [67Sav1] reported V₃In and V₃Pb phases to exist with the A15 structure in the V-In and V-Pb systems, but Leger and Hall [74Leg] found, in agreement with theory [75Mie, 76Mie], no intermediate phase in either system at any temperature at ambient pressure. Further, Savitskii *et al.* [64Sav], in an initial investigation of the V-Ga system, reported V₃Ga to have a narrow range of homogeneity and to melt peritectically and, in a second investigation [67Sav2], reported V₃Ga to undergo a polymorphic transformation. Subsequent investigations have shown V₃Ga to have a rather broad range (~12 to 14 at.%) of homogeneity and to transform congruently to the bcc terminal solution. The combined work of Flükiger *et al.* [76Flu], Meissner and Schubert [65Mei], and Maier *et al.* [66Mai] showed that the report of a high-temperature polymorph was attributable to oxygen contamination. Other examples could be cited, but it appears that the V-base metal that was used in the Savitskii investigations may have been less pure than desired. On this basis, no MgZn₂-type polymorph is included in Fig. 1.

In defining the boundaries of the intermediate phase in Fig. 1, the data of Eremenko *et al.* [62Ere] were not weighted because they assigned a range of homogeneity of

13 at.% to V₂Ta. Such a broad range of homogeneity seems inconsistent with the fact that Laves phases form primarily because of atomic size considerations, whose associated packing constraints require restricted ranges of homogeneity in binary systems. The congruent transformation at 33 at.% Ta was placed at 1310 ± 10 °C, the midpoint between the Carlson [59Car1] and Nefedov [64Nef] transition temperatures, with the uncertainty encompassing both values. Also in agreement with the Carlson and Nefedov investigations, the range of homogeneity of V₂Ta was drawn not to exceed 3 at.%. The V-rich solvus at 900 °C was placed between 9 and 10 at.% Ta, in accord with the results of three investigations [54Ros, 59Car1, 64Nef], and both the Nefedov V-rich and Ta-rich solvus values were accepted for 1250 °C. A value of 53 at.% Ta was selected for the Ta-rich solvus at 900 °C, in accord with the Rostoker and Nefedov reports [54Ros, 64Nef]. Though Carlson *et al.* [59Car1] placed this latter solvus boundary near 60 at.% Ta on the basis of microstructures, their text indicates that X-ray lines from V₂Ta disappeared almost completely in a 53 at.% Ta alloy after annealing for 1000 h at 900 °C. This X-ray observation supports the choice of the lower solvus composition.

Crystallography

Crystal structure data are given in Table 1.

bcc Phase. Lattice parameters were measured as functions of composition across the entire system by Eremenko *et al.* [60Ere] and by Rudy [69Rud]. Their data are plotted in Fig. 4, and values from the smooth curve are listed in Table 2. These data show a positive deviation from Vegard's law. Lattice parameters at a limited number of compositions also were reported by Heiniger *et al.* [66Hei] and by Schippnick and Lawson [74Sch] and are in good accord with the tabulated values. In all instances where it was necessary to suppress the formation of V₂Ta, lattice parameter measurements for the bcc phase were made on quenched alloys.

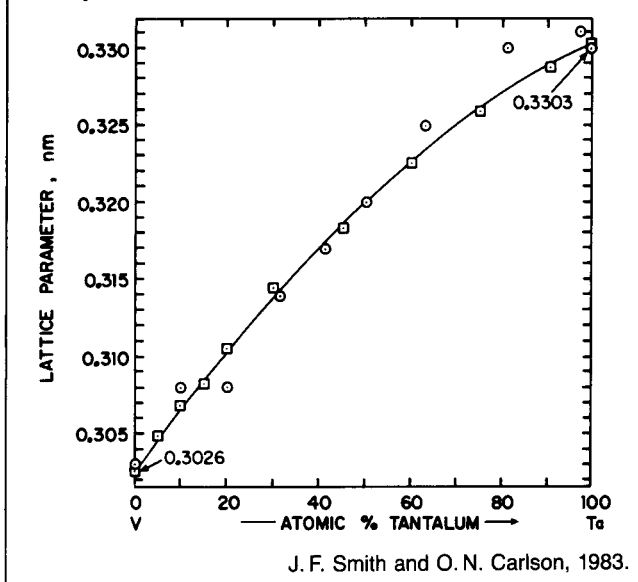
Laves Phase. Rostoker and Yamamoto [54Ros] indexed a diffraction pattern of the intermediate phase as tetragonal. They concluded that it was a σ phase or a member of the σ -phase family. Eremenko *et al.* [62Ere] also reported tetragonal symmetry for the intermediate phase and referred to it as a σ phase. Carlson *et al.* [59Car1] initially indexed a powder pattern from the intermediate phase as hexagonal with $a = 0.5065$ nm and $c = 1.1703$ nm. After examining Carlson's data, Elliott [59E11] suggested that the hexagonal indexing of the phase did not utilize the full symmetry of the diffraction pattern and that better accord with the characteristic extinctions could be obtained by indexing the phase as cubic, with $a = 0.7163$ nm, and by postulating a structure of the fcc Laves type. Confirmation that the phase had the cubic Laves structure was obtained by Carlson [59Car2], whose re-examination of his diffrac-

Table 1 V-Ta Crystal Structure Data

Phase	Approximate composition range(a), at.% Ta	Pearson symbol	Strukturbericht designation	Space group	Prototype
α or (V) or (Ta)	0-100	<i>cI2</i>	A2	<i>Im3m</i>	W
V ₂ Ta(b).....	33	<i>cF24</i>	C15	<i>Fd3m</i>	Cu ₂ Mg

(a) From the phase diagram. (b) A high-temperature polymorph of V₂Ta was reported by Savitskii *et al.* [72Sav] to be a hexagonal MgZn₂-type structure, with $a = 0.496$ and $c = 0.811$ nm, *hP12*, *C14*, and *P6₃/mmc*.

Fig. 4 Room-Temperature Lattice Parameters for the bcc Solid Solution as a Function of Composition



tion patterns showed that the relative intensities of observed reflections were in excellent agreement with those of another Cu_2Mg -type Laves phase, the low-temperature form of Cr_2Ta , with the X-ray scattering power of Cr and V being almost the same.

The tetragonal lattice parameters of Rostoker and Yamamoto [54Ros] were reported as $a = 0.6116$ nm and $c = 0.8851$ nm, and those of Eremenko *et al.* [62Ere] were reported as $a = 0.616$ nm and $c = 0.887$ nm, which indicates that the diffraction patterns from the two investigations were closely comparable. Greenfield [54Gre], however, compared the diffraction data of Rostoker and Yamamoto with corresponding data for the σ phases in the Fe-Co, V-Mn, and V-Fe systems and showed that the V-Ta intermediate phase was not isomorphous. Table 3 shows a comparison of the d spacings and visually estimated intensities of the diffraction pattern of Rostoker and Yamamoto [54Ros] with similar data from Carlson *et al.* [59Car1]. The comparison shows a strong correlation with all of the more intense lines of the Rostoker-Yamamoto pattern, matching the lines of the Carlson pattern in position and with good qualitative accord in relative intensities. Only those lines of the Rostoker-Yamamoto pattern with very weak to very, very faint intensities are absent from the Carlson diffraction pattern. On the basis of the matching in both position and relative intensity of the more intense lines of the Rostoker-Yamamoto pattern with those of the Carlson pattern, it may be inferred that the alloy of Rostoker and Yamamoto contained the cubic V_2Ta Laves phase plus a small amount of a contaminant phase or phases. The last column in Table 3 shows that the residual lines in the Rostoker-Yamamoto pattern cannot in their entirety be attributable to untransformed bcc phase, though such phase may have been present to some degree. Overall, the comparison indicates that reports of a σ phase by Rostoker and Yamamoto [54Ros] and Eremenko *et al.* [62Ere] can be discounted and their data rationalized as being compatible with the existence of a V_2Ta Laves phase.

Table 2 V-Ta Lattice Parameter Data

Phase	Lattice parameter, nm a	Note	Reference
α or (V) or (Ta)	0.3026	Elemental Ta	(a)
	0.3067	10 at.% Ta	
	0.3106	20 at.% Ta	
	0.3139	30 at.% Ta	
	0.3170	40 at.% Ta	
	0.3200	50 at.% Ta	
	0.3226	60 at.% Ta	
	0.3250	70 at.% Ta	
	0.3271	80 at.% Ta	
	0.3289	90 at.% Ta	
V_2Ta	0.3303	Elemental Ta	[75Wen] [74Sch]
	0.7155	...	
	0.7153	...	

(a) Smoothed data from Fig. 4.

Later investigators [72Sav, 74Sch, 75Wen] concurred that the intermediate phase has the cubic Cu_2Mg -type Laves structure. An isolated exception is the report of Nefedov *et al.* [64Nef], who performed an X-ray analysis on an annealed specimen of 34 at.% Ta and found tetragonal lattice parameters of $a = 0.5041$ and $c = 0.6702$ nm, which are significantly different than those of Rostoker and Yamamoto [54Ros] and Eremenko *et al.* [62Ere]. The report of Nefedov *et al.* was terse and gave no d spacings or intensities. Without such intensities, and particularly extinctions, calculated d spacings from the Nefedov lattice parameters are so numerous that extensive matching with the Carlson diffraction pattern is almost guaranteed. It may or may not be fortuitous that the value of $a = 0.5041$ nm is very close to the distance between the origin point and an fcc lattice point of the cubic V_2Ta Laves structure and to the a parameter of Savitskii's [72Sav] hexagonal polymorph. No similar correspondence in distance is evident for $c = 0.6702$ nm. The preponderant evidence is that V_2Ta has the Cu_2Mg -type structure, and the Nefedov report has, therefore, been considered aberrant.

The high-temperature polymorph of V_2Ta , which was reported by Savitskii *et al.* [72Sav], was indicated to be hexagonal, with $a = 0.496$ and $c = 0.811$ nm, with the MgZn_2 Laves structure. Other Laves phases crystallize in two polymorphic forms; this may be true of V_2Ta , but there is no other corroborative evidence.

Thermodynamics

The only experimental thermodynamic data for V-Ta alloys are low-temperature heat capacity measurements [64Hak, 66Hei, 70Cor, 74Sch, 75Wen]. Heat capacity values for the normal states have been fitted to the standard relationship:

$$C = \gamma T + \beta T^3 \text{ J/K} \cdot \text{mol Ta}_x\text{V}_{1-x}$$

where γ is the electronic specific heat coefficient and $\beta = 1922 \theta_D^3$, with θ_D being the Debye temperature. Values for γ and θ_D are listed in Table 4. Included in Table 4 are superconducting transition temperatures and related superconducting parameters. It may be noted from the compositional dependence of T_c that the heat capacity data indicate a minimum for the bcc phase near the center of the system. The existence of such a minimum is confirmed by other types of superconducting measurements [71Rap].

Table 3 Comparison of *d* Spacings and Visually Estimated Intensities

Rostoker and Yamamoto		Carlson <i>et al.</i> (for V ₂ Ta)		Calculated bcc <i>d</i> spacings(c), nm
<i>d</i> spacings(a), nm	Visually estimated intensities(b)	<i>d</i> spacings(a), nm	Visually estimated intensities(b)	
...	...	0.4118	w	...
0.2707	vf
0.2510	m	0.2520	s	...
0.2353	vvf	0.2262
0.2143	s	0.2151	vs	...
0.2054	vf	0.2059	w	...
0.1948	vf
0.1877	vvf
0.1783	vf	0.1784	vvw	...
0.1636	f	0.1638	vw	...
0.1559	vvf	0.1600
0.1515	vw
0.1453	wm	0.1456	ms	...
0.1416	vf
0.1371	wm	0.1374	ms	...
0.1331	vvf	0.1306
0.1260	w	0.1262	ms	...
0.1240	vw
0.1221	vvf
0.1204	vw	0.1208	w	...
0.1174	vvf	0.1131
0.1126	w	0.1129	m	...
0.1087	w	0.1090	w	...
0.1074	f	0.1079	vw	...
0.1047	vf	0.1011
0.0998	f	0.10018	vw	...
0.09608	vw	0.09566	m	...
0.09526	wm	0.09320	ms	...
...	...	0.08949	w	...
...	...	0.08747	vw	...
0.08413	wm	0.08439	m	...
...	...	0.08270	ms	...
...	...	0.08215	vw	...
...	...	0.08008	vvw	...
...	...	0.07862	w	...

(a) From the diffraction patterns. (b) vvf = very, very faint, vf = very faint, f = faint, vvw = very, very weak, vw = very weak, w = weak, wm = weak medium, m = medium, ms = medium strong, s = strong, vs = very strong. (c) For a bcc solid solution with $a = 0.3200$ nm.

Savitskii *et al.* [72Sav] reported their Cu₂Mg-type phase not to be superconducting above 4.2 K. This is in accord with the findings of other investigations [74Sch, 75Wen] of a value of 3.6 K for the superconducting transition temperature of this phase. Savitskii also reported that the quenched MgZn₂-type polymorph had a superconducting transition temperature of 10 K, which dropped to 8.2 K at the V-rich composition limit and to 9.2 K at the Ta-rich composition limit. Even if this polymorph is not an equilibrium phase in the pure binary system, it is interesting to note that the hexagonal structure has a transition temperature so much higher than either the cubic structure or the constituent elements.

Addendum

After reading the manuscript for this paper, Dr. R. Krishnan of the Bhabha Atomic Research Center was kind enough to call to the author's attention two additional Russian references. The first of these is by Kolygin *et al.* [75 Kol], who, on the basis of thermal and metallographic analyses, also reported the existence of azeotropic melting at 12 at.% Ta. The second is by Pushin *et al.* [80 Pus], who confirmed the structure of TaV₂ as being a cubic Laves structure with $a = 0.714$ nm.

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Table 4 Values for Heat Capacities and Superconducting Parameters for V-Ta Alloys at Various Compositions

Composition, at.% Ta	Electronic specific heat coefficient ($\gamma \times 10^3$), J/(K ² · mol V _{1-x} Ta _x)	Debye temperature (θ_D), K	Super- conducting transition temperature (T_c), K	Electron- phonon coupling parameter, λ	Critical field(a), $H_c(0)$, 10 ⁵ A/m	Reference
bcc solid solution						
0	9.45	377	5.17	0.60	106.3	[70Cor]
5	9.20	357	[64Hak]
10	8.78	341	4.47	0.60	87.0	[70Cor]
24	8.48	300	3.58	0.58	67.0	[70Cor]
25	8.3	285	2.8	[66Hei]
33.3	9.0	325	2.8	[74Sch]
50	7.5	264	2.35	[66Hei]
50	7.62	273	2.73	0.56	46.5	[70Cor]
70	6.41	256	3.00	0.58	45.6	[70Cor]
75	6.7	250	2.65	[66Hei]
90	5.70	250	3.58	0.61	50.0	[70Cor]
100	5.85	251	4.33	0.65	61.2	[70Cor]
V₂Ta cubic Laves phase						
33.3	11.0	278	3.6	[74Sch]
33.3	8.2	279	3.6	[75Wen]

(a) At 0 K.

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*Indicates key paper.

#Indicates presence of a phase diagram.

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