Experimental study of the Fermi surface of vanadium*

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The Fermi surface of vanadium has been studied experimentally using magnetothermal-oscillation techniques in the {100} and {110} planes in fields of 7–11 T. The Fermi surface has qualitatively the same topology as those of tantalum and niobium: a set of six distorted ellipsoidal surfaces centered at N in the Brillioun zone, and a multiply connected jungle-gym surface consisting of interconnecting arms along the ⟨100⟩ directions with intersections at Γ and H. The data, taken on samples supplied by the U.S. Bureau of Mines, are qualitatively consistent with recent augmented-plane-wave (APW) calculations, and agree excellently with previous experimental data. The observed extremal areas near the symmetry directions are: ⟨100⟩, 33.1 nm^{-2} attributed to the jungle-gym necks, 50.5 and 57.6 nm^{-2} attributed to the ellipsoids; at ⟨111⟩, 47.9 nm^{-2} attributed to the ellipsoids; at ⟨110⟩, 64.1, 50.4, and 53.1 nm^{-2} attributed to the ellipsoids. Effective masses, measured using the de Haas–van Alphen effect, ranged from 1.7 to 2.2 times the free-electron mass for the ellipsoids, while the orbit around the jungle-gym arms at ⟨100⟩ was found to have an effective mass of 3.0. The data are consistent with the existence of necks along the Γ-N direction, connecting the ellipsoids to the jungle gym.

I. INTRODUCTION

In the past several years, the considerable interest in the electronic structure of the vanadium-group (Vb) transition metals has prompted a number of theoretical calculations of the band structure and experimental studies of the Fermi surfaces. A preliminary indication of the over-all features of the Fermi-surface topology of the metals as a group was given by Mattheiss[1] in a paper primarily concerned with the band structure of tungsten. In a later paper Deegan and Twose concentrated on the band structure of niobium.[2] Following this, Mattheiss[3] predicted Fermi-surface topologies, extremal cross-sectional areas, and effective masses for both niobium and tantalum. Experimental studies of these two elements have been pursued by Halloran *et al.*,[4] and by Scott and Springford,[5] and the results are in general agreement with the calculations of Mattheiss.

Theoretical studies of vanadium, generally believed to have an electronic structure similar to tantalum and niobium, have been made only recently. Anderson *et al.*[6] gave a preliminary report on non-self-consistent calculations. Yasui *et al.*[7] used a self-consistent method for two values of the exchange parameter, and also presented a calculated Fermi-surface topology. Most recently, Papaconstantopoulos *et al.*,[8] in an extension of earlier work,[6] have completed self-consistent calculations for several values of the exchange constant and for several different lattice spacings. In addition, they predicted Fermi-surface topologies, extremal cross-sectional areas, and effective masses for some orbits. Little experimental work on vanadium has been reported, the earliest work being an inconclusive magnetoresistance study by Alekseevskii and Egorov.[9] A subsequent study using the same technique revealed an anisotropy in the Fermi surface,[10] which was interpreted as being consistent with the early model.[1] The first report of a study yielding specific Fermi-surface information was that of the author.[11] These results were in agreement with a simultaneous independent study by Phillips, who investigated only one crystallographic plane using the technique of impulsive fields.[12] Phillips measured effective masses in addition to cross-sectional areas, but the uncertainty in the mass data was fairly large.

The present paper reports the results of an extended experimental study of the Fermi surface of vanadium in two planes, using the observation of the de Haas–van Alphen effect (dHvA) and of magnetothermal oscillations (MTO). The various verifiable theoretical predictions will be discussed, and then they will be compared to the present experimental work. The experimental results of Phillips and the data for the other two elements of the group will also be examined in light of these new results.

II. EXPERIMENTAL DETAILS

Experimental work on vanadium has been greatly hampered by the lack of pure samples. Unlike either tantalum or niobium, vanadium is extremely difficult to purify. It is equally difficult to grow pure single crystals with few defects, as required for Fermi-surface studies. Residual resistance ratios of 200 are typical for vanadium, compared to 10 000 for niobium. However, in the past few years Sullivan has succeeded in preparing very pure vanadium in the form of dendritic crystals by electrorefining from a molten bath of KCl-LiCl-

VCl_2.[13] The samples used in the present work were prepared from a batch of double-refined crystals obtained from Sullivan.

The actual dendrites were small crystallites attached together much in the manner of rock salt. Each individual crystallite was in turn composed of many smaller platelike crystals, all oriented in nearly the same direction and arranged so as to form the faces of the larger crystallite. The platelike structures were {100} planes, and the dendrite strings appeared to have grown along ⟨111⟩. The size of the larger faces was a function of the growth rate, with the less pure dendrites having the larger faces. Owing to the nature of the growth process, small internal voids were present in the crystals, but were found only when spark cutting the material. These voids were filled with electrolyte, but comprised only about 200 ppm by volume.[14] These dendritic crystals were of very high purity. They contained only 18-ppm O_2, and less than 20-ppm total Ni, Cr, and Fe. Total metallic impurities were 37 ppm.[14] Resistance ratios have been measured on similar material, and found to be in the range 1000-1600.[15] One sample was cut from the largest single crystal among the dendrites. A quantity of the smaller dendrites was melted and formed into an ingot under vacuum. Slices from this ingot were then zone refined under high vacuum. Several samples were cut from the purest section of the zone-refined bar. The resistance ratio of these zone-refined samples was 210.

The dendritic crystal sample was 2 mm on a side, while the zone-refined samples were 3 mm in diameter and 6 mm long. The samples were oriented using Laue x-ray techniques to within 1°, and this accuracy was preserved with special jigs during mounting into the sample holder.

The experiments were conducted in the field of an 11-T Nb_3Sn superconducting solenoid, which was driven by a specially built sweep controller[16] that permitted sweeps linear in H or $1/H$. The field range used for all experiments was 7–10 T. The inhomogeneity of the field in the sample volume was measured with a cryogenic Hall probe and found to be less than 0.005%, about an order of magnitude better than necessary for the frequencies observed.[17] The field was measured by a copper-wire magnetoresistance coil[18] counterwound to minimize inductance effects. This coil was wound on the tail of the sample Dewar, and was calibrated using a rotating-coil gaussmeter. Initial calibration was by means of a Hall probe at the factory. The resolution of this magnetoresistance probe was of the order of 10^{-4} T, since the current source used to drive it was stable to several parts in 10^5.

The sample holder was a modification of a type reported previously.[19] Two sets of pulleys and braided BeCu cables allowed the adiabatic rotation of the sample 270° about a principal axis, and ±15° about an axis perpendicular to the principal axis, with a total accuracy of better than 1°. For MTO measurements, the sample temperature was sensed with a carbon resistance thermometer that had a sensitivity of 2×10^6 Ω/°K at 1.2 °K. This resulted in an ultimate detection sensitivity of better than 1.8 microdegrees Kelvin. For dHvA measurements, the change in magnetic moment of the sample was detected with a small set of balanced coils. In both cases, the sample was thermally isolated on a hollow graphite post, the thermal contact to the He^4 bath being provided by a low pressure of helium gas in the sample chamber. For MTO, a pressure of 50–80 mtorr was used to minimize temperature variations. For dHvA, a pressure of several hundred mtorr was used. The relaxation time sample to bath was of the order of 1 sec for MTO and 0.1 sec for dHvA. All MTO experiments were run with the He^4 bath between 1.2 and 1.13 °K, while the dHvA experiments were run with bath temperatures in the range 1.1–2 °K. Temperature control was achieved by controlling the pressure above the bath.

The solenoid field was modulated so that signals from either effect could be detected using a low-frequency version[20] of a method first proposed by Shoenberg and Stiles.[21] The modulation field was supplied by a small superconducting solenoid wound inside the main bore of the magnet. Modulation frequencies near 11 Hz were chosen, since the best signal to noise was found at those points. The signals were detected by conventional phase-sensitive methods. Data points, consisting of the voltage from the magnetoresistive field probe and MTO or dHvA data, were processed by a data acquisition system and stored on magnetic tape.

The data thus recorded were computer analyzed, using a Fourier-transform method. Modulation ripple was removed from the magnetic field data using a least-squares smoothing routine. Frequency calibration independent of solenoid calibration was provided by the comparison of intervals between zeros in the data with similar intervals in calibration data, which consisted of sweeps on the same apparatus of well-known frequencies in copper and tungsten.[22] The accuracy of the Fourier analysis was in general $1/N$, where N is the number of periods of the frequency analyzed; the analysis programs determined the specific accuracy for each sweep relative to the calibration frequency.

III. THEORETICAL MODELS

There have been three relatively complete treatments of the band structure and Fermi-surface topology in vanadium, those of Mattheiss,[1] Yasui

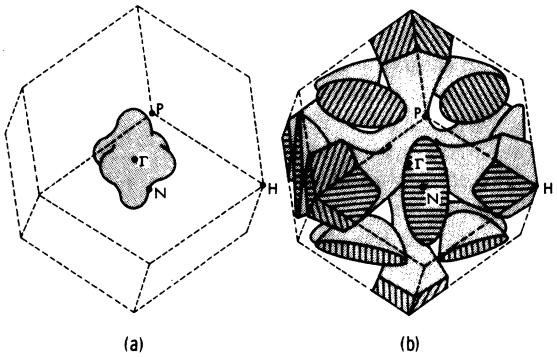

FIG. 1. Fermi surface of the vanadium-group transition metals, showing the (a) second- and (b) third-zone hole surfaces.

et al.,[7] and Papaconstantopoulos *et al.*[8] In this section the three treatments are examined, and those points admitting to experimental verification in these experiments are brought out for later comparison with the data.

Mattheiss's nonrelativistic augmented-plane-wave (APW) treatment of the vanadium-group metals allowed him to predict a general Fermi-surface topology which has been accepted by the other workers. Figure 1(a) is his closed second-zone hole surface centered at Γ, and Fig. 1(b) is his complex third-zone hole surface. This latter surface consists of multiply connected hole tubes along the ⟨100⟩ directions plus large distorted half-ellipsoids centered at N. The hole-tube section of this surface has been termed the "jungle gym."

Mattheiss made three sets of non-self-consistent calculations with full Slater exchange, using a different set of atomic potentials each time. He expected that the Fermi surface would be quite sensitive to the details of the band structure calculations, since he expected the Fermi level to fall near the Γ_{25}' state.[1] This in fact was the case

Two qualitatively different Fermi-surface topologies resulted from the calculations. The surface most like that of Fig. 1 was obtained from either of two sets of tungsten functions; it is shown in Fig. 2(a). A slightly different topology, Fig. 2(b), was obtained when the iron functions of Wood[23] were used. The greatest qualitative difference is that in Fig. 2(b) the ellipsoids are connected to the jungle-gym surface by narrow necks along the Γ-N directions, while in Fig. 2(a) no such connections exist. Unfortunately, Mattheiss did not predict extremal cross-sectional areas or effective masses.

Using a combination of orthogonalized-plane-wave (OPW) and tight-binding methods, Yasui *et al.*[7] did a self-consistent calculation for two different values of the exchange constant. For the case of full exchange the results differed greatly from other work, but the surface for reduced exchange ($\gamma = 0.725$) is similar to one shown previously by Mattheiss, Fig. 2(a). The authors felt that the reduced-exchange calculation was more valid than the case of full exchange, since the results of the calculations then matched experimental density of states and bandwidth data. No predictions of extremal cross-sectional areas or effective masses were made.

Using the self-consistent nonrelativistic APW technique, Papaconstantopoulos *et al.*[8] have recently made an extensive theoretical investigation of vanadium for several values of the Slater exchange and at two different lattice spacings. Their band-structure results are in qualitative agreement with those of Mattheiss and of Yasui *et al.* Papaconstantopoulos *et al.* found that for both full and $\frac{2}{3}$ exchange the Fermi level falls below the Γ_{25}' level. They felt that on the basis of photoemission and soft-x-ray results better agreement with experiment was obtained using reduced exchange. The Fermi surface for reduced exchange and normal lattice parameter corresponds to Mattheiss's tungsten function surface, Fig. 2(a). The Fermi surface for reduced exchange and 5% reduction in lattice parameter is similar to Fig. 2(b), found by Mattheiss from iron potentials. For this second case, they estimate that the degeneracies along Γ-N should appear at a hydrostatic pressure of 135 kbar. Most interestingly, Papaconstantopoulos *et al.* actually calculated extremal cross-sectional areas and effective masses along symmetry directions, and compared the results to the experiment of Phillips[12] in the {100} plane. The results to be presented in Sec. IV will be compared to these calculations and experimental data.

To draw together these treatments, let us examine the points of agreement. All agree that the

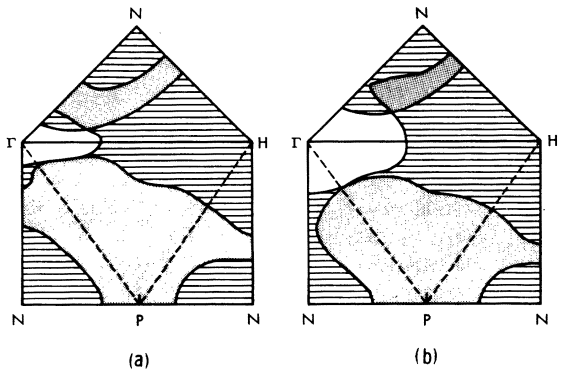

FIG. 2. Central {110} and {100} cross sections of the vanadium-group Fermi surface, calculated by Mattheiss (Ref. 1) using energy bands for (a) tungsten and (b) iron.

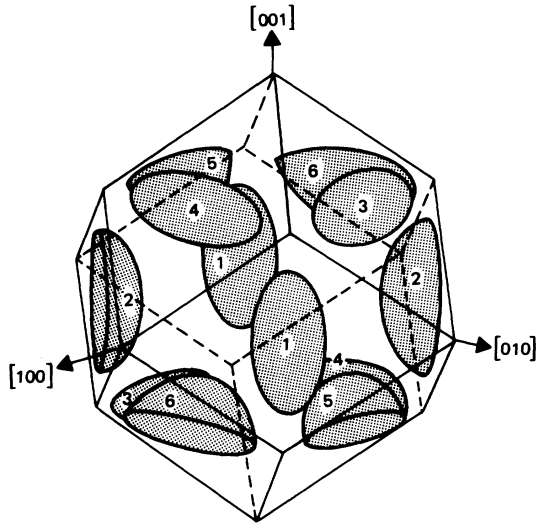

FIG. 3. Arbitrary numbering scheme for distinguishing among the six equivalent distorted ellipsoids at N.

Fermi energy falls below the Γ_{25}' energy. Yasui *et al.* and Papaconstantopoulos *et al.*, though using different methods, find closest agreement with experiment for a reduced Slater-exchange parameter. The Fermi surface predicted from this treatment has the general features of Fig. 2(a). Finally, there are the interesting results of Fig. 2(b), from Mattheiss's calculation using iron potentials and from the calculation of Papaconstantopoulos for 5% reduced lattice constant, which indicate degeneracies along the Γ-N directions, connecting the ellipsoids to the jungle-gym structure.

IV. EXPERIMENTAL RESULTS

It is convenient to adopt the numbering scheme of Mattheiss to distinguish among the equivalent ellipsoids; this scheme is shown in Fig. 3. Orbits attributed to the ellipsoids will be labeled E, and those attributed to the orbits around the ⟨100⟩ arms of the jungle gym will be designated JG. Many other orbits are possible on this Fermi surface, as Halloran *et al.*[4] have described, but these other orbits were not observed in vanadium. On the charts that follow, areas are displayed, rather than the equivalent frequencies; these data were all taken using MTO. The effective-mass data were taken using dHvA, since in this experimental arrangement the MTO mass data had been found to be erroneous. The errors introduced were due to the loading of the thermometer by the lock-in amplifier when data at temperatures lower than 1.5 °K were being taken. Three samples, one dendritic and two zone-refined, were employed.

Ellipsoid orbits

The data collected from the various samples are shown in Fig. 4 for both the {110} and {100} planes. The accuracy of the area determinations is better than 1 part in 100 near the ⟨100⟩ axis and better than 1 part in 300 near the ⟨110⟩ axis, the change being due to the greatly increased signal amplitude observed near the ⟨110⟩ axis.

A rough semiquantitative idea of the size and shape of the ellipsoids may be obtained from the semiaxis dimensions, which are calculated with the assumption that the ellipsoids are regular. For vanadium, these figures are 4.632, 4.405, and 3.649 nm^{-1}, for the N-P, N-Γ, and N-H directions, respectively.

Because the axes N-Γ and N-H of any one of the ellipsoids are always parallel respectively to the axes N-H and N-Γ of one of the other five ellipsoids in the zone, it is not possible to experimentally assign the two corresponding semiaxis val-

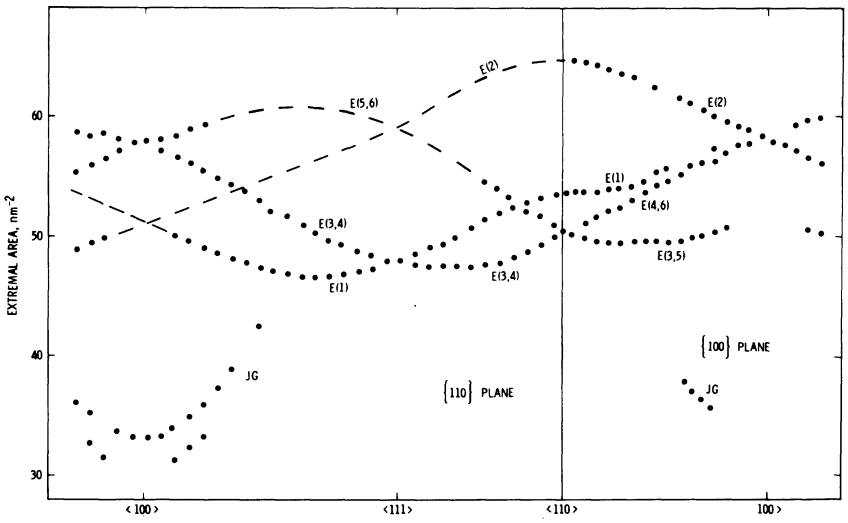

FIG. 4. Experimental cross-sectional areas in vanadium. Ellipsoid branch numbering corresponds to Figure 3. Unobserved branches are indicated by the dashed lines.

ues.[5] The results of the calculation of Papaconstantopoulos *et al.*[8] have therefore been used to choose N-$\Gamma > N$-H. When these data are compared with the results found for niobium,[5] it is evident that the ellipsoids in vanadium project farther toward the zone center relative to their size than the ellipsoids in niobium. The N-Γ/N-P and N-Γ/N-H ratios for vanadium are 0.946 and 1.21, and for niobium 0.842 and 1.14, respectively.

However, the splitting of the $E(1)$ and $E(2)$ orbits in the {100} plane indicates that the ellipsoid surfaces are not figures of revolution, for if they were, the orbits would be expected to be degenerate. These results are consistent with those found for Nb and Ta. Furthermore, there is an off-axis minimum in the {110} plane for the $E(1)$ branch at 37° from ⟨100⟩ toward ⟨111⟩, farther than in either Nb or Ta. This indicates that the ellipsoids in vanadium are more severely distorted than in the other two elements. Evidently, the sample orientation was excellent, since there was no scatter in the doubly degenerate branches, as had been reported in Nb and Ta when the sample was slightly tipped out of the symmetry plane.[5]

The gaps in the $E(1)$ branch near ⟨100⟩ in the {110} plane and in the $E(3, 5)$ branch near ⟨100⟩ in the {100} plane are most likely due to spin splitting. It is thought that the term $gm^*/2m_0$, the argument of a cosine in the equation for MTO,[24] approaches a multiple of $\frac{1}{2}\pi$ near ⟨100⟩ for those branches, due to the slight variation of effective mass and electronic g factor with angle. This results in the extinction of the first (and only observable) harmonic. These gaps were seen in the other two elements[4,5]; Phillips observed the gap in $E(3, 5)$ in vanadium, but interpreted it as being due to experimental difficulties.

The gap in the $E(2)$ branch in the {100} plane about midway between symmetry directions was reported by Phillips[12]; he found the gap to extend over 10°. In the present data, the gap extends over only 3°, the decrease probably being due to different samples and experimental set up. This gap was observed in both Ta and Nb, and it is believed that it is due to variations in local curvature of small regions on the Fermi surface which reduce the density of coherent electron orbits in those regions.

In the {110} plane, the $E(5, 6)$ branch vanishes near ⟨111⟩, and the $E(2)$ branch is not seen at all. The gap in $E(5, 6)$ is consistent with observations in Ta and Nb, but the $E(2)$ branch is observed in those elements. One explanation for these two gaps in the data is that the necks along the N-Γ direction, found by Mattheiss in one calculation and by Papaconstantopoulos *et al.* for reduced lattice spacing [Fig. 2(b)], may be present in vanadium. If this were the case, the orbits that would be expected to vanish are precisely those that are not seen.[4] A particularly thorough search for signals from these orbits was made with no result. The MTO signal strength depends upon only three parameters of the material, the electron effective mass, the electronic g factor, and the Dingle temperature. Since these parameters do not change abruptly over the rest of the surface of the ellipsoids, one would expect to see at least some faint signals from $E(2)$ near the symmetry directions if the orbit existed.

An observation tending to support the neck hypothesis is that the $E(2)$ branch in the {100} plane is very strong, but disappears abruptly very near the ⟨100⟩ axis. This result is at variance with the report of Phillips, but since he took points every few degrees perhaps he straddled the gap. It would appear that the size of the neck, if there is one, is quite small, since for a large neck the orbit would be expected to become nonextremal farther from the symmetry direction. In fact, it is possible to estimate the size of the neck, using the N-Γ dimension of the Brillouin zone and the angular range over which the $E(2)$ branch vanishes. Since in the present data the orbit vanishes about 2° from the axis, the neck must be about 500 times smaller in cross-sectional area along the ⟨100⟩ direction than the ellipsoid. The frequency from an orbit around the smallest part of the neck would thus be about 10 T, but the effective mass might be very large, due to the high curvature of the neck. Frequencies as small as this are difficult to observe with a modulated-field experiment, since sweeps over large field ranges must be used to give the Fourier analyzer sufficient data. A search has been made for this frequency without result.

If the ellipsoids are connected to the jungle-gym surface, other, more complicated, orbits are also possible, and some orbits on the jungle-gym surface would vanish. For example, one would expect large orbits similar to the "dog bone" in copper around the inside of the surface formed by the jungle gym and the connection to the ellipsoids. Furthermore, the central orbits around H of the jungle-gym surface would be expected to vanish. It was not possible to confirm experimentally any of these predictions.

In Table I are presented the extremal areas and effective masses along symmetry directions for the theory of Papaconstantopoulos *et al.*,[8] the experiments of Phillips,[12] and the current work. Phillips's work was in one plane only, but for those directions in which he measured areas his data are in excellent agreement with those of the present work, the largest deviation being a mere 0.6%. Agreement with the APW calculation is not nearly so good, the observed frequencies being about 18% larger than the calculations. Papaconstantopoulos

TABLE I. Comparison of experimental and APW Fermi-surface results.

Direction	Surface	Area/nm^{-2} MTO	dHvA[a]	APW[b]	Effective Mass m^*/m dHvA	APW[b]
⟨100⟩	E (3, 4, 5, 6)	57.6	57.5	49.4	2.08±0.18	
	E (1, 2)	50.5	50.2	42.9	1.77±0.07	0.86
	JG	33.1			3.2±0.5	
⟨111⟩	E (1, 3, 4)	47.9			1.77±0.08	
⟨110⟩	E (3, 4, 5, 6)	50.4	50.7	45.8	1.72±0.07	
	E (1)	53.1	53.2	44.4	2.20±0.07	0.92
	E (2)	64.1	64.1	56.8		1.3

[a]Data from Phillips (Ref. 12).
[b]Data from Papaconstantopoulos *et al.* (Ref. 8).

et al. observed that by using an exchange of 0.715 the theoretical results approached the experiment more closely, but the difference was still 8%. The difference between the measured[4,5] and calculated[3] areas in Nb and Ta was between 5 and 10%.

Jungle-gym orbits

The jungle-gym orbits are shown in Fig. 4 near the ⟨100⟩ direction in both the {110} and the {100} planes. These branches are qualitatively similar to those reported for Nb and Ta, and are attributed to orbits around the ⟨100⟩ arms of the multiply connected third-zone hole surface. The majority of the data describe a smooth curve intersecting the symmetry direction at about 32 nm^{-2}, but there are a few other points which tend to describe a second branch intersecting the axis at 30 nm^{-2}. These points were all as strong as the signals from the ellipsoids, but were seen only over very small angular ranges. The same sorts of signals were seen in Nb, and they were tentatively ascribed to local bumps on the arm surface. These data are the first evidence confirming the existence of the jungle gym in vanadium, but no theoretical estimates have been made for quantitative comparison.

That the orbit results from an area minimum is easily seen from Fig. 5 where the {110} plane JG data and the extremal area for a cylinder of 32 nm^{-2} cross-sectional area are plotted together. The measured area increases more rapidly than the cylindrical model, and is therefore associated with a concave surface.

Many other orbits are possible on the jungle gym besides this observed orbit. These other orbits are about the intersection of the arms at Γ, and have been seen with difficulty in Nb and Ta.[4,5] Signals from these orbits were sought but not detected in vanadium. The effective masses for these orbits are thought to be large, resulting in low signal strength. In addition, since the frequencies are probably larger than those of the ellipsoids, the field homogeneity of the experimental apparatus may not have been good enough to permit observation.

Effective masses

Measurements of effective masses have been made using the temperature dependence of the amplitude of the de Haas–van Alphen effect at the symmetry points in each plane. The experimental data and the APW predictions are shown in Table I; Phillips reported only that the masses ranged from 1.7 to 2.1. It is obvious that these masses are larger than the APW calculations. This mass enhancement is generally believed to be due to the phonon enhancement of the bare electron mass, as pointed out by McMillan.[25] His results are expressed in terms of a constant λ, such that

$$m^*_{\text{exp}} = (1+\lambda)\, m^*_{\text{calc}}.$$

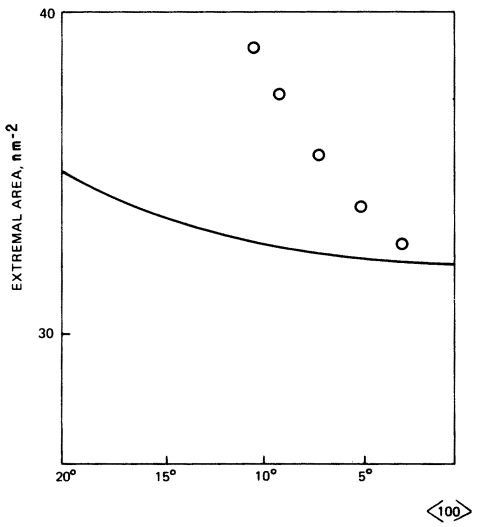

FIG. 5. Comparison of jungle-gym data to the extremal cross-sectional area of a cylinder. Solid line is the extremal cross-sectional area of a cylinder of 32-nm^{-2} diameter.

In vanadium McMillan's value for $1+\lambda$ is 1.60. For those two points where both experimental and APW results exist, the ratio m^*_{exp}/m^*_{calc} is found to be 2.05 and 2.40 for $E(1, 2)$ at $\langle 100\rangle$ and $E(1)$ at $\langle 110\rangle$, respectively. The accuracy of the experimental results is good, and they are consistent with the measurements of Phillips.[12]

However, Scott and Springford found for niobium[5] that the measured enhancement factor was 15–20% larger than that calculated by McMillan. This difference was thought to have resulted from the use by McMillan of an average isotope shift constant for transition metals (this constant has not been measured in either Nb or V). If the same explanation is valid for vanadium, it is not unreasonable to assume that $1+\lambda$ is closer to 2.0 and that the isotope shift constant in vanadium is higher than the average used by McMillan.

Nevertheless, so few calculated masses were presented that no firm conclusion regarding their validity can reasonably be drawn.

V. CONCLUSIONS

The Fermi surface in vanadium has been found to be qualitively similar to those of tantalum and niobium, and to the model of Mattheiss for the V*b* group metals. The data admit the possibility that the ellipsoids and the multiply connected hole surface of the third zone are connected by necks along the N-Γ direction, as postulated by Mattheiss and Papaconstantopoulos *et al.* for some conditions. However, it was not possible to confirm the existence of such necks by measuring signals from orbits around them. The effective masses have been measured, and agreement with other experiment has been found. The enhancement factor has been found to be greater than 2.0. General agreement with the APW calculations is good for an exchange constant of 0.715, except for the possible existence of the necks, which the calculation predicts only under high pressure.

The obvious interesting problem arising from these experiments is the confirmation of the existence of the necks along N-Γ. Several possible experimental approaches could be used to prove or disprove this theory. Superconducting magnet facilities capable of 18 T or more are now available, so one approach would be to repeat these experiments in a much higher field range than used here (7–10 T). One might attempt to grow purer samples for work in either field range, although we believe this would be very difficult. Finally, if the degeneracies along Γ-N exist, one would expect to see open orbits along the $\langle 110\rangle$ directions in a magnetoresistance experiment.

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