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Radio-Frequency Size Effect and the Fermi Surface of Molybdenum[†]

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Fermi-surface dimensions for the (110) plane for molybdenum are determined from caliper dimensions obtained through radio-frequency size-effect measurements. These measurements were taken over a frequency range of 6.5–26 MHz on high-purity molybdenum approximately 0.13 mm in thickness. Studies of the frequency dependence of the extrema in the resonance line shape were used to determine the magnetic field values for resonances along major crystallographic directions. The Fermi-surface dimensions are compared with recent theoretical and experimental work on molybdenum.

I. INTRODUCTION

The Fermi surface and energy-band structure of molybdenum have been the subjects of several investigations in recent years. The Fermi-surface model for Mo was first proposed by Lomer. 1 In a subsequent augmented-plane-wave (APW) calculation Loucks² determined Fermi-surface dimensions along the major symmetry directions, which verified the Lomer model. However, a detailed calculation of the Mo band structure and Fermi surface has not been published. Experimental studies of the Mo Fermi surface have been reported on the anomalous skin effect, 3 the dc size effect, 4 the magnetoresistance, 5,6 the de Haas-van Alphen (dHvA) effect, 7-10 the magnetoacoustic effect, 11,12 cyclotron resonance, ¹³ and the radio-frequency size effect (RFSE). ^{14,15} In the present publication we present the results of a RFSE investigation on the (110) plane in Mo in which the resonance field values were determined on the basis of frequency studies of the RFSE line shapes. A preliminary report of this work has been given. 16 Reviews of the experimental aspects of the RFSE technique have been written by Gantmakher¹⁷ and by Walsh. ¹⁸ The theoretical aspects are discussed in a review by Kaner and Gantmakher 19 and in recent papers by Juras. 20,21 In RFSE experiments, a flat single-crystal metal plate, sufficiently pure that the electron mean free path is on the order of the thickness of the plate at helium temperatures, is placed in the presence of

a magnetic field. For electrons executing trajectories such that $\vec{\mathbf{v}} \cdot \vec{\mathbf{n}} = 0$ ($\vec{\mathbf{v}}$ is electron velocity and $\vec{\mathbf{n}}$ is normal to plate surfaces) at the two surfaces of the plate, anomalies (RFSE resonances) occur in the surface impedance of the plate. For the magnetic field directed parallel to the plate surfaces, these anomalies yield Fermi-surface caliper dimensions $|\Delta \vec{\mathbf{k}}|$ given by $\Delta \vec{\mathbf{k}} = (e/\hbar)t(\vec{\mathbf{n}} \times \vec{\mathbf{B}}_{res})$, where t is the sample thickness and B_{res} is the magnetic field value at the resonance. For central orbits $\Delta \vec{\mathbf{k}} = 2\vec{\mathbf{k}}_E$.

Because the RFSE resonance linewidth ΔH extends over a field range 5-20% of the magnetic field magnitude, the assignment of accurate resonance field values is difficult. The question therefore arises as to what are the proper criteria to use to determine the correct resonance field value. In an investigation on potassium, Koch and Wagner²² assigned B_{res} to a point close to the first discernible departure of the resonance from the background. This assignment was made on the basis of a study of the frequency dependence of the linewidth and from the known Fermi-surface dimensions for potassium. Krylov and Gantmakher²³ found in a similar study on In that the resonance linewidth reduced to zero with extrapolation to infinite frequency at a field value corresponding to a position quite close to the low-field side of the resonance. They concluded that the field value obtained with this technique is the proper value to assign to the resonance. A result similar to that of Krylov and Gantmakher was obtained by the present authors. 16 In all three

investigations, 16,22,23 the resonance linewidth ΔH was found to vary as $\omega^{-1/3}$, which is to be expected under the conditions of the anomalous skin effect regime since $\Delta H \sim \delta$, the characteristic penetration depth of the radio-frequency field.

To properly assign the resonance field values, it is necessary to determine the position of the first discernible departure of the trace from the background. Accurate assignment based on visual examination of the X-Y recordings is difficult, however. In the present investigation, it was found that the first departure of the signal from the background could not be ascertained visually to an accuracy of less than 3% for the relatively strong signals, 5% for weak resonances, and 7-10% for complicated signals where RFSE resonances interfere. We therefore assigned the resonance field values on the basis of frequency studies of the resonance line shapes to achieve greater accuracy.

II. EXPERIMENTAL TECHNIQUES

A. Sample Preparation

The samples used in this investigation were prepared from a single-crystal rod having a residual resistance ratio $(R_{300\,k}/R_{4.2\,k})$ of 5000 as determined by the eddy-current decay method. 24 Plates 0.7-0.8 mm thick were cut from the rod such that each plate normal was within 2° of a (110) axis and then electropolished (6% solution of perchloric acid in methanol at - 70 °C). Each plate was mounted with beeswax on a lapping tool (designed so that the lapping plane could be adjusted 5° in any direction), oriented to within $\pm 1^{\circ}$ of a $\langle 110 \rangle$ axis, and lapped flat with No. 600 grit paper. To ensure parallel sides, a fixed lapping tool was then used to lap the second side, after which each plate was again electropolished. All of the samples had highly polished surfaces with a few faint scratches. RFSE resonances were detected for samples of thickness 0.075-0.25 mm prepared with these techniques. The bulk of the data discussed in this paper, however, was obtained with one sample of approximate thickness 0.13 mm. To reduce any possible effects of electron mean-free-path deterioration with thermal cycling, each sample was kept at liquid-nitrogen temperature (77K) or below for the entire duration of RFSE measurements on it.

B. Sample Thickness Determination

The sample thickness was measured by three different methods, the results of which were consistent with each other. First, the thickness was determined by means of a Leitz optometer with the vertical stage calibrated in microns. Second, the thickness was determined from a knowledge of the sample area, weight, and the known density of molybdenum, 10.22 g/cm³. The area of the sample was determined by means of a shadowgraph tech-

nique, and the weight of the sample was determined with a precision balance. The third method was found to be the most precise of the three: The sample was placed in the spring-loaded exit slit of and optical monochromator, and the slit-width control dial adjusted such that the slit blades clamped down slightly on the sample. By passing the 6328-A radiation of a He-Ne laser through the slit, a clearly defined diffraction pattern could be observed on a distant surface. From a knowledge of the distance between equivalent minima in the pattern and of the distance between the slit and pattern, the slit width, and hence the sample thickness, could be determined. The slit width was calculated from the diffraction pattern as a function of dial setting. A sharply defined break was found to occur when the slit width exceeded the sample thickness. 26 The optometer, shadowgraph, and slit-width methods yielded thickness determinations of 130 ± 2 , 127 ± 4 , and 129.4 \pm 0.2 μ , respectively. With 90% confidence limits, the sample thickness is taken to be 129. $4 \pm 0.3 \mu$. A Laue back-reflection x-ray picture indicated that the sample normal was within $\pm 30'$ of a $\langle 110 \rangle$ axis.

C. Apparatus

The RFSE resonances observed in this investigation were detected by placing the sample in the tank coil of a variable frequency oscillator at liquid-helium temperatures and detecting frequency changes with field modulation techniques. This method was first used by Gantmakher²⁷ and subsequently by others. ^{22,28} During most of this work, a modulation frequency of 80 Hz and a modulation amplitude of 5 Oe were used. For the detection of very weak signals, it was necessary to make use of continuous signal averaging techniques to enhance the signal-to-noise ratio. Such techniques are described in detail elsewhere. ^{29,30}

III. RESULTS

The data for the (110) plane were recorded by rotating the dc magnetic field in the plane of the sample. It was necessary to rotate the sample in the coil only once because very strong signals could be detected with the magnetic field directed at large angles from the coil axis. Representative traces are shown in Figs. 1 and 2. The assignment of these resonances to specific Fermi-surface calipers is discussed below. The rapidly varying background upon which the RFSE resonances were superimposed was found to be independent of the sample. The resonance occurring at 2000 Oe in Fig. 1 and labeled NMR represents the nuclear magnetic resonance of the H* protons in the GE 7031 varnish used on the oscillator coil. This signal was used as an internal calibration check on the magnetic field strength.

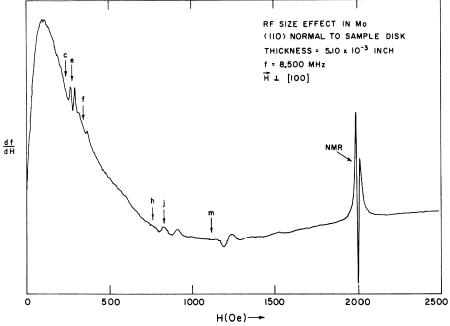


FIG. 1. RFSE resonances for Mo.

To provide more accurate estimates of $B_{\rm res}$ in this investigation, the RFSE line shapes were studied as a function of frequency for the magnetic field directed perpendicular to major crystallographic axes. The frequency study and analysis method used was that described by Krylov and Gantmakher. The magnetic field value for each extremum in the line shape (Fig. 3) was determined as a function of frequency f and plotted as a function of $f^{-1/3}$. Straight lines were fit to the data by means

of the least-squares fitting techniques with the non-constant precision of the data being taken into account. These techniques were used to study the line shapes for resonances detected with \overrightarrow{H} directed perpendicular to a $\langle 110 \rangle$ axis, a $\langle 111 \rangle$ axis, and a $\langle 100 \rangle$ axis

Typical results are shown in Figs. 4 and 5. In Fig. 4 we note that lines I and II converge to approximately one field value while lines III-VI to another, clearly demonstrating the existence of two reso-

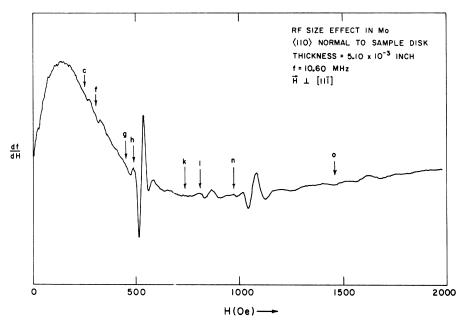


FIG. 2. RFSE resonances for

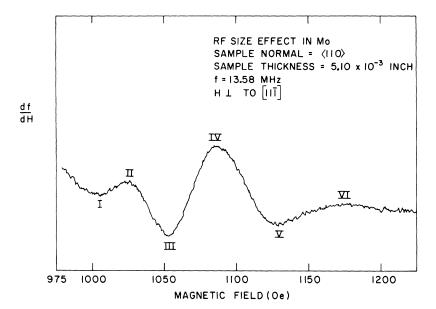


FIG. 3. Expanded trace of RFSE resonance in Mo.

nances. The intercept of lines I and II is indicated by the arrow at g and that for lines III-VI by the arrow at h in Fig. 2. The data for resonance n of Fig. 2 is shown in Fig. 5. All six lines converge to approximately the same field value, which is indicated by the arrow at n. This field value is double that determined for resonance h.

The resonance field values for all the resonances observed are plotted in Fig. 6 as a function of the direction perpendicular to the applied magnetic field. For all of the data shown, the field values

were corrected for the delay time in recording the data induced by the lock-in amplifier time constant.

The Fermi-surface dimensions calculated from the knowledge of the sample thickness and the resonance field values are shown in Fig. 7. Thermal contraction corrections were not made since they were negligible (0.1%) compared with other experimental uncertainties.

IV. DISCUSSION OF RESULTS AND CONCLUSIONS A. Fermi-Surface Parameters

We first consider the assignment of the data for

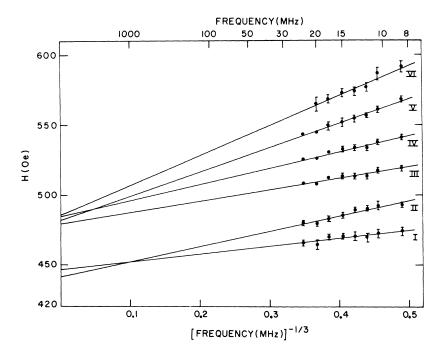


FIG. 4. Frequency dependence of the extrema in the RFSE line shapes for Mo as depicted in Fig. 3 for resonances g and h of Fig. 2.

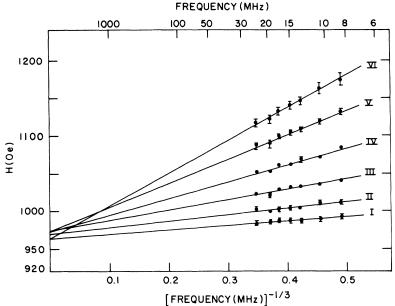


FIG. 5. Frequency dependence of the extrema in RFSE line shapes for resonance n of Fig. 2.

the smaller Fermi-surface pieces which are labeled a through f in Figs. 6 and 7. Such resonances arise from orbits on the hole ellipsoids, the electron lenses, and the knobs on the electron jack. The interpretation of these results was difficult because the resonances interfered with each other, and because some of the resonances were observed only over narrow angular ranges. The dimensions a are tentatively assigned to indicate the lens diameter, although, as shown in Table I, this dimension does

not agree with that of other investigations. For the hole pieces at N we refer to the dHvA^{7,8,10} results where the interpretation is made in terms of ellipsoids with semiaxes A, B, and C along the NP, $N\Gamma$, and NH directions, respectively, such that A > B > C. The best agreement with the existing data is obtained by taking C determined by the dimension b along the $\langle 110 \rangle$ directions and by taking A determined by the dimension of A along the [110] direction. Knowing the values of A and C thereby permits the

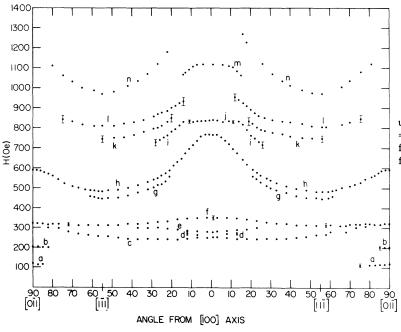


FIG. 6. Resonance magnetic field values for RFSE resonances in Mo with $t=129.4\pm0.3~\mu$ and $\hat{n}\parallel\langle110\rangle$ plotted as a function of direction normal to the applied field.

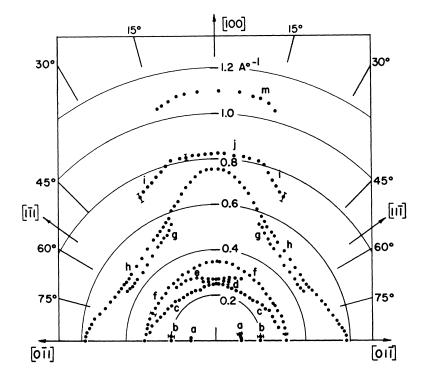


FIG. 7. Experimental Fermi-surface dimensions for the (110) plane for Mo.

calculation of B. The results shown in Table I for A, B, and C agree within the experimental error with the values obtained in other investigations.

The assignment of the results labeled g, h, and m can be made without ambiguity because of the signal strength and because of the frequency scaling. The results labeled g are thought to arise from orbits around the body of the electron jack while those labeled m are thought to arise from orbits around

two knobs and the body of the electron jack. Figure 8 shows the g-h line complex as a function of \overline{H} in the (110) plane. As \overline{H} is rotated toward [01 $\overline{1}$], the first minimum and maximum of the line complex disappear. This effect reaffirms the results of Fig. 4 that there are two resonances present in this strong signal complex. The results (see Figs. 6 and 7) exhibit discontinuities near 20° and near 60° because the extremal orbits for the electrons with

TABLE I. Experimental and theoretical values for the K vectors of the Fermi surface of molybdenum (in Å-1).

Fermi-surface piece	Direction	Present investigation	Other RFSE results ^a	Theoryb	Other investigations
Electron jack	⟨100⟩	1.098 ± 0.005°	1,16	1.149	$1.20\pm0.10,^{d}1.19,^{e}1.13^{f}$
	(111)	0.439 ± 0.014^{c}	0.47	0.438	0.47 ^f
	(110)	• • •	0.52	0.486	0.58 ^f
Knobs	(110)	0.32 ± 0.02	0.35	0.32	$0.33, ^{f}0.365, ^{g}0.33 \pm 0.03^{h}$
Electron lenses (diameter)	⟨110⟩	0.24 ± 0.02^{i}	0.31	•••	0.30, f 0.32 ^g
Hole octahedra	⟨100⟩	0.751 ± 0.009^{c}	0.79	0.841	0.86 ^f
	(111)	0.476 ± 0.004^{c}	0.51	0,533	0.50 ^f
	(110)	0.580 ± 0.006^{c}	0.60	0.627	0.60 ± 0.05 , $^{d}0.60^{f}$
Hole ellipsoids	NP: A =	0.35 ± 0.02	0.38	0.368	$0.40, 10.39 \pm 0.01, 20.28^{h}$
	$N\Gamma$: $B =$	0.32 ± 0.04	0.29	0.266	$0.35, 10.30 \pm 0.01$
	NH: C =	0.20 ± 0.02	0.22	0.189	$0.20, 10.23 \pm 0.01, 20.21$

See Ref. 15.

^bSee Ref. 2.

 $^{^{\}rm c}$ The indicated uncertainty represents a 90% confidence limit. An additional 1% uncertainty exists due to possible inaccuracies in the magnetic field.

dSee Ref. 11.

^eSee Ref. 14.

See Ref. 10.

See Ref. 8.

hSee Ref. 12.

^{&#}x27;Tentative assignment; see text.

the magnetic field directed in the range $0^{\circ}-20^{\circ}$ from the [01 $\overline{1}$] axis and in the range $0^{\circ}-30^{\circ}$ from the [100] axis are around the knobs on the electron jack. These discontinuities arise because the dimensions for such orbits are larger than those for the body of the electron jack. Similar discontinuities have been

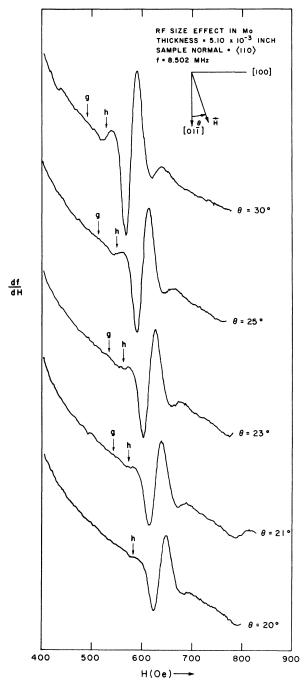


FIG. 8. Recordings of RFSE resonances g and h showing the disappearance of the electron jack resonance g and H directed 20° from the $[01\overline{1}]$ axis for a $\langle 110 \rangle$ sample normal.

observed in dHvA results 10 and in cyclotron resonance results 12 for Mo. The results labeled h in Figs. 6 and 7 are assigned to the hole octahedron. As shown in Fig. 4 of Ref. 16, the dimensions for the electron jack and hole octahedron give the shape predicted by the Lomer model for the (110) section. The dimensions obtained for these two pieces, however, are smaller than those of other investigations as shown in Table I.

The data labeled i and j are believed to arise from noncentral orbits on the electron jack. For j the electrons pass around one knob, over the body, and at the neck for the opposite knob. This conclusion corroborates that for similar data in Ref. 15. The exact orbit corresponding to the data labeled i is not clear, but is not considered to be a continuation of j since no such resonances were detected in the $12^{\circ}-15^{\circ}$ range where the two sets of data appear to join (see Figs. 6 and 7).

The results labeled k and l in Fig. 6 are thought to arise from chains of orbits from the hole octahedron and from the smaller Fermi-surface pieces such that k = h + c and l = h + f.

One of the features of the Lomor model¹ is that the electron jack and hole octahedra almost touch along the ΓH directions. From the APW energyband calculations of Loucks, 2 the separation between these two Fermi-surface pieces is $\Delta k = 0.008 \text{ Å}^{-1}$. Boiko, Gasparov, and Gverdtsiteli¹⁵ estimated the gap to be $\Delta K = 0.05 \pm 0.04 \text{ Å}^{-1}$. From the results of the present investigation, however, we estimate the gap to be $\Delta K = 0.15 \pm 0.04 \text{ Å}^{-1}$, which amounts to 7.5% of the ΓH dimension. In a previous publication 16 we attributed this gap to spin-orbit coupling effects of the 4d electrons which split the degenerate Δ_5 energy band into the central Δ_6 and Δ_7 bands.³¹ Because a detailed energy-band calculation for Mo is not available, a quantitative explanation of a gap of this magnitude cannot be made. We note, however, that the gap dimension is inversely proportional to the slope of the central Δ_6 and Δ_7 bands at the Fermi energy and hence inversely proportional to the d bandwidth. 26 Since a narrow bandwidth is expected in Mo because of the more localized nature of its d-wave functions, an enhancement of the gap dimension in Mo could occur.

The cross-sectional area in the (110) plane for the octahedron is estimated to be 1.01 ± 0.03 Å $^{-2}$ from the measurements of the present investigation and 1.08 Å $^{-2}$ from a previous RFSE study. 15 Areas obtained in dHvA investigations are 1.13, 9 1.08, 10 and 1.11 Å $^{-2}$, 32 which are significantly smaller than the area 1.24 Å $^{-2}$ predicted by Loucks. 2 Preliminary high-field dHvA results of Hoekstra 33 obtained subsequent to the completion of the present investigation yield an area of 1.08 ± 0.01 Å $^{-2}$, which is approximately 7% larger than the value obtained in the present investigation.

Because the measurements of the present investigation yield results which are at variance with those of other investigations, we consider possible sources of experimental errors. Since the normal to the sample was aligned within $\pm 30'$ of a $\langle 110 \rangle$ axis, the uncertainty in the orientation will introduce a negligible error. In the determination of the sample thickness, each value obtained for t is consistent with the other two. In order that the results of the present investigation match those of Ref. 15, the thickness needs to be approximately 138 μ , which is several standard deviations from the measured value. Another possible source of error is that the plane of the sample may have been inclined at an angle to the plane of rotation of the magnetic field. Because of the care exercised in constructing the sample holder and coil, we do not believe inclination errors to be significant.

Another possible source of error is the magnetic field calibration. NMR data appear on a number of the RFSE graphs (as in Fig. 1) and yield H values to better than $\frac{1}{3}\%$ of that presumed from the high-dial setting. A systematic error of more than 1%, though possible, seems unlikely.

B. Frequency Studies

A visual examination of the line shape to determine where the first deviation from the background occurs may not be reliable. In recent calculations, ^{20,21} it is shown that the initial features of the line shape are strongly influenced by the ratio of the electron mean free path to sample thickness as well as by the type (specular or diffuse) of surface scattering by the electrons. Tsui and Gantmakher³⁴ have shown experimentally that the electron mean free path does influence the line shape. As suggested by Haberland, Cochran, and Shiffman, ³⁵ we used the frequency dependence of the line shape to determine the resonant field values.

To show that the results in Figs. 4 and 5 actually yield a $\omega^{-1/3}$ dependence, the data in Fig. 5 were fit to the straight line y = b + mx, where $x = f^{\alpha}$, using the least-squares fitting technique. This resonance represents the doubling of the octahedron signal. The 90%-confidence limits for the estimate of error of the mean intercept were determined for -1.0 $\leq \alpha \leq -0.2$. Figure 9 demonstrates that for an exponent of $-\frac{1}{3}$ the minimum deviation from the mean intercept is obtained. The resonance linewidth thus indeed varies as $\omega^{-1/3}$ and hence scales with the radio-frequency penetration depth for the anomalous skin effect regime. We would therefore expect the frequency dependence of the line shape to yield the appropriate resonance field values. However, since the results of the present investigation do not agree with those of other studies, it is possible that such is not the case, although this seems unlikely.

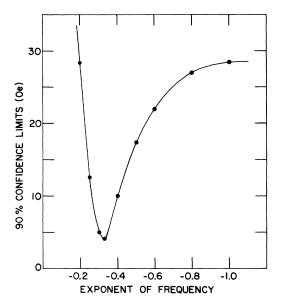


FIG. 9. Confidence limits for the mean value of the intercepts for the six lines in Fig. 5 as a function of the exponent for the frequency. The minimum confidence limit occurs for an exponent of $-\frac{1}{3}$.

The difference between assigning the field values at the onset of the resonance and that determined from the frequency dependence of the line shape is indicated in Ref. 16. The field values at the onset of resonances h and m along [100] yield Fermi-surface dimensions of 0.77 and 1.13 Å⁻¹ for the octahedron and jack, respectively. Although these values are still smaller than those reported for other investigations, the agreement is better. For the resonance corresponding to the octahedron dimension along $\langle 110 \rangle$, however, the field value at the onset of the line shape and that determined from the frequency dependence coincide. For this dimension, agreement with the results of other investigations is good.

By choosing the field values at the onset of the resonance, the agreement with existing data is better but smaller caliper dimensions are still obtained along $\langle 111 \rangle$ and $\langle 100 \rangle$. We note that, in a RFSE investigation in Cu in which a different technique was used, caliper dimensions smaller than the accepted values were obtained along certain directions. ³⁶

V. SUMMARY

Measurements of the caliper dimensions for the Fermi-surface pieces for the (110) plane for Mo have been obtained using the RFSE technique. The resonance field values were determined from the frequency dependence of the line-shape extrema for resonances along major crystallographic directions.

We believe this technique should yield more accurate estimates of the resonance field values than a visual examination of the resonances because the linewidth scales with the radio-frequency penetration depth. We show that a $\omega^{-1/3}$ frequency dependence best explains the linewidth. However, although there is agreement between the results of the present investigation and those of earlier investigations concerning the shapes of these larger pieces, the Fermi-surface dimensions obtained do not agree. The reasons for the disagreement are at present unknown.

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