GUREVICH MAGNETOMORPHIC OSCILLATIONS IN SINGLE CRYSTALS OF ALUMINUM AT HELIUM TEMPERATURES

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THESIS

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By

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CHAPTER I

INTRODUCTION

In thin metal films where the mean free path is comparable with some other characteristic length in the metal, one must consider the effects of boundary scattering in problems of electron transport. Boundary scattering under these circumstances is known as a size effect. The limitation of the free path by the boundaries of the specimen was considered as early as 1901 by J. J. Thomson (24). Effects of this type have attracted much attention in the last few years (8, 13, 14, 18). Sondheimer (22) has treated the case of electrical conductivity in thin metal films with a transverse magnetic field. He predicted oscillations in the Hall effect and transverse magnetoresistance as a function of magnetic field with the field applied perpendicular to the plane of a thin conducting metal film. Since his prediction a limited number of experiments have been reported confirming these oscillatory effects both in single crystals and in polycrystalline samples (1,2,4,5,14,17,20,25,26).

In particular, Sondheimer has calculated the conductivity tensor for the case of an infinite, isotropic, crystalline film with a magnetic field perpendicular to the plane of the material. He assumed a spherical Fermi surface, diffuse reflection at the crystal boundaries, and the existence of a relaxation time τ, and solved the Boltzmann equation for the

perturbation to the equilibrium Fermi distribution. The Boltzmann equation for the distribution function f of the conduction electrons is formed by equating the rate of change in in f due to external fields to the rate of change due to the collision mechanism, which is assumed given by

$$\left[\frac{\partial f}{\partial t}\right] = -\frac{f - f_0}{\tau} .$$

$$f_0 = \left\{ \exp(E - E_F)/kT + 1 \right\}^{-1}$$

is the Fermi-Dirac equilibrium distribution function with E the energy of a particle in the system under consideration, E_F the Fermi energy and kT Boltzmann's constant times the absolute temperature. In the presence of an electric field E and a magnetic Field H, the Boltzmann equation takes the form

$$-\frac{e}{m}\left(E + \frac{1}{c}\vec{v} \times \vec{H}\right) \cdot \operatorname{grad}_{\vec{v}} f + \vec{v} \cdot \operatorname{grad}_{\vec{r}} f = -\frac{f - f_0}{\tau}$$

with e the charge of an electron, m the electronic mass and c the speed of light. Sondheimer considered a metal film of thickness a with the Z axis perpendicular to the plane of the film, and the surface of the film being the planes Z = 0, Z = a. When the film is subjected to an electric field (E_x, E_y, 0) in the plane of the film and a magnetic field (0, 0, H) along the Z axis, the Boltzmann equation takes the form

$$\frac{\partial f_1}{\partial z} + \frac{f_1}{\tau v_z} - \frac{eH}{mcv_z}\left(v_y \frac{\partial f_1}{\partial v_x} - v_x \frac{\partial f_1}{\partial v_y}\right) =$$

$$\frac{e}{mv_z}\left(E_x \frac{\partial f_0}{\partial v_x} + E_y \frac{\partial f_0}{\partial v_y}\right).$$

Sondheimer finds upon applying the complex electric field

$$E = E_x - iE_y$$

$$f = f_0 + f_1(\vec{v}, z) = f_0 + (v_x c_1 + v_y c_2)\frac{\partial f_0}{\partial v} \qquad (4a)$$

with

$$c_1 - ic_2 = \frac{e\tau E}{mv[1 + (ieH\tau/mc)]} \times$$

$$\left[1 + h(v)\exp\left\{-\left(1 + \frac{ieH\tau}{mc}\right)\frac{z}{v_z\tau}\right\}\right] \qquad (4b)$$

where

$$h(v) = 1 \quad \text{for } v_z > 0 \qquad (4c)$$

and

$$h(v) = -\exp\left\{\left[1 + (ieH/mc)\right]\frac{a}{v_z\tau}\right\} \text{ for } v_z < 0. \qquad (4d)$$

The conditions (4c) and (4d) are introduced by applying the boundary conditions such that $f_1(\vec{v}, 0) = 0$ for all v such that $v_z > 0$ and $f_1(\vec{v}, a) = 0$ for all $\vec{v}$ such that $v_z < 0$.

Defining a complex current density $J = J_x - iJ_y$, one obtains

$$dJ(v_z) = dn(v_z)\ (e^2\tau'/m)\left\{1 + [v_z\tau'/a]\left[\exp\left(-\frac{a}{v_z\tau'}\right) - 1\right]\right\} E \qquad (5)$$

where $dJ(v_z)$ is the average, over the thickness of the crystal, of the contribution of the electrons $dn(v_z)$ belonging to a slice of the Fermi sphere bounded by the planes v_z, v_z, + dv_z. The quantity τ' is defined by $1/\tau' = 1/\tau + i(eH/mc)$. Defining

$$t = v_F/v_z \quad , \quad \lambda = v_F\tau \quad , \quad H_o = mv_Fc/ea$$

$$s = (a/\lambda) + i(H/H_o) = K + i\beta$$

$$J_x - i J_y = (\sigma_{11} + i\sigma_{12})(E_x - i E_y) \qquad (6)$$

and integrating Equation (5) over the Fermi sphere, one obtains the result

$$\sigma_{11} + i\sigma_{12} = 3/2\ \sigma_b \int_1^\infty (t^{-2} - t^{-4}) \chi \left\{1 + (st)^{-1} [\exp(-st) - 1]\right\} dt \qquad (7)$$

where the bulk conductivity is

$$\sigma_b = nec(H_i - iH) / (H^2 + H_i^2),$$

and the saturation field is

$$H_i = mc/e\tau \quad .$$

λ is the mean free path of the electrons and v_F is the velocity of the electrons on the Fermi sphere.

Grenier (9) has shown that if an expansion of Equation (7) is made using the method of integration by parts, it is found that the first term gives a good approximation to the results for the high magnetic field oscillatory phenomena.

Grenier found:

$$\tilde{\sigma}_{11} + i\tilde{\sigma}_{12} = (\pm 3 |\sigma_{12_b}| \beta^{-3} e^{-K}) \chi [\cos\beta + i\cos(\beta + \pi/2)] \qquad (8)$$

where $\tilde{\sigma}_{11}$ and $\tilde{\sigma}_{12}$ denote the oscillatory components of the magnetoconductivity σ_{11} and the Hall conductivity σ_{12} respectively. The upper sign refers to a sphere of electrons

and the lower sign refers to a sphere of holes. The oscillations in $\tilde{\sigma}_{11}$ and $\tilde{\sigma}_{12}$ are attributed the following properties by Equation (8):

(1) period = $P_o = 2\pi H_o = 2\pi mv_F c/ea$,

(2) maxima for $\tilde{\sigma}_{12}$ should occur when $H = H_n$ $= (n - \delta/2\pi) P_o$ with $\delta = \pi/2$, and maxima should occur for $\tilde{\sigma}_{11}$ when $H = H_n = nP_o$ where n is an integer,

(3) the amplitude should decay as H^{-4} at high field,

(4) at constant H, the amplitude should vary with temperature as $e^{-a/\lambda(T)}$.

Grenier also extended the Sondheimer theory to the case of Fermi surfaces which are axially symmetric about the z axis. In particular, a lens-shaped pocket of the form which occurs in the third Brillouin zone in cadmium has been considered. The differences in the results of these calculations as compared to Equation (8) are a reduction in the predicted amplitude of the oscillations by a factor of 0.58 and the substitution of the radius of curvature of the lens apex for the radius of the Fermi sphere which reduces the expected period by a factor of 0.97 with no change in phase.

Gurevich (10) has treated arbitary energy surfaces and, specifically for the case of convex surfaces, the radius which enters is given by $1/\sqrt{\alpha}$ where α is the Gaussian curvature at the elliptic point where the Fermi surface normal is parallel to the magnetic field. The period P_o, is given by

$$P_0 = \frac{2\pi c}{ea\sqrt{\alpha}} \tag{9}$$

where a is the sample thickness.

Forsvoll (5) used polycrystalline aluminum films in a transverse magnetic field and found the size effects to be in good qualitative agreement with Sondheimer's theory. Forsvoll used several cold-rolled samples of aluminum ranging in thickness from 2.43 mm to 0.0125 mm. It was found that ρ_{11} showed no tendency to fall towards the bulk value in high magnetic fields as required by the Sondheimer theory.

Balcombe (2) made low-temperature measurements of transverse magnetoresistance on 24 single crystals of pure aluminum in magnetic fields up to 20 Kg, and found that neither complete saturation of magnetoresistance nor quadratic variation with field were observed in any orientation. In the best specimens at 1.4°K, Balcombe calculated the mean free path of conduction electrons to be not more than 0.1 mm on the basis of a free-electron model.

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CHAPTER II

EXPERIMENTAL PROCEDURE

The Sondheimer theory was tested by looking for oscillatory phenomena in a group of single crystals representing a range in dimensions from matchbox geometry to thin-film geometry. The single crystals were identical with respect to impurity content, strain, orientation, surface condition, and probe placement.

The initial single crystal was spark machined from an aluminum ingot obtained from Cominco Products, Inc., Spokane, Washington. It was X-ray oriented (Figure 1) and spark machined into parallelepiped geometry of dimensions 20.0 x 5 x 2 mm. The (111) axis was perpendicular to the large faces to within 1°. The large faces were spark planed parallel to within 1°. In order to obtain a surface free of the polycrystalline layer produced by spark planing, the large faces were electropolished until approximately 100 microns were removed from each surface. After removal of 100 microns by electropolishing the large faces were found to be free from damage by the study of X-ray back-reflection Laue pictures.

The electropolishing solution consisted of 131 ml of 60 per cent Ac_2O (acetic anhydride) in 69 ml of 60.8 per cent $HClO_4$ (perchloric acid). Diluting 430 grams of 97 per cent Ac_2O up to 660 ml with 99.5 per cent HOAc (glacial acetic acid) yields 660 ml of 60 per cent Ac_2O. 350 ml of 60.8 per cent $HClO_4$ was prepared by diluting 505 grams of 70.5% $HClO_4$

up to 350 ml with distilled water. Upon mixing the polishing solution one should add the $HClO_4$ to the Ac_2O with the latter kept below 20°C. The addition of $HClO_4$ to the Ac_2O should be dropwise, for each drop of $HClO_4$ that comes into contact with the Ac_2O causes a temperature rise of approximately 2°C. The solution should be stirred gently while adding the $HClO_4$. After approximately 25 ml of the $HClO_4$ solution has been added there is no further danger of heat being released and the remaining 44 ml can be poured in. Once the solution is mixed it should be kept in a closed glass container, below 20° C, and in a safe place for perchloric acid is extremely active chemically. For additional precautions to be taken when using perchloric acid solutions Tegart's (23) book is a good reference.

A thin aluminum cylinder that just fits inside a 1000 ml beaker was used as the cathode. Experiment showed the best geometry for optimum polishing was with the aluminum crystal surface to be polished located at the center of the cylindrical cathode and horizontal. Miccroshield (21) was used to isolate the other surfaces from the electrolyte. Polishing was carried out in a 1000 ml beaker with the cell operated at 19 volts with a current density of 11 ma/cm^2, establishing a removal rate of 12.6 microns/hr. Prior to electropolishing concentrated HNO_3 or H_2CrO_4 was used as a cleaning agent for the aluminum crystal with distilled water being the final washing agent. Ten hours of electropolishing produced a mirror

finish. Repeated use of the electrolyte increased the polishing character of the solution.

Current, magnetoresistance, and Hall probes were soldered to one polished face. These wires were all soldered to the crystal at one time using Alutin 51-S solder and Alutin-51 flux. Both solder and flux were obtained from Eutectic Welding Alloys Corporation distributor in Dallas, Texas. After tinning the ends of the wires (34 gauge copper) with the solder and allowing a small amount of solder to flow down on the ends of each wire they were placed in a rectangular plate similar to aluminum base in Figure 1 with the trenches excluded. Masking tape held the wires in place while the crystal was placed on a hot plate with eletropolished surface facing up and heated to a temperature of $180^{o}C \pm 10^{o}C$. When this temperature was reached the hot plate was turned off and the wires, having been dipped in the flux, were lowered by using a three-dimensional goniometer until contact with the surface was made. The solder flowed and upon reaching room temperature the excess flux was washed away with distilled water. To facilitate subsequent spark planing and electropolishing, the crystal was cemented rigidly to an aluminum base, probes side down. A thin coat of Miccroshield and a piece of thin paper were used to effect electrical isolation from the base. The aluminum base was used as a precaution against thermal strain associated with repeated cycling between room temperature and helium temperatures. Finally, the aluminum base was

attached to a phenolic holder to form a rigid unit. Trenches were milled into the aluminum base to provide an exit for the twisted probe leads (Figure 1). This unit could be conveniently mounted in the spark machine for further planing, and was small enough to fit in the electropolishing cell for further polishing.

External leads were brought into the dewar system via an epoxy-sealed, thin-walled stainless steel tube, fitted with a phenolic holder. The aluminum base was attached to the phenolic, and the short probe leads were soldered to the external leads prior to data runs. Care was taken to insure that the helium level remained above all solder joints to prevent thermal emf's during the experiments. After each run the external leads were unsoldered and the aluminum base detached from the phenolic holder. Laue pictures were taken to insure absence of strain (Figure 2). The sample was then planed and electropolished to smaller thicknesses. In order to insure obtaining the same orientation of the crystal when placed back in the dewar system a plane mirror was attached to the perimeter of a brass plate that supported the tubing holding the crystal. The plane of this mirror was parallel to the plane of the crystal and directly above it. This allowed the orientation of the crystal by placing a plane mirror on the flat surface of the North pole of the electromagnet. Then the planes of the two mirrors were made parallel by use of a telescopic cathetometer. The final samples were not

spark machined but were thinned by electropolishing only.

The samples were examined with a Unitron Depthscope equipped with x - y translation drums calibrated directly in microns, and Z translation calibrated to five microns. The potentials were measured by recording the off-balance of a Honeywell Six-Dial Thermal-Free Potentiometer, amplified by a Backman Model 14 Breaker Amplifier (Figure 3). Current through the crystal was supplied by a 12 volt battery, and kept constant by adjusting variable rheostat while monitoring the voltage drop across a 1 Ω (1 per cent) precision resistor with a student potentiometer. Data were taken for normal and reverse directions of the magnetic field, and the results appropriately averaged to eliminate the effects of probe misalignment.

In general the current density is expressed as $\vec{J} = \hat{\sigma}\vec{E}$ where

$$\hat{\sigma} = \begin{pmatrix} \sigma_{11} & \sigma_{12} \\ -\sigma_{12} & \sigma_{11} \end{pmatrix}$$

is the conductivity matrix (19) for the motion of electrons in the xy plane with the magnetic field perpendicular to the sample. The resistivity is defined to be $1/\sigma$ which implies that in two dimensions $\hat{\sigma}\,\hat{\rho} = \hat{1}$, the identity matrix. This gives the following:

$$\sigma_{11} = \frac{\rho_{11}}{\rho_{11}^2 + \rho_{21}^2} \qquad \sigma_{12} = \frac{\rho_{21}}{\rho_{11}^2 + \rho_{21}^2} .$$

The resistivities (ρ_{11} - longitudinal, ρ_{21} - transverse) were programmed using

$$\rho = \frac{RA}{L} ,$$

where R is the resistance of the sample, A the cross sectional area and L the distance between the ρ_{11} probes. The data for $\rho_{11}(\pm H)$ and $\rho_{21}(\pm H)$ were averaged in the following manner.

$$\rho_{11} = \frac{\rho_{11}(+H, +J_x) + \rho_{11}(-H, +J_x)}{2}$$

$$\rho_{21} = \frac{\rho_{21}(+H, +J_x) - \rho_{21}(-H, +J_x)}{2} .$$

Corrections were added to the program to eliminate any drift in the recorder or amplifier while taking data.

Two crystals (No. 1, No. 2) were used with three samples being used from each. Electropolishing problems developed in the 0.15 mm to 0.07 mm region of sample thickness. In this area of thickness it was difficult to coat the perimeter of the samples with Miccroshield and as a result some of the polishing solution was able to penetrate under the sample and etch the solder spots. This problem did not show up until after the samples were placed back in the helium, where contraction separated the solder spot from the crystal. Erroneous data were obtained in that large oscillations appeared in ρ_{11} and ρ_{21}. The error was eliminated by resoldering the wires and repeating the run.

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CHAPTER III

RESULTS AND CONCLUSIONS

The magnetoresistance of both samples was observed to increase monotonically in fields up to 24 kG, contrary to the results of Forsvoll and Holwech, who found ρ_{11} saturated at 2-5 kG. The gross Hall resistivity, ρ_{21}, was positive and increased nearly linearly as a function of magnetic field with slope corresponding to a carrier density of approximately $1.2 \times 10^{23} cm^{-3}$. The result of subtracting a least-squares quadratic from each pross ρ_{21} is presented in Figure 4. It is seen that a very low amplitude oscillatory component, apparently periodic in the magnetic field, is present in both samples. The oscillation in the thinner sample is larger in amplitude and longer in period than in the thicker sample. Although theory gives expressions for the oscillatory conductivity $\tilde{\sigma}$, one may show (16) that the oscillatory Hall resistivity may be expressed as

$$\tilde{\rho}_{21} = \left| \tilde{\rho}_{21} \right| \cos\left(2\pi H/P_0 + \delta\right).$$

Then the field values for which maxima occur may be written as

$$H_m = \left(n - \frac{\delta}{2\pi}\right) P_0 \quad . \tag{10}$$

In order to normalize the data belonging to different thicknesses one may substitute P_0 from Equation (9) into Equation (10) to obtain

$$H_m a = \left(2\pi c/e\sqrt{\alpha}\right) n - c\frac{\delta}{e\sqrt{\alpha}} \quad . \tag{11}$$

Equation (11) shows that a plot of $H_m a$ against the integers should yield a straight line if δ is independent of field. The slope of the line determines the Gaussian curvature α, and the vertical intercept determines the phase δ. Figure (5) is such a plot for the two samples studied. Maxima have been plotted at the integers and minima at the half integers. The line chosen to represent the data corresponds to a radius $1/\sqrt{\alpha} = 5.3 \times 10^{-19}$ g-cm/sec. Because of the long period only a few maxima and minima were observed up to 24 kG. The horizontal separation of the data belonging to the two samples indicates δ may depend on a. The line indicating the free-electron slope and the line representing the slope corresponding to the data of Forsvoll and Holwech (7) are exhibited for comparison. The radius of Gaussian curvature of the Fermi surface in the (111) direction is seen to be about three times the radius of the Fermi sphere. This difference may be expected due to the lifting of the central symmetry points on the free-electron second-zone surface toward the zone faces (Figure 6), and the lowering of points near the zone corners and edges, due to the lattice potential (11, 12, 21).

The observed long period oscillations (11kG for a sample of thickness a = 0.022 cm and 18kG for the 0.007 cm sample) were quasi-periodic in the magnetic field with the period proportional to the reciprocal thickness. The slopes of the lines (Figure 5) yield the radius of curvature of the Fermi surface (5.1×10^{-19} g cm/sec) in the (111) direction which

is about three times the radius of the Fermi sphere in free-electron calculations. Forsvoll and Holwech's (7) Hall resistivity data showed oscillations having periods of 7kG and 10kG for sample thicknesses of 0.007 cm and 0.005 cm respectively, with saturation in large magnetic fields. The radius of curvature of the Fermi surface from their data (1.33×10^{-19} g cm/sec) was slightly less than the free-electron prediction. These differences in radii may be expected due to the lifting of the central symmetry points on the free-electron second-zone surface toward the zone faces (Figure 6), and the lowering of points near the zone corners and edges, due to the lattice potential. Forsvoll and Holwech's use of poly-crystalline samples prohibits the determination of any specific details of the Fermi surface due to the averaging effect of randomly oriented crystallites.

In order to make a further study of aluminum monocrystlas it is necessary to use very thin samples, since the amplitude of the size effect grows exponentially as thickness is reduced. However, reduction in thickness means a corresponding increase in the period of oscillation necessitating the use of very high fields.

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APPENDIX

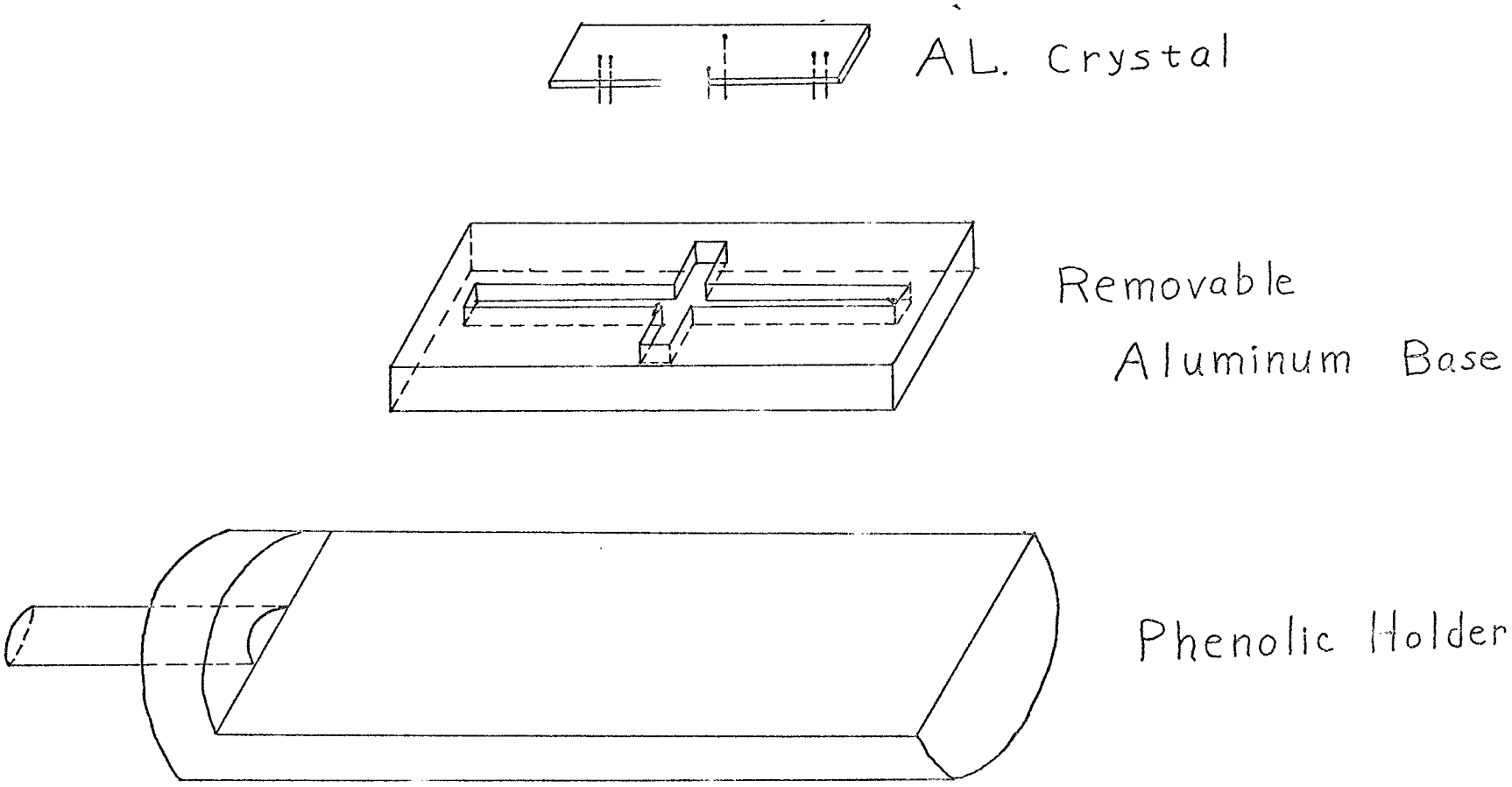

Fig. 1--The Crystal Assembly

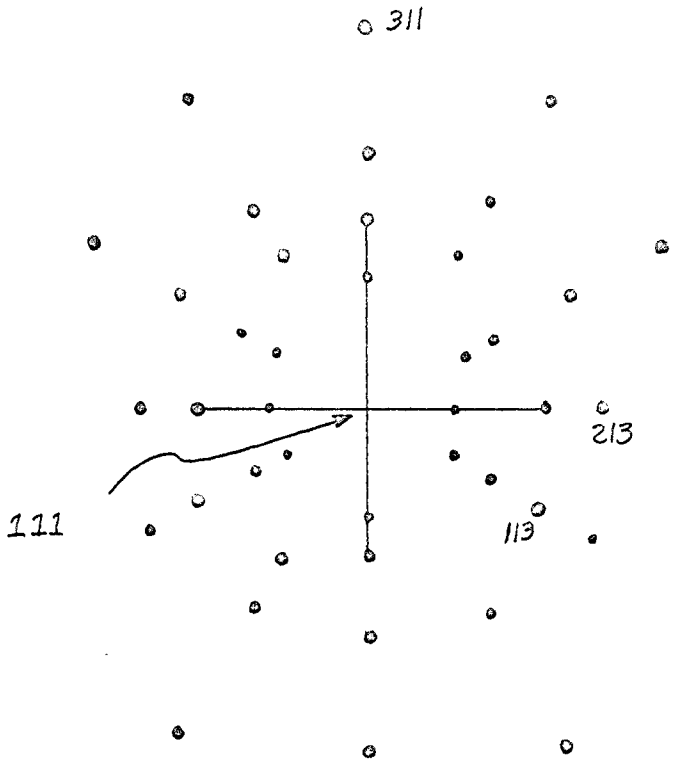

Fig. 2--Laue Picture of Aluminum Single Crystal

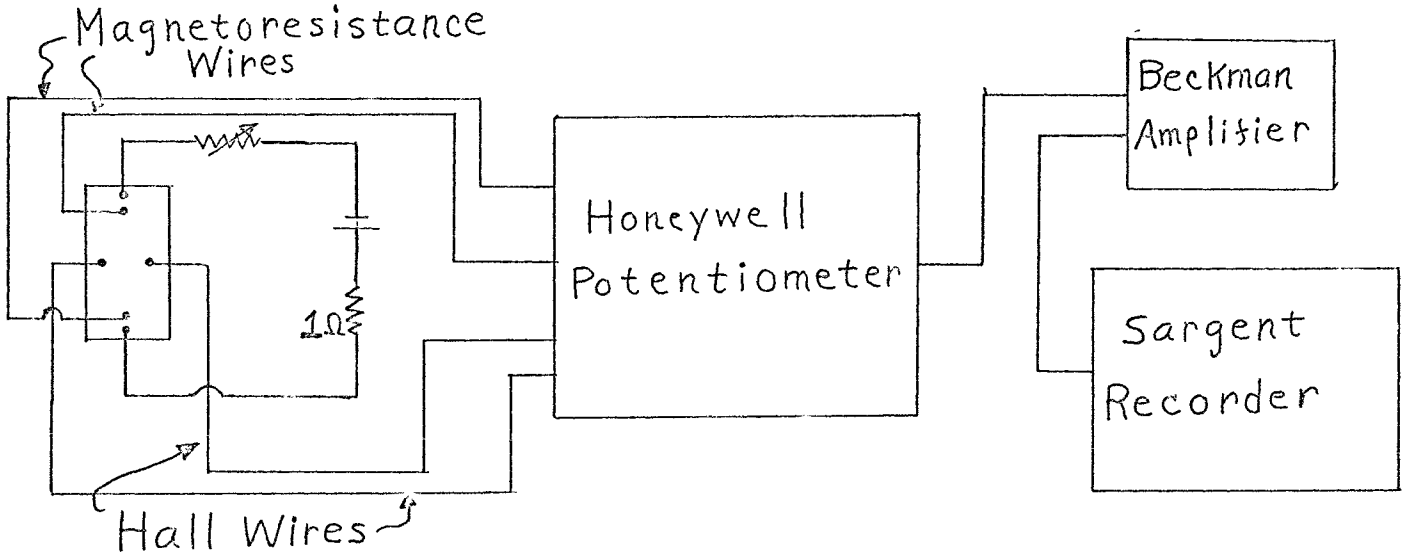

Fig. 3--D. C. Measuring System

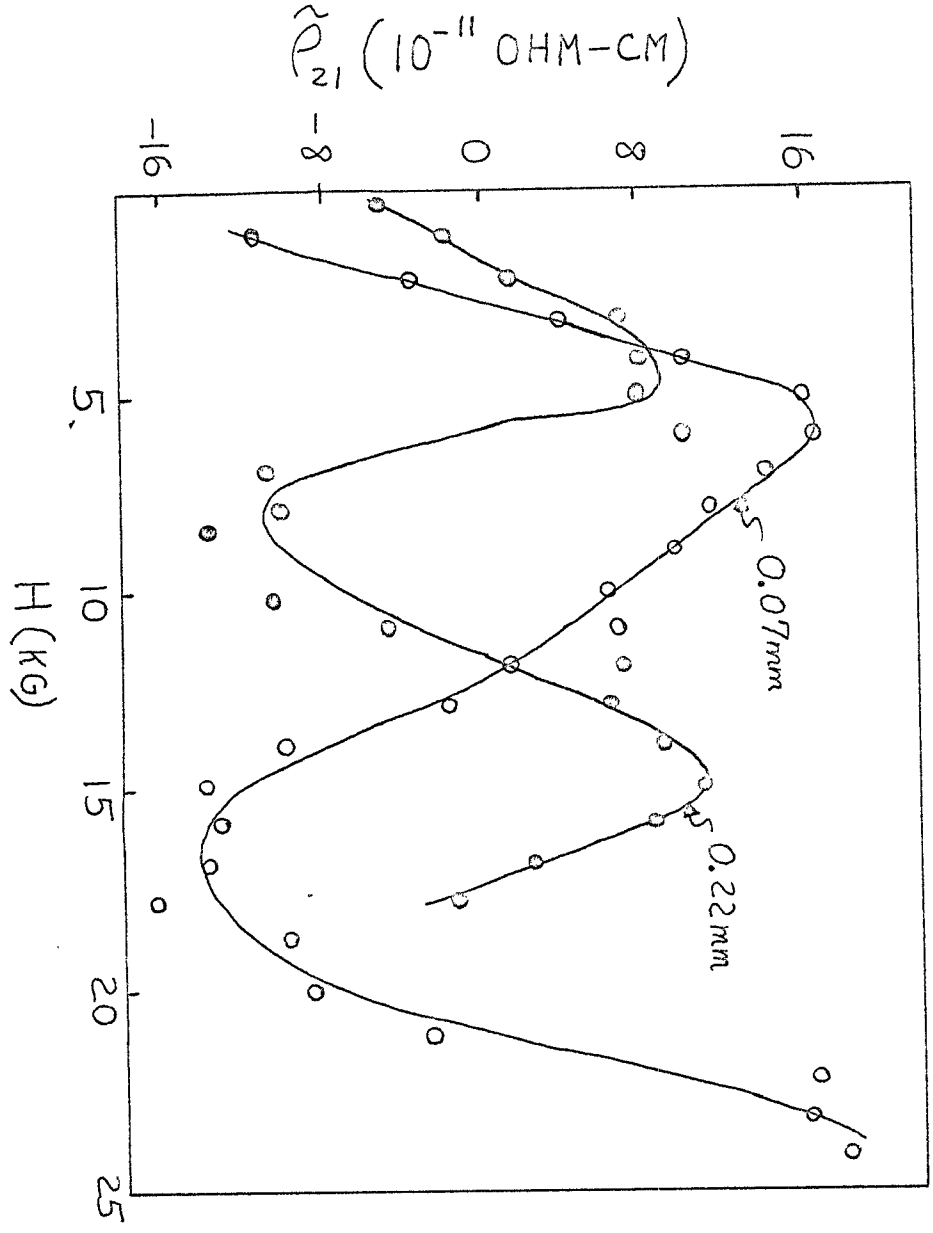

Fig. 4 -- ρ_{21} vs. H

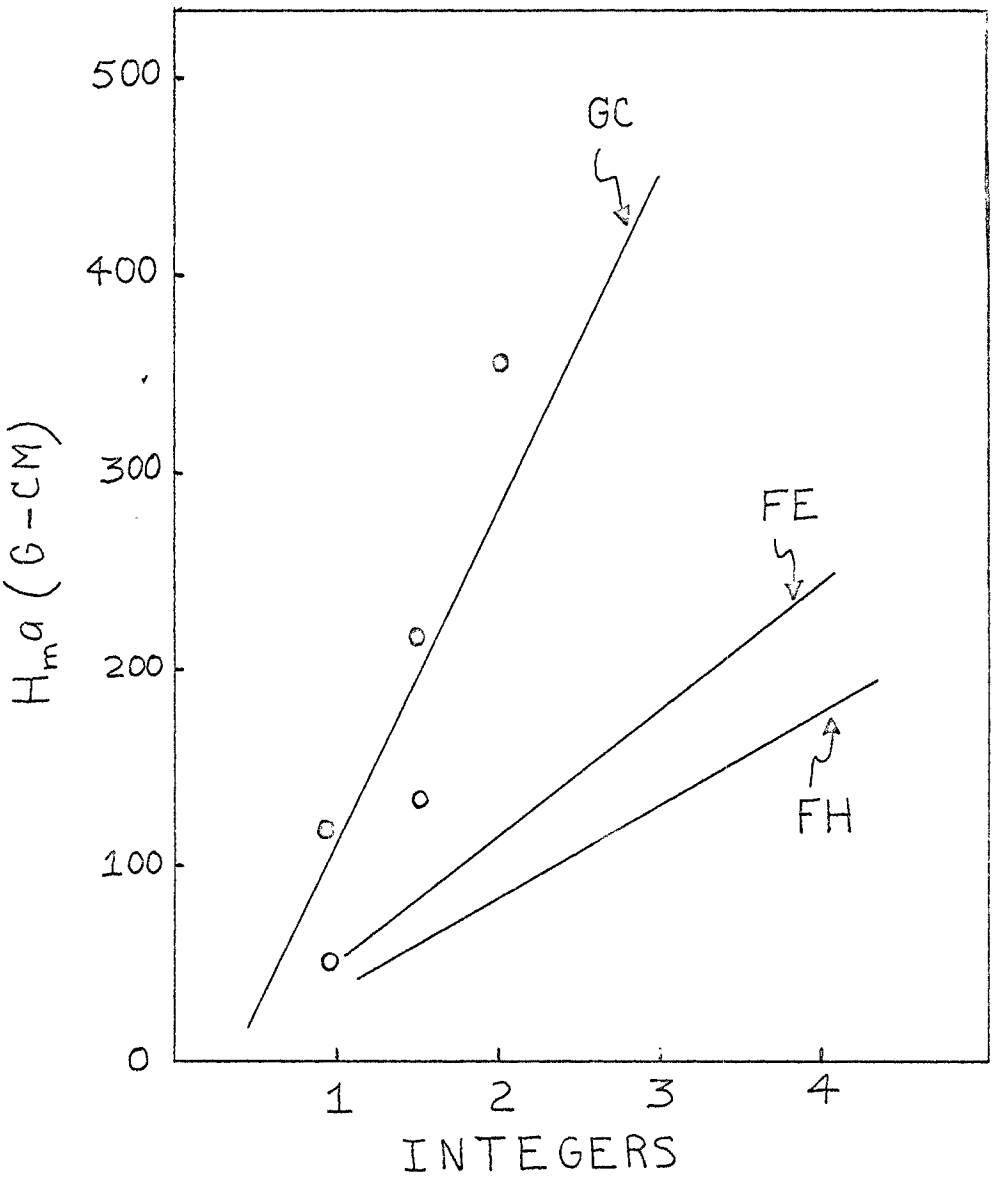

Fig. 5--$H_m a$ vs. Integers

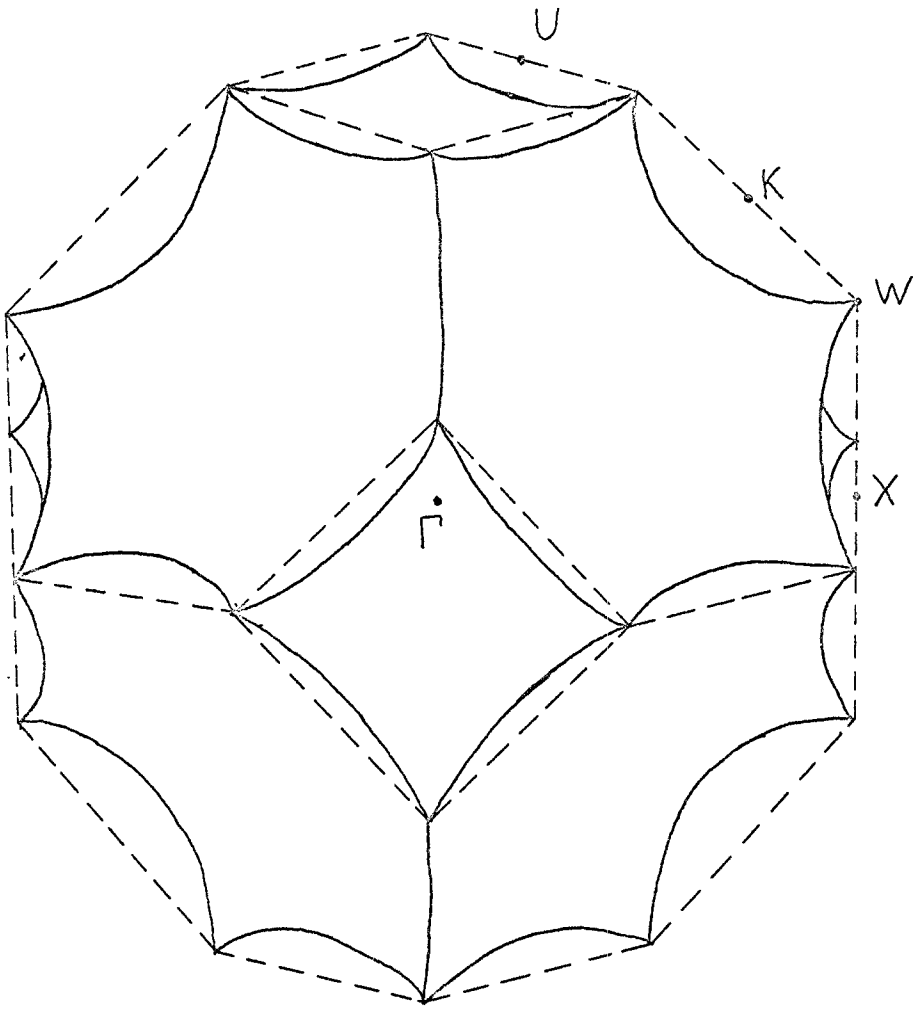

Fig. 6.—The Second Zone Hole Surface

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