

### III. SOLID STATE PHYSICS

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#### RESEARCH OBJECTIVES

##### 1. Soft X-Ray Spectroscopy

The soft X-ray spectroscopy program has as its objective the experimental study of the structure of the conduction band of electrons in a series of metals, particularly the alkalis, alkaline earths, and some of the transition metals. The filled portion of such a band can be studied by observing the emission spectrum produced by transitions from this band to the nearest available sharp levels below this band. In most metals this corresponds to an energy in the range 15-250 ev (wavelengths in the range 50-900 A), so that the technique of extreme ultraviolet vacuum spectroscopy is applied. The energy widths of these bands usually lie in the range 2-10 ev.

Work on copper and nickel, recently completed by R. G. Newburgh, and reported in Quarterly Progress Report No. 54, July 15, 1959, pages 44-46, has raised some fundamental questions regarding the structure of these bands. As indicated there, Newburgh found that by using very clean samples of the solid metal (as contrasted with evaporated films used in the earlier work) and a better vacuum, the band structure is considerably altered. Where single peaks were obtained from the evaporated targets, the solid targets give double peaks, with a total width approximately twice as great. Further, it was possible to reproduce the earlier results by using evaporated targets and a worse vacuum, so that the effect appears to be real.

While we do not claim that Newburgh's results give the "correct" band structure, it is apparent that the whole field is again opened up for a more careful investigation. As a result, we are, at present, looking into the matter of greatly improving the vacuum technique, as well as the analysis of the emitted spectrum.

G. G. Harvey

##### 2. Elastic and Thermal Properties of Solids

In recent years there has been a revival of interest in lattice vibrations and the theory of lattice dynamics. The dispersion curves, vibrational frequency spectrum, and the bulk thermodynamic properties of a crystalline material can be computed by a Born-von Kármán calculation based on interatomic force constants. The adiabatic elastic constants of a single crystal may be measured with high precision, and provide data for evaluating these force constants. Since many properties are more sensitive at low temperatures to the parameters of the force model employed, calculations based on the elastic constants at liquid-helium temperature are of great interest.

The elastic constants of a crystal may be computed from the velocities of ultrasonic waves propagated along various crystallographic axes. Pulsed-circuit techniques provide a convenient method for measuring such velocities. Low-temperature measurements are being carried out on metals, intermetallic compounds, and inorganic salts. Thus far, special emphasis has been given to studies of hexagonal close-packed metals, some of which are highly anisotropic.

In connection with calculations based on lattice-dynamical models, it is vital to have precise heat capacity data in the region below 20°K. Calorimetric work is being carried out in the region 1°K-20°K with liquid helium and liquid hydrogen used as refrigerants. This region is also of particular interest for studying magnetic transitions, electronic specific heats, the nature of surface contributions to  $C_v$  for high-area solids, and anisotropic lattice effects.

C. W. Garland

## A. A SIMPLE TRANSISTOR-OPERATED OVEN-TEMPERATURE REGULATOR

While we were constructing apparatus with which to observe soft X-ray emission from an atomic beam (reported in Quarterly Progress Report No. 53, pp. 13-19) the need arose for an electronic circuit to regulate the temperature of a small oven that dissipates approximately 100 watts and operates in the neighborhood of  $1000^{\circ}\text{C}$ . Many circuits for similar applications have been described by several authors, but the advent of the power transistor, working at low voltage and high current, seemed to offer new possibilities for this purpose. A particularly simple circuit has been developed and preliminary tests indicate that a high degree of regulation is attainable. The circuit is shown in Fig. III-1.

The heating element of the furnace is designated  $R_L$ . The transistors  $T_1$ ,  $T_2$ , and  $T_3$  are operated without the use of dc bias elements and, as a consequence, their operating points vary with the instantaneous value of the supply voltage. The circuit operation may be described briefly as follows.

First, consider the negative half-cycle of input voltage. Over this half-cycle the transistors are biased negatively with respect to their emitters – the correct polarity

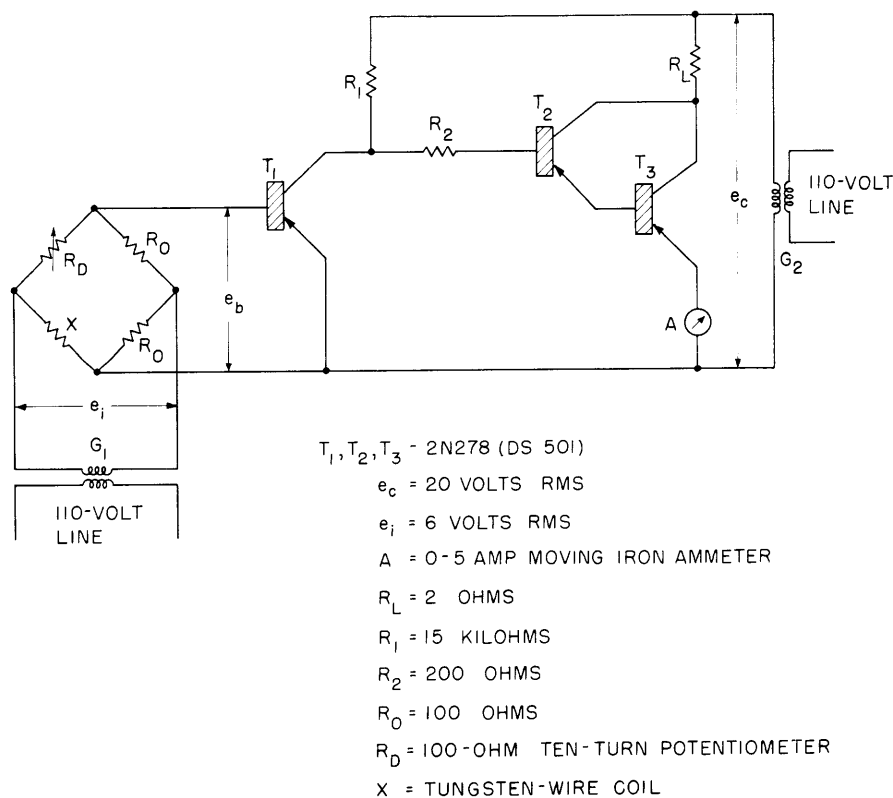


Fig. III-1. Circuit diagram for the oven-temperature regulator.

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for p-n-p transistor action – so that the combination  $T_2$  and  $T_3$  supplies a current to the furnace whose magnitude is dependent upon the output signal from the amplifier stage  $T_1$ .  $T_2$  is an emitter follower that serves to reduce the base current from the output transistor  $T_3$ . The input signal to  $T_1$  is derived from a Wheatstone bridge in which one arm is a temperature-sensitive resistance element,  $X$ , mounted in the furnace.  $R_D$  is preset to the resistance to be attained by  $X$  at the desired operating temperature. It is necessary to choose the phase of the bridge supply,  $e_i$ , so that when the furnace is too cold the off-balance signal,  $e_b$ , is in antiphase with  $e_c$ , that is, so that over the negative half of the input voltage the transistor  $T_1$  is biased toward cutoff, and hence  $T_3$  is biased to pass a large current. When the furnace becomes too hot, the signal from the bridge changes phase by  $180^\circ$ , so that  $T_1$  draws an increasingly larger current and biases  $T_3$  toward cutoff. As a consequence of this regulating action, the circuit maintains the furnace at a temperature that is such that the resistance of  $X$  is approximately the same as that of  $R_D$ .

Over the positive half-cycle of input voltage the collector-base junctions in all three transistors become biased in the direction of easy current flow. Nevertheless,  $T_3$  draws practically no current through the furnace because in series with its base is the high resistance presented by the emitter-base junction of  $T_2$  biased in the direction of hard conduction. Transistors  $T_1$  and  $T_2$  do, however, pass a current limited by the furnace resistance, the bridge resistance, and  $R_2$ . The resistor  $R_2$  serves to limit this back current to a value that is not harmful to the transistor junctions.

Final tests will be made presently with an improved oven, and we intend to submit full details of the regulator and its performance for publication to the Review of Scientific Instruments.

E. R. Pike, J. F. Cochran

### B. SOFT X-RAY DETECTION BY MEANS OF Be-Cu PHOTOMULTIPLIERS

Be-Cu photomultipliers have been used for several years for the detection of spectra in soft X-ray vacuum spectrographs. As such multipliers cannot have envelopes, their treatment before evacuation of the spectrograph is of the utmost importance. In order to gain a better understanding of the operation of these multipliers a series of tests was carried out.

A 16-stage Be-Cu unit, such as is used by the Atomic Beams Laboratory and the Soft X-Ray Laboratory of the Research Laboratory of Electronics, was assembled in a demountable vacuum system containing a low-energy X-ray source.

Photomultipliers incorporate, of course, two basic physical phenomena, the secondary electron effect, and the photoelectric effect. The reproducibility and permissible exposure-to-air times of the Be-Cu electron multiplier have been thoroughly investigated by J. S. Allen. Therefore only a few trial runs were necessary in our experiment

to check the characteristics depending on the secondary electron effect. To obtain the best results, we found it desirable to operate at approximately 400 volts per stage, instead of at 270 volts per stage used in the R.L.E. spectrograph. This operation was achieved with the same power supply as the one used for the spectrograph's multiplier, by simply shorting the last four plates and using them as a single collector. By operating on the comparatively flat peak of the secondary yield curve, a gain higher and more stable than that achievable with the 16 stages at 270 volts per stage was attained. Incidentally, the greatly increased area of the collector over the hairpin-shaped wire of the original Allen design was itself a contributing factor in the increase of gain.

For satisfactory counting rates, the aluminum target was held at +380 volts with respect to the front plate of the 100-volt electron gun. The anode current was maintained at 8 ma during measurements of the photoelectric yield. All readings given below are the counting rates for an arbitrary standard setting of the amplifier. All readings of the yield are therefore relative; no attempt was made to obtain absolute yield determinations. Instrumental gain fluctuations were approximately  $\pm 5$  per cent during an individual run, and approximately  $\pm 50$  per cent from one run to the next (which includes changes in gain of the multiplying section caused by variable times of exposure to air).

The tests carried out can be considered in two parts: (i) Those in which a particular sample was subjected to pressure or temperature variation during the yield determination. (ii) Those in which an attempt was made to reproduce identical operating conditions, but in which different metals were used for the first plate, in order to look for a more suitable electrode material than Be-Cu.

We shall now discuss the first set of results. The yield variation of Be-Cu samples with temperature was extremely great but, aside from the general trend of approximately 10 counts per second at 200°C to approximately 5000 counts per second at room temperature, was not accurately reproducible. Yields from well-cleaned OFHC copper specimens were much more consistent, especially after these specimens had been cleaned by rf vacuum firing in their position at one end of the multiplier. Stability depended markedly on the pressure. At pressures higher than  $1.5 \times 10^{-5}$  mm Hg, curves of types (a) and (b), shown in Fig. III-2, were obtained. In case (b) the equipment cooled off at approximately half the rate for case (a) because of the bakeout heating used in the (b) run. At pressures below  $1.5 \times 10^{-5}$  mm Hg, the yield was much lower, and became very unstable as the specimen approached room temperature. See curves (c) and (d) in Fig. III-2.

The explanation offered for the form of these curves is as follows. Immediately after firing, the specimen has a very nearly clean surface. At pressures above  $1.5 \times 10^{-5}$  mm Hg it soon acquires stable layers of adsorbed gas. The layers form at characteristic temperatures for a given pressure. Gases have an extremely high absorption coefficient for radiation in the soft X-ray region. The photon absorption results in the

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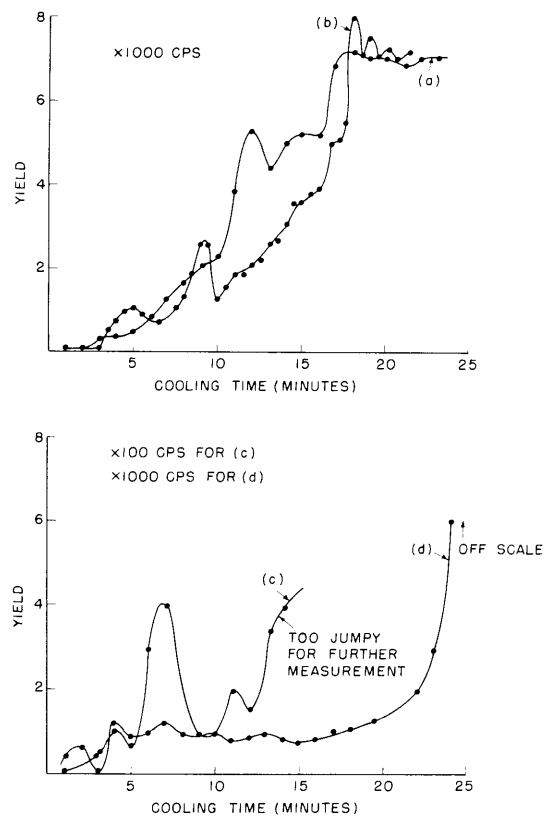


Fig. III-2. Typical yield curves.

ejection of L-shell electrons in the cases of oxygen and nitrogen. Hence, as the temperature of the specimen drops after firing and the surface adsorbs layers of gas, the yield of photoelectrons from the surface concentration of gas increases. At pressures below  $1.5 \times 10^{-5}$  mm Hg, the first layer of adsorbed gas forms in the same way as before. At the lower pressure, however, the other layers do not seem to adhere so well, and the electric field between the multiplier stage causes instability to arise.

In the second set of results, Be-Cu, Cu, oxidized Be-Cu, stainless steel, Al, and Ta were compared immediately after firing, and after they had acquired a gas layer. Stainless steel containing Fe and Cr gave a yield of approximately 200 counts per second, immediately after firing, as compared with 25-50 counts per second for all other specimens. The higher yield for stainless steel may be ascribed to  $M_{I, II, III}$ -level electrons from the Fe and Cr; Be, Cu, and Al M-levels fall outside our energy range. The most important result from the second part of the experiment was that none of the specimens gave a volume photoelectric yield of the same order as, or greater than, that of surface adsorbed gas. Furthermore, of the metals tested, pure Cu acquired the most satisfactory surface gas layer for our purposes.

To summarize, we can say that as the yield comes almost entirely from the gas

layer, the important thing in treating a photosensitive surface for soft X-ray detection is to maintain similarity of treatment of the surface after cleaning, from one run to the next, in order to obtain consistent counting rates. There is no evidence from this research that higher yields can be obtained by using metals other than Cu, or by oxidizing their surfaces. The methods of this experiment offer possibilities for further study of the formation of gas layers on metal surfaces.

K. R. Dawber

### C. ELASTIC CONSTANTS OF CADMIUM SINGLE CRYSTALS

The five independent elastic constants of cadmium have been measured from 4.2°K to 300°K by a 10-mc ultrasonic pulse technique. Three directions of propagation were used (parallel to the c-axis, perpendicular to the c-axis, and a direction at 22° to the c-axis), and seven wave velocities were measured. The results are shown in Figs. III-3 and III-4. The quantities  $\rho U^2$  for quasi-longitudinal and pure transverse waves in the 22° direction were used as checks. The smooth curves shown for these two are calculated from the data on the other five waves. Figure III-5 gives the temperature dependence for  $c_{12}$  and  $c_{13}$ ; these quantities are not obtained directly from a single velocity measurement. The values at 0°K, obtained by extrapolation, are:  $c_{11} = 13.08$ ,  $c_{33} = 5.737$ ,  $c_{44} = 2.449$ ,  $c_{66} = 4.516$ ,  $c_{13} = 4.145$ , all in units of  $10^{11}$  dynes/cm<sup>2</sup>. The relation  $c_{66} = (c_{11} - c_{12})/2$  gives  $c_{12} = 4.05$ .

A study of the room-temperature velocity as a function of sample path length over

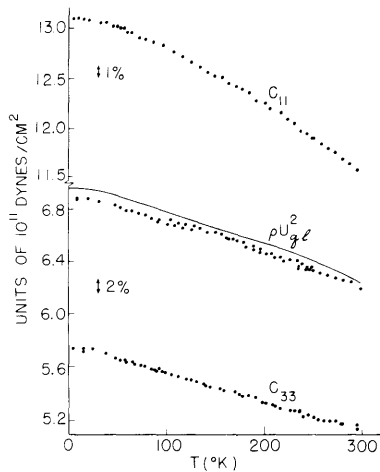


Fig. III-3. Adiabatic elastic constants  $c_{11}$ ,  $c_{33}$ , and  $\rho U^2$  quasi-longitudinal.

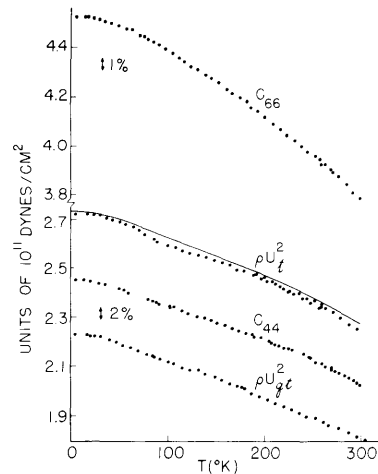


Fig. III-4. Adiabatic elastic constants  $c_{44}$ ,  $c_{66}$ , and  $\rho U^2$  transverse,  $\rho U^2$  quasi-transverse.

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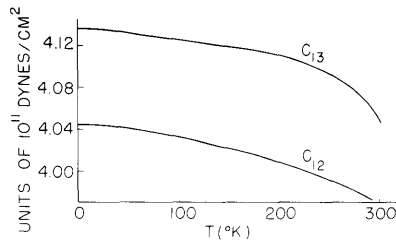


Fig. III-5. Adiabatic elastic constants  $c_{12}$  and  $c_{13}$ .

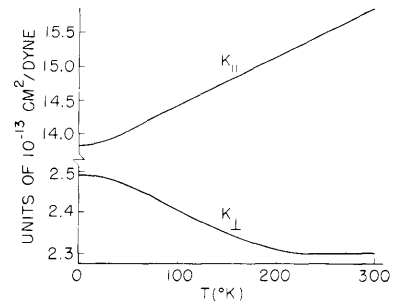


Fig. III-6. Adiabatic compressibilities  $K_{||}$  and  $K_{\perp}$  for cadmium.

the range 1.25 cm-7 cm gave a seal correction of +0.04  $\mu$ sec for longitudinal waves, and +0.045  $\mu$ sec for transverse waves. These seal corrections, which were applied to all data points, cause changes of less than 0.5 per cent. Corrections were also made for thermal expansion changes in the path length.

The elastic properties of cadmium are quite similar to those of zinc (1); that is, both are very anisotropic. At 0°K for cadmium,  $c_{66}/c_{44} = 1.84$  and  $c_{11}/c_{33} = 2.28$ . Also there is an interesting temperature dependence for the adiabatic compressibility parallel to the c-axis ( $K_{||}$ ) and perpendicular to the c-axis ( $K_{\perp}$ ) as shown in Fig. III-6.

Work is in progress on an atomic force model for both zinc and cadmium; such a model must be more complex than the central force model used for magnesium (2).

J. Silverman, C. W. Garland

#### References

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2. C. W. Garland and L. J. Slutsky, J. Chem. Phys. 26, 787 (1957); 28, 331 (1958).

#### D. RECOMBINATION OF ELECTRONS AND DONORS IN GERMANIUM

We have tried to determine quantitatively the characteristics of the "phonon drag" described in Quarterly Progress Report No. 53, pages 27-33. In that report the signal that was detected was associated with the unbalance of a hybrid Tee. The cavity was mounted (see Fig. III-7) on one of the symmetrical arms (No. 2), and on the other symmetrical arm (No. 1) there was a load of such a value that at equilibrium none of the signal fed into the H-arm would come out through the E-arm. When there was a change in the Q of the cavity, an unbalanced signal would appear from the E-arm. This signal, however, was not directly proportional to the change in the properties of the cavity. To

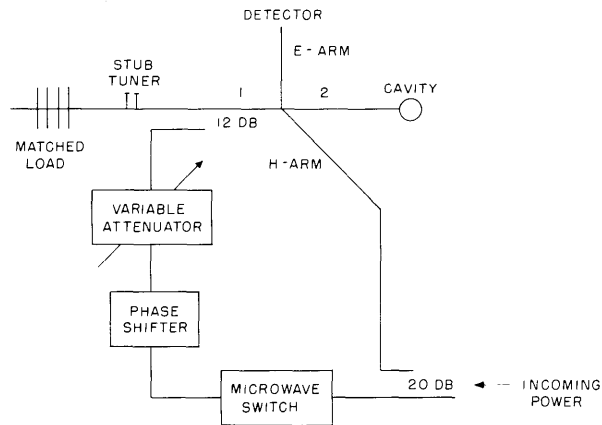


Fig. III-7. Block diagram of apparatus for measuring very small changes of  $Q$  of the microwave cavity in which the sample is mounted.

avoid this difficulty, a new null method was devised.

The circuit was modified as shown in Fig. III-7. We intended to rebalance the Tee by introducing in arm No. 1 a signal equal in magnitude and phase to that reflected by the cavity because of changes in its properties. If the change of the  $Q$  of the cavity takes place within a short time, a short pulse is injected in arm No. 1. This short pulse must have phase coherence with that applied to the H-arm of the measuring Tee so that a null can be achieved. This is done by diverting some power from the stabilized klystron that is used for measuring purposes, and by using a fast microwave switch (Fig. III-8). If only the  $Q$  of the cavity changes while its frequency remains constant, no change of phase of the injected signal is needed, once the correct phase is obtained; only the magnitude of the injected signal has to be adjusted to compensate for changes in  $Q$ . If only the resonant frequency of the cavity changes, a change in the frequency of the incoming power rebalances the bridge (1).

The microwave switch is made with the help of another hybrid Tee whose symmetrical arms carry a variable load and a microwave crystal (1N21), respectively.

At first, the Tee is balanced so that none of the power fed into the H-arm flows out of the E-arm. If there is a ground return, one can observe a rectified microwave current through the crystal. If this current can be observed by means of an ammeter in series with the crystal, the balance of the Tee depends on the microwave power level; otherwise, it is independent of it. When a small dc pulse (approximately 0.5 volt) is applied to the crystal, its impedance is changed so that some power comes out of the E-arm of the Tee. In this way, it is easy to obtain fast

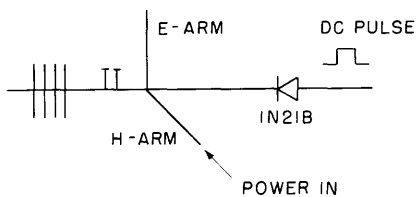


Fig. III-8. Microwave switch.



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pulses (rise time, approximately  $10^{-7}$  sec) with a signal-to-noise ratio of at least 40 db. The phase shifter and attenuator that follow the switch determine the phase and amplitude of the injected signal.

To obtain best results, it is useful to "pretreat" the crystal in order to increase its change of resistance with the applied voltage pulse. This was done by passing a current that is an order of magnitude larger than the maximum specified by the manufacturer. This current was applied for a few seconds. When it was necessary, the treatment was repeated.

By suitable delays of the dc pulse, the instant of injection of the microwave pulse could be varied in time. The results of the previous report were partially confirmed. The "phonon" component was seen, but it was also found that the effect of nonohmic contacts at either A or B could be such as to obscure the results completely. When phonons that produce free electrons in the portion of the sample in the cavity diffuse from the region AB of the sample into the portion BC that is mounted inside the cavity, the drift velocity of the phonons is parallel to that of the electrons. A dc field applied to the portion BC of the sample does not affect the signal seen out of the E-arm of the measuring Tee. Furthermore, in view of the direction of the drift velocity of the electrons in AB, when A is positive with respect to B, the signal observed out of the E-arm of the Tee is smaller than it is when the polarity is reversed. If we want to observe this effect, the portion AB of the crystal must be rather long (approximately 1 cm). The reason is that the majority of the phonons that are detected by means of their secondary effects in the portion BC of the sample is produced in the neighborhood of B. If the region AB is small, the electric lines of force in the neighborhood of B will diverge

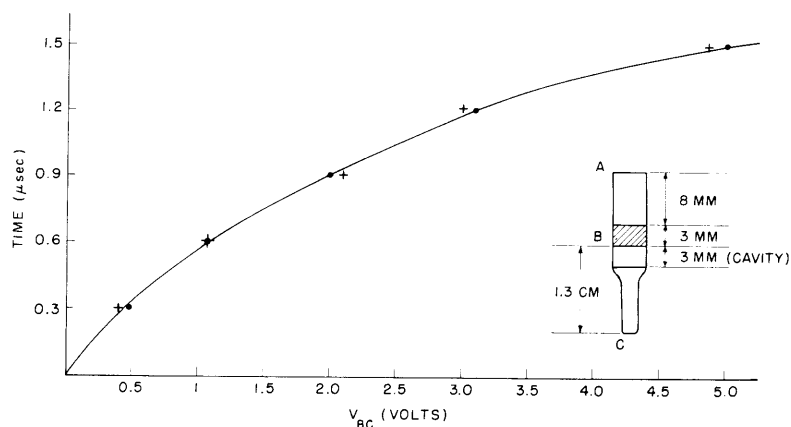


Fig. III-9. Time increase for reaching an arbitrary unbalance as a function of the dc voltage applied across BC. C is positive with respect to B (which is grounded); pulse applied to AB is 15 volts; A is positive with respect to B. (Sample, BTL-7.)

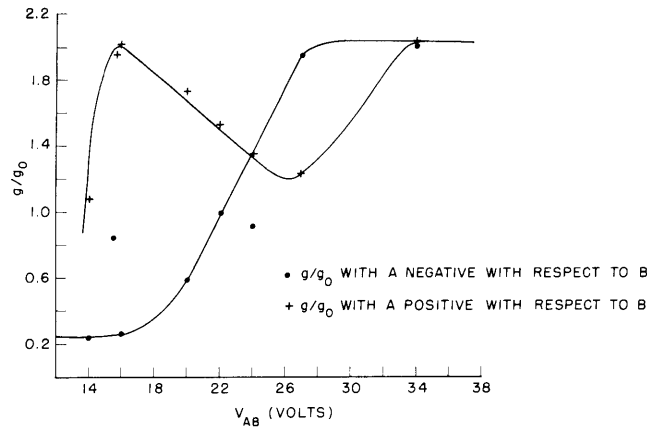


Fig. III-10. Changes of the cavity admittance as a function of the voltage applied to AB. There is no voltage across BC. Notice the inversion of the relative magnitude of  $g/g_0$  with increasing voltage.

appreciably, and the phonons emitted in the direction of motion of our accelerated electrons will form a beam that is not well collimated, and thus the electrons are lost at the walls of the sample.

The effect of injecting contacts is easily understood. Assume that either A or B is nonohmic so that the contacts will inject holes. The signal out of the E-arm will then be sensitive to a voltage applied to BC, increasing or decreasing accordingly as B is positive or negative with respect to C (Fig. III-9). If A is the injecting contact, holes will be accelerated from A to B, will overshoot B, and diffuse into the cavity. In such a case, the pulse out of the magic Tee will be larger when A is positive with respect to B, in contrast to prediction when phonons are the dominant effect, and to actual observations. The decay times of the density will also be larger because of the loss of minority carriers whose lifetime is longer than that of majority carriers. Surfaces can play an important role here.

Figure III-10 shows the combined effect of phonons and injection. For low fields injection is important, and the changes in conductivity produced by positive pulses applied to AB are greater than they are when negative pulses are applied to it. The effect is reversed at higher fields when phonons become predominant. Finally, at still higher fields, bulk heating becomes the predominant phenomenon, and both conductivities become equal.

The curves shown in Fig. III-10 are not reproducible, and their shapes depend very much on the contacts.

Pure phonon effects were obtained with samples of the BTL series when the room temperature and liquid-air temperature resistance of part AB of the sample appeared to be ohmic, at least within one part in a thousand. The difficulty of obtaining ohmic

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contacts with tin was the principal source of trouble. The microwave null method is repeatable and sufficiently accurate to enable the determination of very small changes in conductivity.

An example of a similar procedure for measuring very small frequency shifts encountered in the measurement of Hall effect with microwaves when  $\omega\mathcal{L} < 1$  has been described by Weber (1). This furnishes a good example of the sensitivity of the method.

G. Ascarelli

#### References

1. R. Weber, Measurement of resonant-frequency shifts of microwave cavity caused by the Hall effect in semiconductor materials, Quarterly Progress Report No. 53, Research Laboratory of Electronics, M.I.T., April 15, 1959, pp. 25-27.