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Memorandum of Project MICHIGAN

EXCITON AND MAGNETO-OPTICAL EFFECT IN STRAINED AND UNSTRAINED GERMANIUM

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Robert L. Hess
Technical Director
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EXCITON AND MAGNETO-OPTICAL EFFECT IN STRAINED AND UNSTRAINED GERMANIUM[1]

ABSTRACT

Measurements have been made of the direct-transition magneto-optical effect in strained and unstrained germanium at 77°K. The results indicate that the absorption peaks correspond to transitions to exciton levels associated with each Landau level in qualitative agreement with the theoretical calculations of Loudon (Reference 12) and of Howard and Hasegawa (Reference 13).

1 INTRODUCTION

As part of its program in basic research, Project MICHIGAN is carrying out experimental and theoretical investigations on the optical properties of semiconducting materials. Included in such materials is the monatomic semiconductor germanium, the subject of this memorandum.

The direct-transition exciton and magneto-optical effect in germanium have been investigated in several recent experiments (References 1 and 2). The samples used were glued to a backing, usually of glass. However, it was subsequently pointed out by Macfarlane, MacLean, Quarrington, and Roberts (Reference 3, hereinafter referred to as MMQR) that samples mounted in this way become strained when cooled because of the difference in contraction of the sample and the backing. This group made transmission measurements without a magnetic field at 77°K on the same germanium sample, first mounted free of any backing, and then glued to a glass substrate. They observed for the backed sample that the exciton absorption, E_x, shifted to

[1]The authors wish to acknowledge their indebtedness to Dr. F. Blatt, Dr. R. R. Goodman, and Dr. G. Weinreich for their helpful discussions and advice, Dr. R. W. Terhune, Dr. C. W. Peters, and Mr. P. D. Maker for their assistance with the instrumentation, and G. Weinreich of the Bell Telephone Laboratories, Dr. R. Petritz of the Texas Instruments Incorporated, and Dr. W. C. Dunlap of Raytheon Manufacturing Company for supplying the intrinsic germanium crystals.

higher energies and that a second absorption appeared at an energy greater than E_x. They concluded that backing the samples on glass produced a compressional strain which distorted the energy band structure. As a further check of this theory, fused silica was used as the backing material to produce a tension on the sample when cooled. The result was a shift of E_x towards lower energy as expected. Edwards and Lazazzera (Reference 4) have also reported transmission measurements without a magnetic field for free-mounted germanium at 77°K with approximately the same results as found by MMQR. The effect of the strain on the energy band structure has been examined by Kleiner and Roth (Reference 5) to obtain information about the deformation potential for the band edges. They concluded that the backing produces a shear strain that splits the valence band into two edges and also increases the energy gap. For the backed samples they associate an exciton level with each valence band edge and thus explain the second absorption. The shift in E_x is associated with the increased energy gap.

These investigations make it clear that a sample must be mounted strain-free for an experiment to represent accurately the intrinsic characteristics of the specimen and not some property of the environment. The problem of a strain-free sample will arise, however, only for the case of the direct-transition magneto-optical effect. Here the samples must be thin (about 5 microns) for the effect to be observed. For the indirect transition, since the samples are several millimeters thick and self-supporting, the question of strain will not arise.

The purpose of this paper is to report the results of direct-transition magneto-optical-effect measurements on strained and unstrained germanium samples at 77°K. An interpretation of these data is made in terms of transitions to exciton levels associated with each Landau level. The instrumentation and experimental technique are briefly described in Section 2. In Sections 3 and 4 are given the experimental results and analysis for the unstrained and strained specimens, respectively.

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EXPERIMENTAL TECHNIQUE

The germanium samples were made from either [100] or [110] wafers cut from intrinsic single crystals. The thin specimens were made by careful grinding and polishing techniques, and a special attempt was made to minimize the adverse surface effects of the grinding. Each wafer was cut to a square of about 2 cm on a side and a thickness of about 1 mm. The trimmings

were then used as material to help maintain parallel surfaces and were termed blocking material. The 2-cm square was glued at the center of a 3-inch-diameter optical flat with an acetone-soluble thermosetting glue.[2] The trimmings were glued adjacent to this piece in approximately the same position as before they were cut off. In addition, three or four blocks of the same thickness were glued equally spaced at the edge of the flat. To help insure parallel surfaces these eight or nine pieces were glued at the same time using a thin layer of glue and held under pressure until the glue hardened. The first surface was then ground lightly, using 3200-grit carborundum on an optical flat to remove any saw marks. A scratch-free, highly reflecting surface was then obtained by polishing with Linde A[3] on a moist Metcloth[4] stretched over an optical flat. About 25 microns of material were removed by the polishing to reduce any layer damaged by the grinding. The resulting surface was optically flat to less than a fringe over approximately the center 90% of the sample. For this first surface, no attempt was made to reduce the thickness of the germanium wafer. The wafer and blocks were then removed, either by gently heating, or by dissolving the glue in a solvent; and each piece was turned over and glued with the polished surface towards the optical flat. Sometimes a section of a microscope slide can be used between the germanium pieces and the flat. The thickness of the wafer was then reduced to about 100 microns, using 400- or 600-grit carborundum. Periodic checks were made to insure that the wafer surfaces remained parallel. The wafer thickness was next reduced to about 25 to 30 microns using 3200 grit. The final 20 to 25 microns were removed using the Linde A polishing compound on Metcloth as before. Once the second surface has been polished the sample thickness can be checked from the interference fringes produced by multiple internal reflections in the transparent spectral region of the germanium. By careful polishing and frequent checking for parallel surfaces, fringes usually can be obtained for samples about 15 to 20 microns thick. The polishing was continued until the samples were about 5 microns thick. The wafer was then masked and cut, using a dental dust blast, into samples about 4 mm x 12 mm, along definite crystallographic directions. The samples were floated from the microscope slides by dissolving the glue in warm dichlorethylene.

The crucial part of the direct-transition measurements was obtaining a sample that is strain-free at all temperatures. To help insure this the samples were mounted free from any backing in the holders of the type shown in Figure 1. The samples were placed between two

[2]NU-C-70, Hugh Courtright & Co., 7600 Greenwood Ave., Chicago 19, Ill.

[3]Linde A, Linde Company, Div. of Union Carbide Corp., New York, N. Y.

[4]Metcloth, Buehler, Ltd., Evanston, Ill.

microscope cover-glass slides and this sandwich was then slipped under the spring clips of the sample holder, as shown at the bottom of Figure 1. This arrangement permitted easy handling of the delicate samples and also permitted the free movement of the sample with respect to the cover glass when the holder was cooled. To insure that the sample was at the temperatures of the coolant, the entire holder, with sample in place, was immersed in the coolant. The sample end of the metal dewar (Reference 6) used for these measurements is shown in Figure 2. The nose piece is a Kovar metal-to-glass seal with the infrared beam passing through the glass. The tips of the pole pieces are part of the Dewar wall and magnetic fields up to 25 kilogauss are possible.

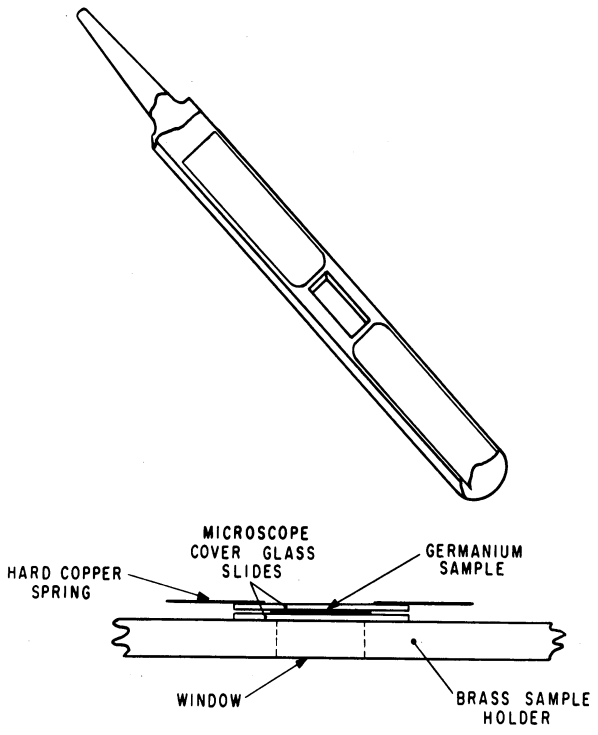

FIGURE 1. SAMPLE HOLDER FOR FREE MOUNTED SAMPLES

A high-resolution Fastie-Ebert grating spectrometer (References 7 and 8) with 3-meter focal-length optics, single pass, and a 127 x 203-mm (300 lines/mm) Bausch and Lomb grating operated in second order was used for these measurements. The detector was an Eastman lead sulfide detector cooled to 77°K. The entire optical path was evacuated to less than 50-microns pressure with the exception of a short distance between the source and entrance to the evacuated spectrometer tank. This path was filled with dry nitrogen. The purpose of the

evacuation and dry nitrogen was to remove all water vapor and CO_2 from the optical path. A combination of an ADP (ammonium dihydrogen phosphate) crystal and a coated silicon crystal were used in the fore optics to remove the unwanted orders of radiation from the incoming beam. A Polaroid type-HR polarizer was also used in the fore optics for the polarized-light experiments. The sample was placed in the aft optics to avoid possible heating by the source. The resolving power of the instrument was not used to its fullest because of the broad nature of the absorption lines. For a resolving power of about 13,000 the spectral slit width was about 1/15 the width of the narrowest line and gave a signal-to-noise ratio of about 100 with the sample in the beam.

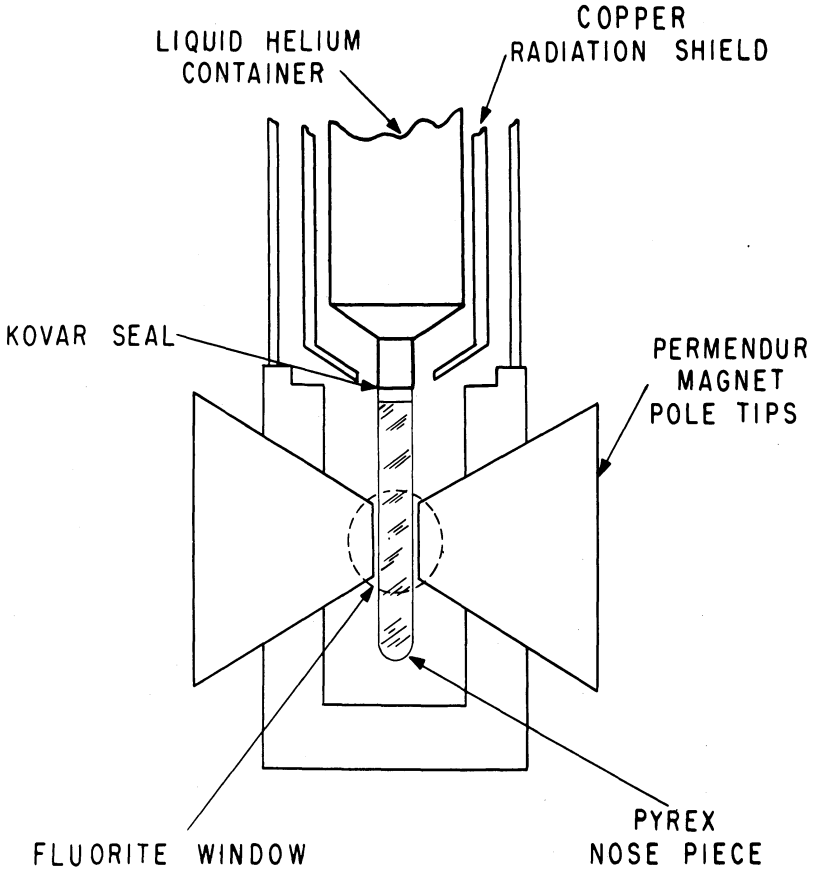

FIGURE 2. SAMPLE END OF LIQUID HELIUM METAL DEWAR SHOWING GLASS NOSE-PIECE AND MAGNET POLE TIPS

3
UNSTRAINED SAMPLE

In our laboratory we have repeated the zero-field exciton transmission measurements of MMQR at 77°K and the results for an unstrained sample are shown as curve TI-91, Figure 3. The exciton absorption line, $E_x = 0.88171 \pm 0.00002$ ev, represents the excitation of an electron from the valence band to the ground state of the direct transition exciton. The spectral slit width for this measurement was 7×10^{-5} ev. For comparison, curve RRE-2 is the data of MMQR for a free-mounted sample at 77°K. They give the position of the exciton as $E_x = 0.8820 \pm 0.0001$ ev. It should be noted for both curves that there is just the single absorption, E_x, followed by the monotonically decreasing transmission; no other absorptions are present.[5] Elliott (Reference 9) has investigated theoretically the direct transition between spherical energy bands including the coulombic effect between the hole and electron. He showed that one should expect a sharp absorption peak for the exciton followed by a region of continuous absorption beginning at the energy gap, E_o, of the form

$$\frac{e^x}{\sinh x}$$

where

$$x = \pi \left(\frac{E_o - E_x}{h\nu - E_o} \right)^{1/2} \qquad (1)$$

By fitting the experimental data of Figure 3 with the theoretical curve, Equation 1, the direct transition energy gap, E_o, can be evaluated. MMQR have done this and find $E_o = 0.8832 \pm 0.0001$ ev. For sample TI-91 the direct transition energy gap was estimated to be $E_o = 0.8834 \pm 0.0001$ ev.

With the application of a magnetic field the continuous absorption for $h\nu > E_o$ becomes a series of lines or oscillations, and is called the magneto-absorption effect. The results of magneto-absorption measurements on the unstrained sample, TI-91 are shown in Figure 4. Here is plotted the energy of each absorption for various values of applied magnetic field. From Figure 4 several points should be noted. First, for zero field only the single absorption, E_x, is observed as was shown in Figure 3. Second, the lowest absorption behaves quadratically

[5]For sample RRE-2 the deviation in the region of 0.8855 ev might be due to a slightly strained sample (see Figure 6) and would thus account for the difference in the E_x value.

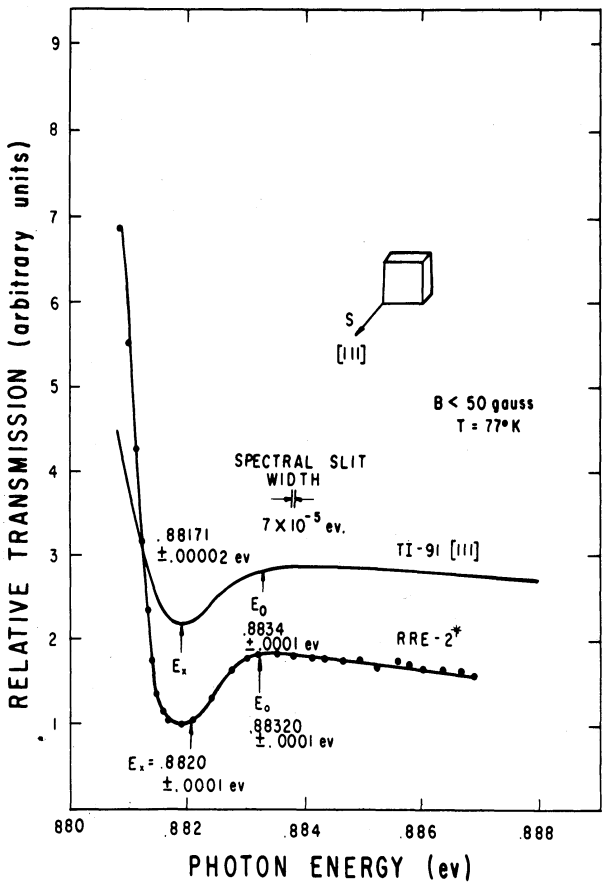

FIGURE 3. ZERO-FIELD EXCITON TRANSMISSION FOR STRAIN-FREE GERMANIUM AT 77°K. See Reference 3.

with magnetic field and can be traced to zero field. The second level is also quadratic in B and appears to originate from the region of E_x, but the absorption peaks are not well defined for fields less than 8 kilogauss. The third point to note is that the higher levels appear to be linear with B. However, if the same change of curvature in going from the lowest to the second level is extended to the higher levels, it is reasonable to believe that any curvature above the second level would not be discernible. If we assume that these higher levels depend linearly on magnetic field, i.e., assume the absorption lines correspond to transitions between Landau levels, then the energy separation between two levels, n and n', will be of the form (References 10 and 11):

$$E_{nn'} = E_o + \left(n + \frac{1}{2}\right)\hbar\omega_{c1} + \left(n' + \frac{1}{2}\right)\hbar\omega_{c2} \qquad (2)$$

with the condition

$$\Delta n = n' - n = 0, -2$$

where n and n' refer to the Landau quantum numbers, and $\omega_{c1} = eB/m_1^* c$ is the cyclotron frequency for band 1, and similarly for band 2. For zero magnetic field, $E_{nn'} = E_o$; thus the extension of the higher levels in Figure 4 to zero field would give the direct transition energy gap $E_o' = 0.88047 \pm 0.00047$ ev, a value less than the exciton energy, E_x. But it is not possible for the energy of the bound electron-hole of the exciton to have a higher energy than the free carriers in the conduction bands. In other words, if the higher levels corresponded to Landau transitions the exciton would have a negative binding energy which is contradictory to the usual definition of an exciton.

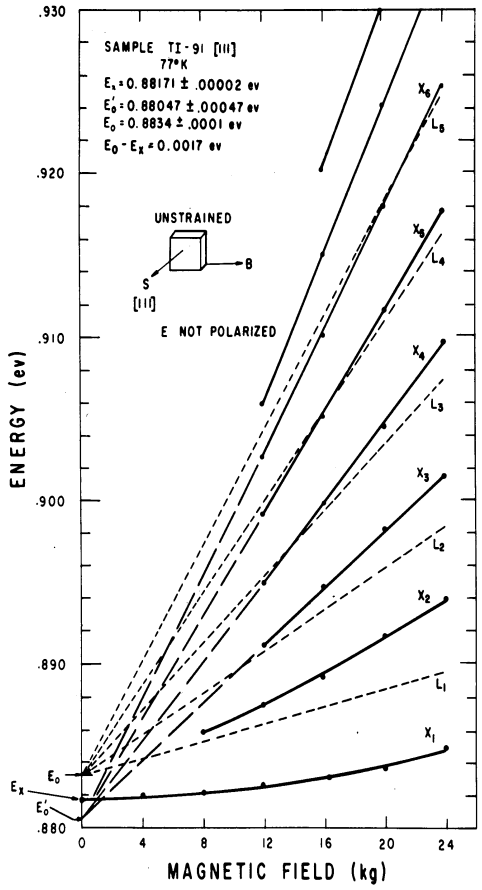

FIGURE 4. MAGNETO-ABSORPTION SPECTRUM FOR STRAIN-FREE GERMANIUM AT 77°K. Levels E_{X_i} correspond to exciton transitions and E_{L_i} correspond to Landau transitions determined from Equation 2.

Loudon (Reference 12) has extended the theoretical calculations of Elliott (Reference 9) to the case of optical absorption in the presence of a magnetic field taking into account the effect of the exciton. He concludes that there is an exciton level associated with each Landau level in the conduction band, and that the most important absorption peaks in the magneto-absorption spectrum correspond to transitions to these exciton levels, with the Landau absorption an insignificant shoulder. This same conclusion was arrived at independently by Howard and Hasegawa (Reference 13), who examined the magneto-absorption spectrum of impurity states. Thus, the conclusion for Figure 4 is that the higher levels, as well as the two lowest levels, correspond to exciton transitions with a nonlinear field dependence, and originate from the region of E_x. The model of this energy band structure is illustrated in Figure 5. For zero magnetic field the exciton absorption, $E_x(0)$, is the strong absorption peak followed by the smooth direct transition absorptions for $h\nu \geq E_o$. For $B \neq 0$ the strong absorptions of the magneto-absorption spectrum are for transitions to the exciton levels, $E_{x_i}(B)$, $(i = 1, 2, \ldots)$, associated with each Landau level $E_{L_i}(B)$. The position of the Landau levels can be estimated using Equation 2 and the cyclotron resonance effective masses, $m_v^* = 0.33m_o$ (heavy hole) and $m_c^* = 0.034m_o$. They are shown in Figure 4 by the dashed lines and are labeled L_i $(i = 1, 2, \ldots)$. We have arbitrarily considered only transitions between the heavy holes and the conduction band with $\Delta n = -2$. The difference between the Landau level and the corresponding exciton level represents the exciton binding energy. At zero field the exciton binding energy, $E_{ex_i} = E_{L_i} - E_{x_i}$, is 0.0017 ev and increases quadratically with magnetic field in qualitative agreement with

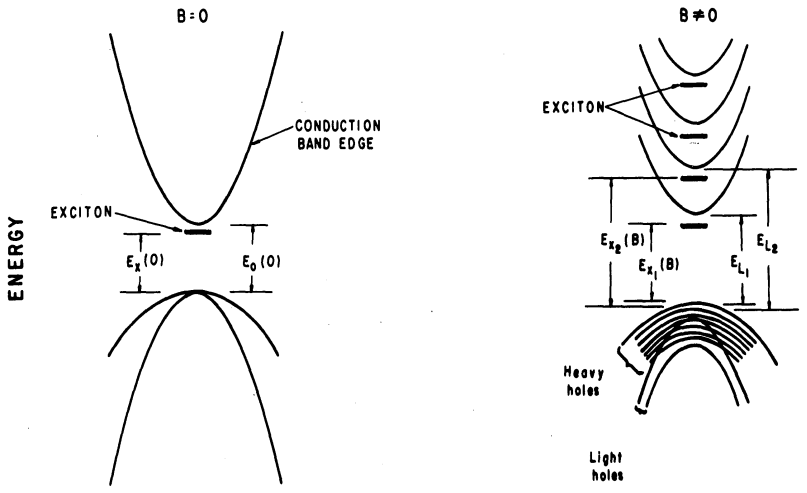

FIGURE 5. ENERGY BAND STRUCTURE FOR STRAIN-FREE GERMANIUM WITH AND WITHOUT A MAGNETIC FIELD

the calculations of Yafet, Keyes, and Adams (Reference 14). For a given magnetic field the E_{ex_i} values increase with the index i. This may be fortuitous, however, because of the manner used in selecting the Landau levels.

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STRAINED SAMPLES

The zero-field exciton transmission is shown in Figure 6 for a few strained samples at 77°K. The curve TI-91 is for the unstrained sample of Figure 3 and is included for comparison. Curve BTL-61 is the transmission of the sample displaying the greatest shift of E_x and presumably greatest strain that we measured. The strain in sample BTL-61 was accidental and was probably produced by localized binding between the sample and the cover-glass slides. Unfortunately, the strain in the samples could not be measured. The curve at the extreme right is that of Zwerdling, Lax, Roth, and Button (Reference 1, hereinafter referred to as ZLRB), and is for a sample glued to a glass substrate. The strain in each of these samples is compressional and is produced by the difference in the contraction of the substrate and the sample when cooled. These curves have been adjusted vertically to avoid confusion. The general

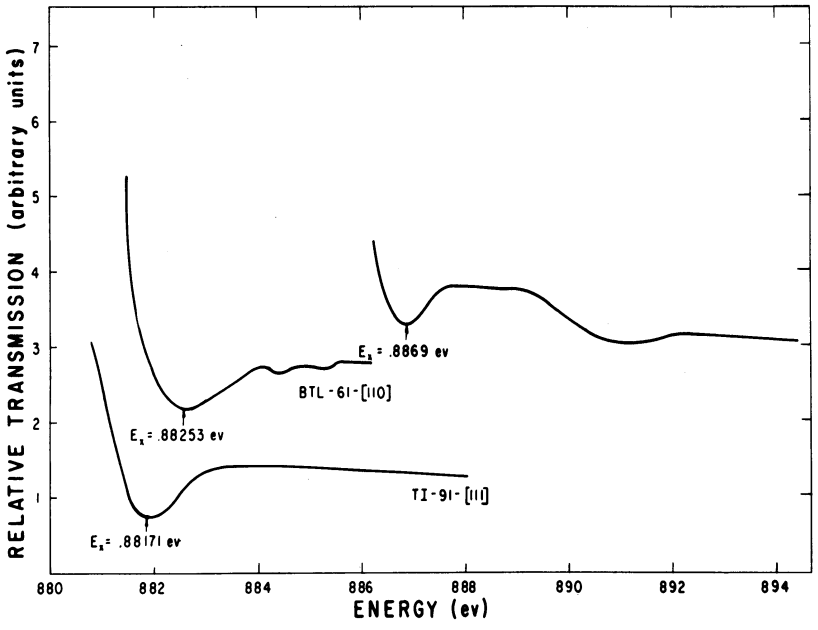

FIGURE 6. ZERO-FIELD EXCITON TRANSMISSION FOR STRAINED GERMANIUM SAMPLES AT 77°K. See Reference 1.

properties of the strained samples as compared to the unstrained sample are the following: (1) the position of the absorption E_x is shifted towards higher energies; and (2) additional absorptions are evident at energies greater than E_x. These effects have been attributed (Reference 5) to the shear strain which splits the valence band edge into two edges and also increases the energy gap.

The magneto-absorption effect has been measured for a few strained samples, and the results are shown in Figures 7 and 8. Both samples have the same general characteristics as for the unstrained sample, Figure 4, with the exception of the shift in energies produced by the strains. The two lowest levels of Figures 7 and 8 behave quadratically with magnetic field, with the lowest level being traced to zero field. The higher levels appear to be linear and extrapolate to a point, the energy of which is less than E_x. It should be noted that sample TI-91A is under a slight tension as demonstrated by the fact that E_x has shifted towards energy lower than for the unstrained sample, Figure 3. Thus it is evident from Figures 7 and 8 that

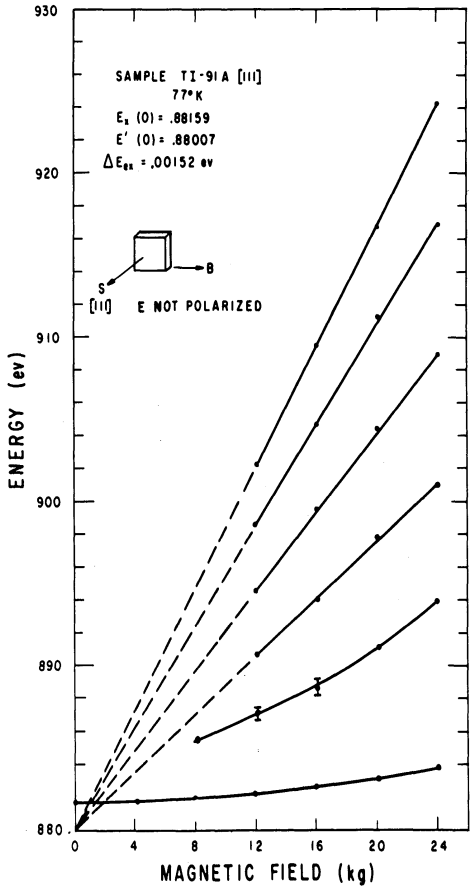

FIGURE 7. MAGNETO-ABSORPTION SPECTRUM FOR A SAMPLE UNDER A SLIGHT COMPRESSIONAL STRAIN

for samples accidentally strained, at least up to a certain amount, the intense transitions are to exciton levels associated with each Landau level the same as for the unstrained sample. For sample BTL-61 the magneto-absorption measurements were made using polarized radiation. Within the limits of the experiment no difference could be detected in the positions of the absorptions for E $\perp$ B and E $\parallel$ B, where E is the electric vector of the polarized beam. The strain was compressional and presumably along a [110] crystallographic direction.

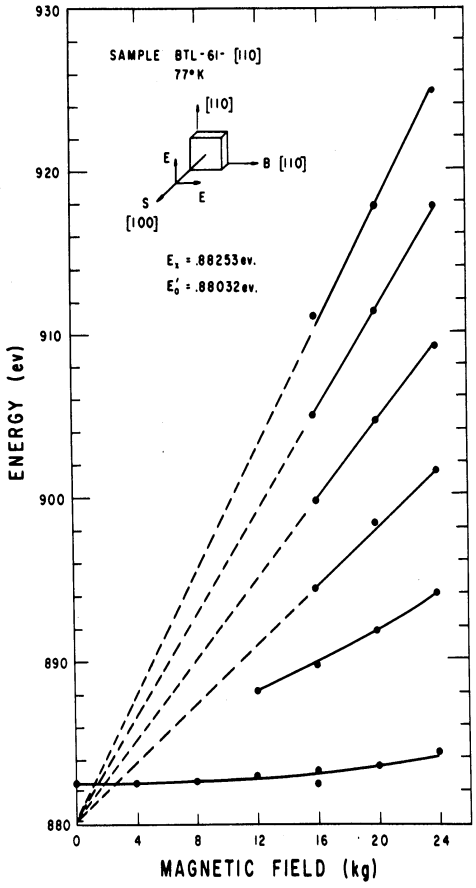

FIGURE 8. MAGNETO-ABSORPTION SPECTRUM FOR A SAMPLE UNDER A SLIGHT TENSIONAL STRAIN

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CONCLUSIONS

Results of the measurements of the direct-transition magneto-optical effect in unstrained and slightly strained samples indicate that the absorption peaks correspond to transitions to

the exciton level associated with each Landau level, and that the Landau absorption is an insignificant shoulder. These experimental results are in qualitative agreement with the theoretical calculations of Loudon and of Howard and Hasegawa. For a sample glued to a glass substrate the strain is relatively large, and the magneto-absorption spectrum becomes rather complicated. An example of this can be seen from the measurements of ZLRB where at $77^{o}K$ at least three zero-field absorptions are observed.

A definitive experiment that would verify that the intense absorptions were due to excitons would be to measure the photoconductivity at $h\nu = E_{x_i}$ $(B \neq 0)$. For a true exciton no photoconductive effect should appear. Although this should verify that the absorption is due to an exciton, it may be a difficult measurement to make because of effects such as impact ionization.

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2. Optics and spectroscopy—Absorption
3. Optics and spectroscopy—Magneto-optic rotation

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