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Hall measurements to 40 kilobars under hydrostatic pressure in a piston-cylinder device

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A technique for Hall measurements under truly hydrostatic pressures to 40 kilobars is described. The method employs the piston-cylinder apparatus with a Teflon cell to contain a suitable fluid. The necessary magnetic field is generated by a coil placed inside the cell. Hall data can be obtained over the full pressure range, at temperatures from 150 to 300 K. Results for an $Al_xGa_{1-x}As$ sample are presented as an example.

INTRODUCTION

Hall effect measurements under high pressure have been done before by several workers. Paul *et al.*[1] have reported Hall measurements on Ge samples up to 10 kilobars hydrostatic pressure using a Cu–Be pressure vessel. Since the pressure vessel was nonmagnetic the magnetic field was supplied by an external source. By immersing the pressure vessel in an ice bath the temperature during the experiment was kept constant at 273 K. Vaisnys and Kirk[2] were able to make measurements at pressures as high as 60 kilobars using the Bridgman anvil technique. The gasket was made out of impregnated epoxy. Silver chloride served as a pressure medium. Tungsten carbide anvils were used and a thin epoxide adhesive layer provided insulation. Two coaxial coils supplied magnetic fields up to 4 kG at the sample location. With this arrangement Vaisnys and Kirk measured the Hall coefficient of Te and Bi as a function of pressure at room temperature. Pitt[3] used essentially the same technique, but had high-speed tool steel anvils hardened to RC69. With the latter he claims to have obtained magnetic fields of 12 kG at the sample and pressures as high as 70 kilobars. Further, it is claimed that the nonhydrostatic components were within 2 kilobars in the pressure range 30 to 60 kilobars, and larger deviations from hydrostaticity occurred at pressures below 25 kilobars. This arrangement was used by Pitt and Lees[4] to measure the Hall coefficient and mobilities in GaAs at room temperature. Vyas *et al.*[5] modified the above anvil technique for low temperatures by circulating liquid nitrogen through copper coils encircling the anvils and were able to obtain Hall data on GaAs at pressures to 50 kilobars in the temperature range 120–300 K.

In all the above-mentioned studies except those reported in Ref. 1 the pressure was not strictly hydrostatic. Semiconductor band structure is extremely sensitive to nonhydrostatic components, and hence it is desirable to make Hall measurements under truly hydrostatic conditions over an extended pressure range—much greater than the 10 kilobars reached in the study quoted in Ref. 1. We report in this paper a technique for performing Hall measurements to 40 kilobars hydrostatic pressure using the piston-cylinder apparatus in conjunction with the Teflon cell technique.[6,7]

I. ARRANGEMENT AND EXPERIMENTAL PROCEDURE

The main problem in using the piston-cylinder device is that the magnetic field has to be generated inside the pressure chamber, since the pressure vessel and other components are mostly of steel which would screen out an externally applied magnetic field. Hence we devised the following arrangement which enables us to perform Hall studies on semiconductors whose mobilities and carrier concentrations are within reasonable limits.

The Teflon cell technique for generating hydrostatic pressures to 50 kilobars has been described in detail.[6,7] For our experiment a cell 30 mm long having an o.d. of 25.4 mm and an i.d. of 19 mm was chosen. To generate a magnetic field we provided a coil inside the cell. The coil has five layers of 0.6 mm thick enameled copper wire, is 16 mm in length, 8 mm i.d., and 16 mm o.d. This leaves a 1 mm gap all around the coil with respect to the cell wall and a space about 8 mm between the bottom of the Teflon cell and the coil. The 8 mm distance is essential to avoid cell bottom-coil contact and consequent nonhydrostatic stress on the coil. The cell with its cap, the lead assembly, and the coil is shown in Fig. 1.

The coil was wound using a suitable former and was impregnated with epoxy under vacuum. We found that unimpregnated coils, when immersed in the fluid medium (isoamyl alcohol) had air trapped in the space between the turns resulting in excessive volume take up during compression. This caused cell rupture at low pressures. With impregnation the problem completely disappeared. The two terminals of the coil were brought to the same end facing the cap of the cell, and then two thinner copper wires were soldered to them. The bent ends were inserted into two blind holes in the cap section to hold the coil in a fixed position as close to the cap as possible. Two nickel wires were threaded through the cap section and were soldered to the thinner copper wires. These two nickel wires served as elec-

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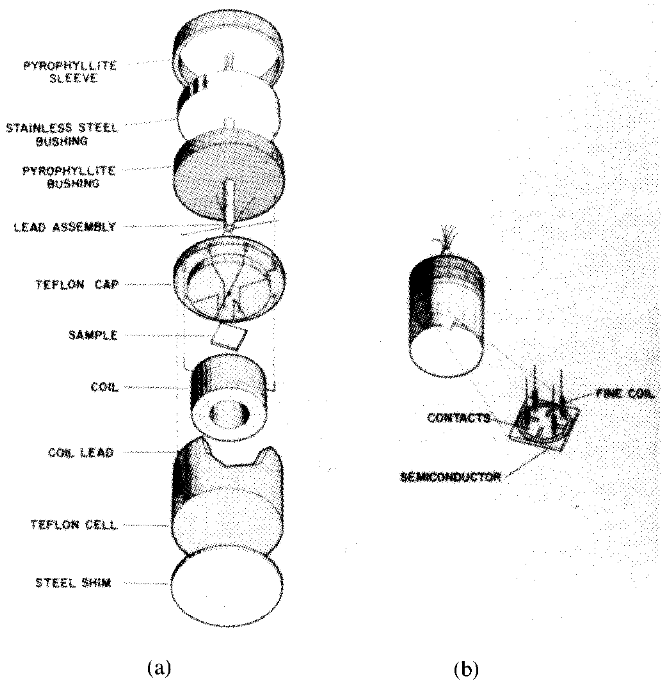

FIG. 1. (a) Schematic diagram of the experimental setup showing the Teflon cell, the copper coil and the cap section with the sample and its leads. Also shown are other details used to bring the lead assembly out of the pressure chamber. (b) The figure on the left shows the position of the sample inside of the Teflon cell. The figure on the right shows details of the sample geometry and its electrical contacts.

trical terminals to the coil. One terminal of the coil was connected to the electrically isolated top end-loading plate of the press through the stainless-steel bushing, and the second terminal was bent along the outer wall of the Teflon cell to contact the main body of the press through the piston end.

The top end-loading plate and the body of the press were connected to the output of a regulated power supply. A magnetic field of 210 G was obtained inside the coil with a current of 3 A. The field was uniform ($\pm 2.5\%$) within the length of 3 mm near the middle section of the coil. A semiconductor sample with four leads and a thermocouple junction were mounted on the Teflon cap as shown in Fig. 1. The sample was located as close to the center of the coil as possible. The leads were brought out of the cell and through the six hole mullite ceramic for outside connection. For measurements below room temperature the pressure chamber was surrounded by thermal insulating material. The pressure chamber was cooled by pouring liquid nitrogen on the top side of the pressure vessel. A 10-mm-thick Transite disk inserted between each of the end loading plates and the vessel provided good thermal isolation of the pressure chamber from the press. In this manner it was possible to reach temperatures down to 150 K. However, during the cooling cycle the pressure could not be kept constant. Hence the following procedure was adopted: the pressure was brought up to a predetermined value, and then the liquid nitrogen was turned on until the temperature dropped to about 150 K. Then the nitrogen supply was cut off, and the warming cycle was started, during which the pressure tended to increase slowly while the temperature increased. Hall data were taken at frequent intervals while maintaining the pressure constant by bleeding the excess piston ram pressure. It is to be noted that freezing of the pressure medium occurs at some temperature during the cooling cycle, depending on the pressure applied at ambient temperature. However, such freezing does not affect the hydrostatic condition, as long as pressure is not applied when the fluid is frozen. It is for this reason we had to take the pressure to a predetermined value at room temperature and then start cooling.

II. RESULTS

With a 25.4-mm-diam pressure chamber we could reach pressures close to 40 kilobars and perform Hall measurements in the temperature range 150–300 K. Hall and resistivity measurements were made on GaAs and $Al_xGa_{1-x}As$ samples ($0 < x < 0.4$) using the above technique. The samples were ultrasonically cut to the shape of the Van der Pauw bridge. Evaporation of Au and Sn and subsequent heating up to 400 °C in hydrogen yielded ohmic contacts to the epitaxially grown semiconductor crystals. Copper coils made from fine wire (0.08 mm in diameter) were bonded to the contacts using In as a bonding material [see Fig. 1(b)]. These coils protected the samples from any strain or fracturing while mounting on the Teflon cap and during pressurization.

Carrier concentrations and mobilities in the samples ranged from 10^{15} to 2×10^{16} cm^{-3} and from 100 to 4000 cm^2 V^{-1} s^{-1} correspondingly. The Hall signal varied from $\sim$0.15 to $\sim$500 mV. Since the applied magnetic field was weak, we compensated the zero magnetic field un-

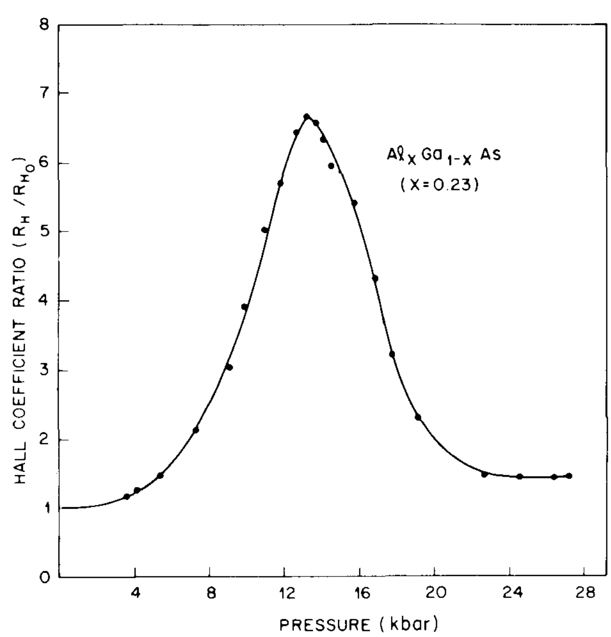

FIG. 2. Hall coefficient of $Al_{0.23}Ga_{0.77}As$ as a function of hydrostatic pressure measured using the setup shown in Fig. 1(a).

balanced voltage between Hall probes due to sample resistivity, using a potentiometer and a battery.

We show in Fig. 2 the Hall coefficient as a function of hydrostatic pressure for a typical sample. The detailed interpretation of the results will be given elsewhere. Because of the limits of the magnetic field obtainable the present technique is useful only with samples having mobilities not lower than 100 $cm^2/V\ s$ and carrier concentration not higher than $10^{17}\ cm^{-3}$. We also believe that by increasing the current it is possible to obtain a field of 500 G. However, increasing the current would cause ohmic heating which will become a problem especially when working at the lower end of the temperature range.

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