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An Apparatus for Microwave Studies at High Pressures and Low Temperatures

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An apparatus for high pressure microwave experiments has been developed which can be operated at pressures up to 2 kilobars at 4.2 K using helium as the pressure transmitting medium. The high pressure chamber contains a rutile microwave resonator oscillating at ~35 GHz. Microwave radiation is coupled into the resonator via a self-sealing conical dielectric waveguide. The apparatus has been used in preliminary measurements of the effect of pressure on cyclotron resonance in bismuth.

INTRODUCTION

FOR cyclotron resonance pressure effect studies in solids a system allowing microwave measurements in a high pressure environment at ~4.2 K is required. Microwave experiments at high pressure have been described as early as 1955.[1–8] Except for Kushida,[2] who conducted experiments at 196 K and 10 kilobars, all the other authors[1,3–8] describe room temperature equipment. Using the design for the conical dielectric waveguide–pressure plug (sapphire) described by Lawson[5] we have developed a system capable of 2 kilobars at 4.2 K which allows microwave measurements. We have obtained the first direct measurements of the effect of pressure on cyclotron resonance in bismuth using this apparatus.

APPARATUS AND PROCEDURES

Helium is pressurized at room temperature using a standard gas pressurization system capable of generating 10 kilobars. The gas pressurization system is constructed from commerically available components and is similar to the one used by Schirber.[9]

The high pressure cell in its experimental position is shown in Fig. 1. Both the pressure tubing and waveguide are introduced into the Dewar through the top. Space limitations impose the requirement of a 180° bend in either the waveguide or the pressure tubing. Because bending the pressure tubing could result in a safety hazard due to weakened material the bend was placed in the waveguide. The main disadvantage of this choice is that it reduces the microwave power delivered to the rutile resonator. In practice this loss has not proven to be important.

The high pressure cell is depicted in Fig. 2. Pressurized helium is delivered to the high pressure cell via the high pressure tubing. A pressure seal is obtained between the pressure tubing and the pressure plug by soldering their common threads. Another pressure seal is achieved by the antiextrusion ring and the lead and indium washers. The final pressure seal between the sapphire cone and the high pressure cell is made by lapping with diamond paste and then applying a thin film of indium to the sapphire cone by vacuum deposition. Although the cell is designed to contain 10 kilobars pressure, the seals usually fail at lower pressures ~2 kilobars.

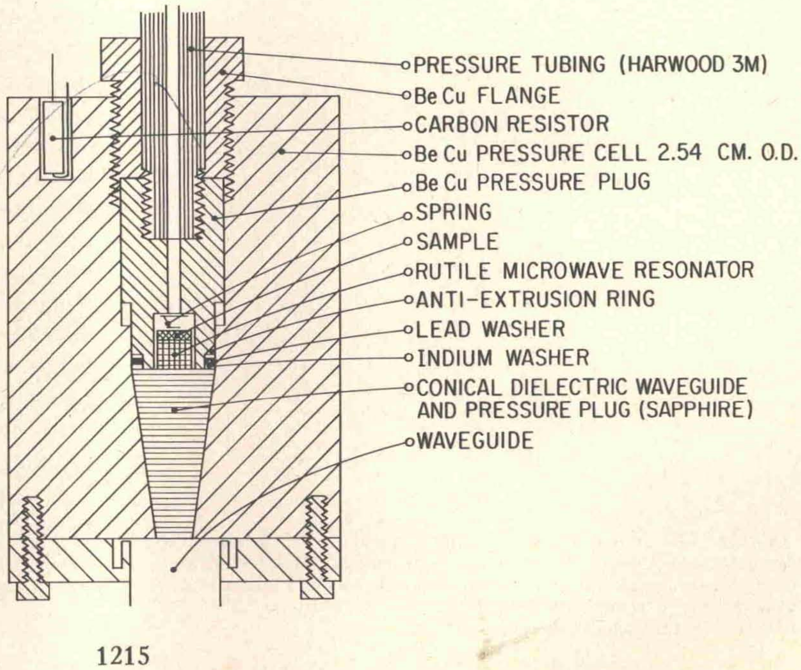

FIG. 1. The high pressure cell is shown in its experimental position. Not shown is a vacuum quick connect which is used to transfer liquid helium.

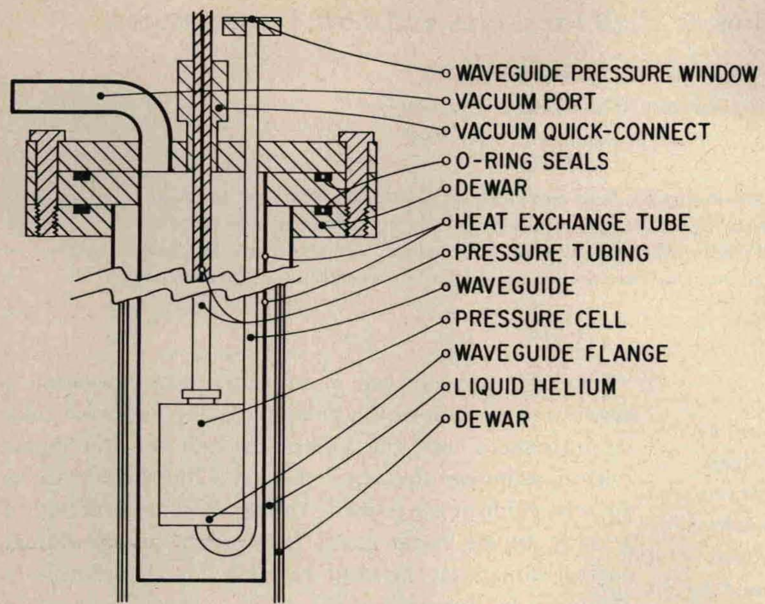

FIG. 2. The high pressure cell is shown in a detailed cross section. Not shown is another carbon resistor mounted similarly to the resistor in the figure.

The sapphire cone acts as a conical dielectric waveguide and couples the microwaves at $\sim$35 GHz into the rutile resonator. Our rutile resonator is a right circular cylinder 0.2286 cm$\times$0.254 cm diameter which theoretically has a fundamental TE_{111} mode at $\sim$8 GHz assuming a dielectric constant of $\sim$135. In the frequency range of our klystron (31.5–36 GHz) the rutile resonator oscillates in many high Q ($\sim$10 000) nonfundamental modes. The frequency of any individual oscillation is not changed significantly by a change in pressure. Nor is there any noticeable change in the Q. No EPR signals were observed from either the rutile or the sapphire for the frequencies and magnetic fields employed. The end of the rutile resonator which contacts the sample is highly polished. The other end and the sides are simply ground smooth. The rutile resonator is glued to the sapphire cone with Eastman 910. Use of rutile as a microwave resonator is described by Okaya.[10]

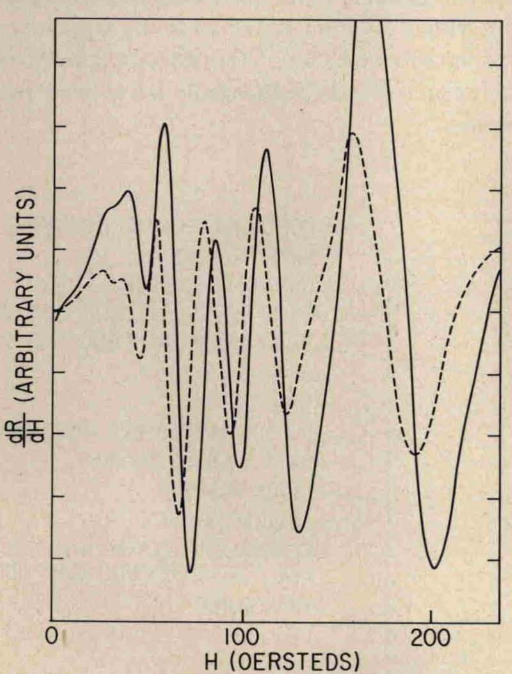

FIG. 3. The derivative of the surface impedance with respect to magnetic field is shown. The solid line represents atmospheric pressure data and the dashed line represents data taken at 1.5 kilobars. The peak with a flattened maximum was due to saturation of the X–Y recorder for that particular range setting.

The temperature of the pressure cell is measured using a carbon resistor following the procedure described by Clement.[11] Another resistor, similarly mounted in a well drilled into the pressure cell, is used to heat the cell before changing pressure. This heating is required because helium at 4.2 K solidifies at $\sim$130 bars. By heating we can maintain a hydrostatic environment to any pressure as long as the temperature is above the melting temperature of helium as determined by Dugdale.[12] The procedure is to evacuate the heat exchange tube, dissipate power in the heating resistor, change pressure at a temperature above the appropiate melting temperature, stop power dissipation, introduce a small quantity of helium into the heat exchange tube, and cool to 4.2 K. The entire sequence requires $\sim$30 min. The pressure in the cell can be determined by using the applied pressure as measured at room temperature by a manganin gauge with a small correction for change in pressure as the solid helium is cooled. This correction is readily accomplished using the isochores of helium published by Dugdale.[12]

As a preliminary test of the apparatus several experimental runs have been made to observe the changes in the cyclotron masses of the carriers in bismuth with pressure. A typical Azbel–Kaner cyclotron resonance experiment was arranged similar to that of Kip.[13] The derivative of the