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Apparatus for Electron Spin Resonance Studies at Very High Pressures*

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Apparatus for studying electron paramagnetic resonance in solids under pressure to 60 kilobars and beyond has been developed. The high pressures are produced between dielectric Bridgman anvils, one of which serves as a microwave resonance cavity. The instrument makes possible the study, by EPR techniques, of paramagnetic atoms or ions subject to stresses which might be expected to induce changes in their electronic structure.

INTRODUCTION

MICROWAVE resonance would appear to provide an ideal diagnostic tool for the determination of the physical changes that take place in materials as pressure is applied, particularly those changes which are not accessible to x-ray analysis. Some work has been done in the intermediate pressure range to about 10 kilobars[1–3] using pressure bombs, but some of the most interesting high pressure effects are those thought to involve a change in the atom itself, and these changes generally occur at much higher pressures. Basic equipment and techniques for producing these higher hydrostatic pressures on a paramagnetic solid in a microwave bridge are described here.

DESCRIPTION OF EQUIPMENT

A block diagram of the system appears in Fig. 1. The system consists essentially of four subsystems: the pressure system; the microwave bridge; the magnetic field modulation, control, and measurement system; and the low temperature system. Each of these subsystems is described below.

Pressure System

The sample cell is a $\frac{1}{8}$-in.-diam$\times$0.030-in.-high boss in a $\frac{3}{8}\times$0.006 in. copper disk placed between a pair of Bridgman anvils, the lower of which forms the bottom face of the cell enclosure and serves as the resonant cavity of the microwave system. The sample cell is filled with Viscasil 100 000 (General Electric Company silicone fluid, viscosity at 25°C is 100 000 centistokes) or mineral oil for the pressure transmitting medium. The cell is surrounded by a multilayered band of pyrophyllite washers 0.010 in. thick $\times$0.125 in. i.d. $\times$0.375 in. o.d. separated by half-hard Berylco-25 beryllium-copper washers 0.005 in. thick with the same i.d. and o.d. (Figs. 2 and 3).

The lower anvil is essentially a right circular cylinder with height and diameter 0.700 in. made of cold-pressed alumina of an especially pure form manufactured by Electroceramics, Salt Lake City, Utah. The anvil is silver-coated by the Brashear process[4] followed by silver-plating to a thickness of about 0.001 in.

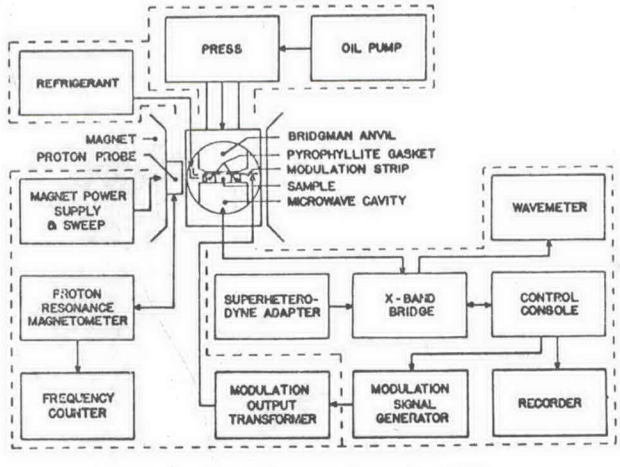

FIG. 1. Block diagram of complete high pressure magnetic resonance system.

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