NOTES

BRIEF contributions in any field of instrumentation or technique within the scope of the journal should be submitted for this section. Contributions should in general not exceed 500 words.

Four-probe single-crystal holder for conductivity measurements

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The development and testing of a four-probe small single-crystal holder for conductivity studies down to liquid-helium temperature is described. A single crystal of the reactive sodide, $Na^{+}C222\cdot Na^{-}$, was successfully mounted, but its resistance was too high to permit determination of the conductivity. The method should be useful for single-crystal conductivity measurements of reactive and/or thermally unstable crystals.

Various four-probe methods have been described for measuring the conductivity of anisotropic materials.[1-7] Montgomery[7] has described a four-probe method which has the advantage that it can be applied to rather small (~1 mm) but rectangular prismatic samples: small electrodes are attached at the four corners of one face. The ratio of the voltage between two adjacent electrodes to the current between the other two electrodes is measured, then a similar measurement is made with all connections rotated by 90°. From these measurements and the sample's dimensions, the conductivity tensor can be calculated.

We developed a technique to use Montgomery's method for the determination of conductivity in single crystals of two new classes of compounds, called alkalides and electrides,[8-11] in which the anions are alkali metal anions or trapped electrons. Since these compounds are extremely air and moisture sensitive and are also thermally unstable, it was necessary to design a system that permits one to mount cooled crystals in an inert atmosphere. We report the development and use of a single-crystal holder for four-probe conductivity studies of small reactive samples down to liquid-helium temperatures.

The single-crystal holder, based on a design described by Phillips *et al.*,[12] consists of an eight-pin integrated circuit package with the top of the can removed. The integrated circuit was replaced by a layer of insulating material such as mica fastened to the base with epoxy glue. The crystal to be examined was held to the surface of the insulating material with a thin layer of Apiezon-N stopcock grease, selected because it does not react with our samples. Thin wires (~0.03 mm diameter) of "bare hard copper[13]" were used to establish the four connections to our small crystals (Fig. 1). Silver paint covered with epoxy was used to make the contacts between the wires and alternate bonding headers. The four wires were attached to the surface of the single crystal by using a locally prepared gold–palladium (80:20) Apiezon-N mixture. Other available contact materials such as silver paint were too reactive for our samples. During the mounting process the crystal was kept cold by a slow flow of cold (200-K) nitrogen gas over the crystal holder.

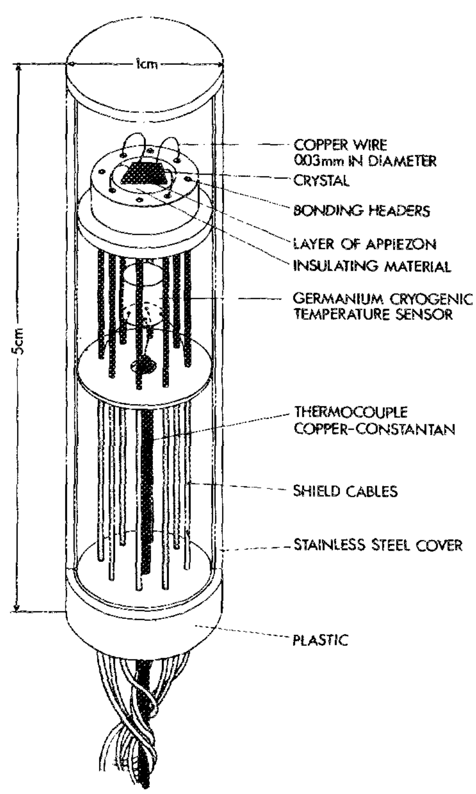

FIG. 1. Single-crystal holder.

The temperature was monitored (Fig. 1) by a copper-Constantan thermocouple for the 300–70-K range, and by a germanium cryogenic temperature sensor, GR-200A (1500), which covers the range of 70–5 K. Variable temperature studies were made by placing the crystal holder with its cylindrical stainless-steel cover, into the internal tube (1.1 cm in diameter) of a partially filled liquid-helium container. The temperature of the sample could be controlled by varying the distance to the surface of liquid helium.

Four out of the eight shielded cables, each about 1 m in length, were used to couple the crystal holder to the conductivity measuring circuit. This system, designed by Phillips *et al.*,[12] is a versatile four-probe ac conductance measuring device which covers the range 10^{-8}–10^{+5} Ω^{-1} over the frequency range 20–2000 Hz. The other four cables were used for the connections to the cryogenic temperature sensor.

The operation of the apparatus was tested at various temperatures with a silicon rectangular crystal, $0.9 \times 1.0 \times 0.3$ mm^3, doped with phosphorous atoms (density $\sim 10^{16}$ cm^{-3}). The resistivity calculations were based on the "thin sample" equations.[7] Two calculated resistivity components ρ_1, ρ_2, at 25 °C are shown in Fig. 2 as a function of frequency. The average value of the two components at low frequencies is in good agreement with known resistivity of 0.33 Ω cm at 298 K. Similar measurements were repeated at various temperatures.

Attempts were made to determine the conductivity of a single crystal of $Na^+C222 \cdot Na^-$, the prototype of the alkalide–electride class of compounds. Under a dry-nitrogen atmosphere a single, almost rectangular, crystal $0.62 \times 0.50 \times 0.25$ mm^3 was selected and loaded into the crystal holder. The conductance of the crystal was found to be less than 10^{-8} Ω^{-1} which is on the borderline of the sensitivity of our apparatus. However, the ability to mount this reactive crystal and to make suitable electrical contacts with it shows that the four-probe method should be applicable to the study, now in progress, of more conductive alkalide salts and of electrides.

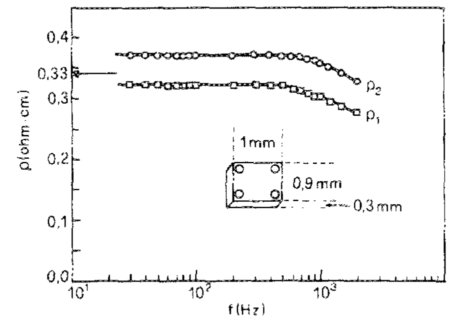

FIG. 2. Resistivity components ρ_1, ρ_2 vs frequency for a single crystal of P-doped silicon at room temperature. The subscripts 1 and 2 refer to the shorter and longer dimensions, respectively.

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Small oven for obtaining temperatures to 1350 °C

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A small, inexpensive oven capable of achieving a temperature of 1350 °C with a power consumption of less than 1 kW is described. The oven has a working volume which is roughly $10 \times 6 \times 12$ cm. It has been used for bonding gold to sapphire substrates, but would be suitable for use in any application which requires elevated temperatures in a working volume of 500–1000 cm^3.

Varmazis *et al.*[1] describe a technique for bonding gold to sapphire which involves no more than placing small pieces of gold on sapphire substrates and then heating the substrate to a temperature of the order of 1500 °C. The technique is easy to implement if an oven capable of reaching temperatures in the region 1300–1500 °C is available. Commercial ovens which work in this temperature region are, however, expensive, and the techniques for constructing and using such an oven do not appear to be widely known among experimental physicists. In this note we describe an oven which can be

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