NASA Technical Memorandum 87256

Apparatus for Electrical Measurements of Thin Films from 77 to 1000 K

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March 1986

NASA

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E-2839-1

SUMMARY

A novel method of mounting thin samples for electrical measurements is described. A vacuum chuck holds a mounting plate, which, in turn, holds the sample. Contacts on the mounting plate establish electrical connection to the sample. The attachment of wires directly to the samples is unnecessary. Measurements can be made at temperatures from 77 to 1000 K. As an application of the apparatus, resistivity and Hall measurements of a thin silicon carbide sample are presented.

INTRODUCTION

The NASA Lewis Research Center, as a part of its effort in high temperature electronics, currently has a research program aimed at developing silicon carbide (SiC). SiC is of interest because of its potential as a semiconductor material for high temperature applications. During this research, electrical measurements are required for the routine characterization of the SiC material. From these measurements, basic electrical parameters such as carrier concentration, resistivity, and Hall mobility are derived. If these measurements are made as a function of temperature, additional information such as the impurity doping concentration, impurity ionization energy, and the band gap energy can also be obtained.

The combination cryostat-furnace apparatus described herein has been built for the purpose of making resistivity and Hall measurements as a function of temperature (77 to 1000 K) on thin samples (the order of 10 μm) of SiC. The measurements were made using the van der Pauw technique (ref. 1). For the Hall measurements, an electromagnet capable of generating 1 T (10 kG) was utilized.

In designing this apparatus, the ease of sample mounting was a primary concern. Direct electrical connection to the sample by wire bonding or similar techniques had been considered but rejected. Bonding to these fragile samples is time consuming and frequently results in destruction of the substrates. Instead, a vacuum chuck concept was adopted which simultaneously holds the sample in place and establishes electrical contact. With this mounting method, measurements on many samples can be made quickly. For measurements as a function of temperature, either cooled or heated nitrogen gas was circulated over the sample and the mounting hardware.

DESCRIPTION OF THE APPARATUS

The photograph in figure 1 shows three elements that make up the cryostat/furnace apparatus. These elements are (1) the tubular vacuum jacket, (2) the sample mounting assembly, and (3) the heating/cooling assembly. Figure 2

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illustrates the three elements in their assembled configuration and gives relevant dimensions of the apparatus. These dimensions were forced by the dimensions of the electromagnet and the SiC samples. The vacuum jacket must fit between the magnet pole pieces (51 mm air gap) and the sample mounting plate must receive the SiC sample. A typical SiC sample is a 5 mm square platelet. For the most part, the apparatus is constructed of nonmagnetic stainless steel.

The sample mounting assembly and the heating/cooling assembly slide into opposite ends of the vacuum jacket such that the cooled or heated nitrogen gas is injected in close proximity to the sample and the sample mounting hardware. The vacuum jacket is mounted between the pole pieces of the magnet and connected to a mechanical vacuum pump. The use of the vacuum jacket minimizes the heat conduction to and from the ambient environment. To further reduce the heat loss from the sample mounting volume, the void spaces in the sample mounting assembly and the heating/cooling assembly are packed with a ceramic fiber insulation.

Low temperature measurements approaching 77 K were obtained by flowing cryogenic nitrogen while electrical resistance heating of dry nitrogen was used to obtain measurements at temperatures approaching 1000 K. By balancing the flows of these two nitrogen gas sources, measurements at any temperature from 77 to 1000 K could be made.

To monitor the temperature, two thermocouples were initially utilized. One thermocouple was connected to the bowl of the vacuum chuck while the second sensed gas temperature near the sample. The original reasoning was that when the temperature was being changed, the chuck temperature would lag behind the gas ambient temperature. In practice, no significant temperature difference was noted. Since the bowl thermocouple did, on occasion, interfere with the electrical measurement leads, the bowl thermocouple was eliminated. All temperature measurements are gas temperatures near the sample.

A unique feature of this apparatus is the method of mounting and maintaining electrical contact to the sample under evaluation. The mounting method had to retain the sample and sustain electrical contact over a wide temperature range and not introduce distortions into the magnetic field during the Hall measurements. Various methods of clamping were considered and rejected. Instead, the mechanical mounting force is provided by the vacuum chuck. The mounting stage is shown in figure 3 as well as the "exploded view" in figure 2. With this mounting method, the vacuum force acts upon raised contacts on both the sample and the mounting plate to provide the necessary electrical connections.

The mounting plate is made of a machinable glass-ceramic insulating material. Both surfaces of the plate, as well as the surface of the vacuum chuck are polished to enhance the vacuum seal between the various elements. The vacuum source for the chuck is a commercial venturi type pump. Holes through the mounting plate communicate the vacuum to the interface between the plate and the sample. Atmospheric pressure, acting upon the sample then provides the mechanical pressure to hold the sample in place.

In this application, the vacuum holes through the mounting plate are concentric with the various electrical contacts so that the contacts on the mounting plate are in the form of conducting annuli. Each sample has a pattern of solid contacts similar to that of the mounting plate. The sample is positioned on the plate so that the contacts are aligned and then the vacuum is applied.

The contacts on both the samples and the mounting plate are sputter-deposited through metal masks. The deposited pattern on the mounting plate includes conductors from the conducting annuli to a second set of contacts near the periphery of the plate. These peripheral contacts are used to connect to the lead wires which, in turn, are connected to the appropriate measurement devices.

For this application, gold contacts were used. In addition, gold wire was used for electrical leads to minimize any thermally induced contact voltages. The van der Pauw measurement requires a pattern of four contacts. For this work, the contact size was 0.5 mm^2, and they were arranged in a square pattern with 2.5 mm center-to-center spacing.

In applications of this technique to other measurements, one must consider the problems of contact material, size, and spacing as well as the effect of contact potentials or resistance. The use of a conducting annulus as a contact does limit the size of the contact. In some applications, gold as a deposited contact may not be appropriate. The influence of contact size and geometry upon the van der Pauw measurement is discussed in reference 1.

APPLICATION

The apparatus described has been used to obtain electrical measurements upon SiC samples. These samples are epitaxially grown upon commercial silicon wafers (ref. 2). With care, thicker (>10 μm) SiC layers can be removed from the silicon and handled without breakage. For thin layers, the measurements must be made with the silicon in place. The resistivity of the silicon decreases rapidly with increasing temperature and, in turn, introduces large errors into any measurement. Obviously, at higher temperatures, meaningful measurements cannot be made with the silicon layer present. The higher temperature measurements can be made only on the thicker samples.

Figure 4 presents typical measurements of carrier concentration, resistivity, and Hall mobility as a function of temperature. These data are from a cubic SiC sample having a thickness of 17 μm.

The apparatus described in this report was constructed for the specific purpose of SiC characterization. It is expected however, that the vacuum chuck concept would be useful in a variety of measurement applications.

REFERENCES

1. van der Pauw, L.J.: A Method of Measuring Specific Resistivity and Hall Effect of Discs of Arbitrary Shape. Philips Res. Rep., vol. 13, no. 1, Feb. 1958, pp. 1-9.

2. Nishino, S.; Powell, J.A.; and Will, H.A.: Production of Large-Area Single-Crystal Wafers of Cubic SiC for Semiconductor Devices. Appl. Phys. Lett., vol. 42, no. 5, Mar. 1, 1983, pp. 460-462.

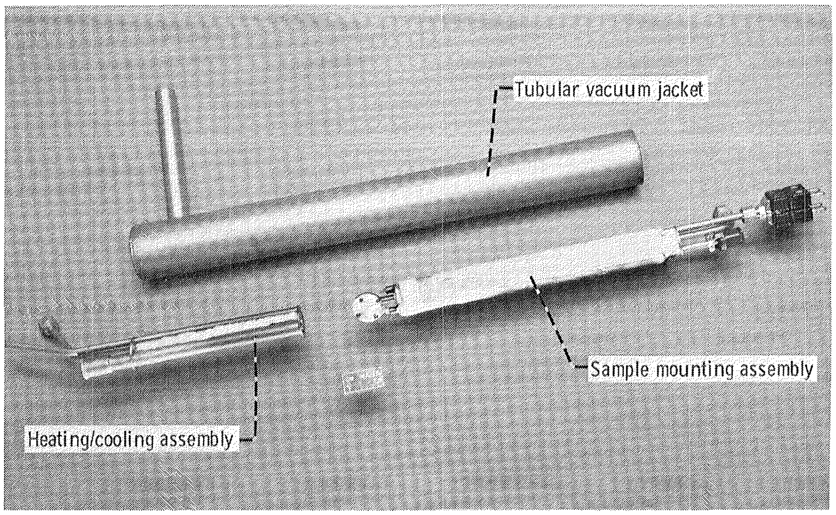

Figure 1. - Elements of cryostat/furnace apparatus.

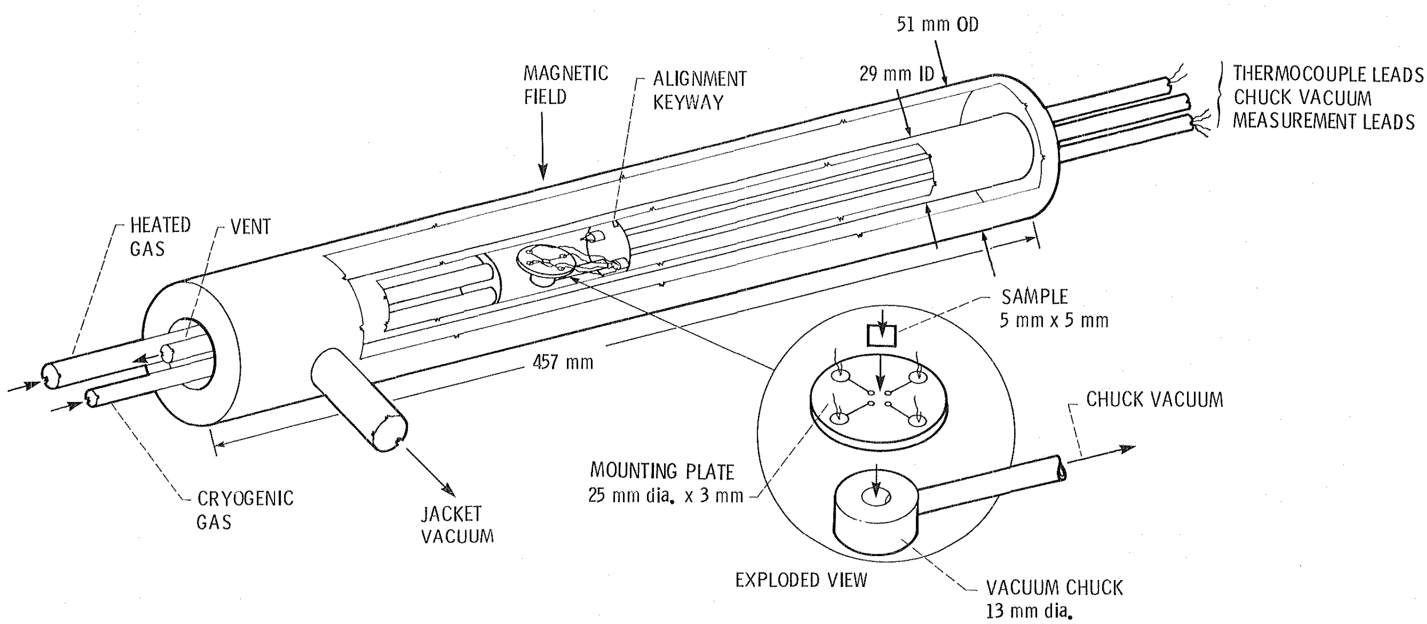

Figure 2. - Assembled configuration of cryostat/furnace apparatus. Exploded view details the sample mounting hardware.

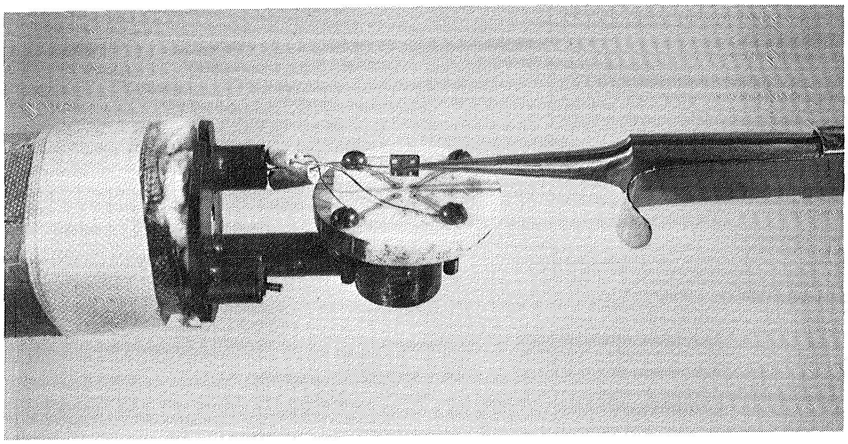

Figure 3. - Sample mounting hardware.

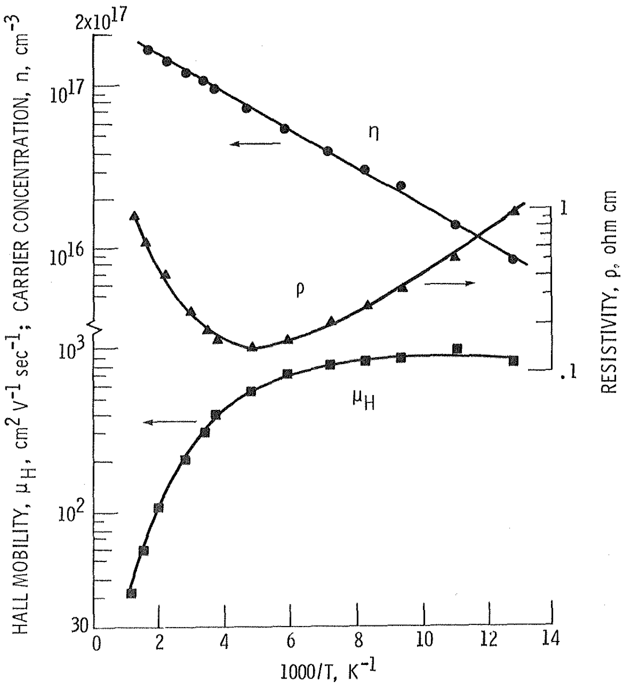

Figure 4. - Electrical measurements for 17-μm-thick cubic SiC sample.

1. Report No. NASA TM-87256	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle Apparatus for Electrical Measurements of Thin Films from 77 to 1000 K		5. Report Date March 1986
		6. Performing Organization Code 505-62-01
7. Author(s) Lawrence G. Matus and Robert L. Summers		8. Performing Organization Report No. E-2839-1
		10. Work Unit No.
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135		11. Contract or Grant No.
		13. Type of Report and Period Covered Technical Memorandum
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

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17. Key Words (Suggested by Author(s)) Apparatus; Electrical measurements Sample mounting; Thin films	18. Distribution Statement Unclassified - unlimited STAR Category 35

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of pages	22. Price*
Unclassified	Unclassified		

*For sale by the National Technical Information Service, Springfield, Virginia 22161

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