SUPERCONDUCTOR TEST STATION

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ABSTRACT

A basic test station, using a modified four point probe was designed and built for measuring resistivity over a temperature range. This test station can now be used to characterize the effects of any subsequent semiconductor processing on transition temperatures and performance of superconductive films.

INTRODUCTION

A short history of superconductors is provide to fully understand the background of this project [1-17]. Superconductors are materials that exhibit a complete loss of electrical resistance as they are cooled below a critical temperature. This phenomenon was discovered in 1911, when physicist Heike Kamerleigh Onnes was conducting experiments with mercury at low temperature. During one of his experiments he noticed that the resistance of a sample all but disappeared at four degrees Kelvin. Little work was done in the early parts of the century, and the work that was done, was limited to metals and metal alloys.

Then in 1973 niobium alloys were found to exhibit superconductivity at around 23 K. These materials found their first applications in medical imaging systems and in particle accelerators. Still, the temperatures needed to obtain superconductivity made most applications impossible. Then in 1983 metal oxides such as BaLaCuO showed transition temperatures of 35 K. This was a major jump from the previous temperature of 23 K, and was discovered by two of IBM's research scientists, Karl Alexander Muller and Georg Bednorz. Early in 1986 another small jump up to 44 K was observed in niobium-germanium-aluminum and oxygen material developed by two Japanese scientists, Oguski and Osono. During the last few months of 1986 various researchers started working with ceramic material with transition temperatures around 70 K. Early in February of 1987 a Houston scientist discovered a ceramic material (yttrium, barium, copper, oxide) with a transition temperature of 95 K. This was probably the most significant development since the discovery of superconductors , because liquid nitrogen could now be used as a coolant instead of the conventional liquid helium coolant. The boiling point of nitrogen is 77 K while the boiling point of helium is 4.2 K. Nitrogen is much cheaper, easier to handle and obtain than liquid helium. Liquid helium has a current price of about four dollars a liter compared to forty cents per gallon for liquid nitrogen. Reports of superconductors with higher critical temperatures continue to come in almost weekly. Sumitomo

Electric claims to have developed a material made up of yttrium, copper and oxygen that shows superconductivity at room temperature, up to 27 C (1). Probably the most promising new development comes from Georgia Institute of Technology. Physicist Ahmet Erbil says that he has developed a material made of copper oxides with a critical temperature of 500 K (2). This report like many others remains unconfirmed, but if true, would revolutionize the superconductor field.

Before tackling the importance and limitations of superconductors in the Microelectronics field it is necessary to consider some background material. Two basic criteria for a material to be accepted as a true superconductor are zero resistance below a critical temperature and the material must show the Meissner effect. The Meissner effect is defined as the expulsion of a magnetic field from a materials interior (4). This phenomenon is easily shown in the floating magnet experiment. The zero resistance effect is shown in Figure 1. Superconductive materials have basically the same resistance versus temperature curve as most metals until the critical temperature is reached, at which point the resistance drops off dramatically.

Superconductors not only have critical temperature criteria but critical current and magnetic field specifications as well. Critical current is the maximum current density a sample may conduct before it's resistance reappears. Critical magnetic field is much the same but not as much of a limitation as critical current. Current densities have been raised to 100,000 A/cm2 in thin films, but is limited to 1,000 A/cm2 in bulk material.

Other problems that plague thin films of the high Tc materials have to do with stability. Superconductors tend to lose oxygen when exposed to atmospheric pressures. The loss of oxygen has drastic effects on performance, since oxygen is a crucial factor in the structure of superconductors. Many of the superconductive materials available today are very sensitive to water, heat and air which degrade their delicate structure and composition.

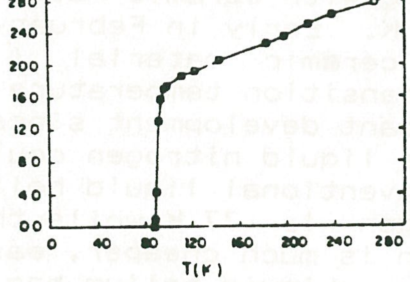


FIGURE 1 Typical response of resistance to temperature for YBaCuO [9]

The most obvious application of superconductors to microelectronics is their use as zero resistance interconnects. Low power dissipation, coupled with faster signal transfer makes this application very attractive. The most important application in VLSI technology has to be Josephson Junction devices. Josephson Junctions are comprised of a thin insulating material sandwiched between two superconductive plates. One of the superconducting layers acts as a gate, when a voltage is applied, a magnetic field changes the current in the other layer. These devices act as superfast, low power, switches. Their low power dissipation also allows for higher packing densities.

Little is known about the reaction of superconductive films to standard semiconductor processing steps. Steps such as lithography (how will prebake and post bake temperatures effect the thin films ?) chemical etching, plasma etching, and a host of other processing steps still need to be characterized for superconductive thin films. Ultimately a complete analysis of semiconductor - superconductor hybrid circuits, and their fabrication, must be done before superconductors become an important part of VLSI technology.

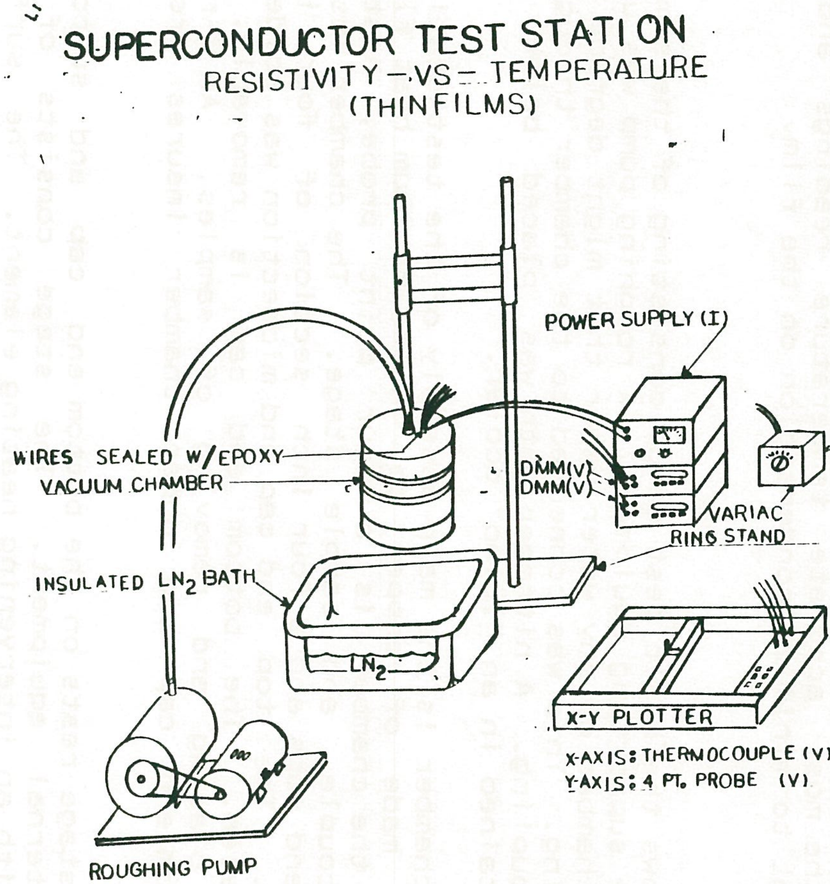
This project involved a special set up to achieve the environmental conditions needed to preform resistivity measurements at low temperatures on thin films. The main concern of this station was to provide accurate and repeatable resistivity measurements without degrading the thin film. Attention was given to uniform cooling and heating of the system so as to obtain the most accurate temperature readings and a vacuum environment to minimize condensation on the film.

EXPERIMENTAL

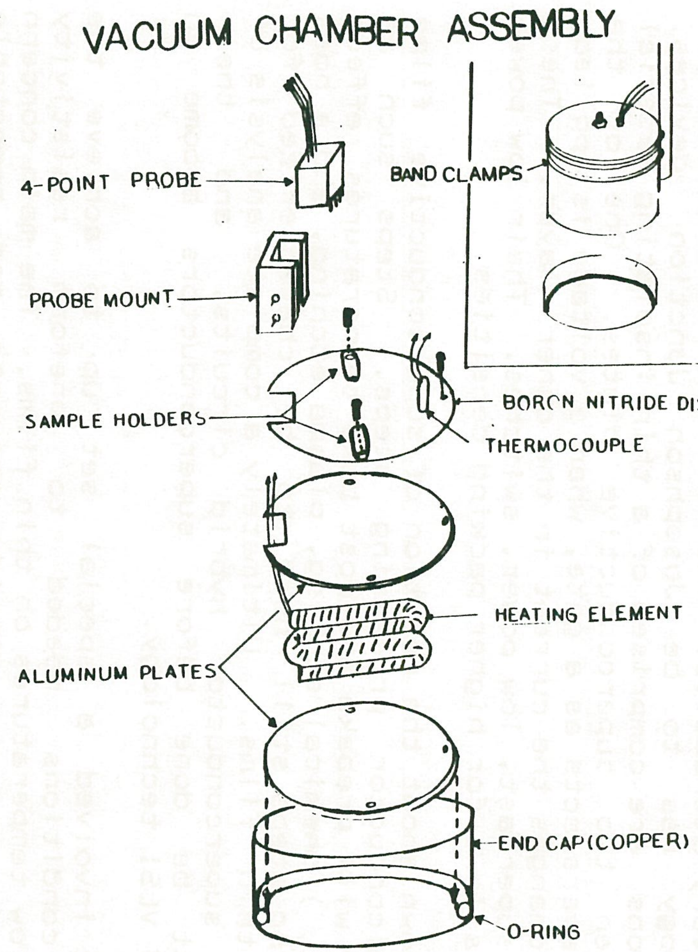
Figure 2 shows the basic test setup consisting of the sample chamber and the supporting equipment. A roughing pump was used to evacuate the chamber of any water vapor that might degrade the film upon cooling. This was connected to the chamber through a threaded brass coupling. A nitrogen bath was placed below the chamber, and contained in an "Igloo" cooler.

The vacuum chamber is the main assembly of the test station. The chamber is made of copper to insure maximum heat flow. Contained within the chamber is a four point probe, heating element, thermocouple, and sample stage. The chamber itself consists of two end caps and a four inch section of four inch diameter tubing. The top end cap and midsection was soldered together and sealed. The bottom end cap is removable, to accomadate the placing and removing of samples. An O-ring between the bottom end cap and upper chamber insures proper sealing.

The sample stage rests on the bottom end cap and supports all of the internal equipment. The stage consists of two aluminum plate with an intervening heating element. The surface of the stage is a Boron Nitride disc, this material was used to



(FIGURE 2)



(FIGURE 3)

electrically insulate the sample from the other elements while still allowing excellent thermal conductivity. A four point probe is also contained within the chamber, the probe is a standard tool for measuring resistivity of thin films . The four point probe is mounted on a teflon probe holder. This holder allows for positioning of the probe, although very limited, and securely holds the probe with three set-screws. The holder is made of teflon to protect the probe from the extreme cold temperatures during the experiment (the probe tips will be directly on the sample). A thermocouple is clipped to the stage and is used to monitor the temperature upon heating. The thermocouple implemented was an Omega " Type-J " thermocouple, which can measure temperatures down to -210 C. Eight wires connect the various components to outside equipment through an epoxy seal.

The thermocouple voltage was connected to a DMM in parallel with the x-axis of a chart recorder. The voltage from the four point probe was connected to a DMM in parallel with the y-axis of the same chart recorder.

Four point probe voltage as a function of temperature is plotted as the sample is heated. Readings from the thermocouple DMM must be taken at two points to accurately calculate temperature. The resulting graph can be converted to a resistivity versus temperature graph by using the thermocouple table (APPENDIX-A) in conjunction with the equation for resistivity as a function of probe voltage (V), current (I) and spacing (S), given by rho :

$$rho=2\pi S(V/I)$$

RESULTS/DISCUSSION

The test station was calibrated by measuring the resistivity of a silicon sample at room temperature and comparing it to another standard four point probe setup.

Problems appeared when cooling started. The Thermocouple never reached the desired temperature, that is to say that the correct voltage was never generated. The temperature achieved was only -10 C. A slight air pocket between the stage and bottom end cap, the insulated heating element between the two aluminum plates, coupled with the fact that a vacuum is present could account for this lack of heat flow, since a vacuum allows for little heat flux. Another problem that resulted upon cooling, was that the four point probe voltage became very erratic and actually went up. This may have been caused by contact problems that resulted from uneven heating. Compressive stress on portions of the stage and chamber could have caused the probes to lift off slightly.

Yet another problem was identified when the chamber was opened after the experiment. Proper vacuum was not achieved and water had condensed on the inner surface.

The solution to the first two problems is to allow for better thermal conduction. The first step is probably to eliminate the heating element and let the sample warm to room temperature without assistance thereby allowing for direct contact of the two plates. The next, and easiest step, is to make sure that the stage is in direct contact with the bottom end cap before placing it in the chamber.

Before a higher vacuum can be achieved a leak check should be done to find the source.

SUMMARY

The test station was designed for resistivity measurments as a function of temperature. The station works at room temperature but needs to be modified in the area of heat transfer and vacuum technology. A couple of suggestions for a second generation test station , if there is to be one, include; enclosing the nitrogen coolant and modifying the probe holder to allow for greater mobility.

ACKNOWLEDGEMENTS

I would like to thank Scott Blondell and Gary Runkle, for assisting me in building and testing, Jim Argana from CVC corp. for supplying samples and advice, Standard oil of Niagra Falls for supplying Boron Nitride discs, and Dr. Richard Lane, Dr.Bob Snyder, Rob Pearson, and Mike Jackson for advice and information.

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