Chapter 5

Experimental Techniques

Introduction

This section outlines all the procedures that were followed in the electrical characterization of defects in ZnO starting with sample cleaning, contact fabrication, contact quality evaluation and defect characterization. It also explains some sample preparation methods that were employed to intentionally introduce defects such as proton irradiation and annealing.

5.1 Sample cleaning

Undoped bulk single crystal n-type ZnO samples obtained from Cermet Inc. were used in this study. The samples were rinsed in an ultrasonic bath, first in acetone for five minutes, then in methanol for another five minutes and then treated with boiling concentrated hydrogen peroxide for three minutes. Without any water rinse, the samples were blown dry using nitrogen gas. Immediately after cleaning the samples were taken for ohmic and Schottky contact fabrication. This procedure was followed for all the samples used in this study.

5.2 Contact fabrication

Ohmic and Schottky contacts were fabricated onto either the Zn- or O-polar face of the samples for comparison purposes. Ohmic contacts of either Ti/Al/Pt/Au or Al/Au were e-beam and resistively deposited, respectively as outlined in the results and discussion section. Pd, Ir and Pt were used to fabricate the Schottky contacts. It must be mentioned that in this study, if the ohmic contact was fabricated on the Zn-polar face, the Schottky was put on the O-polar face and vice-versa. The fabrication of the ohmic and Schottky contacts was

performed in vacuum. Since it was observed that e-beam deposition introduces defects in semiconductors, Schottky contacts for the control sample were fabricated using the resistive evaporation system shown in Figure 5.1.

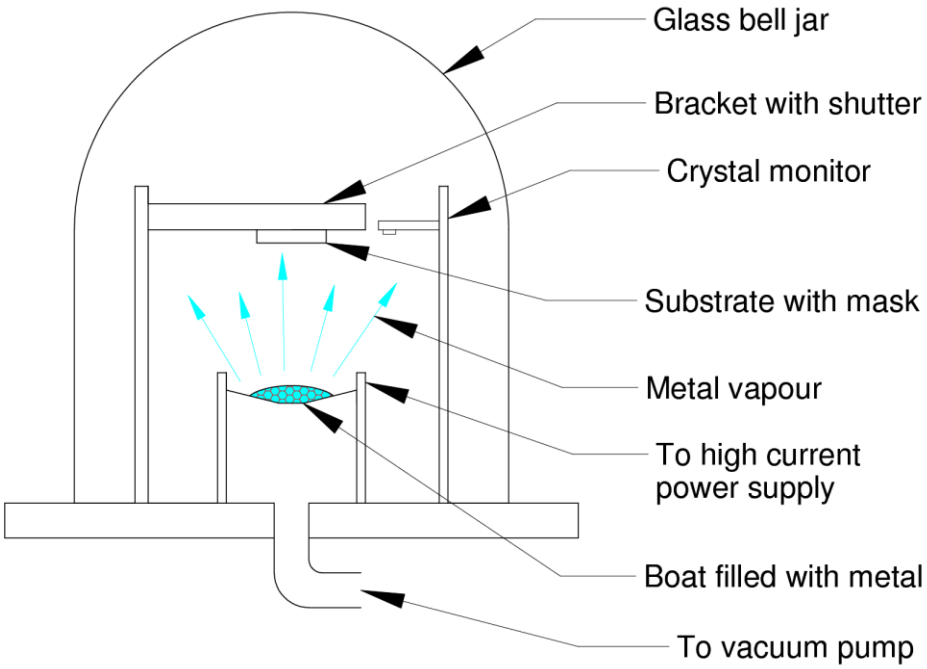

Figure 5.1: Schematic diagram of the resistive evaporation system for contact fabrication.

This method of metal evaporation uses the thermal resistance of the material to melt and evaporate it. A current is supplied and flows through the crucible containing the metal. This crucible heats up because of the metal resistance until the melting point of the metal is reached where it starts evaporating, depositing onto the sample. The amount of current supplied to the material after the melting point has been reached determines the deposition rate. This evaporation technique is ideal for metals with low melting points, $\leq 1600°C$.

In studying e-beam induced defects, Schottky contacts were fabricated using the e-beam deposition technique. Figure 5.2 shows a typical e-beam deposition system.

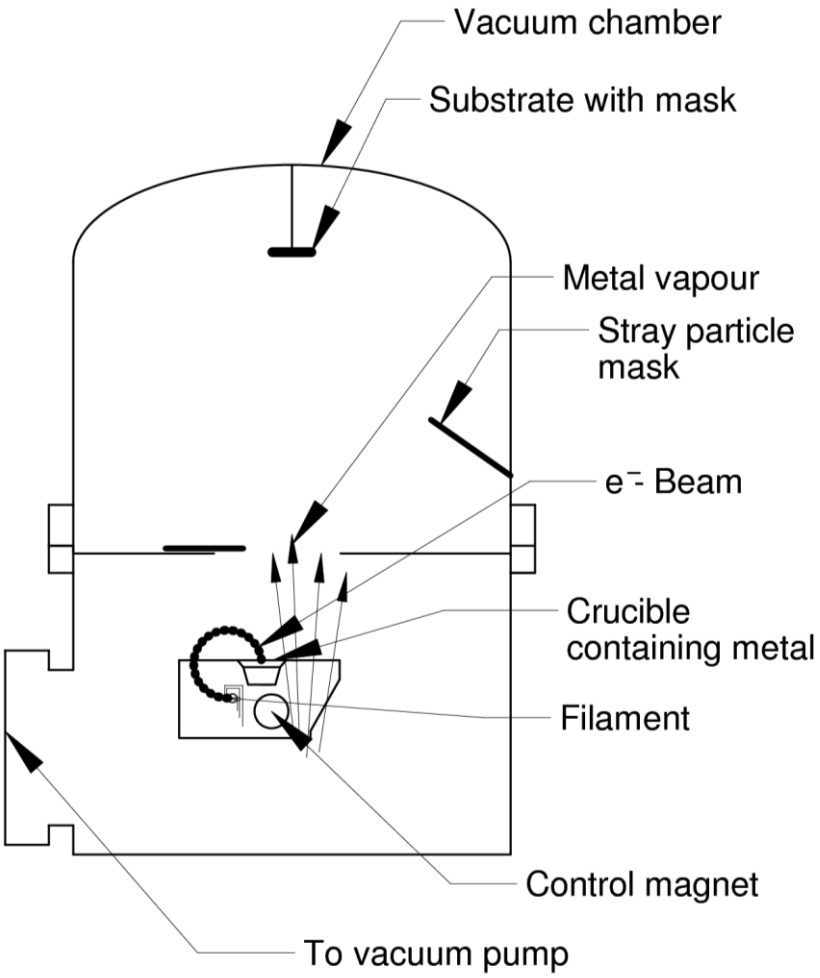

Figure 5.2: Schematic diagram of the electron-beam (*e-beam*) *evaporation system for Schottky contact fabrication.*

A beam of electrons emitted by a hot filament is used to heat and evaporate the metal. Electrons are accelerated by a high voltage and bent by the magnetic field towards the metal. The current supplied to the filament controls the rate at which the metal deposits onto a sample. This technique is ideal for high melting point metals, and can also be used for low melting point metals.

During the fabrication of Schottky contacts with either the resistive or the e-beam system, a mask with circular holes was used to produce circular contacts on the sample surface. Figure 5.3 shows a sample containing the ohmic (back contact) and Schottky (circular dots) contacts.

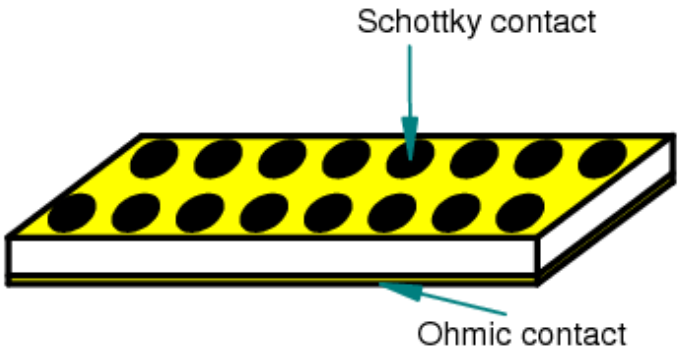

Figure 5.3: A schematic diagram showing the ohmic and Schottky contacts fabricated on ZnO

5.3 Current-Voltage and Capacitance-Voltage measurements

The Current-Voltage (IV) measurement technique is ideal for checking the quality of the Schottky contacts. By measuring the current that flows through the contact under forward and reverse bias conditions, one can deduce whether the contact is a good rectifier or is an ohmic contact. This helps in obtaining the reverse bias conditions which can be applied to a particular contact when one uses capacitance based techniques e.g. DLTS to determine other electronic properties of the contacts such as defects. An analysis of the IV characteristics of a contact can give other contact parameters such as the barrier height, ideality factor and series resistance. These characteristics can help one deduce the dominant current transport mechanisms. In this study, current-voltage measurements were performed in the dark at room temperature conditions. A schematic diagram of the IV and CV station is shown in Figure 5.4. An HP 4140B pA meter/ DC voltage source with a current limit of 10^{-14}A was used to determine the IV characteristics of the contacts.

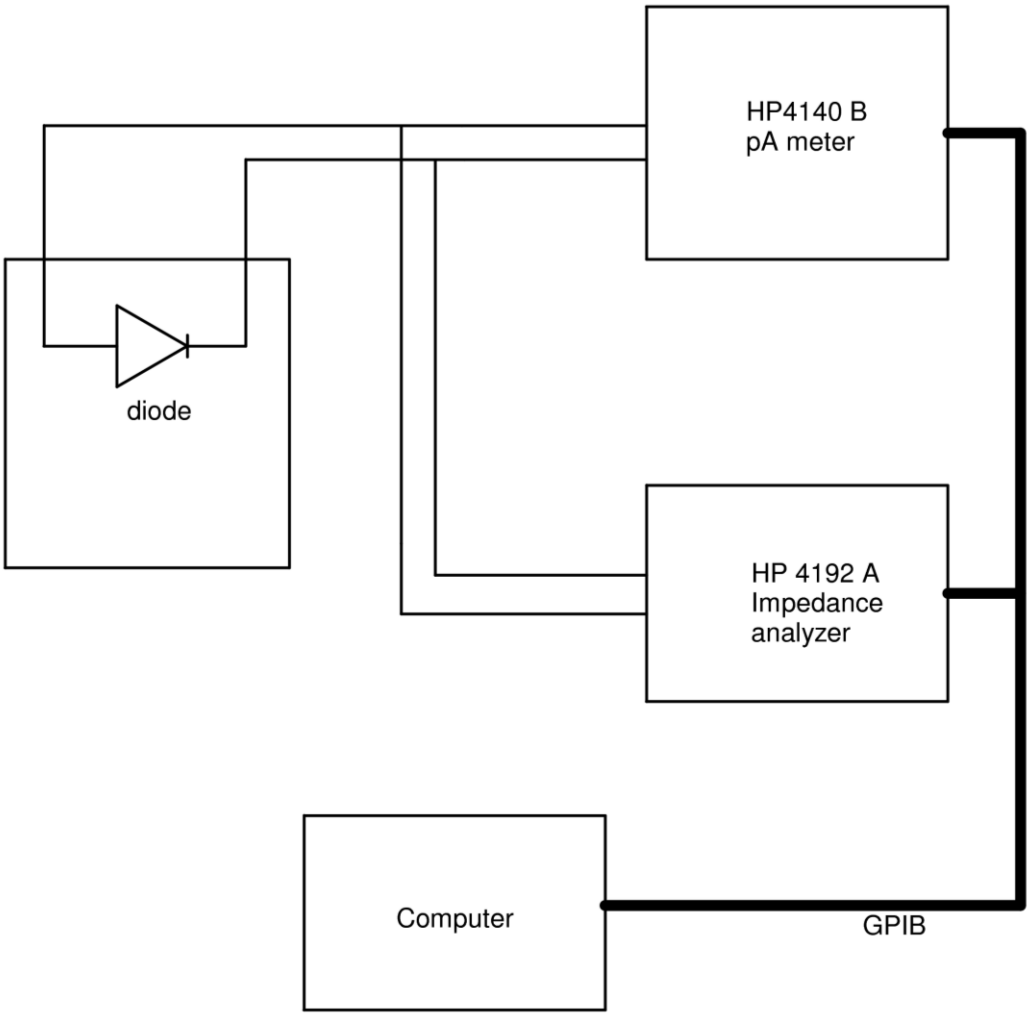

Figure 5.4: A schematic diagram of the IV and CV station that was used in this study.

The Capacitance-Voltage (CV) technique is also used to deduce important parameters of the contact that include the barrier height, the built-in-voltage and free carrier concentration. The use of the CV technique for depth profiling can also yield the net doping concentration of the metal-semiconductor conductor as a function of depth. An HP 4192A LF impedance analyser was used to determine the CV characteristics of the contacts. These measurements were also performed in the dark.

5.4 Deep Level Transient Spectroscopy (DLTS) and Laplace-DLTS

Conventional DLTS and high resolution Laplace DLTS were used to characterize the defects in ZnO. Measurements were performed in the 30 K – 350 K temperature range. Figure 5.5 shows a schematic diagram of the DLTS and Laplace-DLTS system that was used in characterizing defects.

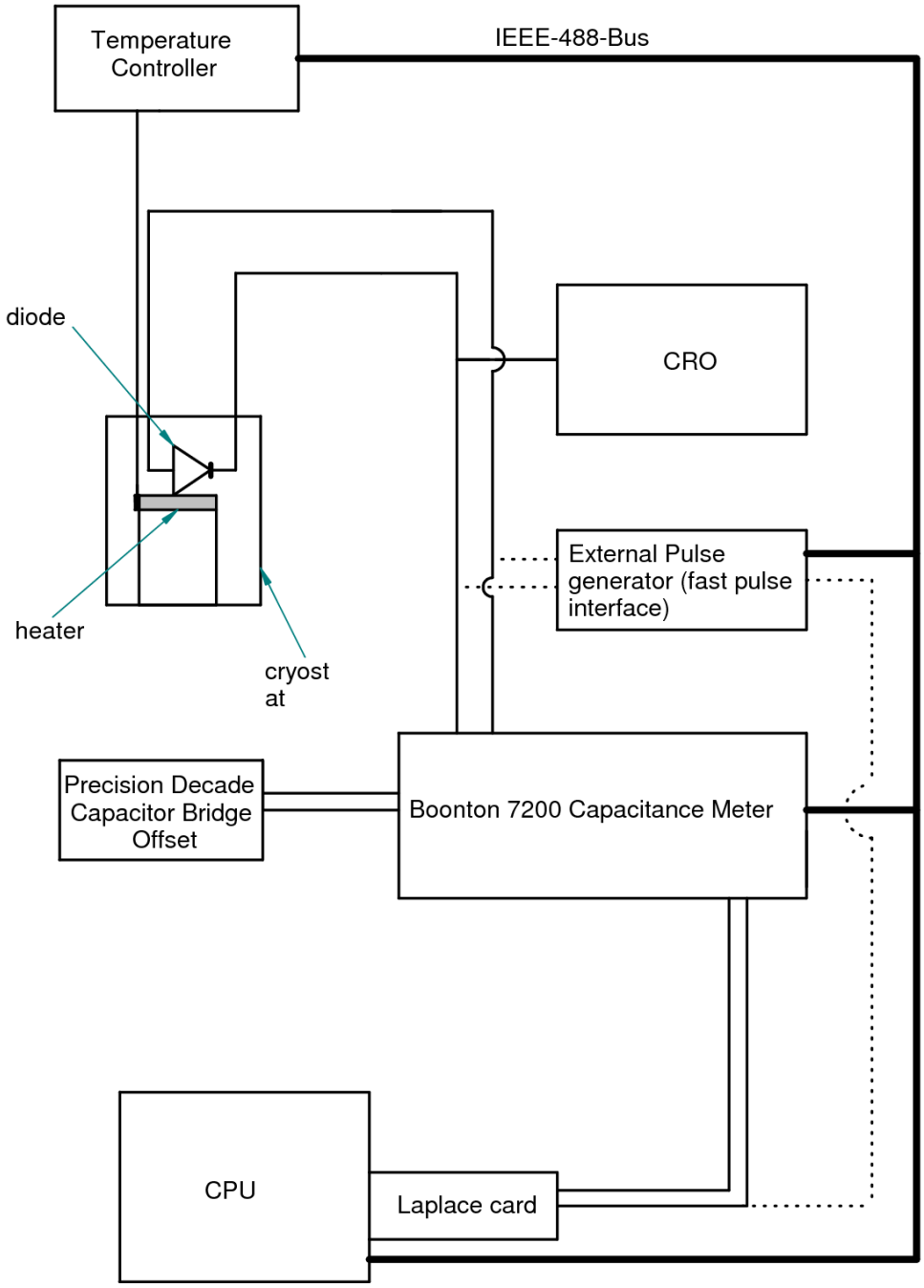

Figure 5.5: Schematic diagram of the DLTS and Laplace DLTS system. The dotted lines show the necessary connections when an external pulse generator is used.

As indicated in Figure 5.5, the sample was mounted in a cryostat. A closed cycle helium cryostat was used in the cooling from high temperatures to low temperatures. A heater located at the tip of the cryostat was used in raising temperature from the low values to high temperature values. The temperature was controlled by a Lakeshore 330 temperature controller. Thermal emission of carriers after excitation by the pulse generator was monitored using the Boonton 7200 capacitance meter with a 100 mV, 1MHz ac voltage signal. A Laplace card [1] with an internal pulse generator was used for generating the desired quiescent reverse bias voltage and pulses. It also contains software that is used for data collection and has a processing system which analyses and averages transients before displaying the spectra for both the conventional and Laplace-DLTS. The Laplace card is also used to record the capacitance-temperature (CT) scans.

Since the Laplace card could not produce well defined signals for short filling pulse widths, an external signal generator (fast pulse interface) was used in measurements that required the use of pulses with shorter filling pulse widths.

Conventional-DLTS and Laplace-DLTS sample excitation parameters are set up using the Laplace program. These include capacitance transient acquisition conditions, measurement initiation, transient acquisition and conversion into a DLTS or Laplace-DLTS spectrum. In the conventional-DLTS mode, the capacitance meter measures the capacitance transients after excitation. These transients are then processed by the Laplace card. Ramping up/down the temperature using a particular rate window, a DLTS spectrum is displayed on a computer. When one is using the Laplace-DLTS mode, the capacitance meter also monitors the capacitance transients after excitation at a fixed temperature. The Laplace card performs transient averaging and implements the inverse Laplace transform to calculate the signal magnitude and emission rates using three different routines; CONTIN, FTIKREG and FLOG [2] before a Laplace spectrum is displayed on the computer.

5.5 Sample annealing

Crystal annealing can at times help reduce defects in semiconductors and in some cases can also introduce them in a case where a certain defect species diffuse along a certain direction within the semiconductor. Annealing of samples was performed in a Lindberg Hevi-duty furnace. Depending on the type of atmosphere/ambient required, the furnace was connected

to a cylinder containing the gas required. The gas was supplied constantly at 3.0 L/min for the entire annealing period. Annealing times for each experiment are given in the results and discussion section.

Vacuum annealing was performed in a Lindberg furnace at a pressure of approximately 10^{-7} Torr. It must be noted that prior to annealing the samples were rinsed in methanol in an ultrasonic bath for five minutes and then blown dry using nitrogen gas. Sample preparation and defect characterization was performed as outlined in sections 5.1 – 5.4. Since it was observed that e-beam fabrication of contacts introduces defects, ohmic and Schottky contacts on annealed samples was performed using the resistive evaporation technique.

5.6 Proton irradiation

Sample irradiation on the deposited Schottky contacts was performed with a van de Graaff accelerator. An energy of 1.6 MeV and a fluence of $1 \times 10^{14}\text{cm}^{-2}$ were used. Immediately after irradiation, the samples were taken for contact evaluation using IV and CV measurements and defect characterization using DLTS and L-DLTS.

References

1. L. Dobaczewski, P. Kaczor, I. D. Hawkins, and A. R. Peaker, J. Appl. Phys. **76**, 194 (1994)
2. L. Dobaczewski, A. R. Peaker and K. B. Nielsen, J. Appl. Phys. **96**, 4689 (2004)