Several preparation techniques were investigated, including (a) dimpling and ion milling, (b) hand-held tripod polishing and (c) automatic tripod polishing, to prepare electron transparent samples of ZnO suitable for transmission electron microscopy (TEM). Thinned ZnO became prone to cracking below a thickness of ~20 microns during the use of each of these techniques. The extension of cracks, supposedly generated at micropores, was believed to be the cause of the brittle behavior of the material. A repeat dimple process was introduced to ameliorate this problem. In this procedure, the sample was dimpled while it is >200 microns, polished from the backside and then redimpled with a felt-polishing wheel. Ion milling with a low beam current (2-4), a low angle (8°-13°) and the use of a liquid nitrogen cold stage to reduce ion mill damage was subsequently employed. The investigation of an additional technique namely, focused ion beam thinning, generated high densities of defects in the ZnO that were extrinsic to the material and which prevented TEM studies of these samples. Micrographs of ZnO produced by the repeat dimple procedure confirmed the presence of dislocations and stacking faults in the material. TEM studies of gold contacts on the ZnO wafers confirmed epitaxial growth of the former and the delamination of the contacts during cooling from annealing in air at 175 degrees for 15 minutes.
Transmission Electron Microscopy Preparation and Characterization of Bulk ZnO and Au(111) / ZnO(000\(\bar{1}\)) Heterostructures

by

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A thesis submitted to the Graduate Faculty of North Carolina State University in partial fulfillment of the requirements for the Degree of Master of Science

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____________________________
Chair of Advisory Committee
Dr. Robert F. Davis
Dedicated to my parents

George and Joanne Kiesel

and to my brother Christopher
Biography

Sharon Kiesel was born in Poughkeepsie, New York on June 30, 1978 to George and Joanne Kiesel. She is four years younger than her brother, Christopher. Sharon grew up in Poughkeepsie until her family moved to Cary, NC in 1990. She attended Apex Middle School and through encouragement from teachers skipped pre-algebra to advance into the algebra class. This only encouraged her interests in math and science as she continued her academics at Apex High School. She was actively involved in Key Club and kept physically active in gymnastics, cross country, and track and field. As a senior, Sharon decided on a future career based on math and science. While visiting several departments of the College of Engineering at N.C. State for an Engineering Open House in the fall of 1996, she was thoroughly intrigued by the Department of Materials Science & Engineering. After two years as an undergraduate, she began working for Dr. K. L. Murty as a lab assistant under the graduate student Edward Preble in August of 1998. In May of 1999 she moved to a different lab under the direction Dr. Robert Davis. Here she worked with Edward Preble and Harmony McLean performing sample preparation of SiC-based samples for the transmission electron microscope (TEM). She began taking graduate courses the summer of 1999 to complement her work in the Davis laboratories. In May of 2000, Sharon received her B.S. in Materials Science & Engineering. Now a graduate student she continued her work for Dr. Davis, concentrating on the development of a TEM sample preparation technique for bulk ZnO and the TEM characterization of these samples. While in graduate school she joined the Materials Research Society, NCSU local chapter and served as Social Chairman in 2001 and 2002 and Treasurer in 2003. She was also very active in the Kiwanis Young Professionals of Cary serving as Fundraising Chairman for several years starting in August of 2001. She earned Chairman of the Year in 2002 and Kiwanian of the Year in 2003. For the duration of her graduate career Sharon served as a teaching assistant for introductory classes to Materials Science and Engineering and the TEM graduate course. She received her Masters of Science degree in December of 2003.
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Chapter 1: Introduction

1.1 Brief Overview of ZnO, Realized and Potential Applications and its Relationship to Other Semiconductor Materials

Zinc oxide occurs most commonly in the hexagonal wurtzite crystal structure \( (a_o = 0.325 \text{ nm} \text{ and } c_o = 0.5207 \text{ nm} \text{ at room temperature (RT)}) \), possesses a direct band-gap of \( \approx 3.4 \text{ eV} \text{ at RT} \) and exhibits spontaneous polarization along the \( \{0001\} \) directions. Realized applications commonly associated with powdered and bulk polycrystalline ZnO include paints, facial powders, sunscreens, gas sensors\(^2\), phosphors\(^3\), surface acoustic wave devices\(^8\), piezoelectric transducers\(^1\), varistors\(^4\) and textured polycrystalline transparent conducting films for the photovoltaic industry\(^5\). This material also has a high free exciton binding energy of 60 meV\(^1\) (compare with 21-25 meV for GaN), and it can be etched using wet chemical processes common in the semiconductor industry. As such, recent interest and research have focused on the growth of single crystal boules;\(^6\)-\(^9\) the acquisition, preparation and characterization of wafers from these boules; and the growth and characterization of thin films of ZnO for future applications in (1) optoelectronics including blue and ultraviolet light emitting diodes and lasers as well as ultra-violet detectors\(^1\) and (2) spintronics via the introduction of Mn to produce oxide-diluted magnetic semiconductors\(^10\).

High-purity crystals of ZnO are typically n-type. This donor characteristic has traditionally been attributed to native defects such as oxygen vacancies and zinc
interstitials\textsuperscript{11}. Supporting evidence for the former as the dominant electron donor has resulted from calculations of reaction rates\textsuperscript{12}, diffusion experiments\textsuperscript{13} and Hall effect measurements\textsuperscript{14}. Support for zinc interstitials as the dominant defect has been derived from considerations of ionic diffusion and the atomic radius of this element in ZnO.\textsuperscript{1, 15-18} However, recent theoretical calculations by Kohan, \textit{et al.}\textsuperscript{19} have shown that none of the native defects exhibit characteristics consistent with a high-concentration shallow donor. Van de Walle\textsuperscript{20} has noted that (1) only vacancies have sufficiently low energies to form in ZnO and (2) Zn and O vacancies act as deep acceptors and deep donors, respectively, and thus do not contribute to the n-type character of the unintentionally doped material. First-principals calculations by the last author have provided strong evidence that H (or OH\textsuperscript{−}) behaves as a shallow donor in ZnO. The OH\textsuperscript{−} radical can substitute for O\textsuperscript{2−} in ZnO in a manner similar to the substitution of fluorine in MO oxides and thus possess an effective charge of +1. This positive charge state allows this radical to become a shallow donor in ZnO and thus cause the n-type behavior. It can be incorporated in high concentrations via the formation of O-H bonds in the oxide crystal during growth and/or as a component of the hydroxide that invariably forms on the surface of this crystal when exposed to the normal laboratory ambient. If this theory is supported by experimental studies, this problem may prove difficult to surmount, as hydrogen is ubiquitous, difficult to remove from the crystal growth environment and produces a strong bond with oxygen.

The availability of n-type ZnO wafers and films makes possible the subsequent growth of acceptor-doped, p-type ZnO films and the formation of pn junctions which are the basis of most semiconductor devices. However, the realization of p-type ZnO has
been a significant challenge and a controversial topic. Several groups have reported the achievement of p-type material; however, at this writing, the mechanism of doping has not been explained, and other groups have not duplicated the reportedly successful experiments.

1.2 Methods of Growth of Boules of ZnO

1.2.1. Overview

Heteroepitaxial growth of ZnO has been accomplished via molecular beam epitaxy (MBE)\textsuperscript{23,24}, pulsed laser deposition (PLD)\textsuperscript{25-27}, laser MBE\textsuperscript{28}, and metal-organic chemical vapor deposition (MOCVD)\textsuperscript{29,30} on different types of substrates such as glass\textsuperscript{30}, $\text{Al}_2\text{O}_3$\textsuperscript{23-26,28,29} and GaN\textsuperscript{27}. However, the availability of single crystal wafers of ZnO is a considerable advantage to the technology in that it allows the homoepitaxial growth and doping of ZnO films. The growth of films of the same chemistry and crystal structure as the substrate dramatically reduces the formation of misfit and threading dislocations that occur during heteroepitaxial growth to relieve strain at the interface and within the growing film and that usually compromise the physical characteristics of the material. Large area wafers of GaN, the principal competitor of ZnO in the optoelectronics arena, have not yet been realized. The following subsections describe in detail the current most successful processes for producing boules of ZnO. One focus of the research of this dissertation has been the determination of the microstructure of these materials, because
of their importance to the future success of active devices fabricated in films grown on wafers derived from these boules.

1.2.2. Growth from a ZnO Melt

Several methods for producing moderately large ZnO boules have been successful including mining crystals from either a cooled skull melt or pulling crystals from this melt via Czochralski or modified Bridgman techniques\textsuperscript{8,9}, seeded vapor-phase deposition\textsuperscript{6}, and hydrothermal growth\textsuperscript{7}. Cermet Inc. produces bulk ZnO via the skull melting process that involves, as noted above, either the mining of crystals from a solidified melt produced from ZnO powder inside a solid ZnO crucible formed against a water-cooled chamber or the pulling of crystals from this melt. In this process, moderately pure (99.9\%) ZnO powder, acquired from KODOX-930 Zinc Corporation of America, Monaca PA, and high purity zinc are placed inside a crucible composed of very closely spaced, water-cooled copper fingers that are surrounded by RF coils. The crucible is contained inside a chamber that is pressurized with oxygen during operation. The metallic zinc initially couples to the RF field and is heated in tandem with the ZnO until the latter is sufficiently hot to also couple to the RF field such that it is subsequently melted. The process parameters include 2-10 atmospheres of oxygen, RF power levels of 2-20kW, a soak time at the highest temperature for 30-60 minutes and cooling at 50°C/minute. The resultant crystals mined from the melt are 3-4 mm in cross-section and 15-25 mm long. They have been reported\textsuperscript{8} to be n-type with a charge carrier density of $5 \times 10^{17}$ cm\textsuperscript{-3} (ZnO grown from the vapor phase usually has a charge carrier density of
Larger diameter crystals can be formed by pulling them from the melt via the use of the Czochralski technique or a modified Bridgeman approach. The personnel at Cermet Inc. believe that these processes are 10-100 times faster than the rates of growth currently achieved via vapor growth techniques and are less expensive\(^8\). They are certainly more rapid than the hydrothermal approach; however, the latter technique is much more easily scaled upward such that boules having both larger diameters and greater lengths can be produced at a lower cost because of the much lower temperatures employed.

### 1.2.3. Seeded Vapor Phase Growth

The Avionics Directorate at Wright-Patterson Air Force Base in conjunction with Eagle Pitcher Industries, Inc. and its recent spin-off company, ZN Technologies, and the University Research Center at Wright State University have produced 2-inch wafers of single crystal ZnO through seeded vapor-phase growth\(^6\). In this technique, ZnO is grown in a nearly horizontal tube having a hot and a cold end. At the hot end pure ZnO powder, formed from high-purity Zn vapor and O\(_2\), is held at 1150\(^\circ\)C. The resulting vapor is entrained in the H\(_2\) that is flowed over the powder, reacts with this carrier gas to form Zn and H\(_2\)O which are transported to the cold side of the tube containing a single crystal seed held at 1100\(^\circ\)C. The aforementioned reduction reaction is believed to proceed in reverse at the cold end of the tube to form ZnO on the seed. A boule of ZnO is created after 150-175 hours of deposition. Polished, “single crystal” 2-inch diameter wafers having a 1
mm thickness are subsequently cut from the boule. The microstructure of these wafers was determined in the research of this dissertation.

1.2.4. Hydrothermal Growth

Hydrothermal autoclaves at the Air Force Research Laboratory at the Hanscom AFB were used to grow boules of ZnO\(^7\). A sealed platinum liner was used to isolate the crystal growth environment from the steel walls of the autoclave. A nutrient consisting of 99.99% ZnO (Alfa Aesar) powder with a particle size <3\(\mu\)m was prepared by sintering in a platinum crucible in air at 1350\(^\circ\)C and placed in the bottom half of the autoclave. Platelets of ZnO seed crystals with the primary faces oriented along the \(<0001>\) directions were collected from previous hydrothermal growth runs and placed in the top of the autoclave. A mineralizer solution of Li\(_2\)CO\(_3\), 4 N KOH, and 4 N NaOH comprised 80% of the resulting vessel. The temperature of the nutrient was held at 355\(^\circ\)C with a declining gradient of 10 degrees to the seed. The gradient allowed for the less dense fluid of the dissolving ZnO powder to rise and precipitate on the cooler seed. Growth occurred along the \(<0001>\) directions at 10 mils per day. The growth period was approximately 30 days. The rate of crystal growth from the (0001) or zinc-rich surface was three times faster than that from the (000-1) or oxygen-rich surface. Photoluminescence data\(^7\) revealed that the optical character of hydrothermally grown ZnO is nearly identical to that grown via vapor transport by Eagle Picher.
1.3 ZnO as a Substrate for the Growth of GaN Films

Gallium nitride-based thin film heterostructures have been successfully fabricated into blue, green and ultra-violet light emitting diodes (LEDs) and blue-emitting laser diodes. The combination of red, green and blue light emitting diodes is currently being used, e.g., for large area displays, video billboards, scanners, optical storage and retrieval, traffic lights and flat panel televisions. This three-color combination of LEDs as well as the device configuration that employs the excitation of selected phosphors by AlGaN-based UV-emitting LEDs is being introduced commercially at this writing as sources of white light. Essentially all III-Nitride films and device heterostructures are currently grown on sapphire (Al₂O₃) or SiC substrates. The mismatch in lattice parameters between the films and the substrates cause high densities of misfit and threading dislocations densities to form. For example, the lattice mismatch in the [11.0] direction of GaN grown on (0001) sapphire is 16%\(^{31}\). This results in a dislocation density as high as 10⁹-10¹⁰ /cm². As such, progress in the development of significantly improved III-Nitride-based optoelectronic as well as microelectronic devices is facing barriers created by the high densities of dislocations present in these materials.

The crystal structure, band gap energy at room temperature, lattice parameters and donor character of unintentionally doped ZnO are similar to the characteristics exhibited by gallium nitride (GaN) (\(E_g = 3.4\) eV) and 4H-silicon carbide (SiC) (\(E_g = 3.2\) eV)\(^1\). There is a 2.2 % lattice mismatch between ZnO and GaN in the \(<11.0>\) directions at room temperature, and ZnO and In\(_{0.22}\)Ga\(_{0.78}\)N have a perfect match in their \(<11.0>\) lattice
constants at room temperature. As such, blue light-emitting diodes have been produced from n-ZnO/p-GaN heterostructures. Thus ZnO would seem to be a better choice for a substrate than either sapphire or SiC. However, Detchprohm et al. and Johnson et al. have determined that defects in the ZnO are produced at the GaN/ZnO interface that thread into the GaN layer. Additional problems with the use of ZnO as a substrate for III-Nitride films include the limited supply ZnO wafers with low densities of micropipes and stacking faults as well as the instability of ZnO in a reducing atmosphere such as H₂ at high temperatures.

1.4 Previous TEM Studies of Bulk ZnO

Transmission electron microscopy (TEM) is an important characterization technique used to identify the crystallographic form as well as the imperfections in a sample including precipitates, dislocations, point defects, and strain contrast, among others. J. Nowok reported TEM investigations of sublimation grown, polycrystalline ZnO. Other investigators have subsequently conducted TEM studies of powdered ZnO and single crystal thin films on sapphire. However, due to the expense and tedious, time consuming preparation steps very few, if any, TEM studies of bulk ZnO have been published at this writing. McLean, et al. have recently submitted for consideration for publication, papers derived from her thesis concerned with TEM studies of bulk ZnO obtained from Eagle-Picher, Inc. and Cermet, Inc as well as heteroepitaxially and homoepitaxially grown thin films of this material. This writer is a co-author on these papers. The TEM images derived from this study reveal stacking faults in the bulk
materials obtained from both sources. This is not surprising, as Sagalowicz has noted from the results of his studies that planar defects are common in materials having the wurtzite structure. A schematic of a stacking fault in this structure is shown in Figure 1. Lower magnification images of the Eagle-Pitcher material revealed strain contrast. High-resolution images of the same material revealed single and double stacking faults with displacements of $1/6 <2\overline{2}03>$ and $1/3<1\overline{1}00>$. The stacking sequence is altered from AaBbAaBb to AaBbAaBb/CcBbCc where the capital letters are anions (e.g. oxygen) and the lower case letters are cations (e.g. zinc). Edge dislocations were also found in this material. Bulk ZnO provided by Cermet, Inc. also showed strain contrast in the low magnification images; however, it was not as prevalent as in the Eagle-Picher material. High densities of edge dislocations and stacking faults, similar to those found in Eagle-Picher material, were revealed in the high magnification images. Although the material from Cermet, Inc. appeared to be more suitable for device fabrication, the Eagle-Picher material was chemomechanically polished and thus provided a better surface for the growth of ZnO thin films. Suscavage et. al. have investigated the PL spectra acquired from their hydrothermally grown material that was polished either mechanically or chemomechanically via a technique developed for $\{0001\}$ surfaces of ZnO by Eagle-Picher, Inc. The amplitudes of the spectra acquired from the chemomechanically polished surfaces were significantly greater than those obtained from the mechanically polished surfaces of the same orientation.
Additional TEM studies by McLean\textsuperscript{39} of ZnO films heteroepitaxially grown by Smith et al.\textsuperscript{42} on GaN films that had been previously deposited on AlN/SiC(001) substrates revealed columnar or textured growth, conical crystallites with similar orientation to their surrounding material and threading dislocations that were derived both from the underlying GaN film and that were generated at the ZnO/GaN interface due to relief of the strain generated by the misfit in the (110) lattice parameters. As noted above, threading dislocations act as recombination centers, reduce electron velocities, degrade lifetimes of optoelectronic devices, and cause premature breakdown and leakage currents at low voltages. As such, homoepitaxial films of ZnO were also grown by T.
Smith and investigated via TEM by McLean. The diffraction patterns revealed double diffraction spots at (0001) and (000 $\overline{1}$) due to the homoepitaxial ZnO epilayer. As expected, the homoepitaxial ZnO films did not exhibit columnar growth and did not contain conical crystallites. Misfit and threading dislocations were also absent in these films. However, the homoepitaxial films contained a significant density of stacking faults. These are thought to be intrinsic due to the low stacking fault energy of ZnO.

1.5 Nature of Dissertation Research

This thesis investigates TEM studies of bulk ZnO and primarily the processing steps in developing a proper sample preparation method. Many approaches were made in creating a thin region of bulk zinc oxide material for analysis in the transmission electron microscope (TEM). Chapter 2 describes all of the methods attempted and the method that worked best. First, the same approach used to create TEM samples for silicon carbide was attempted. However, ZnO tends to be mechanically weaker perhaps due to microporosity so alternative approaches were attempted. These include hand held tripod polishing, in which a separate stage for the optical microscope was created; automatic tripod polishing; focused ion beam and a repeat dimple method that produced useful TEM samples. Chapter 3 will show results of defects in Eagle Picher, Inc. seeded vapor phase grown bulk ZnO wafers obtained in the optical microscope as well as the TEM. Optical images show micropipes, the source of microporosity in bulk ZnO and TEM images show defects in bulk ZnO. Gold contacts deposited on ZnO (000 $\overline{1}$) and annealed gold contacts on ZnO (000 $\overline{1}$) will be presented in Chapters 4 and 5. Investigations to
sample preparation techniques as well as conclusions on defects and TEM images are included in Chapter 6. Suggestions for future TEM sample preparation of bulk ZnO wafers is presented in Chapter 7.
1.7 References


42. T. Smith, et. al., submitted to *J. Crystal Growth*. 

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Chapter 2: Experimental Procedures

2.1 Introduction

Cross-sectional TEM samples are achieved via several different process routes. Ultramicrotomy\(^1\) involves the use of a knife, usually diamond, to cut electron transparent sections from a larger sample. This technique is mainly used for “soft” samples including biological tissue, catalysts, polymers and some metals. Other techniques used for much harder materials including semiconductors include focused ion beam (FIB) sectioning\(^2\), acid etching\(^3\), wedge cleaving\(^4\), and the tandem procedures of surface grinding and dimpling\(^6\). The focused ion beam technique is also useful for integrated circuits when one particular area of interest must be analyzed. However, it is a slow process, very expensive and can cause artifacts in some materials. Acid etching or chemical thinning is a convenient technique used to reduce or eliminate ion milling time, artifacts and other defects created during mechanical thinning. Twin-electrojet polishing is the most common technique. Etching rates must be known for the material; however, the rate by which it attacks the damaged areas differs from that of damage-free regions making it difficult to control the thickness of the sample\(^3\). Wedge cleaving is neither as time-consuming nor as expensive as the previous techniques; however, it requires considerable practice to achieve a good sample.

For most semiconductor materials the conventional method of preparation involves cutting specimens of the sample and support material to form a sandwich in which the film of interest is in the middle. These sandwiches are then polished on one
side, dimpled from the backside and ion milled until electron transparency and perforation are achieved, as indicated by the transmission of light through the hole to a photocell which activates a relay that stops the milling process. For materials such as silicon this is very useful; however, for optically transparent materials such as silicon carbide, electron transparency must be determined by other methods, unless the wavelength of the illumination is above the energy of the band gap of the milled material. An alternative method involving Nomarski interference contrast microscopy has been employed in conjunction with different thinning methods for the preparation of TEM samples of GaN/AlN/SiC heterostructures as described by E. A. Preble et al. This method produces large electron transparent areas.

2.2 Dimpling and Ion Milling of SiC- and ZnO-based Materials

The initial technique employed in the research of this dissertation for the preparation of TEM samples of ZnO-containing materials mirrored that used for silicon carbide (SiC). In general, this process involves sectioning, sandwiching, polishing, placing the sample on a copper TEM ring, dimpling and ion milling. Two pieces of the material of interest are cut into 0.1-0.15 cm X 0.29-0.38 cm pieces. The average thickness of a sample is less than 1 mm. Two pieces of the material under study are sandwiched together with the surfaces of interest placed facing inward and in opposition to each other. Pieces of silicon or sapphire are subsequently glued on the outside faces of the sandwich for support and a total of four layers. Between each of these layers is a 10:1 resin and hardener epoxy mixture. The G2 Epoxy Kit was purchased from Gatan, Inc.,
part number 601.07270. The sandwich is subsequently heated to 130°C for 30 minutes to cure the epoxy mixture and cooled to room temperature.

In the particular case of the preparation of SiC samples, each assembly is lapped on one side with a rough grit paper to flatten all four material phases. The sample is then sequentially polished using 30 micron, 15 micron, 6 micron, 3 micron and 1 micron diamond papers and a colloidal silica slurry on a Buehler Micro-cloth paper. Each sample is then inverted with the polished side down, attached to a copper ring with the 10:1 resin and hardener epoxy mixture. The samples are again cured at 130°C for 30 minutes and flattened with either a rough grit paper or 30 micron diamond paper to a thickness of 70-100 microns.

Figure 2.1: (a) Optical micrograph showing interference fringes in GaN/Al/6H-SiC where repeated green and pink rings represent a thicker region and the white/grey region represents a thinner part of the sample. (b) The color pattern is indicative of the sample thickness. For example, the blue ring is approximately 6 microns thick.\textsuperscript{5}
Each sample is then dimpled using a Model 656 Gatan Dimple Grinder, 3 micron diamond paste and a starting weight on the sample of 20 grams. The approximate thickness of the sample is monitored on a dial indicator. The actual thickness of the material is determined using an Olympus BX60 light microscope in the differential interference contrast (DIC) mode. This mode allows one to observe the Nomarski color fringes of a transparent material, which vary with thickness. The color progression as a function of thickness of single crystal GaN/AlN/6H-SiC is shown in Figure 15. In the specific case of SiC substrates, the weight is changed to 10 grams when the color in the sample is approximately at the first yellow/orange range or approximately 9-10 microns thick. When the SiC sample is approximately 2 microns thick, i.e., within the second yellow/orange range it is polished with a felt wheel containing 3 micron diamond paste. When as many scratches possible are eliminated the sample is removed from the dimpler, cleaned with acetone and methanol and placed in the ion milling. A Gatan Dual Ion Mill set at 6 kilovolts and at an angle of 13 degrees was employed for the GaN/AlN/6H-SiC studies until a hole was produced near the interface.
Figure 2.2: (a) Interference fringe palette in ZnO. (b) Optical micrograph of a dimpled sample. Note the central gray area, which is indicative of a sample thickness of less than 2 microns. The sample becomes thicker as you move away from the center. This sample was produced using the final sample preparation procedure described in subsection 2.6.

The grinding, dimpling and ion milling procedures used for the preparation of ZnO-based TEM samples in the present study were similar to those described above for SiC. Figure 2 shows the interference fringe pattern for ZnO used in all of the procedures to determine the thickness of a sample below 100 microns. However, the ZnO samples contained flaws, which made them more susceptible to cracking and failure during processing than the SiC samples. For this reason, the ZnO samples were flattened using 6 micron diamond paper and polished on the 3 micron and 1 micron diamond papers and
on the Buehler polishing Micro-cloth containing colloidal silica slurry. Moreover, a smaller starting weight of ~10 grams and a subsequent weight of 5 grams or less were used in the dimpling process for the ZnO samples. A lower gun voltage, usually around two-to-three kilovolts, and a liquid nitrogen stage were used in the ion milling process. However, the ZnO samples still cracked under the pressure applied to the diamond-impregnated ball used to dimple the sample, and the ion milling showed little advancement in thinning the material. Moreover, ion milling-induced damage occurred in the samples after long milling times, which caused the TEM results to be misleading. As such, other thinning techniques were investigated, as described below.

### 2.3 Hand-Held Tripod Polishing

The initial step in this investigation involved thinning the sample into a wedge by hand using a South Bay Technology, Model 590W hand held tripod polisher. As only one side of the cross-sectional ZnO sample was of interest, an attempt was made to create a wedge along the width of the sample. This caused the sample to crack and break. Therefore, the sample was thinned along its length. Prior to this step, polishing was employed to level the three legs of the polisher. The equipment was held with the legs downward over the 8-inch automatic polishing wheel of the Buehler Ecomet III Polisher Grinder. The wheel contained a special 600-grit Buehler Carbimet SiC grinding paper. A piece of Pyrex was then attached in front of the front leg and was leveled with all three of the legs. To check the planarity of the Pyrex, a glass slide was used to insure alignment. The sample was subsequently waxed onto the Pyrex, and the front leg raised
such that it would not interfere with the polishing. The back two legs were then adjusted 15-25 marks or 150 – 250 microns to angle the sample as it was being polished. The hand-held polisher was placed on the 8-inch automatic wheel with the Pyrex and back legs facing downward. Running water was used as the lubricant, and the sample was sequentially polished using 6 micron, 3 micron and 1 micron diamond Buehler paper and a colloidal silica slurry on a Buehler Microcloth. To obtain uniform thickness across the width of the sample, the Pyrex side of the hand-held tripod polisher was swept from the middle to the outside of the wheel as it rotated. To determine the thickness of the ZnO sample, the tripod polisher was turned upside down such that the legs and the Pyrex were facing upward, and the sample was observed under an Olympus BX60 light microscope. Nomarski fringes were used to monitor thickness. The color pattern for ZnO is shown in Figure 2.

Many attempts were made to produce uniform thickness across the sample; however, this was not achieved. One reason may be that the rate of removal of material from the sample was much faster than from the back legs of the tripod polisher. Moreover, one of the legs of the latter was polished on the inner region of the 8-inch polishing wheel while the other on the outer region of the wheel; thus, the legs were polished at different rates and, consequently, were at different heights when the process was completed. Another reason could be that the tripod polisher was held by the operator, and the angle of the sample, in comparison to the wheel rotation, was not uniform even when it was swept back-and-forth across the radius of the wheel. Whatever the reason(s) the polished sample was not level from either front-to-back or side-to-side. Figure 3(a) shows an example of one sample that was not uniformly polished. This made
it difficult to produce a thin area on the desired part of the sample. It was also very
difficult to discern the amount of sample removed after a given amount of time. Crack
formation occurred, as shown in Figure 3(b), when the thickness of the sample was <1
micron.

![Figure 2.3: Optical micrographs of (a) ZnO sample containing fringes indicative of
thickness non-uniformity produced during the use of the hand-held tripod polisher, (b)
completely fractured ZnO sample produced during the use of the hand-held tripod
polisher to achieve a sample thickness of ≤1 micron, and (c) ZnO sample containing
multiple cracks produced during the use of the automatic tripod polishing.](image)

2.4 The New Microscope Sample Stage

The original configuration of the sample stage supplied with the microscope
included two parts: the support piece and the stage. The support piece was attached to
the back of the microscope and was moved up-and-down with the focus knob. It also extended from the microscope and contained a circular piece that allowed the attachment of the stage. The original stage had two levels. The bottom level was immobile and attached to the support piece. The top level held the sample having a maximum allowed height of ¾ inch and allowed easy x- and y-axis movements of the stage. Because of the limited sample height that could be viewed, a new stage was fabricated that (1) supported the hand held polisher underneath the objective lens such that the sample was flat and (2) allowed the sample to be shifted in-and-out of focus as the focus knob was turned with sufficient clearance underneath the objective lens. Since there were several users of the microscope, the new stage would have to be easily interchangeable with the original stage.

Measurements were taken of the original support piece, including the dimensions of its attachment to the back of the microscope, the length to the circular piece, and the dimensions of the circular piece. To keep the new stage attached to the backside of the microscope, a hole for a screw was drilled with the same dimensions as the original. Measurements were also taken of the height of the original stage in relation to the support piece. This information was used in conjunction with the height of the hand-held polisher on its holder to determine the distance that the new support piece would hang. At this point the "stage" is a block of material that attaches to the back of the microscope. Dimensions for material removal were calculated with respect to the height of the hand held-tripod polisher and its holder. Viewing the thickness fringes is easiest at a 45-degree angle; thus, the sides of the support piece were eliminated along parts of the edges. A hole similar to that in the original stage was drilled into the bottom of the
support piece to allow back-lighting of the sample. Finally, since the new support piece was longer than the original support piece, part of the microscope was blocking the new support from descending completely when adjusting the focus knob. Therefore, a hole was drilled underneath the back of the new support piece to allow sufficient clearance for the desired range of motion.

A middle piece was also fabricated to allow the hand-held polisher to be interchanged within the microscope with samples having a height less than ¾ inches. The middle piece has the same circular dimensions as those of the original support piece. It extends towards the microscope and contains wings on the bottom that slide into the new support piece. The middle piece stays attached to the original stage making it easy to attach and detach the original stage. Small samples can now be viewed on the original stage with the new support piece. Samples on the hand-held tripod polisher are also easily viewed by simply sliding the middle piece out of the new support piece.

Photographs of the original microscope, the drawings for the new support piece and the final microscope arrangement are presented in Appendix A.

2.5 Automatic Tripod Polishing

The principal problems inherent in the use of the hand-held tripod polisher were related to human error. As such, the use of an automatic tripod polisher was investigated. A Multiprep and Techprep 8 produced by Allied High Tech Products, Inc. was used to produce a wedged-shaped sample. Selected ZnO samples were sandwiched with another ZnO piece or sapphire; however, most were not prepared in this manner due to lack of
material. Initially, the machine was set-up by leveling the polishing wheel and polishing the sample holder such that it was level with the wheel. The two micrometers were adjusted 15 marks or 150 microns, which did not change during the polishing process. The sample was attached to the front of the Pyrex piece with super glue. The ZnO samples were polished using 6 micron, 3 micron and 0.5 micron diamond-impregnated papers acquired from Allied High Tech Product, Inc. The thickness of the sample was estimated using a digital dial indicator, which determined the amount of material removed by measuring the decrease in the height of the apparatus. The Pyrex piece allowed the removal and replacement of the sample for analysis of the Nomarski fringes under the light microscope without the new microscope attachments. When the sample reached the desired thickness it was removed from the Pyrex sample holder with acetone and cleaned in methanol. Ideally, it was desired to thin the sample to a thickness of less than one micron.

A copper ring was used as a support for the TEM sample. The sample was placed on the ring with a 10:1 resin and hardener epoxy mixture and cured in an oven at 130°C for 30 minutes. A wedged-shaped sample having a thickness of less than one micron at one end of the sample was placed into the TEM. This sample was not transparent to electrons at any location. Also, as the sample was progressively thinned, it became more susceptible to cracking. The use of the Multiprep machine has allowed researchers to achieve sufficiently thinned SiC samples. However, most of the thinned ZnO samples investigated in this research did not have sufficient mechanical strength, due to microporosity and susceptibility to the extension of flaws under low stress. Figure 3(c) shows the cracks that formed in a sample during the use of the automatic tripod polisher.
2.6 Focused Ion Beam

Selected ZnO samples were prepared at NCSU for sectioning into very thin samples via the use of a focused ion beam. They were initially cut into 0.1-0.15 cm X 0.29-0.38 cm pieces. The faces of these sections were polished using 6 micron, 3 micron and 1 micron diamond papers acquired from Buehler, Inc. and a felt wheel containing colloidal silica. Each sample was then glued polished side down to a copper ring that had been cut into a horseshoe shape; the desired region of the sample was located in the opening in the ring. An epoxy mixture of 10:1 resin and hardener, cured at 130°C for 30 minutes, was used as the adhesive. One sample was then placed on a flattened and leveled Mutliprep sample holder with Super-glue®. The Mutiprep micrometers were not changed; thus, the sample remained flat during polishing. Each sample was sequentially polished along its length to a thickness of 20-30 microns on 6 micron, 3 micron and 0.5 micron diamond papers obtained from Allied High Tech Products, Inc. The samples were then polished using colloidal silica slurry on a red felt wheel. The digital dial indicator and the Nomarski fringes in the Olympus optical microscope were used to monitor the thickness. The samples were then removed from the Pyrex sample holder with acetone, cleaned in methanol and shipped to Dr. Robert Fitch at the Wright Patterson Air Force Base (WPAFB) in Ohio for additional thinning using the focused ion beam (FIB) technique.

The sample in the Dual Beam System from FEI, model Strata DB235 was thinned to a very narrow 10 um x 10 um or smaller section using a gallium source for the ion
beam which was oriented at 52 degrees from the e-beam column. The FIB technique is demonstrated in Figure 4. Gallium ions form a focused ion probe used to sputter the material. The ion beam current used was 20,000 pA to rough out material on both sides of the final section, and was then reduced to smaller and smaller values as the sample grew thinner, each time polishing the surface of both sides of the section. It is routine for a metal coating to be placed on the sample to protect it from damage at the surface. The initial samples sent to WPAB were not coated with any metal protecting layer therefore promoting more damage to the surface of the sample and to the section of interest. Later in the research efforts, some samples were coated with Pt. Pt was deposited using a gas injection source. This source was introduced into the chamber via a small needle close to the sample. As the gas flowed into the ion beam it was vaporized and deposited onto the sample per a predefined pattern.

![Ion Beam Diagram](image1)

(a) Sample is mounted onto a copper ring with the desired region facing outward to be thinned by the ion beam. 

![Metal-Coated ZnO Sample](image2)

(b) A metal-coated ZnO sample prepared for TEM studies using the FIB technique. Too much material was removed in the FIB process to obtain useful information regarding the microstructure of the sample.

Figure 2.4: (a) Sample is mounted onto a copper ring with the desired region facing outward to be thinned by the ion beam. (b) A metal-coated ZnO sample prepared for TEM studies using the FIB technique. Too much material was removed in the FIB process to obtain useful information regarding the microstructure of the sample.
Three significant problems were encountered in this process. There were considerable delays between the times of the receipt of the samples at (WPAFB) and the use of the FIB. Secondly, although the amount of material removed using the FIB was relatively unimportant for the unimplanted ZnO samples, too much material was removed from the ion-implanted samples and from the samples on which a metal film had been deposited, as shown in Figure 4. Finally, milling damage was present in all the samples, including those coated with Pt, which significantly obscured the internal microstructure of the samples during the TEM studies.

2.7 Repeat Dimple TEM Preparation Technique for ZnO Based Materials

A final technique was devised such that a portion of a ZnO sample could be thinned to electron transparency without cracking. In this process route a sample is sandwiched between two sapphire support pieces using either the 10:1 resin and hardner epoxy mixture from Gatan, Inc. or the M-Bond from Allied High Tech Products, Inc. As noted above, the former is cured at 130°C for 30 minutes. The latter is cured at 120°C for 2 hours. The sandwich is then placed on a glass peg with a generous amount of wax and flattened on one side using 9 micron diamond paper. The sample is thinned on the flat side to a depth 30-35 microns using an automatic dimpling procedure on the Gatan Dimple Grinder, Model 656, 3 micron diamond paste and 10 grams of weight on the sample holder. The sample is then polished on a felt wheel embedded with 3 micron diamond paste to eliminate as many scratches as possible and the four corners surrounding the dimple are flattened with 3 micron diamond paper.
A dot of wax is subsequently applied to a clean glass peg heated on a hot plate at approximately 80°C. An even smaller amount of wax is placed on the acetone-cleaned sample in the middle of its dimple. When all of the wax has liquefied the sample is inverted onto the glass peg with the side containing the dimple and flat corners facing downward. Upon removal from the hot plate, the flat end of a wooden stick is used to apply light pressure to the top of the sample to ensure the least amount of wax underneath the sample. The sample is then polished using 9, 6, 3 and 1 micron diamond papers to a thickness of 30-50 microns, as determined using the colors of the Nomarski fringes in the manner described above. A copper ring is then glued onto the flat, polished side of the sample using the resin and hardener epoxy mixture or M-bond. After the sample is inverted with the copper ring and the flat side of the sample facing the glass peg, it is again dimpled using 1 micron diamond paste on a felt wheel and a weight of 10 grams until a white/grey Nomarski fringe is achieved. As a final step, the sample is placed on a liquid nitrogen-cooled stage in the ion mill. Material is removed using a gun voltage of 3 keV at an angle of 8 degrees for 0.5-3 hours.

Using this technique eliminated cracking as long as no lap polishing occurred below a thickness of 20 microns. By dimpling the material while it is fairly thick allows more support for the material so that it can withstand more pressure. As the sample grew thinner it also grew weaker. Also, by polishing on the dimpler one can eliminate any scratches, which can be a source of crack propagation. After the sample is thinned to around 30-50 microns a dimple is already present so the sample will dimple easily to its newly defined shape. Polish dimpling at this point proved crucial when removing material without inducing scratches. One problem that arose was the separation of the
ZnO sandwich during the thinning step. It is important to create a tight sandwich so that (1) the sample can protect itself from ion mill damage and (2) a glue line is maintained so that the surface of the material can be identified in the TEM. This proved important to begin the dimpling process for a second time at around 30 microns in order to maintain a tight sandwich and to allow for the attachment of the copper ring, which added support.
2.8 References


Chapter 3: Optical and Transmission Electron Characterization of Bulk ZnO

3.1 Introduction

The formation of microporosity in single crystal boules of semiconductor materials during growth can seriously compromise the properties of devices subsequently fabricated on wafers of this material or on films grown on these wafers wherein the porosity is manifest in the former. Frank\(^1\) predicted that a hollow core would form if the Burgers vector of a screw dislocation exceeded a critical value in a crystal with a large sheer modulus. Dudley et al.\(^2\) subsequently limited the definition of a micropipe to those that form in hexagonal close packed (HCP) materials. In this modification of the definition the Burgers vector of the hollow core can only be multiples of a crystal’s c-axis lattice parameter. Screw dislocations with small Burgers vectors are neither hollow nor as destructive to the characteristics of devices.

Micropipes are common in boules of HCP materials such as SiC and ZnO. A better understanding of their behavior has been determined via synchrotron white beam x-ray topography (SWBXT)\(^3\), scanning electron microscopy (SEM)\(^3,4\), Nomarski optical microscopy\(^3\) and transmission electron microscopy (TEM)\(^5-8\). The progenitors that cause these defects in SiC have been determined to be, e.g., second phase inclusions on the growth surface including silicon droplets\(^9\) and carbon inclusions\(^10\) and metallic deposits\(^11\) and stacking related defects.\(^7,12,13\) Micropipes can also form due to cross-slip of partial dislocations onto hexagonal prism planes\(^12\) and the recombination of screw dislocations...
that originate at polytype boundaries\textsuperscript{13} and at the interface of domains and grain boundaries\textsuperscript{7}.

Transmission electron microscopy (TEM) is used to characterize atomic structure as well as imperfections in a sample including precipitates, dislocations, point defects and strain contrast, among others. TEM studies have been performed on polycrystalline\textsuperscript{15}, powdered\textsuperscript{16} and single crystal films\textsuperscript{17-19}. However, very little TEM studies have been reported on bulk ZnO. Studies performed by McLean\textsuperscript{20} revealed the microstructures with wafers of ZnO received from Eagle Picher, Inc and Cermet, Inc\textsuperscript{21,22} as well as heteroepitaxially and homoepitaxially grown thin films of this material. The high and low resolution images derived from this study revealed numerous stacking faults and dislocations; the former are common in materials having the wurtzite structure\textsuperscript{23}.

3.2 Experimental Procedures

ZnO wafers investigated in this research were received from Eagle Picher, Inc. The ZnO boules were created via the seeded vapor-phase technique.\textsuperscript{14} In this process ZnO powder is held at 1150°C at one end of a nearly horizontal tube. A single crystal ZnO seed is positioned at the opposite end and heated to 1100°C. Hydrogen is flowed over the powder to form Zn and H\textsubscript{2}O that are transported to the lower temperature end of the tube where the reverse of the above reaction occurs to form ZnO on the seed. A two-inch diameter boule of ZnO is created after 150-175 hours of deposition. The wafers used in this research were cut from such a boule and polished to a thickness of \~0.50-0.80 mm. Two grades of material were received from Eagle Picher, Inc. Grade I was believed
to contain no micropipes. Grade II material, which contained many micropipes was used in this research project.

Selected ZnO wafers were cut into ~0.01 x 0.03 cm pieces using a Buehler low speed saw and viewed in cross-section in the Olympus BX60 optical microscope. The differential interference contrast (DIC) and dark focus (DF) settings were used. A Kodak DC120 Zoom digital camera was attached to the microscope with a Kodak MDS 120 universal adaptor. Digital pictures were captured using Adobe Photoshop 5.0.2 and Kodak MDS software.

TEM samples were prepared by sandwiching two ZnO pieces, each having the dimensions of ~0.1 x 0.3 cm, between two sapphire support pieces of the same size using M-Bond epoxy from Allied High Tech Products, Inc. The epoxy was cured at 120°C for 2 hours. The sandwich was then flattened on one side using 9 micron diamond paper and dimpled to a depth 30-35 microns on the Gatan Dimple Grinder, Model 656 with 3 micron diamond paste and 10 grams of weight on the sample holder. The sample was subsequently polished on a felt wheel embedded with 3-micron diamond paste to eliminate as many scratches as possible. The four corners surrounding the dimple were flattened with 3-micron diamond paper.

The sample was then inverted onto a glass peg with wax and polished using 9, 6, 3 and 1 micron diamond papers to a thickness of 30-50 microns, as determined using the colors of the Nomarski fringes for ZnO shown in Table 3.1. A copper ring was then glued onto the flat, polished side of the sample using M-bond. As a final thinning step, the sample was placed in an ion mill. Material was removed using Ar+ ions generated using a gun voltage of 3 keV at an angle of 8 degrees for 3 hours. Each sample was also
mounted in a liquid nitrogen-cooled stage that lowered its temperature and decreased the damage caused by the impinging beam of ions.

Table 3.1: Nomarski thickness fringe pattern for ZnO.

The JEOL 2010F TEM operating at 200kV at the North Carolina State University was used to analyze the samples with the assistance of Dr. Alex Kvit. Bright field and dark field imaging was used to capture low magnification pictures in a 2-beam condition. Selected area electron diffraction (SAED) was utilized to capture diffraction patterns.
3.3 Results

3.3.1 Microporosity in ZnO and Related Materials

During the course of cross-sectional TEM sample preparation it was concluded that only low pressures, i.e. 10 grams of weight on the dimpler, must be applied to the sample to markedly reduce the possibility of crack nucleation from the walls of the micropores. Optical microscopy of micropipes found in the bulk material are shown in Figures 1 and 2. Both figures were acquired from wafers that had been polished on both sides by Eagle Picher, Inc. No further investigation of these defects was performed. Wurtzite ZnO has a crystal structure that is related to those of the 6H and 4H polytypes of alpha-SiC, in which the origin and the nature of the micropipes have been extensively investigated. However, similar investigations of these defects have yet to be conducted in bulk ZnO.
Figure 3.1: Optical microscope images of micropipe-like features (marked by yellow arrows) found in cross-section at different depths in a double polished bulk ZnO wafer from Eagle Picher, Inc. These images were acquired in (a) the Nomarski DIC mode and (b) dark field. (c) A higher magnification image of the lower micropipe-like feature acquired in the Nomarski DIC mode. Only two micropipe-like features were found in this sample piece with an approximate area of 16 mm² after cross sectioning.
Figure 3.2: An optical image of an Eagle Pitcher sample polished down to a micropipe-like feature. These images were acquired at (a) low magnification (100X) and (b) higher magnification (200X).

3.3.2 TEM of Bulk ZnO

Bright field and dark field low magnification images acquired through a two-beam condition are shown in Figure 3. In two-beam condition one beam other than the central beam is strong providing good contrast. Thickness fringes were common in this part of the sample, however, stacking faults and dislocations are also apparent. The orientation of the specimen for these images is (1120). Bright field images are obtained from the direct beam of electrons traveling through the sample. Dark field images are acquired by the collection of scattered electrons. Both show strong contrast, which reveals defects.
Stacking faults and dislocations were found throughout other parts of the sample as well. Figure 4 emphasizes some of these defects. Stacking faults are classified as those that alter the local stacking sequence from the normal $\text{AaBbAaBb}$ to $\text{AaBbAaBb/CcBbCc}$. The capital letters indicate anions (e.g., oxygen) and the lower case letters indicate cations (e.g., zinc). Only a portion of the defects visible are highlighted. No further investigation of the types of defects present or their displacement directions was performed.
Figure 3.4: Stacking faults and dislocations visible in a bright field image.
3.4 References


5) *Philosophical Magazine A (UK)*, 81, No. 12, 2885 (2001).


Chapter 4: TEM Characterization of Au (111)/ZnO(000\(\bar{1}\)) Contacts

4.1 Introduction

According to the Schottky-Mott model, metals with a high work function are prime candidates for Schottky (rectifying) contacts on n-type semiconductors\(^1\). Metals investigated at this writing for Schottky contacts on n-type ZnO include Ag\(^2,3,4\), Pt\(^2\), Pd\(^2\) and Au\(^2,5,6\). Rabdanove et. al.\(^2\) considered Pt, Pd and Au to be the principal candidate metals, since Ag has an affinity for oxygen. The reported high leakage currents\(^2,4,7,8\) and the relatively high ideality factors\(^6,9\) for these metals, e.g., gold\(^2,5,6\) are believed to be due to surface contamination that was not removed prior to deposition of the contacts.

The thermodynamic mode of growth of Au on the oxygen-terminated (000\(\bar{1}\)) face of remote plasma cleaned, single crystal wafers of ZnO has been investigated\(^10\) in the Davis laboratories using sequential depositions to achieve a metal film of increasing thickness and X-ray photoelectron spectroscopy (XPS) to determine the percent coverage of the metal as a function of time. In general, the surface energies of the semiconductor and the metal contact as well as the interfacial energies between these materials determine the mode by which the deposition will occur. The contact metal may deposit in (1) the Frank-van der Merwe (FM) mode or 2-D layer-by-layer growth; the (2) Volmer-Weber (VM) mode or 3-D island growth; or the (3) Stranski-Krastanov (SK) mode that is a combination of the other two modes wherein the transition from FM growth to VM growth is governed by the total elastic stress in the metal. Although there was scatter in the data, an analysis of the XPS data indicated that the growth mode most
likely followed the VW mechanism. Similar results were obtained in analogous experiments concerned with Au contacts on the Zn-terminated (0001) face of ZnO.

Epitaxial growth of Au(111) on polar surfaces of ZnO is supported by LEED patterns reported by Wassermann and Polacek\textsuperscript{11}. Well-defined (1x1) hexagonal LEED patterns indicated a highly ordered surface crystal structure in Au films having a nominal thickness of 20 nm and 100 nm and deposited on ZnO(0001). Results of XPS, TEM and XRD studies confirmed an abrupt, well-ordered interface. Yoshino, et al.\textsuperscript{12} as also reported a clean interface between Au and ZnO. ZnO films grown on Au foils by the last investigator were reportedly also epitaxial and had a better surface microstructure compared to ZnO films grown on Ni, Cu, and Al.

4.2 Experimental Procedures

The wafers of ZnO used in this research were grown via the seeded vapor-phase technique\textsuperscript{13} by Eagle Picher, Inc. In this process, ZnO is grown in a nearly horizontal tube containing ZnO powder held at one end at 1150°C and a single crystal seed at the other end, which is held at 1100°C. Hydrogen is flowed over the powder to form Zn and H\textsubscript{2}O, which are transported to the end of the tube having the lower temperature. The reverse of the aforementioned reduction reaction is believed to proceed at the lower temperature end of the tube to form ZnO on the seed. A two-inch diameter boule of ZnO is created after 150-175 hours of deposition. The wafers used in this research were cut from such a boule and polished to a thickness of ~0.50-0.80 mm. The 2-inch wafers were
cleaved into smaller sections greater than 1cm\(^2\), rinsed in methanol for 5 seconds and dried in flowing nitrogen.

A 40nm thick titanium layer was then deposited from a 99.999% pure metal source by e-beam evaporation onto the backside (0001) face. The purpose of the Ti layer was to (1) absorb radiation from the Pt-Rh heater and conduct heat into the sample and (2) to create an ohmic contact. The front (000\(\bar{1}\)) surface was exposed to a 20-W, 20 vol\% \(\text{O}_2/80\) vol\% \(\text{He}\) remote plasma at 525±20 °C and 0.050±0.001 Torr for 30 minutes in an attempt to completely remove the hydrocarbon and hydroxide contaminants; the sample was then cooled in oxygen to ~25 °C. The XPS studies of the cleaned surface revealed that all detectable hydrocarbons were removed and only ~0.4 ML of hydroxide remained on the surface\(^\text{10}\). Atomic force microscopy (AFM), low energy electron diffraction (LEED), and ultraviolet photoelectron spectroscopy (UPS) affirmed smooth, highly-ordered, stoichiometric, step and terrace surface microstructures. The in-situ plasma cleaning step proved to be a crucial process for the achievement Schottky contacts having low leakage currents and ideality factors essentially equal to ~1.0\(^\text{9}\).

Electron-beam evaporation from a 99.999% pure gold source was used to sequentially deposit very thin regions of increasing thickness onto the ZnO(000\(\bar{1}\)) surface. Base and process pressures used within the 3-kW Thermionics e-beam system were ~7 x 10\(^{-10}\) Torr and ~7 x 10\(^{-9}\) Torr, respectively. Deposition rates were regulated with a Sycon Instruments STM-100MF film thickness/rate monitor and varied from 0.1-1 angstroms/sec. Thickness measurements were determined from a quartz crystal deposition rate monitor. The thickness of the gold analyzed in the TEM was 100 nm.
TEM samples were prepared by sandwiching two ZnO pieces, each having the dimensions of ~0.1 x 0.3 cm, between two sapphire support pieces of the same size using M-Bond epoxy from Allied High Tech Products, Inc. The epoxy was cured at 120°C for 2 hours. The sandwich was then flattened on one side using 15 micron M/B Diamond Disc (50-60130) from Allied Technologies, Inc. and dimpled to a depth 30-35 microns on the Gatan Dimple Grinder, Model 656 with 6 micron diamond paste and 10 grams of weight on the sample holder. The sample was then polished on a felt wheel embedded with 1 micron diamond paste with a weight of 20 grams to eliminate as many scratches as possible. The four corners surrounding the dimple were flattened with 6 micron diamond paper.

The sample was then inverted onto a glass peg with wax and polished at a 45° angle from the interface using a 15 micron M/B Diamond Disc. A thickness of 75 microns was reached according to a hand-held caliper by subtracting out the height of the glass peg and the estimated wax thickness. Subsequent polishing occurred on lap papers from Allied Technologies, Inc with a 6µm diamond paper to ~59µm and a 1µm diamond paper to ~7µm. A copper ring was then glued onto the flat, polished side of the sample using M-bond. As a final thinning step, the sample was placed in an ion mill. Material was removed using Ar+ ions generated using a gun voltage of 4 keV at an angle of 17 degrees for 5 hours. The temperature of the sample was also lowered by mounting it in a liquid nitrogen-cooled stage; this ameliorated the damage caused by the impinging beam of ions.

The TEM samples were analyzed using a JEOL JEM4000EX TEM operating at 400kV. Dr. David Smith at the Center for Solid State Science at Arizona State
University in Tempe, AZ assisted this writer with the use of the microscope and with the analysis of the results. The samples were investigated using bright field imaging, selected area electron diffraction (SAED), and high-resolution imaging (HRTEM). An objective aperture was also employed to filter out the inelastically scattered electrons and therefore create more contrast.

4.3 Results

The cross-sectional TEM micrographs shown in Figure 1 confirm the XPS results regarding the VM or island mode of initial growth of gold on the (000\bar{1}) surface of ZnO. These micrographs also confirm coalescence of some of the islands after growth of 100nm of gold, as shown in Figures 1 and 2. A clean, unreactive interface is also observed which is important for the achievement of rectifying contacts with low leakage currents and ideality factors of \(~1.0\), as noted above.
Figure 4.1: Cross-sectional TEM image of Au(111) islands electron beam evaporated onto the (000̅1) surface of ZnO.
Figure 4.2: Cross-sectional TEM micrograph that reveals (1) the clean interface between an electron beam evaporated Au film and the (000\bar{1}) surface of ZnO and (2) the coalesced gold islands.

The TEM image shown in Figure 3(a) also demonstrates the clean interface that was formed between the deposited gold film and the zinc oxide wafer. Figure 3(b) reveals a high resolution TEM image of this abrupt, unreactive interface. Epitaxial growth is proven by the diffraction pattern in Figure 3(f). Double diffraction spots are noted in the diffraction pattern due to twinning in the gold. Twinning occurred due to the strain obtained from the 11% mismatch of gold to ZnO. The fast-fourier transformations (FFT) of the Au twinning region and the ZnO substrate shown in Figure 3 (c), 3(d), and (e) correlate with the double diffraction spots shown in Figure 3(f). The XRD data (not shown) is in agreement with the results gleaned from the TEM images that bulk Au
(100nm) contacts are crystallographically matched to clean (000\bar{1}) surfaces of ZnO wafers from which the contamination has been removed.

Figure 4.3: (a) Moderate and (b) high-resolution TEM images of the plasma cleaned-and-oxygen adsorbed ZnO(000\bar{1})/(100nm)Au interface, (c) Fast-Fourier transformation (FFT) of gold on left-hand side and (d) right-hand side of twin boundary. (e) FFT of ZnO(000\bar{1}). (f) Diffraction pattern of ZnO(000\bar{1}) and each side of twin boundary in Au.
4.4 References


Chapter 5. TEM Characterization of Annealed Au(111) Contacts/ZnO(000\bar{1}) Wafers

5.1 Introduction

The achievement of rectifying (Schottky) contacts on n-type semiconductors requires materials with a high work function\(^1\). Metals investigated for such contacts on ZnO at this writing include Ag\(^2,3,4\), Pt\(^2\), Pd\(^2\) and Au\(^2,5,6\). Rabadanov et. al.\(^2\) considered Pt, Pd and Au to be the principal candidate metals, since Ag has an affinity for oxygen. The achievement of abrupt, well-ordered interfaces between epitaxial, highly ordered Au(111) films having a nominal thickness of 20 nm and 100 nm and ZnO(000\bar{1}) surfaces has been reported by Wassermann and Polacek\(^7\) and confirmed by well-defined (1x1) hexagonal LEED patterns as well as XPS, TEM and XRD studies. Yoshino, et al.\(^8\) also reported a “clean” interface between Au and ZnO. However, annealing studies of Au(111) on ZnO polar faces have not been reported at this writing.

5.2 Experimental Procedures

Bulk ZnO wafers from Eagle Picher, Inc. were used in this research. They were produced by seeded vapor phase growth\(^9\) which involves a nearly horizontal tube containing ZnO powder held at one end at 1150°C and a single crystal seed at the other end, which is held at 1100°C. Hydrogen is flowed over the powder to form Zn and H\(_2\)O, which are transported to the end of the tube having the lower temperature. The ZnO
crystal grows in the cooler end of the tube on the seeded crystal via the reverse reaction. A two-inch diameter boule of ZnO is created after 150-175 hours of deposition. The wafers used in this research were cut from such a boule and polished to a thickness of ~0.50-0.80 mm. The 2-inch wafers were cleaved into smaller sections greater than 1cm², rinsed in methanol for 5 seconds and dried in flowing nitrogen.

Electron-beam evaporation was used to deposit a 40nm thick Ti layer from a 99.999% pure metal source onto the (0001) face. The purpose of the Ti layer was to both absorb radiation from the Pt-Rh heater and to create an ohmic contact. The front (000\(\bar{1}\)) surface was exposed to a 20-W, 20 vol% O\(_2\)/80 vol% He remote plasma at 525±20°C and 0.050±0.001 Torr for 30 minutes in an attempt to completely remove the hydrocarbon and hydroxide contaminants; the sample was then cooled in oxygen to ~25 °C. The XPS studies of the cleaned surface revealed that all detectable hydrocarbons were removed and only ~0.4 ML of hydroxide remained on the surface\(^{10}\). Atomic force microscopy (AFM), low energy electron diffraction (LEED), and ultraviolet photoelectron spectroscopy (UPS) affirmed smooth, highly-ordered, stoichiometric, step and terrace surface microstructures.

Au was deposited from a 99.999% pure source through electron beam evaporation. Sequentially very thin regions of increasing thickness were deposited onto the ZnO(000\(\bar{1}\)) surface. Base and process pressures used within the 3-kW Thermionics e-beam system were ~7 x 10\(^{-10}\) Torr and ~7 x 10\(^{-9}\) Torr, respectively. Deposition rates were regulated with a Sycon Instruments STM-100MF film thickness/rate monitor and varied from 0.1-1 angstroms/sec. Thickness measurements were determined from a quartz
crystal deposition rate monitor. After a thickness of 100 nm was deposited the sample was annealed in air at 175 degrees for 15 minutes.

TEM samples were prepared by sandwiching two ZnO pieces, each having the dimensions of ~0.1 x 0.3 cm, between two sapphire support pieces of the same size using M-Bond epoxy from Allied High Tech Products, Inc. The epoxy was cured at 120°C for 2 hours. The sandwich was then flattened on one side using 9 micron diamond paper and dimpled to a depth 30-35 microns on the Gatan Dimple Grinder, Model 656 with 3 micron diamond paste and 10 grams of weight on the sample holder. The sample was then polished on a felt wheel embedded with 3-micron diamond paste to eliminate as many scratches as possible. The four corners surrounding the dimple were flattened with 3-micron diamond paper.

The sample was then inverted onto a glass peg with wax and polished using 9, 6, 3 and 1 micron diamond papers to a thickness of 30-50 microns, as determined using the colors of the Nomarski fringes for ZnO shown in Table 1 of Chapter 3. A copper ring was then glued onto the flat, polished side of the sample using M-bond. As a final thinning step, the sample was placed in an ion mill. Material was removed using Ar+ ions generated using a gun voltage of 3 keV at an angle of 13 degrees for 135 minutes. Mounting it in a liquid nitrogen-cooled stage also lowered the temperature of the sample and therefore decreased the damage caused by the impinging beam of ions.

The JEOL JEM4000EX TEM operating at 400kV at the Center for Solid State Science at Arizona State University in Tempe, AZ was used to analyze the samples with the assistance of Dr. David Smith. Bright field imaging was used to capture low magnification pictures and selected area electron diffraction (SAED) was utilized to
capture diffraction patterns. High magnification images were captured through high-resolution imaging (HRTEM). EELS was preformed in the JEOL 2010F at North Carolina State University with the assistance of Dr. Alex Kvit. Data was obtained from the bulk ZnO, the interface and the gold contact in both the as-deposited and annealed samples.

5.3 Results

The growth of electron beam evaporated gold films on cleaned ZnO(000\(\overline{1}\)) wafer sections via the coalescence of islands was discussed in Chapter 4. Distinct islands remained after annealing these assemblies in air at 175°C, as shown in Figure 5.1. Figure 5.2 shows a TEM selected area diffraction pattern that indicates that the Au(111) areas remained crystallographically matched to the ZnO(000\(\overline{1}\)) sections, which agrees with the XRD data\(^{11}\). However, the TEM images of the annealed gold contacts shown in Figure 5 reveal an interface layer that appears thin in some areas, as shown in Figures 5.3 and 5.4 and thick in other areas, as shown in Figure 5.5. Micrographs of the interface layer acquired at higher magnification shown in Figures 5.4 and 5.5 suggest order and disorder, respectively.
Figure 5.1: Distinct islands of gold on a ZnO(000\overline{1}) surface after annealing the assembly in air for 15 minutes at 175°C.

Figure 5.2: TEM selected area diffraction pattern of annealed Au(111)/ZnO(000\overline{1}) interface.

Figure 5.3: Interface layer discovered between gold and zinc oxide after annealing.
Figure 5.4: High resolution TEM micrograph suggests order in the thin interface layer between Au and ZnO(000 $\bar{T}$).
Figure 5.5: High resolution TEM micrograph suggests an amorphous structure in the thin interface layer between Au and ZnO(000 $\bar{T}$).

One possibility for the interface layer could be a reaction between zinc oxide and gold. A much more likely possibility could be delamination of the contact during cooling from the annealing step in which this layer consists of glue used to create the TEM samples. Electron energy loss spectroscopy (EELS) was performed in an attempt to determine the elements present at the interface and any irregularities between the as-deposited and annealed samples. Figure 5.6 shows the EELS data obtained for the annealed sample as well as analogous data for the as-deposited sample at the aforementioned region as well as in the bulk gold and bulk zinc oxide regions.
Figure 5.6: EELS data obtained from the as-deposited gold contact in the (a) bulk gold, (c) at the interface and (e) in the ZnO substrate. EELS data of the annealed sample (b) in bulk gold, (d) at the interface and (f) in the ZnO substrate. Note change in scales of the x- and y-axis.
The energy loss for oxygen, zinc and carbon are 532eV, 1020eV, and 284 eV. Gold appears at 54eV with a weak peak at 334 eV as well. Although there is a slight change in behavior around 54eV in Figure 6 (a) and (b) no gold could be detected by the system. However, the zinc to oxygen and carbon to oxygen ratios were analyzed. Their values are shown in Table 5.1.

Table 5.1: Relative elemental ratios determined in EELS analysis.

<table>
<thead>
<tr>
<th></th>
<th>As-Deposited Sample</th>
<th>Annealed Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Zn:O</td>
<td>C:O</td>
</tr>
<tr>
<td>At the interface</td>
<td>0.98±0.22</td>
<td>0.0±0.051</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>In the ZnO substrate</td>
<td>0.6±0.091</td>
<td>0.0±0.0016</td>
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These ratios are not the actual ratios but can be used as a comparison. The Zn/O ratio is markedly greater at the interface of the annealed sample than at the interface of the as-deposited sample indicating more oxygen at the interface of the former. Also, there is more carbon present at the interface of the annealed sample when compared to the as-deposited sample, which appears to be absent in the latter interface. All of the ratios from the bulk ZnO remained consistent from both samples.

Coppa\textsuperscript{11} found that a slight degradation occurred in the electrical properties of these contacts during annealing at 80±5 °C and at 150±5 °C. Gold is not believed to react with ZnO during annealed at these low temperatures. With an increase in carbon content
at the surface it is possible that the interface consists of epoxy used to hold the TEM sandwich together during sample preparation. This would also explain why the interface layer is thicker in some regions than in others. However, the presence of carbon is inconclusive due to the nature of carbon present throughout the testing apparatus during the EELS analysis. As a chemical reaction between gold and ZnO even at 150°C is very unlikely, it therefore is concluded that the interface layer consists of epoxy.
5.4 References


3)  Y. Liu, C.R. Gorla, S. Liang, N. Emanetoglu, Y. Lu, H. Shen, and M. Wraback,  


Chapter 6: Summary

Several thinning techniques were investigated to determine the best approach to create TEM samples that did not crack during processing. These included a dimpling process similar to that used for SiC, hand-held tripod polishing, automatic tripod polishing, focused ion beam removal of a slice of material and a repeat dimple process. Cracks developed easily in the ZnO during thinning, especially below 5 microns. The source of these cracks has not yet been defined; however, they likely originate at the surfaces of the micropipes that are common in the materials studied in this research. However, cracks did not develop if the sample was polished with a felt surface even below a thickness of 5 microns. The repeat dimple process worked the best by (1) creating a dimple when the sample was thick (greater than 150 microns), (2) utilizing a dimple polish with a felt wheel and 1 micron diamond paste below 20-30 microns and (3) taking advantage of the Nomarski interference fringes to monitor the thickness of the sample.

Optical and TEM images of bulk ZnO revealed intrinsic defects. Micropipes produced in the boules of material during the initial growth process were observed via optical microscopy to occur along the c-axis. TEM investigations of bulk wafers of this material have revealed numerous stacking faults and dislocations. Bright field and dark field images in two-beam condition at low magnification (40KX) at the (11 2 0) zone axis depict several stacking faults and dislocations. No further investigations of these defects were performed.
TEM diffraction patterns, supported by previous XRD data, revealed the epitaxial
growth of Au(111) on remote plasma-cleaned ZnO. Gold was shown to grown as islands.
Bright field images in low magnification and high magnification of the Au/ZnO assembly
showed a well-ordered, interface. Gold contact samples were then annealed in air at
175°C for 15 minutes. Gold remained crystallographically matched to the ZnO. Though,
annealing under these conditions did result in an interfacial reaction. EELS analysis was
unable to analyze gold content in any part of the sample. Nevertheless, results showed
that this layer was comprised of more oxygen and less zinc.
Chapter 7: Recommendations for Future work

Cracking of samples during thinning to achieve materials for TEM investigations due to microporosity in the material must be completely avoided. The use of the repeat dimple method described in Chapter 2 as the processing route of choice to achieve crack-free samples below a thickness of five microns is recommended. In producing thinned samples suitable for subsequent ion milling to achieve electron transparency, sapphire supports are not always necessary; as, they only benefit the support of the sample during polishing steps of the preparation. Whenever possible cleave or ultrasonically trepan your samples rather than cutting them with a low speed saw, as the latter can introduce flaws into the sides of the sample that may be detrimental to the success of a subsequent process step. The use of a glass peg is recommended during sample preparation of samples for analysis of Nomarski color fringes in the optical microscope. These color patterns are crucial for the determination of the thickness of a sample, especially when it is very thin. A substantial amount of wax should be applied to the glass peg, as it both holds and provides support for the sample. The glass peg fits firmly into a Gatan Disc Grinder 623, which is recommended to prevent the formation of an uneven surface on the samples during lap polishing. One major problem that occurred during lab polishing was the delamination of the sandwich. Polishing should occur at an angle or parallel to the sandwich line and should not be continued below 30 microns at the center of the dimple.

Creating a dimple in the material when it is extremely thick leaves sufficient material surrounding the dimple that can withstand the applied pressure required for additional thinning and decreases the probability of crack formation and the loss of the
sample. After dimpling the sample while it is extremely thick a felt polishing wheel embedding with 3 micron diamond paste is utilized to decrease as many scratches as possible. After the sample is thinned through lap polishing to around 30 microns in the center of the dimple a repeat of the initial dimple occurs with 1 micron diamond paste on a felt wheel. The polishing of the sample on the dimpler decreases the thickness of the sample while not promoting any scratches. To decrease the time of this polish the removal of material through lap polishing below 30 microns was investigated. However, additional pressure applied to the material and to the sandwich was more than it could support and separation of the sandwich occurred. It is important to maintain a completely bonded sandwich to protect the surface of the material to be analyzed during ion milling. The glue line in the bonded samples must remain intact for the same reason. Moreover, if the glue line is still intact then the actual surface of the sample can be observed in the TEM, and this is useful in the determination of the thickness of a film or contact. Therefore the samples must be around 30 microns thick before polishing it on the dimpler in the final steps with 1 micron diamond paste.

The use of low angle in the ion milling step (8°-13°) in tandem with a low ion beam energy (2-4 keV) is recommended for preparing electron transparent ZnO-based samples. Furthermore, the utilization of a liquid nitrogen stage is beneficial during the milling procedure to ameliorate damage caused by the impingement of the ion beam.

Two types of epoxies were mentioned in Chapter 2; however, I found that the resin-hardener 10:1 mixture from Gatan remained in contact in the sandwich glue line during sample preparation for a longer time than the M-bond sold by Allied High Tech Products, Inc. Buehler polishing films are the best that I have used but they are also the
most expensive. Southbay Technology, Inc. sells diamond, lapping films (abrasive films) of the same quality but at a more competitive price. Allied polishing films were the least expensive but did not hold up as long as the other brand names. Collodial polishing steps proved essential but the name brand did not play a critical role.

Hand-held tripod polishing may not be the best option due to human error, but automatic tripod polishing would be recommended. However, the before the sample exceeds the 30 micron thickness at its thinnest point a felt wheel containing the colloidal silica would have to be incorporated to prevent crack propagation. Focused ion beam techniques may also be an option. With an appropriate metal protecting layer, samples could be made more easily without crack propagation. However, it is very difficult to discern damage acquired during ion implantation of potential dopant elements from FIB damage. Although expensive, the FIB and lift technique developed at MAS would create a thin region with limited sample preparation.

For samples on which metal contacts have been deposited, a special vise is recommended to keep the sandwich intact during the use of the repeat dimpling approach. It was also observed that the gold layer in a gold/ZnO assembly could be easily removed with a Q-tip® at just room temperature. The purpose of the vise is to hold the glue line, contact metal and substrate intact even at the moderate temperatures used to melt wax and cure epoxy. The vise also adds support to the sample as it is thinned via dimpling and polishing.

Additional transmission electron microscopy of zinc oxide wafers and films would provide addition information regarding defects in the bulk material. Low magnification bright images would provide an overview of the types of defects present.
Two-beam conditions provide the best contrast to see defects. By tilting the sample one can determine what types of defects are present. Stacking faults are planar defects, which will change into a sequence of white and black lines whereas dislocations will remain. The Burgers vector of each dislocation could also be determined through continuous tilting of the sample to different two-beam conditions. High resolution would be useful to image a disruption of stacking sequences of stacking faults in atomic planes. Plane view samples would also be beneficial as to obtain a dislocation density at low magnification.

Additional TEM studies should also be conducted to determine the nature of the interactions, if any, of the oxygen- and Zn-terminated \{0001\} surfaces of ZnO with deposited metals other than gold, e.g., platinum or palladium and the formation of new phases at these interfaces in the as-deposited state and after annealing to progressively higher temperatures. The results of these studies would provide a broader base of understanding in terms of the selection of and the optimum procedures for the deposition and annealing of the principal candidate metal for Schottky contacts as well as facilitate the fabrication of Schottky diodes.
Appendix A: The Development of a New Stage on the Optical Microscope for Investigations of Samples Polished on the Hand-held Tripod Polisher

The 3.5 inch tall hand-held tripod polisher described in Section 2.3 did not fit in the ¾” allowed space of the optical microscope. Therefore, a new stage was created for the Olympus BX60 microscope to allow clearance under the objective lens. The new stage was fabricated such that it (1) supported the hand held tripod polisher underneath the objective lens while allowing for the sample to remain flat and parallel to the floor and (2) allowed the sample to be shifted in-and-out of focus as the focus knob was turned with sufficient clearance. Moreover, since there were several users of the microscope the new stage was created to be easily interchangeable with the original stage.

Figure A-1 shows the original stage on the Olympus BX60 optical microscope. There are two pieces in the original set up including the bottom level and top level. Note that the back of the bottom level is attached such that the stage will move up and down when the focus knob is rotated. The bottom level attached to the microscope is shown in Figure A-2. The top level sits on the bottom level on a circular piece and allows for x- and y-axis movement of the sample platform. Images of the top and bottom level members are shown in Figures A-3 and A-4.
Figure A-1: The original stage on the Olympus BX60 optical microscope. Note the position of the objective lens, sample platform and focus knob.
Figure A-2: The original bottom level attached to the microscope with circular dimensions for attachment of top level. Note the height in relation to the objective lens.
Figure A-3: (a) Right-side-up and (b) up-side-down images of original top level. The ruler shows one-inch increments up to six inches.

Figure A-4: (a) Right-side-up and (b) up-side-down images of original bottom level. The ruler shows one-inch increments up to six inches.
Measurements from the original members as well as the height of the hand-held tripod polisher resting in its stand were used to create the new stage. Two pieces were designed and created. The new bottom piece was designed to (1) connect to the back of the microscope through two vertical grooves, (2) allot a large enough platform area to hold the hand-held tripod polisher in its stand and (3) allow suitable clearance for focusing under the objective lens. In order to easily transform back to the original sample stage a new top piece was created. Two important aspects of the new top piece design included (1) horizontal grooves intended to attach securely to the new bottom piece and (2) a circular section enabling the attachment of the original sample stage. The original CAD drawings for the new stage are shown in Figures A-5, A-6 and A-7.
Figure A-5: Original CAD drawings of the (a) top view and (b) front view of the top piece from the new stage. Note: dimensions were altered slightly in final product.
Figure A-6: Original CAD drawings of the (a) top, (b) front and (c) side view of the new bottom piece from the new stage. Note: dimensions were altered in the final product.
Several slight adjustments in the original design including the depth of the new bottom piece were incorporated after the first construction. The sides of the bottom piece were partially eliminated to allow a 45-degree rotation of the hand held tripod polisher. Nomarski fringes of transparent material appear best at this angle thus allowing proper thickness estimates of the sample as described in Chapter 2.2. Images of the new stage are shown in Figures A-8 and A-9.
Figure A-8: (a) Right-side-up and (b) up-side-down images of the top piece of the new stage. The ruler shows one-inch increments up to six inches.

Figure A-9: (a) Right-side-up and (b) up-side-down images of the bottom piece of the new stage. The ruler shows one-inch increments up to six inches.
The new bottom piece can now be attached to the back of the microscope as shown in Figure A-10. The new design allows proper clearance to focus a sample on the hand-held tripod polisher without damaging the objective lens as shown in Figure A-11.

Figure A-10: Attachment of the bottom piece of the new stage to the microscope.
Figure A-11: The use of the new stage with the hand-held tripod polisher sitting in its stand. Note the allowed clearance under the objective lens and the 45-degree rotation of the stand for improved Nomarski contrast of the sample.

Easy transformation from viewing samples on the hand-held tripod polisher to those less than ¾” tall, i.e. those on a ¾” tall glass pegs used during other polishing steps, was made possible by the addition of the new top piece. It fits securely into the grooves of the new bottom piece as shown in Figure A-12(a). The original top level member which contains a platform for samples less than ¾” tall fits properly onto the new top piece as shown in Figure A-12(b). The final set-up with the newly created pieces and the original top piece member is shown in Figure A-13.
Figure A-12: The new top piece fits snug into (a) the new bottom piece and (b) the original top-level member.

Figure A-13: New stage set-up for viewing samples less than ⅛” tall.