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U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Semiconductor Measurement Technology:

Automated Scanning Low-Energy Electron Probe (ASLEEP) for Semiconductor Wafer Diagnostics

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PREFACE

This study was carried out at the Naval Research Laboratory as a part of the Semiconductor Technology Program in the Electronic Technology Division at the National Bureau of Standards. The Semiconductor Technology Program serves to focus NBS efforts to enhance the performance, interchangeability, and reliability of discrete semiconductor devices and integrated circuits through improvements in measurement technology for use in specifying materials and devices in national and international commerce and for use by industry in controlling device fabrication processes. The work was supported by the Defense Advanced Research Projects Agency* through the National Bureau of Standards' Semiconductor Technology Program, NBS Order Nos. 501718 and 711782. The contract was monitored by R. L. Raybold as the Contracting Officer's Technical Representative (COTR). Drs. W. M. Bullis and K. F. Galloway provided technical review of this report for the National Bureau of Standards.

Certain commercial equipment, instruments, or materials are identified in this report in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

Larger scale drawings of the mechanical parts and detailed listings of the computer programs and subroutines are available on request from the COTR, TECH-A-361, National Bureau of Standards, Washington, DC 20234.

*Through ARPA Order 2397.

AUTOMATED SCANNING LOW-ENERGY ELECTRON PROBE (ASLEEP) FOR SEMICONDUCTOR WAFER DIAGNOSTICS

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This report summarizes the results of a three-year effort in the development of a computer Automated Scanning Low-Energy Electron Probe (ASLEEP) for semiconductor wafer diagnostics. Experiments designed to explore the measurement capabilities of ASLEEP for measurements on silicon, gallium arsenide, and indium phosphide are described. Four areas were emphasized: 1) semiconductor resistivity, 2) semiconductor defect density, 3) lateral inhomogeneities, and 4) oxide uniformity. Although oxide charging problems limit the utility of ASLEEP in its present form for silicon, defects and lateral inhomogeneities could be readily detected in gallium arsenide and indium phosphide.

Key words: Automated scanning low-energy electron probe; lateral inhomogeneity; oxide uniformity; scanning low-energy electron probe; semiconductor defect density; semiconductor resistivity; semiconductor wafer diagnostics.

I. INTRODUCTION

The scanning low-energy electron probe (SLEEP) is an electron beam probing technique whereby an electron beam is first accelerated (to provide beam definition) and then decelerated by a grid placed in front of the sample to be probed [1]. The sample under investigation is scanned by the electron beam in the retarding field region. Only those electrons whose energies are sufficient to overcome the local sample potential barriers are collected. This collected current is measured in the sample-cathode circuit. Similarly, electrons with insufficient energy to overcome sample surface potential are reflected from the sample and may be collected to form the mirror mode operation. Thus, either the directly collected current or reflected current provides a surface potential map of the sample. In the work reported here, only the collected current mode was utilized.

The SLEEP technique is inherently simple. The basic apparatus consists of a low-energy gun structure (800 to 900 V) and a standard vidicon electromagnetic beam focusing and deflection system. Depending on the type of data, precision, accuracy and detail required, control and collection can be carried out by manual techniques or by minicomputer. Similarly, depending on the sophistication of the measurement required, vacuum equipment may range from the rudimentary (10^{-6} torr) to the more advanced oil-free ultra-high vacuum (10^{-11} torr).

In the present investigation a Data General Nova 800 computer has been interfaced to the scanning low-energy electron probe to control data collection and signal processing, thus forming an Automated Scanning Low-Energy Electron Probe (ASLEEP) system. An ultra-high vacuum system was also utilized in order to provide for a contamination free system. Figure 1 shows the vacuum system for the ASLEEP experiments while figure 2 shows the computer and interface electronics. The ASLEEP experiments have been designed to explore

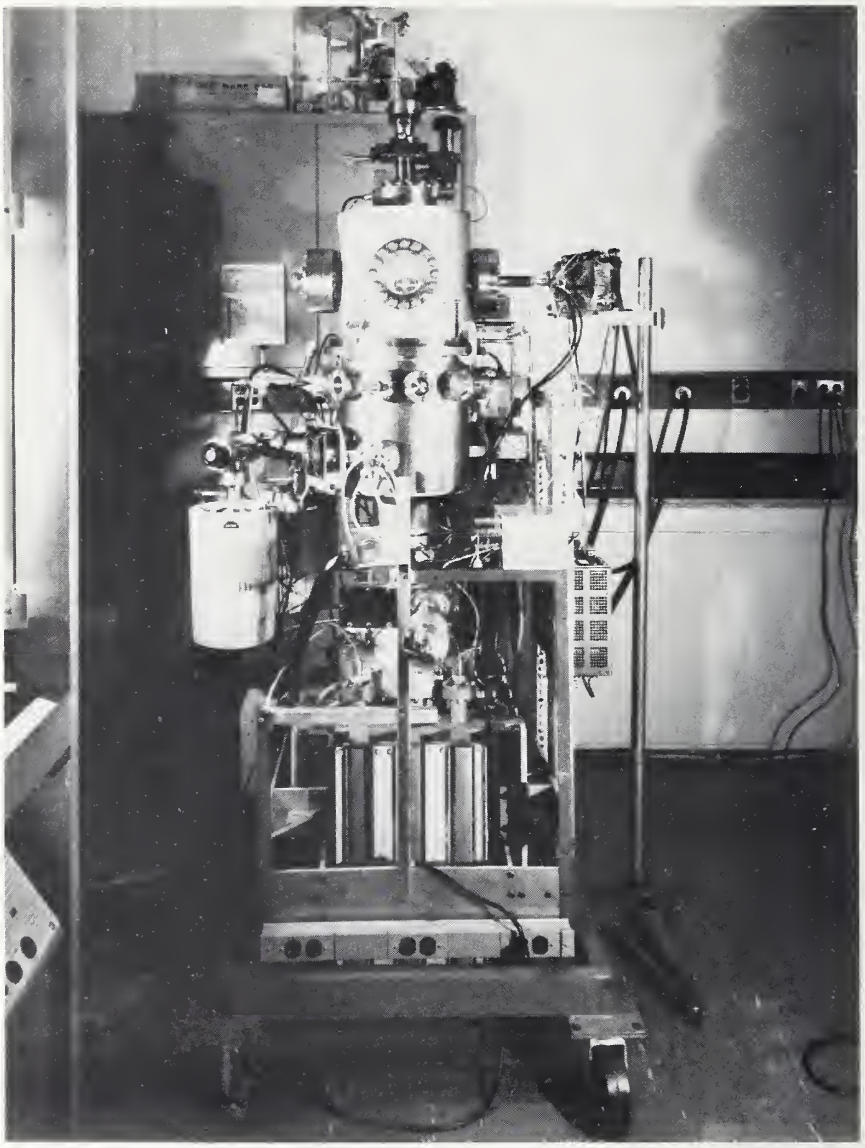


Figure 1. The ASLEEP vacuum system showing the high vacuum, oil-free pump station.

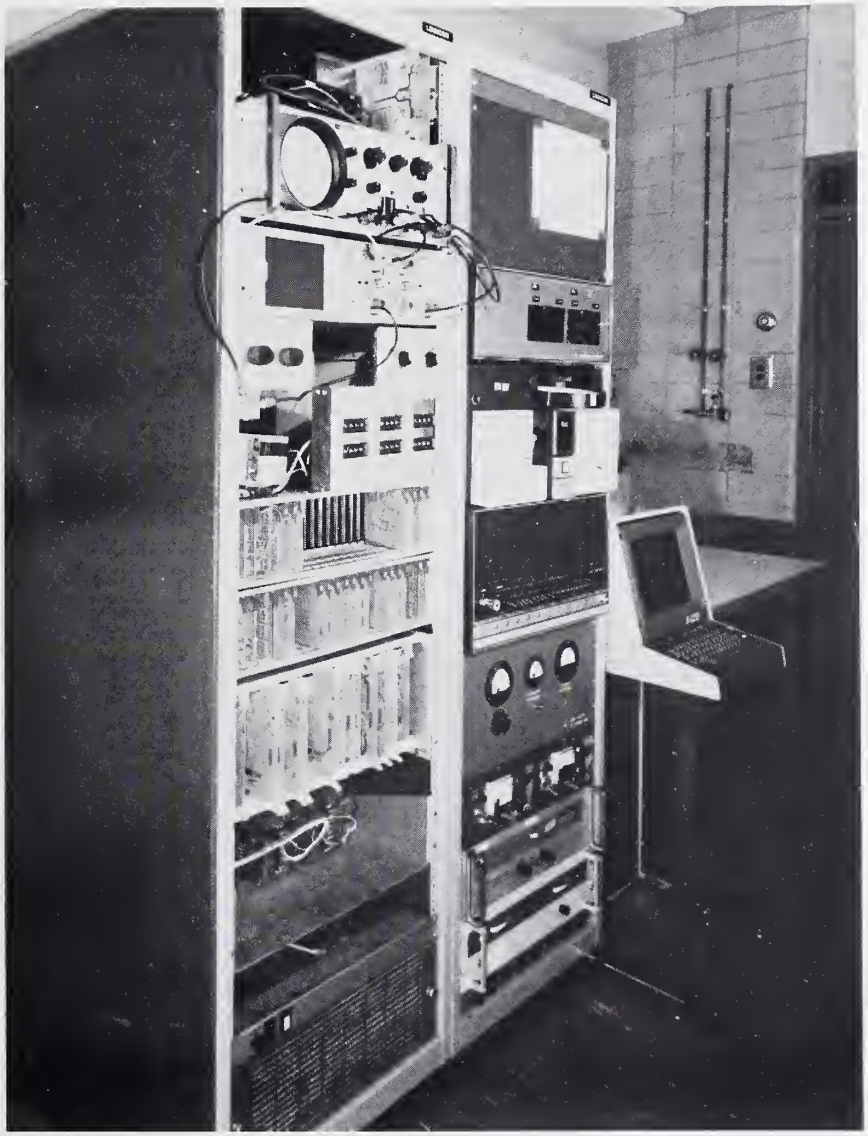


Figure 2. Photograph of the computer and interface electronics.

the measurement capabilities of the system and the applicability of ASLEEP to the solution of problems in the two major semiconductor materials technologies: silicon and III-V compounds (gallium arsenide and indium phosphide). In the silicon technology, ASLEEP was utilized for wafer resistivity-homogeneity studies and dielectric homogeneity studies. In the III-V compound technology, ASLEEP was utilized to investigate lateral inhomogeneities resulting from stoichiometry variations and semiconductor defects.

2. INSTRUMENTATION

2.1 Electron Generation

The electron gun design chosen for ASLEEP was one which had been used by other researchers [1] in work function studies. A schematic of the assembled gun structure is shown in figure 3, while figure 4 shows the interconnections of the electron gun to the rest of the system. The gun structure is similar to that of a commercial vidicon tube and consists of a drift tube, three grids, and a cathode. The target end of the drift tube is covered with a 1000-mesh nickel screen having a 40% transparency.

The cathode is an iridium ribbon 500 μm thick and 4 mm wide. A thoria coating is plated onto the ribbon in the region where high electron emission is required. The plating bath consists of 5 g thorium oxide, 75 mg thorium nitrate, and 100 mL ethyl alcohol. The iridium was biased as the positive lead at a level of 5 to 8 mA at 200 V during the coating operation. A total coating thickness of 50 μm was required. Thorium nitrate is added to increase the conductivity of the bath. On portions of the ribbon where high electron emission is not desired, the thoria may be scraped off with a cleaned razor blade. The cathode is activated by quickly raising the filament temperature above its normal operating value. The filament current required to obtain a given electron emission will then decline and after about 30 min the emission will stabilize at a filament current of 4 to 5 A.

Directly heated thoria-coated iridium emission sources are used for ASLEEP rather than the standard indirectly heated oxide cathode primarily because of the nature of the application. As a diagnostic tool of general utilizability, it is expected that the gun system will be exposed to air, and therefore to water vapor, upon each sample insertion. "Oxide" cathodes degrade rapidly under such conditions while thoria-coated iridium has been found to give good repeatable emission characteristics [1].

Fundamental to the utilization of this system is the measurement of the retarding field current over the target area. This current is given by

$$I = I_m \exp\left(-\frac{qV}{kT}\right), \tag{1}$$

where V is the applied voltage, k is Boltzmann's constant, q the electronic charge and T is the absolute temperature. The minimum current, I_m, is given by the Richardson equation (2)

$$I_m = A_0 T^2 \exp\left[\frac{q\phi_c}{kT}\right], \tag{2}$$

where ϕ_c is the cathode work function and A_0 a constant. Equation (1) is shown in a semilog plot in figure 5 for two different values of target work function. The region below the horizontal line marked I_{TH} is the deep retarding region.

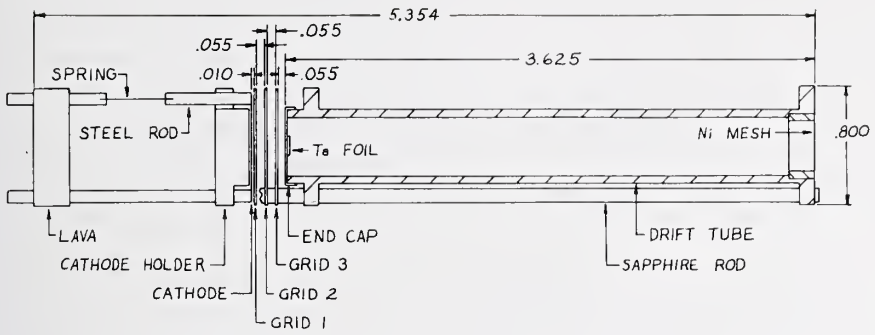


Figure 3. Schematic showing the electron gun assembly for the ASLEEP system.

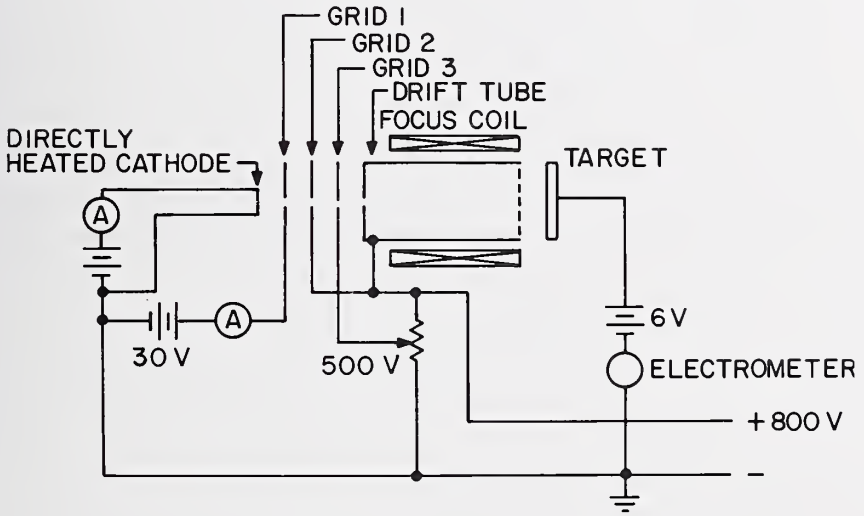


Figure 4. Schematic diagram showing the electron source connections.

The electrons from the cathode are accelerated by the grids and directed onto the end cap of the drift tube. In the drift tube, standard vidicon magnetic deflection and focus techniques image the defining aperture on the target under study. The 700 V applied to the drift tube and nickel mesh screen creates a field of 4000 V/cm in front of the target which decelerates the electrons. A tantalum aperture defines the electron beam size. The target specimen is connected to ground by a current amplifier with the result that the target is virtually at ground potential. Any electrons collected by the target are amplified by the current amplifier and fed to the electronic control circuitry. To make a surface potential measurement, the cathode potential is swept with a saw tooth which starts increasing the beam energy from the deep retarding region. When the beam energy is high enough, such that a predetermined threshold current is collected by the target, the sawtooth is turned off. The magnitude of the sawtooth voltage at turn off is a direct measurement of surface potential at a particular beam position for a constant (known) cathode work function.

The application of the electron gun to the complicated semiconductor diagnostic problems has revealed two important weaknesses in the gun design which must be corrected before ASLEEP can become a routine diagnostic tool. These weaknesses are: a) energy filtering of the electron beam due to the magnetic field of the directly heated cathode, and b) limited electron emission density from the cathode.

a) *Energy Filtering of the Electron Beam:* Energy filtering of the electron beam arises in the following way. A directly heated thoriated iridium cathode was chosen for the electron gun because, as stated above, it is not poisoned by exposure to water vapor. However, the thoriated cathode must operate at a higher temperature than a barium oxide cathode and, consequently, it is directly rather than indirectly heated. A direct current of 4.5 A is required to attain emission temperature. This current results in a large magnetic field which deflects the emitted electrons sharply away from the centerline of the electron gun. External alignment coils are used to compensate for this field, but since the magnetic field from the cathode varies inversely as distance from the filament and the field from the alignment coils is uniform, the compensation is not complete. The uncompensated field gives all of the electrons a radial component of velocity which directs them into the walls of the electron gun. This effect is strongest on the low-energy electrons and they tend to be filtered out before the electron beam reaches the target. If the log of the target current is plotted as a function of bias voltage between the cathode and the target, a plot is obtained which contains two regions. At high bias voltages the current is independent of voltage. This is the saturation region. At lower bias voltages the log of current is proportional to bias voltage. This is the retarding field region. In the case with no electron filtering the slope in the retarding field region is the temperature of the cathode. The removal of the low-energy electrons reduces the beam current undesirably and makes the cathode appear hotter. This apparent increase in temperature manifests itself as a reduction in the slope of the retarding field plots which results in a reduction in the accuracy of the surface potential measurement as the surface potential is defined by the point where the saturation region and the retarding field region intersect on the plot.

The magnetic field could be reduced by redesigning the filament holder so that the current followed a straight bifilar loop, which provides self-canceling of the magnetic field. Indirectly heated cathodes would also be a desirable solution to this problem as the heater current required is much smaller and the heating windings may be arranged to minimize the magnetic field. Field emission sources could also be used as described in reference [2].

b) *Limited Electron Emission Density:* The ASLEEP system requires a small monoenergetic beam of electrons. It is impossible to extract electrons from a large area of the

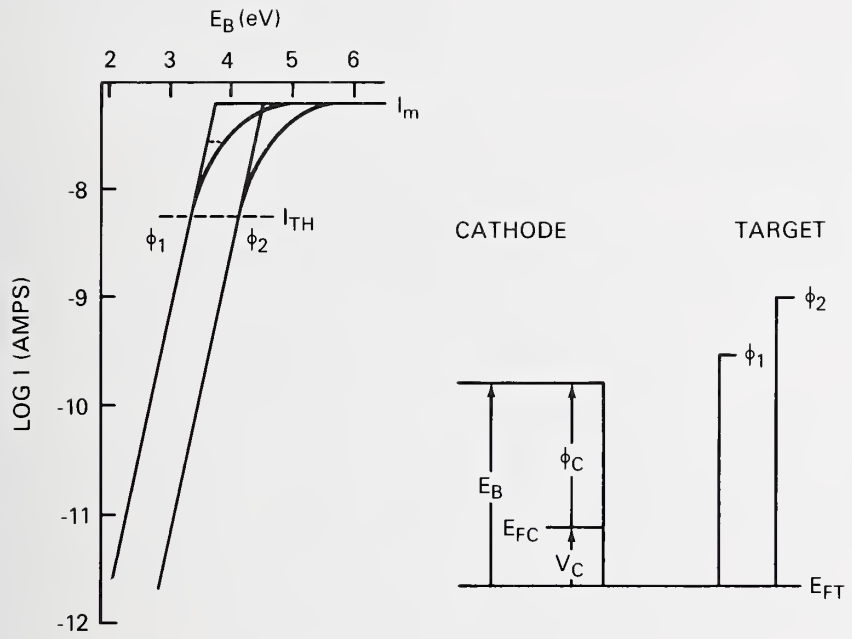


Figure 5. Definition of surface potential relationships and semilog plot for two different work functions.

cathode and focus them into a tight spot and still meet this requirement because the focusing action would give the electrons a radial component of velocity which spreads their energy distribution. Consequently, ASLEEP uses a pinhole aperture and deflection and focus techniques similar to a vidicon tube. Thus, the electrons which participate in the surface potential measurement originate from a region of the cathode approximately the size of the pinhole aperture. Due to the focusing technique used, the electron spot size at the target is also approximately the same as the pinhole aperture. Thus, reducing the aperture size to increase resolution results in reduced beam current and reduced signal to noise ratio. Thus, it is essential that the current density emitted by the cathode be as large as possible.

2.2 Analog and Interface Electronics

A complete block diagram of the ASLEEP system is shown in figure 6. The electron beam is deflected by signals provided to the deflection amplifiers. The x and y deflection channels are identical; thus the discussion is confined to the x axis. There are two possible sources of x-axis deflection information: the computer, or a 12-bit word set by front-panel-mounted octal-thumbwheel switches (x-axis manual). The computer communicates through a series of interfaces which were purchased from the computer supplier. These interfaces provide a 16-bit word, of which 12 bits are fed to the x-axis input select board which provides solid-state switching on a front panel input to determine which source of data will be used. The x-axis D/A converter converts the digital signal to an analog voltage which drives the x-axis deflection amplifier and an oscilloscope. The deflection amplifier drives the deflection yoke. The deflection amplifier has a step response of 1.9 μs/A and a small signal bandwidth of 1.5 MHz. It is a direct-coupled feedback amplifier which operates as a voltage-to-current converter. The x-axis signal is also fed to the x axis of an oscilloscope which provides an analog presentation of the surface potential.

The cathode input provides a selectable voltage to the cathode of the electron gun. There are two possible sources of cathode voltage data: the computer, or a 12-bit word set by front-panel-mounted octal-thumbwheel switches (cathode manual). The cathode input select board provides solid state switching on a front panel input to determine which source of data will be used. The cathode D/A converter converts the digital signal to an analog voltage which is applied to the cathode. When in the computer mode of operation, it is possible to apply a voltage ramp or any other function to the cathode via software. The system also has a 4-digit LED display which can display singly the quantities: x-axis beam coordinate, y-axis beam coordinate, and cathode voltage. In order to reduce cabling, the data are multiplexed and the multiplexed data are sent to the display demultiplexer, timing, and display.

Those electrons which have enough energy to overcome the surface potential barrier of the target constitute a current which is amplified either by a current-to-voltage converter or by a nanovoltmeter amplifier operating as a shunt picoammeter. The output of the current amplifier is fed to the threshold detector. When the target current reaches a preselected value, a selected bit on a specified interface is set. Computer software tests this bit and when it is set, stops the cathode voltage ramp. The value of the ramp at that instant is the relative surface potential of that point on the target. The output of the current amplifier is also fed to the z axis (intensity control) of an oscilloscope to provide a TV-type picture of variation in surface potential.

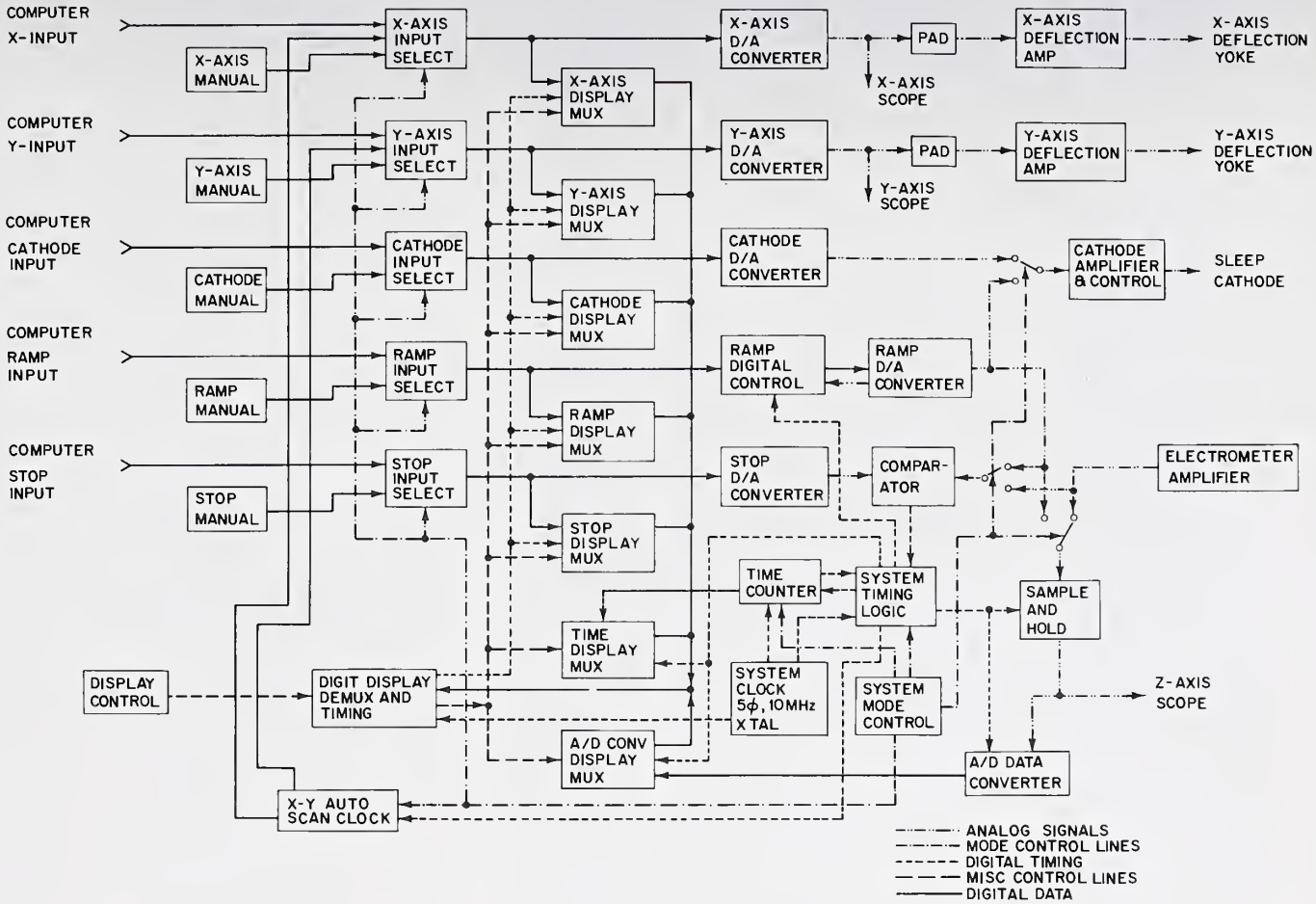


Figure 6. Block diagram of computer automated scanning low-energy electron probe.

The programming language chosen for ASLEEP was BASIC since it is widely used and programs may be interactively written and debugged at the computer terminal. BASIC also supports calls to user-written assembly language subroutines. This feature is essential to the NRL implementation of ASLEEP, because it is through the assembly language subroutines that the computer controls the electron beam position, cathode voltage, and data output.

2.3 Spatial and Voltage Resolution

Utilizing an interdigitated test structure of aluminum on silicon dioxide and consisting of 6-μm-wide lines on 12-μm centers, the minimum spatial resolution attained was 6 μm. To minimize charging effects, a special test structure with minimum exposed oxide was constructed for voltage resolution measurements. A gold-metallized alumina substrate was divided into two large pads separated by an 80-μm gap. An adjustable voltage was applied between the two pads and its effect on the video signal was studied. A single line was scanned across the pads and the target current was displayed on the oscilloscope. The potential on each pad was adjusted so that the currents on each were equal and less than 1 nA. Then the potential of one pad was adjusted until the target current was displaced by the width of the noise in the scope. A 300-mV change was adequate to do this with a target current amplifier bandwidth of 20 kHz. The persistence of the phosphor gives a filtering effect which allows a greater resolution in the TV scans. At a target current of less than 1 nA and even with careful shielding and grounding, the bias box which applies the voltage between the pads acts as a source of 60-Hz pickup. In addition, it adds noise by increasing the shunt capacitance on the input of the target current amplifier. A 20-Hz low-pass filter was inserted in the video circuit to remove these noise sources and the voltage resolution experiment repeated. In this case, the voltage resolution measured was 25 mV.

The computer was used to measure voltage resolution by applying the cathode ramp, threshold detector, and signal averaging. Measurements made over a period of less than a minute were compared and showed standard deviations of 1 mV, but long term drifts increased the standard deviation to 10 mV. The long term drifts were due to the beam wandering around on the target surface and variations in heater current.

3. EXPERIMENTAL INVESTIGATIONS

The results, to be more fully described below, indicate that oxide charging problems limit the utility of ASLEEP, in its present form, for silicon. However, in the case of gallium arsenide and indium phosphide, by utilizing *in situ* oxide desorbing anneals, native oxides may be removed, thus allowing ASLEEP to study a wide range of problems. The problems studied by ASLEEP include surface contamination effects and lateral inhomogeneities due to stoichiometry variations. In addition, defect and fault detection can be readily attained on both the gallium arsenide and indium phosphide without the obscuring effects of oxide charging.

3.1 Oxide Uniformity

Oxide uniformity was studied by utilizing MOS capacitors. Large scale effects such as capacitor shorting to the substrate by defects in the oxide were readily identified by

ASLEEP. Figure 7 shows a three-dimensional surface potential map of 0.7-mm diameter aluminum capacitor dots on 0.89-mm centers on silicon dioxide. Increasing surface potential is directed downward. The oxide and some of the dots are charged to −10 V. The dots which are raised are over regions of thinner oxide where the capacitance is large enough that the signal is capacitively coupled to the target current amplifier.

However, charging of the capacitors by the electron beam made it difficult to extract quantitative information. During the mapping operation, to determine the coordinates of each capacitor, the capacitors were scanned and charged. The oxide of the samples was of high enough quality that over a week was required for them to discharge. This problem could be avoided by using a sample which has a reference mark on it to which the capacitor coordinate could be related. Then only the reference mark would be scanned to determine the coordinates of all of the capacitors. The procedure for obtaining a quantitative measurement is outlined as follows:

a. The uncharged capacitor is probed with a small current to measure the surface potential. It is important in the case of high resistivity oxide capacitors to use a current small enough that the charge deposited during the surface potential measurement is negligible compared to the charge deposited in step b.

b. Then the sample is charged by scanning with a few volts applied to the cathode. The charge placed on the sample is $Q = jAt$ where j = charging current, A = capacitor area, and t = charging time.

c. After charging, the sample is probed and the surface potential is measured again. The capacitance may be calculated from $C = Q/\Delta V$, where ΔV is the change in surface potential.

d. If the thickness, h, of the oxide dielectric in the capacitor is known and assumed to be constant, the dielectric constant may be determined from $\epsilon = Ch/A$. By scanning a number of capacitors the variation in ϵ will determine the homogeneity of the oxide.

3.2 Silicon

3.2.1. Semiconductor Defect Density

Edge dislocation pipes and oxidation stacking faults were investigated by ASLEEP. Edge dislocations were induced by oxidizing a (100) oriented silicon slice at 950°C for 1 h in order to induce dislocation climb to the oxide-silicon interface. The oxide was stripped from the sample before analysis. Typical edge dislocation pipes are shown in figure 8 as indicated by the arrow. Also shown are the electrically active region of the dislocation pipe and the recombination contrast of the radial defect structure which surrounds the core of the dislocation pipe. The broad contrast zone has been attributed to correspond to an increased minority carrier flow surrounding the dislocation core [3]. This effect results from the annular depletion region predicted from electron-beam-induced-current (EBIC) images [4]. At the center of each defect a surface micropit was observed by scanning electron microscopy (SEM) analysis. The above analysis illustrates the applicability of ASLEEP to defect density studies if the defects to be analyzed are larger than the 6-μm spatial resolution limit of the instrument.

The image depicted in figure 8 was formed by initially collecting the electron current and then differentiating it. Differentiation of the signal de-emphasized low frequency

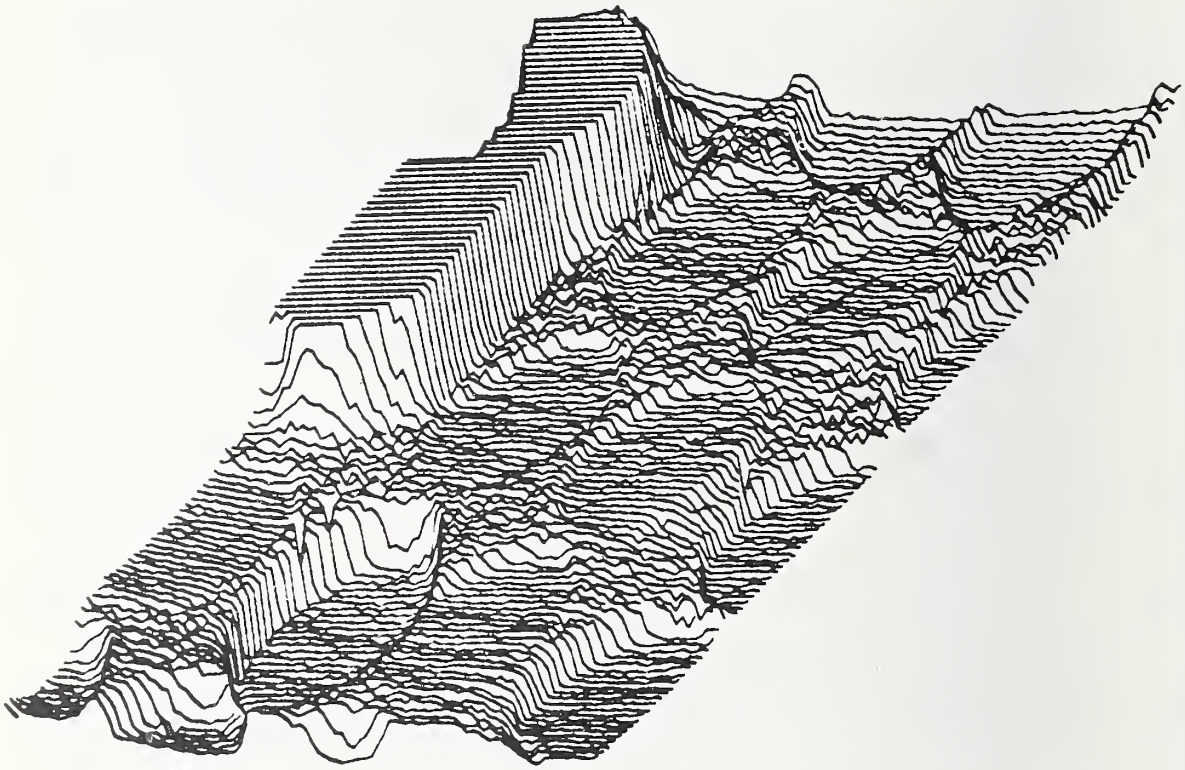


Figure 7. Three-dimensional surface potential map of capacitors.

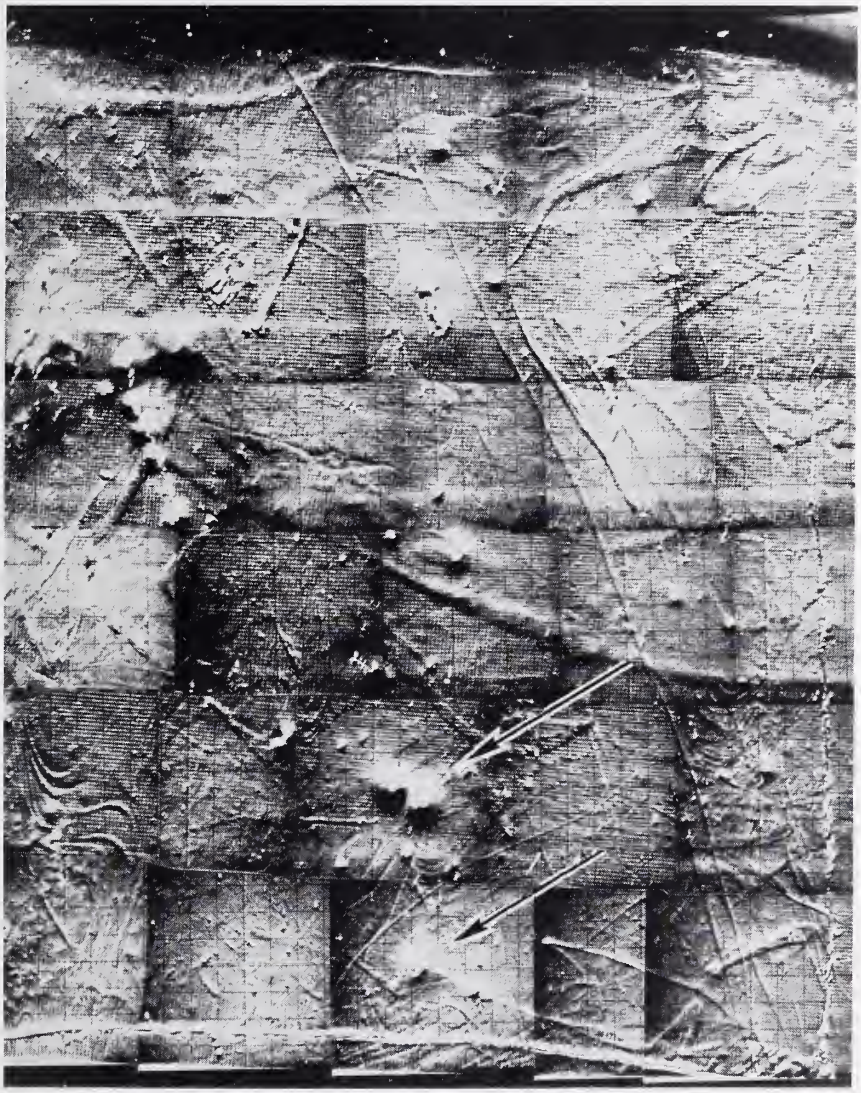


Figure 8. ASLEEP micrograph showing dislocation pipes in silicon wafer as denoted by arrows. The dimensions of area examined are 2 mm by 2 mm.

variations in the target signal due to decreasing electron beam intensity off axis of the electron gun. Differentiation also removed the dc component of the target current and emphasized small variations superimposed on the dc level. The mosaic (6 mm × 7.5 mm) was assembled by scanning a region of the target and then repositioning the target to bring another area into view of the electron gun. Experiments on sputtering to remove native oxides which have been attempted on other samples indicated that the fine lines in the mosaic are produced by variations in the native oxide and by surface contamination.

The second silicon defect density experiment conducted was the analysis of a (100) wafer which contained process-induced defects. The wafer had been examined by X-ray topography and showed the characteristic Lomer-Cottrell dislocation networks (figure 9). Examination of the sample by ASLEEP indicated the presence of dislocation lines (figure 10) with the expected 60-deg angle of intersection, but it was impossible to establish a one-to-one correlation with the features in the X-ray topograph. Further examinations of other regions of the same wafer did not reveal additional instances of these dislocations in regions where the X-ray topograph indicated they would be found. These results can be explained as follows: (1) if the surface defects have an associated surface potential variation of less than ± 10 mV and/or a lateral extent of less than 6 μm, ASLEEP cannot resolve the defect; and (2) if the oxide/contamination effects described above dominate surface potential variations associated with the dislocations, the defect structure cannot be correlated with the X-ray topographs. These results do not rule out this application for ASLEEP, but further support the need for highly controlled *in situ* surface cleaning techniques. The general applicability of ASLEEP for examination of silicon is dependent upon the development of such processes.

3.2.2. Semiconductor Resistivity-Impurity Profiling

Changes in semiconductor surface potential resulting from a variation in impurity concentration were investigated using ASLEEP imaging. Figure 11 shows a hypothetical energy band diagram showing the position of the Fermi level, E_F, the intrinsic Fermi level, E_i, the band gap, E_g, the electron affinity χ, the surface potential ϕ_n in the n-type region and the surface potential ϕ_p of the p-type region. From figure 11,

$$\phi_n = \chi + \frac{E_g}{2} - (E_F - E_i) \tag{3}$$

and

$$\phi_p = \chi + \frac{E_g}{2} + (E_i - E_F). \tag{4}$$

Substituting for the electron and hole concentrations yields the final two expressions for surface potential:

$$\phi_n = \chi + \frac{E_g}{2} - kT \ln \frac{n}{n_i} \tag{5}$$

$$\phi_p = \chi + \frac{E_g}{2} - kT \ln \frac{p}{p_i}. \tag{6}$$

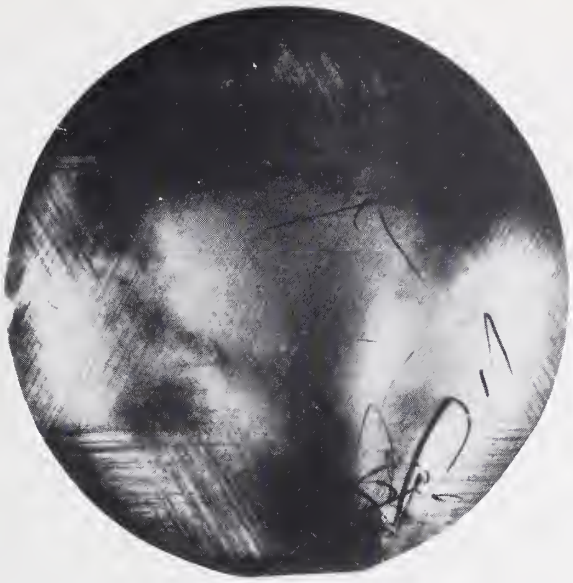


Figure 9. X-ray topograph showing Lomer-Cattrell dislocation networks.

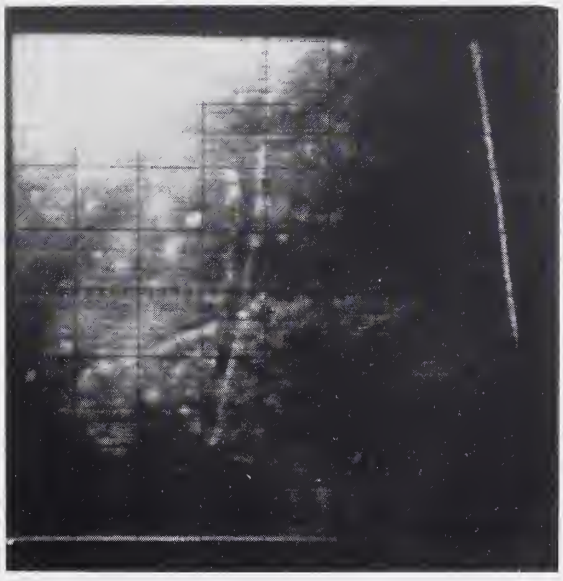


Figure 10. ASLEEP image of X-ray topograph wafer. The dimensions of the area examined are 2 mm by 2 mm.

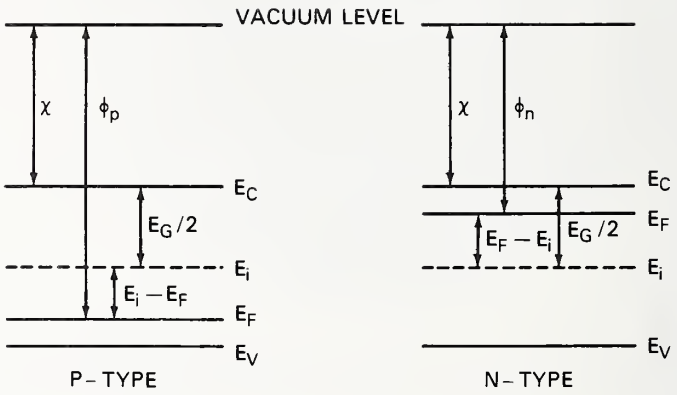


Figure 11. Energy band diagram for silicon showing the position of the Fermi level, the intrinsic Fermi level, the band gap, electron affinity, and surface potential.

The difference in surface potential between two n-type regions of doping densities n_1 and n_2 is given by

$$\Delta\phi_n = \chi + \frac{E_g}{2} - kT \ln \frac{n_1}{n_i} - \chi - \frac{E_g}{2} + kT \ln \frac{n_2}{n_i} = kT \ln \frac{n_2}{n_i} . \quad (7)$$

It should be emphasized that this simple analysis does not take into account the effect of surface states on the surface potential measurements discussed below.

Semiconductor resistivity was investigated by utilizing ion-implanted calibration samples. A p on p^+ wafer of silicon was implanted with phosphorus to create regions with donor densities of 1×10^{20} cm^{-3}, 1×10^{19} cm^{-3}, 1×10^{18} cm^{-3} and 1×10^{17} cm^{-3}. The p-type material was doped to 2×10^{15} cm^{-3}. After the sample was implanted and annealed at 1000°C for 30 min, the oxide which grew during the anneal was stripped and the resistivity of the sample was measured by the four-probe method to verify that the expected carrier concentrations were present. The sample was then given an HF dip and placed in the ASLEEP system. The UHV system was pumped until a pressure of 3×10^{-8} torr was reached, the electron gun system was operated for 2 h until the emission stabilized, and then surface potential measurements were made.

Figure 12a shows the surface potential variations expected for the implanted impurity concentrations as based on a simple one-donor level model. Figure 12b shows the surface potential variation following the work of Allen and Gobeli [5] for a clean silicon surface dominated by surface states. It may be seen that in either case the surface potential variation, over the concentration range chosen, is well within the resolution of ASLEEP. Figure 12c shows typical experimental observations in the implanted samples. It was postulated that oxide/contamination effects were dominating doping effects since the potential shifts are not consistent with figures 12a or 12b and in the area of the junction between the 10^{18} cm^{-3} and 10^{17} cm^{-3} doped n-type regions and the p-type region all traces of doping fade out and the three regions became indistinguishable.

The sample was then sputtered with 2-kV argon ions to remove the surface contamination layer and native oxide. Work function measurements were made after sputtering, but the results were similar to those shown in figure 12c. The sample was then annealed in a vacuum of 2×10^{-7} torr for 2 h at 600°C. Work function measurements were made again and the results were similar to those shown in figure 12c. These results lead to the conclusion that native oxide and/or vacuum-processing-related contamination dominated the surface potential variation. The NRL ASLEEP system has a demonstrated resolution of 10 mV. If the silicon surface were clean either prior to sputter/anneal or subsequent to sputter/anneal, the work of Allen and Gobeli [5] indicates that the surface potential variations should have been easily resolved. It was suspected, then, that the vacuum system sputter/anneal processing was inadequate to completely remove native oxides and/or contamination layers *in situ*. This limitation was verified by subsequent anneal/sputter experiments utilizing low-energy electron diffraction (LEED) and Auger electron spectroscopy.

3.2.3. Integrated Circuit Testing

The final application of ASLEEP to silicon was in the area of contactless integrated circuit testing. The ASLEEP system can be used to analyze integrated circuits provided access tabs are made available to critical portions of the circuit. The low-energy beam

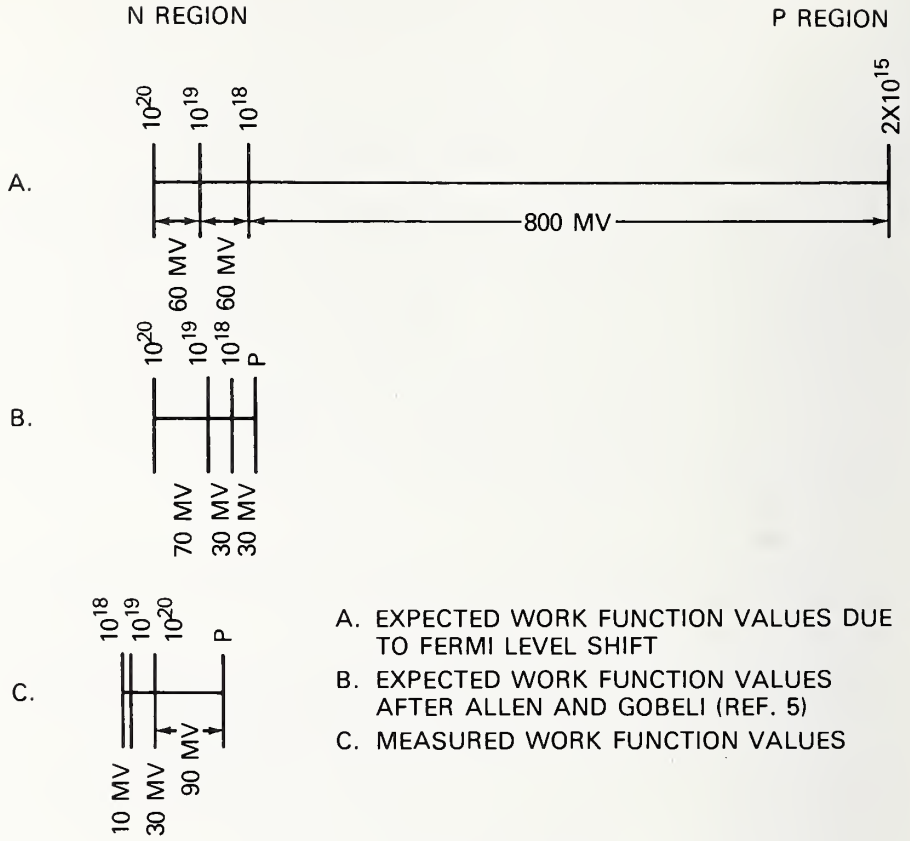


Figure 12. Work functions of ion-implanted silicon.
(A) Expected work function values from Fermi level shifts.
(B) Expected work function values after Allen and Gobeli [5].
(C) Measured work function values.

provides a nondestructive electrical contact to the device and can be programmed to provide acceptance testing of electrical performance on an on-line application.

Three integrated circuit chips were pulled from the production line before the glass overcoat was applied; hence, all of the metallization was exposed (fig. 13). At first examination of the circuit, 1.5-V electrons possessed adequate energy to cross the surface potential barrier of the metal interconnects (fig. 14b shows the ASLEEP image). However, as scanning continued, the oxide visible between the interconnects charged to the extent that tangential fields created at the sample surface prevented any electrons from being collected by the metallization. Increasing the electron energy brings the image back until the oxide charges further. Once the sample has charged, irradiation with high-energy electrons or ultraviolet light was required to discharge the sample. This problem makes it impossible to view a surface potential image of the integrated circuit for more than a few minutes. In an ASLEEP system for production line testing of integrated circuits, it would be possible to mechanically index the circuits so they could be precisely positioned in front of the electron gun. The computer could then steer the beam so that only the metal interconnects would be proved with low-energy electrons.

3.3 ASLEEP Investigations on III-V Compounds

Annealing III-V compounds in an ultrahigh-vacuum chamber has been shown to produce a perfectly clean and well ordered surface [6, 7]. Thus, annealing compounds such as gallium arsenide (GaAs) and indium phosphide (InP) in the ASLEEP vacuum surface should eliminate the oxide charging problems encountered with the silicon experiments.

3.3.1. Indium Phosphide

ASLEEP experiments were conducted as a function of anneal temperature on (100) *n*-type indium phosphide grown on semi-insulating (SI) surfaces. Areas on the indium phosphide surface which exhibited variations in surface potential were analyzed by Auger electron spectroscopy capable of 100-nm resolution. The indium phosphide surface was initially rinsed in acetone and polished by a 1% (by volume) solution of bromine in methanol for 5 min. The same surface was then etched in a dilute solution by hydrofluoric acid for 1 min, rinsed in de-ionized water, and then etched with the MB-etching solution which consists of 10 mL of hydrochloric acid, 10 mL of hydrofluoric acid and 5 drops of hydrogen peroxide in 40 mL of water [8] for 10 min (the entire procedure is denoted as the MB etch). For purposes of comparison, two indium phosphide samples were mounted side by side for ASLEEP examination. One sample had the acetone rinse with no further etching, while the second sample had the MB etch described above. Prior to the ASLEEP investigation, an *in situ* Auger-anneal experiment indicated that desorption of hydrocarbons from indium phosphide surfaces takes place at 250°C. Further annealing above 250°C results in the desorption of phosphorus oxide at 275°C leaving behind an oxidized indium-rich layer which is further desorbed in the vicinity of 310°C to 330°C. Decomposition of indium phosphide was found to occur at 420°C.

The ASLEEP images of the acetone-cleaned and MB-etched indium phosphide surfaces are shown in figures 15 and 16. Typical regions analyzed with Auger electron spectroscopy (AES) are also indicated on the figures. Regions which showed variations in surface potential were invariably identified with a high oxygen content as indicated by the oxygen transition at 510 eV. The phosphorus $L_{2,3}$ VV transition [9] at 120 eV was reduced in magnitude from the expected phosphorus LVV peak height and was also characterized by new peaks at 100 eV and 110 eV. These peaks are phosphorus oxide peaks denoted as $PL_{2,3}OL_{2,3}OL_{2,3}$ at 110 eV and $PL_{2,3}OL_1$ V at 100 eV (not shown on photo-

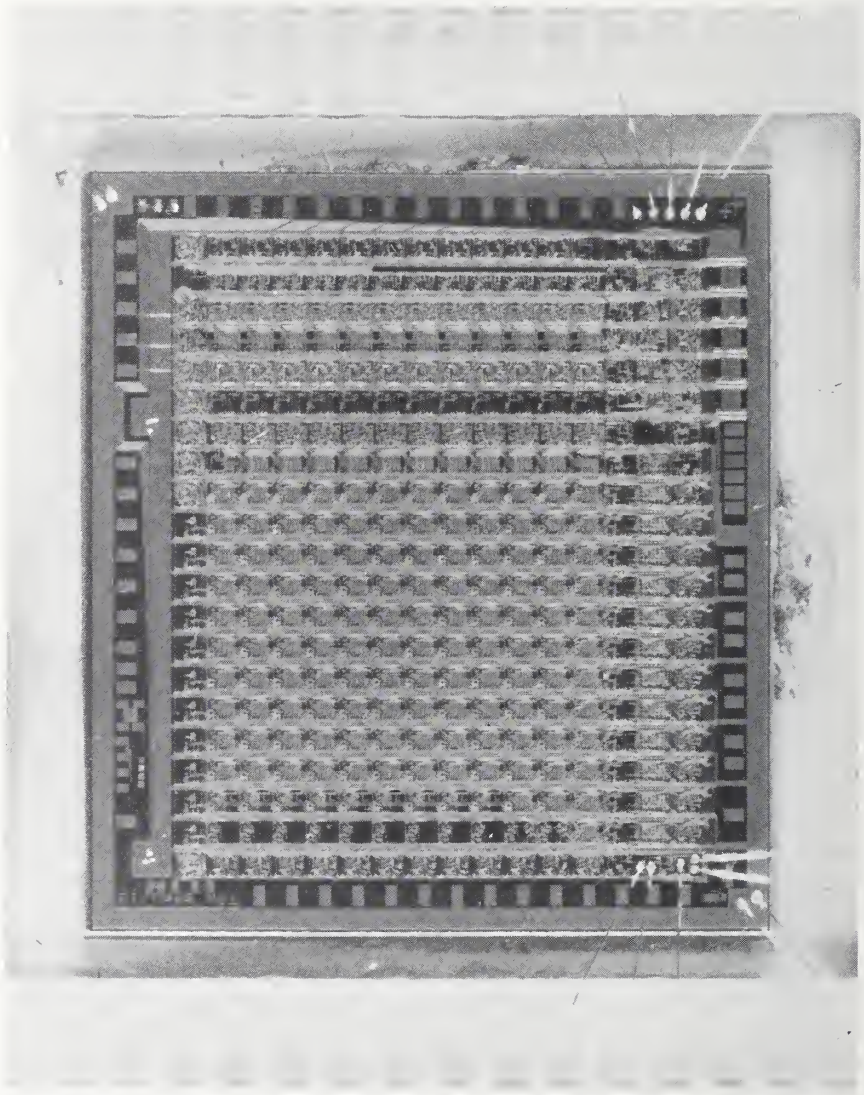
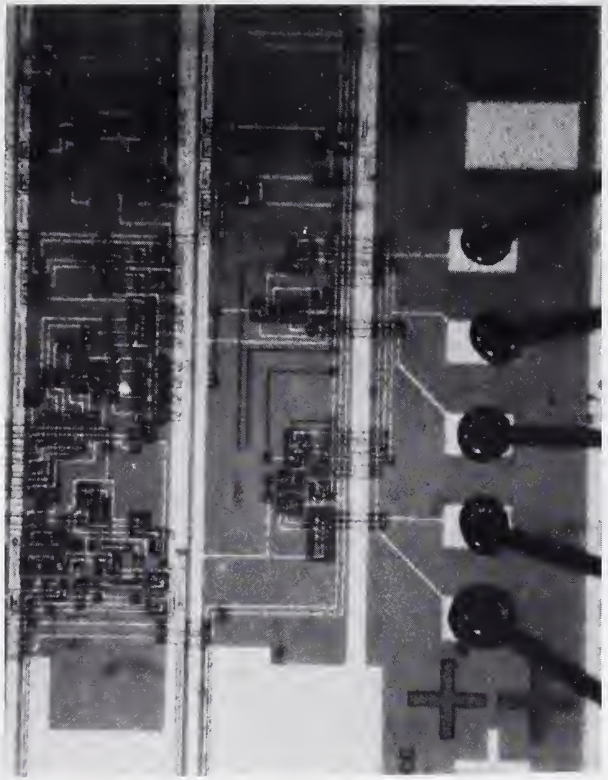


Figure 13. Optical photomicrograph of integrated circuit utilized in the ASLEEP experiment. The dimensions of the integrated circuit are 120 mm by 120 mm.



(A) Optical photomicrograph of test circuit.

(B) ASLEEP image of test circuit.

Figure 14. Micrographs of integrated circuit showing specific area (5 mm by 5 mm) analyzed.

graph). The indium transition at 404 eV was also reduced in magnitude from the expected indium peak height. The extra peaks and above characteristics were only observed in areas A and B and are associated with the desorption of phosphorus compounds. The above characteristics were not apparent in other regions analyzed and are not consistent with transitions involving indium and indium compounds. A small dip in the spectra at 84 eV was also observed, which indicates the presence of the $PL_{2,3}OL_1OL_1$ transition. The MB-etched sample had a limited number of similar regions as did the acetone-cleaned sample as shown in figure 16. The Auger spectrum of each region analyzed on the MB-etched sample was also characterized by the large oxygen peak at 510 eV and phosphorus oxide peaks at 110 eV and 84 eV.

The samples shown in figures 15 and 16 were subsequently annealed at 330°C for 15 min. The ASLEEP images of figure 17 showed a perfectly uniform surface potential map for the MB-etched surface and a relatively well behaved surface potential map for the acetone-cleaned surface. The Auger spectra of regions shown indicate that oxygen has been desorbed probably in the form of the phosphorus and indium oxides. The 510-eV oxygen transition has disappeared while the indium and phosphorus transitions have recovered the peak-to-peak height expected for uncontaminated indium phosphide. All of the observed features of the Auger spectra may be attributed to indium and phosphorus, together with their characteristic energy loss peaks. Immediately after attaining the ASLEEP images, LEED experiments were conducted on the annealed surfaces. It is noted that prior to annealing, the LEED pattern was obscured completely. Upon heating to 330°C, the LEED pattern appeared at 35 eV and consisted of two sets of 4 × 1 patterns rotated 90 deg. This pattern is in general agreement with the reconstructed indium phosphide surface observed by McRae [7]. From this experiment we have concluded that ASLEEP can successfully map surface potential variations which arise from local changes in material stoichiometry.

In order to further test the previous observations that surface stoichiometry variations in indium phosphide resulted in surface potential differences, controlled oxidation experiments were conducted on the originally desorbed indium phosphide surface. The ASLEEP image prior to oxidation is shown in figure 18. Random AES profiles also shown in figure 18 indicate features attributed to indium and phosphorus. The indium:phosphorus peak ratio is approximately 0.33. Oxygen was allowed to leak into the UHV ASLEEP system until the pressure approached 1×10^{-3} torr, while the substrate was maintained at 125°C. After an exposure of 10 min at 10^{-3} torr, the system was evacuated to the 10^{-9} torr range. The ASLEEP image after oxidation (fig. 19) shows irregular regions with one well defined flat region. The Auger spectrum is typical of contaminated indium phosphide with carbon and oxygen present; the peak-to-peak height of the phosphorus transition at 120 eV is drastically reduced from that expected for uncontaminated indium phosphide. The irregular regions, in addition to the phosphorus $L_{2,3}$ VV transition at 120 eV, also showed the previously discussed phosphorus oxide peaks at 110 and 100 eV. . Exposure at 10^{-3} torr also decreased the phosphorus:indium peak ratio which is indicative of oxygen penetration into the bulk. Therefore, from the oxidation experiment, localized surface potential variations can be attained which are attributed to indium, phosphorus, and oxygen variations.

3.3.2. Gallium Arsenide

ASLEEP imaging was applied to the study of gallium arsenide (1) surface contamination and (2) surface etchants. State-of-the-art etchants for gallium arsenide utilize halogens such as chlorine and bromine which segregate at select surface sites resulting in surface potential variations. Figure 20 shows a comparison between the ASLEEP image, SEM

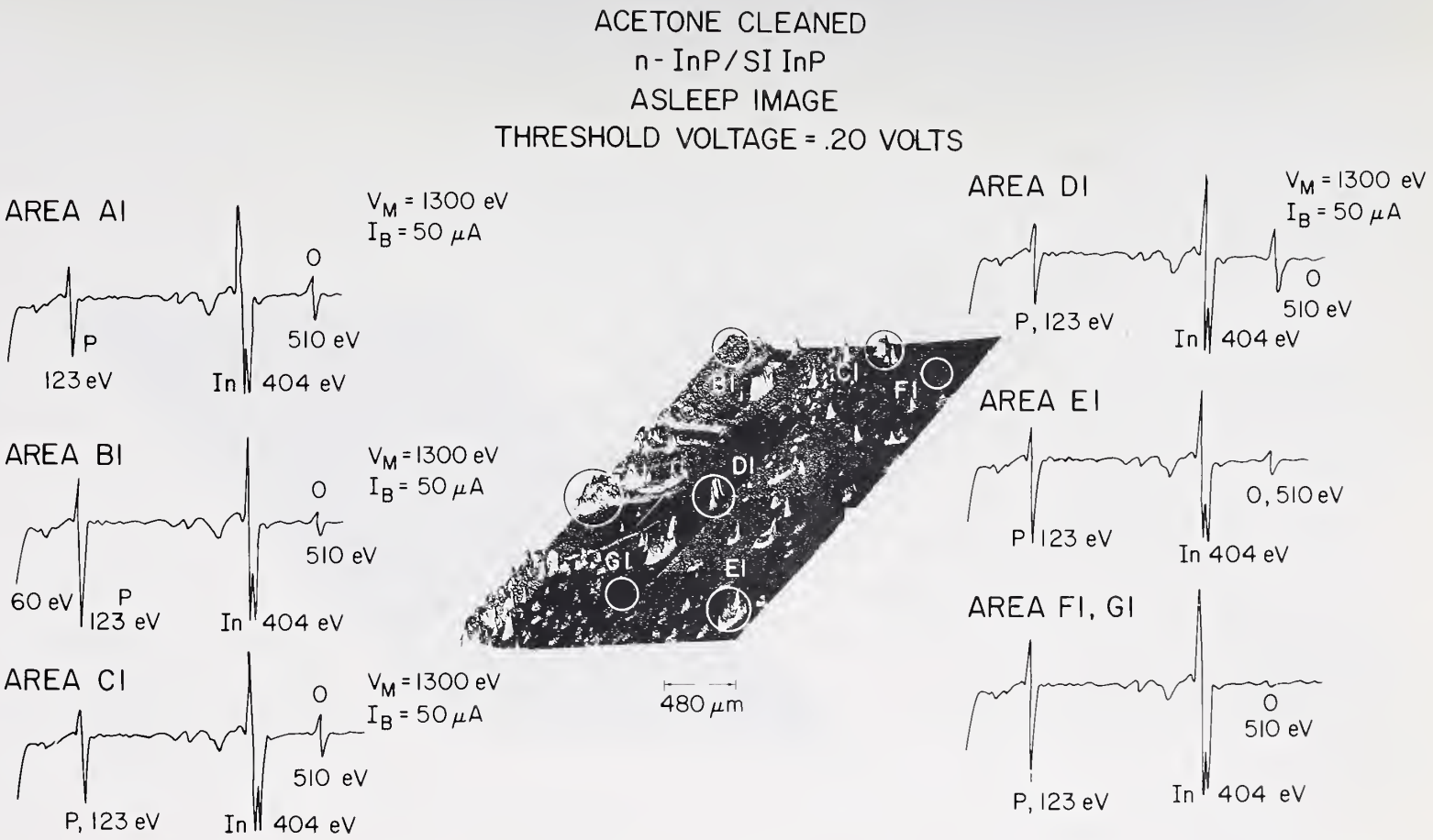


Figure 15. ASLEEP image and Auger spectra of indium phosphide surface after an acetone rinse.

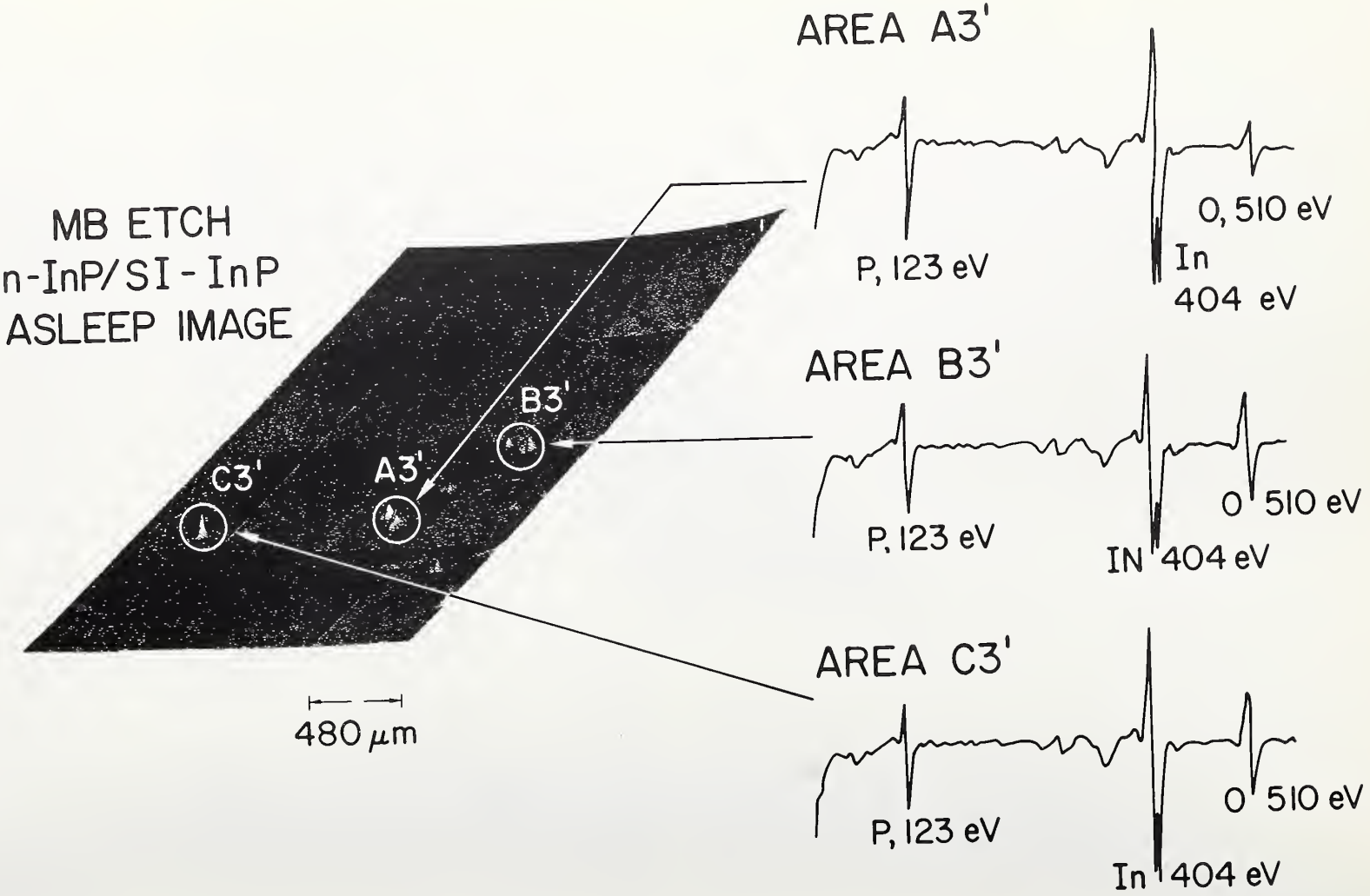


Figure 16. ASLEEP image and Auger spectra of MB-etched [8] indium phosphide surface.

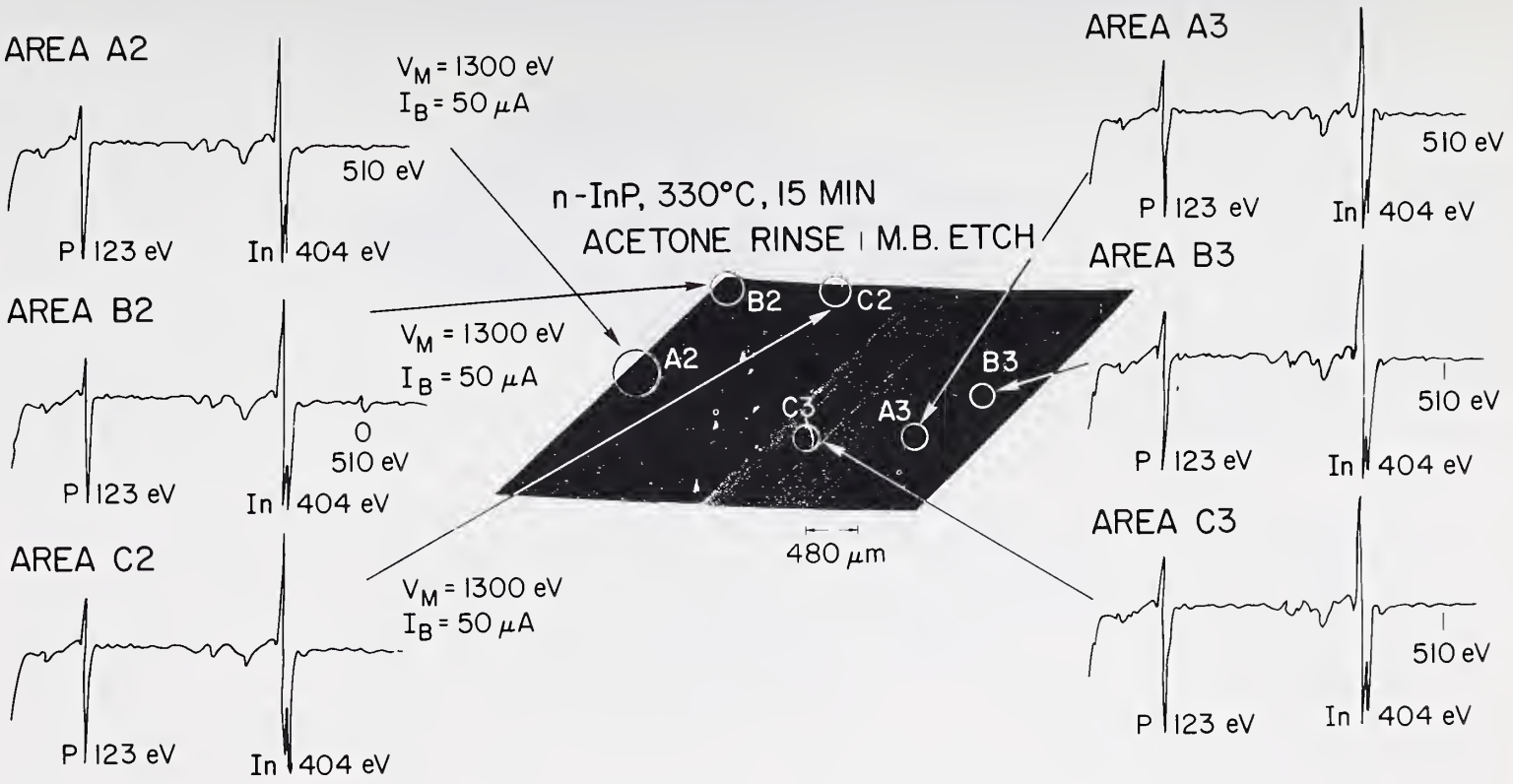


Figure 17. ASLEEP image and Auger electron spectra of indium phosphide annealed at 330°C for 15 min.

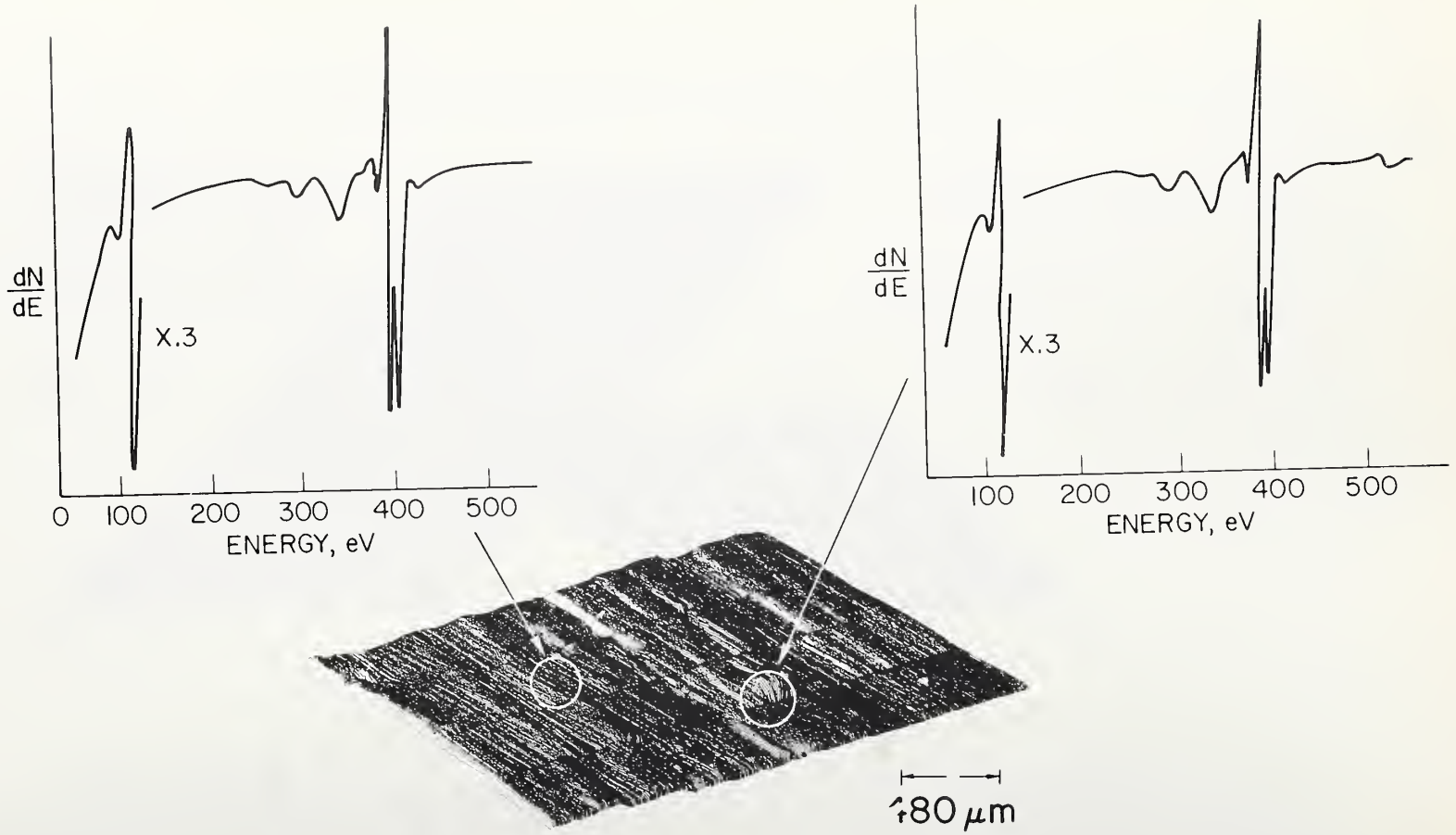


Figure 18. ASLEEP image of indium phosphide prior to oxidation treatment.

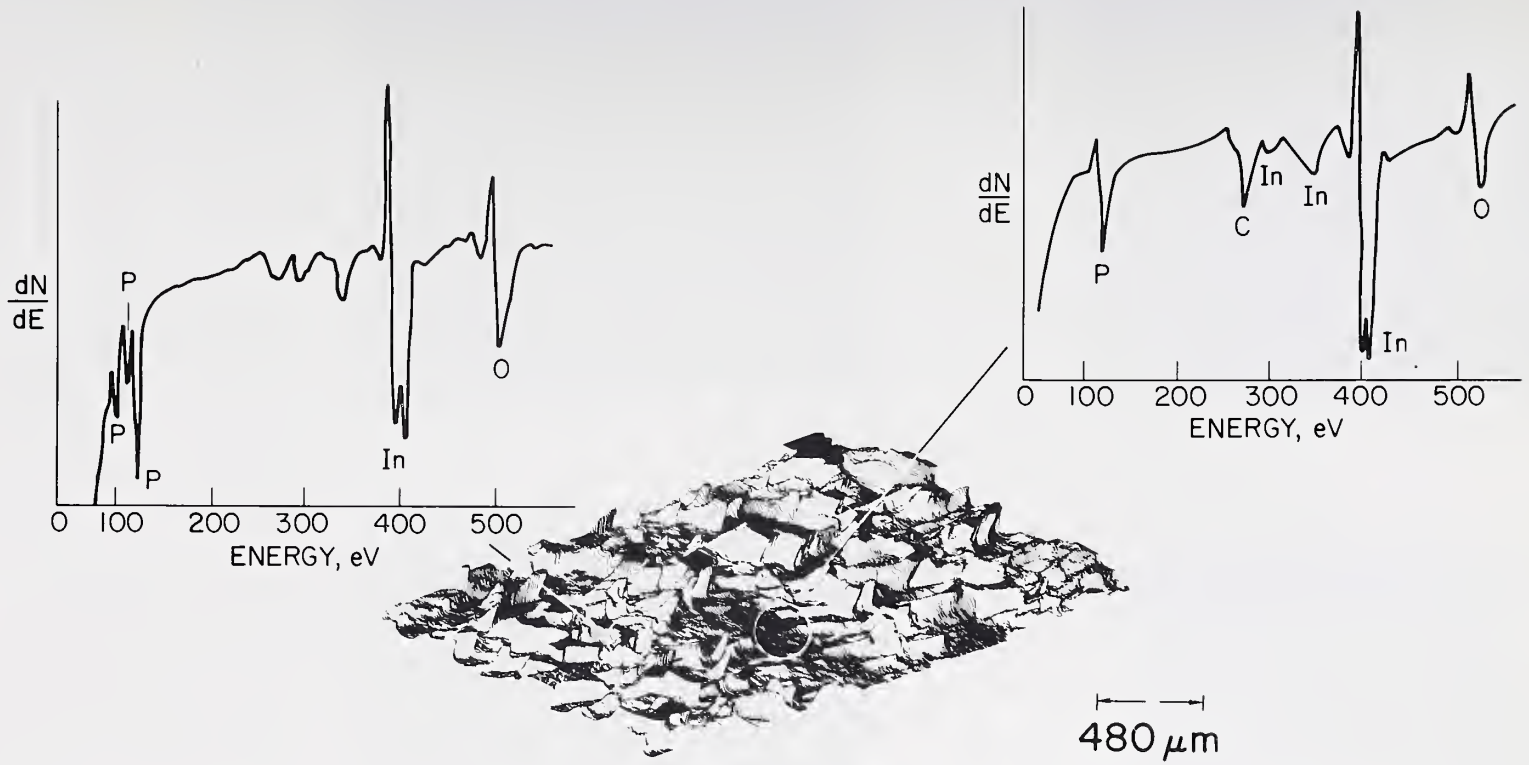


Figure 19. ASLEEP image of indium phosphide after oxidation.

image and scanning spot Auger oxygen and chlorine images on n-type gallium arsenide taken after a standard etch treatment. The wafer was processed as follows: 2 min in MB etching solution followed by a dip in 10 mL of hydrofluoric acid for 10 s and a cascade rinse. The round features of the ASLEEP image are shown to consist of adsorbed oxygen and chlorine, while the elliptical feature is probably due predominantly to adsorbed chlorides. In this case the correlation of ASLEEP with Auger and SEM imaging has clearly explained the features observed in the ASLEEP image.

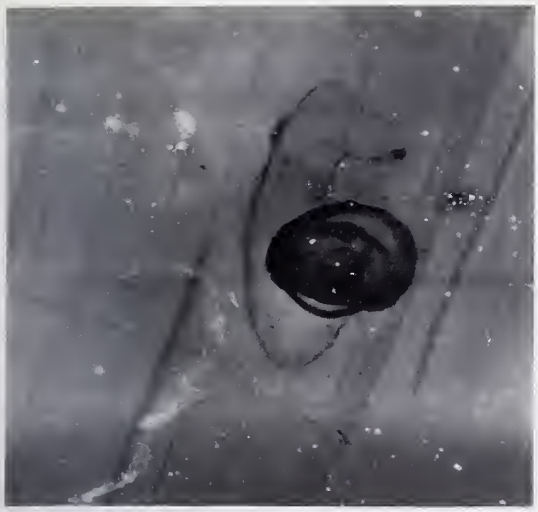
In a second experiment an n-type gallium arsenide epitaxial layer grown on an n^+-type gallium arsenide substrate was utilized for comparison of two candidate etchants. The gallium arsenide sample was divided in half and both halves were degreased, rinsed in methyl alcohol, and silver indium germanium contacts were evaporated on the back. The small half was process as follows: dipped in 10-mL hydrofluoric acid for 2 s, followed by a deionized water rinse; etched in 10-mL hydrochloric acid for 2 min; dipped in 10-mL solution of hydrofluoric acid fo 10 s, followed by a cascade rinse. The second half was processed as follows: dipped in 10-mL hydrofluoric acid solution for 2 s; rinsed in deionized water; dipped in 10-mL solution of hydro chloric acid for 30 s; rinsed in deionized water; dipped in 10-mL hydrofluoric acid solution for 10 s, and etched in MB-etching solution for 10 min, followed by a cascade rinse. The samples were dried in nitrogen and immediately mounted in the ASLEEP system. The MB-etched half as shown in figure 21 has a lower density of topographical features and a lower overall surface potential than the routinely treated half. Statistical data were taken in order to determine the surface potential difference between the MB-etched side and the standard side. The surface potential difference is 1.18 V, and it is clear that the distribution function for the MB-etch is tighter than for the standard etch. An optical inspection of the sample was made and it became obvious that most of the features seen in the ASLEEP image were not present in the optical images. In particular, the swirl which appears on the MB-etched side was not observed by optical microscopy nor by the scanning electron microscope. These experiments were performed in order to demonstrate the utilizability of ASLEEP for optimizing semiconductor processing.

4. CONCLUSIONS

This investigation has revealed the following points in connection with the application of ASLEEP to the silicon investigations.

1. ASLEEP has the capability to measure quantitatively the surface work function variations associated with the doping level normally occurring in the silicon device technology. This is so, even taking into account the effect of surface states on compressing the Fermi level shift. As shown in the work of Gobeli and Allen [5], for an n-type material the work function variation is from approximately 4.82 to 4.70 eV ($\Delta\phi \simeq 0.12$ eV) for doping density changes from approximately 10^{15} to 10^{20} cm^{-3}. A similar variation occurs for p-type material up to $\sim 2 \times 10^{19}$ cm^{-3}. The resolution of ASLEEP is 10 mV and thus this remains an important application of the ASLEEP technology. However, the present investigation including utilization of LEED and Auger analysis has shown that native oxides and processing related contamination factors must be controlled in order to allow meaningful measurement of the surface potential.

2. The question of the utilizability of ASLEEP for defect and fault detection in silicon was resolved during the contract period. Large defect structures such as dislocation pipes were observed and correlated with SEM and optical microscopy. In the case of oxidation-induced stacking faults which are below the spatial and surface potential resolution



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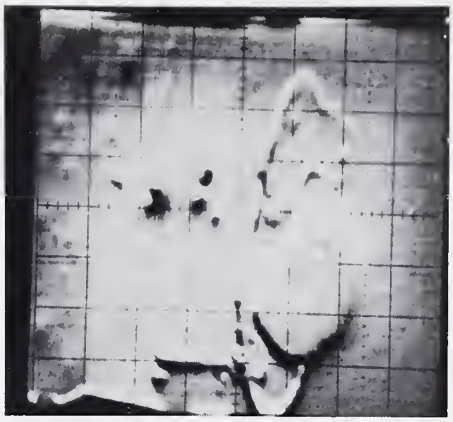
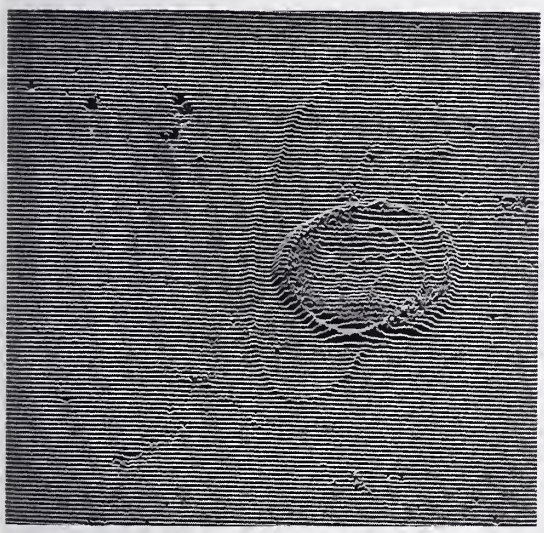


ASLEEP IMAGE 90X



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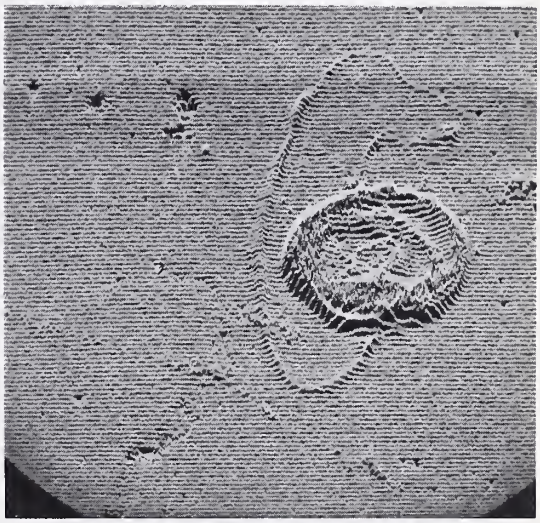

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Figure 20. A comparison of ASLEEP image, SEM image, and scanning Auger images of gallium arsenide contamination.

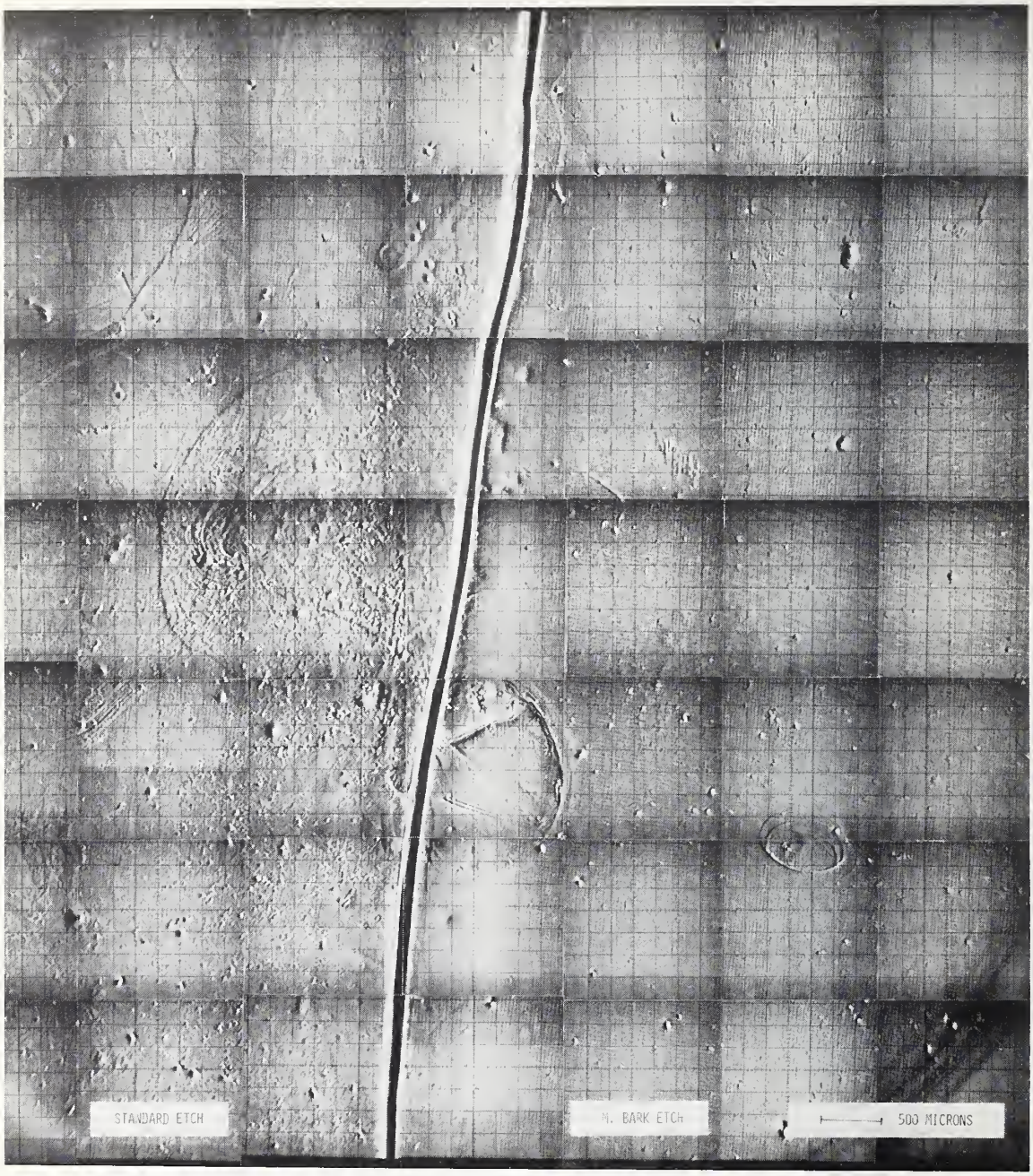

Figure 21. ASLEEP image of gallium arsenide wafer showing the effects of two different etch treatments.

limits of the system, ASLEEP is clearly not applicable. In the case of certain surface and sub-surface faults, it is believed that the surface contamination problem described above is dominant and must be solved before these defects can be observed.

3. Studies of the uniformity of oxide thickness and dielectric constant proved to be impractical since provision was not made for routine discharging of the oxide surface after initial charging by the scanning electron beam. However, discharging could be accomplished by pulse sampling techniques.

4. In order to avoid unwanted variations in electron energy, the retarding field must be uniform, which means that the target must be flat.

5. Improved gun design is required to reduce beam size, increase current density, and reduce the loss of low-energy electrons from the beam.

6. Oxide charging, spatial resolution limitations, and deflection amplifier drifts make examination of state-of-the-art integrated circuits marginal with the present equipment. However, further development effort could solve these problems.

The application of ASLEEP to indium phosphide and gallium arsenide proved successful since oxides and contaminants were successfully desorbed by *in situ* annealing. ASLEEP was therefore successfully utilized to study the following problems to provide data which could be used to optimize indium phosphide and gallium arsenide processing.

1. Surface potential variations in indium phosphide and gallium arsenide due to lateral inhomogeneities.

2. Surface potential variations in indium phosphide and gallium arsenide due to adsorbed contaminants from processing.

3. Effects of various processing steps on surface potential variations.

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REFERENCES

1. Haas, G. A., and Thomas, R. E., Electron Beam Scanning Technique for Measuring Surface Work Function Variations, *Surface Science* 4, 64-75 (1966).

2. Strayer, W., Mackie, W., and Swanson, L. W., Work Function Measurements by the Field Emission Retarding Potential Method, *Surface Science* 34, 225-235 (1973).

3. Ravi, K. V., and Varker, C. J., Edge Dislocations in Silicon, *J. Appl. Phys.*, 45, 263-271 (1974).

4. Ravi, K. V., Dislocation Pipes in Oxidized Silicon, *J. Appl. Phys.* 43, 1785-1791 (1972).

5. Allen, F. G., and Gobeli, G. W., Work Function, Photoelectric Threshold, and Surface States of Atomically Clean Silicon, *Phys. Rev.* 127, 150-159 (1962).

6. Chang, K. H., and Meijer, P. H., Surface Properties of Indium Phosphide, *J. Vac. Sci. Technol.* 14, 789-796 (1977).

7. "MacRae, A. U., LEED Examination of Indium Phosphide and Gallium Arsenide, *J. Vac. Sci. Technol.* 4, 247-255 (1966).

8. Bark, Marvin, NRL, private communication.

9. Siegbahn, K., "ESCA-Atomic, Molecular and Solid State Structure Studied by Means of Electron Spectroscopy," in Nova ACTA REGIAE Soc., Ser. 4, Vol. 20, North Holland Publishing Co., Amsterdam-London (1967).

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16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

This report summarizes the results of a three-year effort in the development of a computer Automated Scanning Low-Energy Electron Probe (ASLEEP) for semiconductor wafer diagnostics. Experiments designed to explore the measurement capabilities of ASLEEP for measurements on silicon, gallium arsenide, and indium phosphide are described. Four areas were emphasized: 1) semiconductor resistivity, 2) semiconductor defect density, 3) lateral inhomogeneities, and 4) oxide uniformity. Although oxide charging problems limit the utility of ASLEEP in its present form for silicon, defects and lateral inhomogeneities could be readily detected in gallium arsenide and indium phosphide.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)* Automated scanning low-energy electron probe; lateral inhomogeneity; oxide uniformity; scanning low-energy electron probe; semiconductor defect density; semiconductor resistivity; semiconductor wafer diagnostics.

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