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TRACE ELEMENT CHARACTERIZATION IN III-V COMPOUNDS SPUTTER BY SPUTTER INITIATED RESONANCE IONIZATION SPECTROSCOPY

Atom Sciences, Inc.

James E. Parks

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The VLSI and VHISC development programs of the Air Force require characterization of III-V compounds with an interference free, highly sensitive element analysis technique having good lateral and depth resolution. Sputter Initiated Resonance Ionization Spectroscopy (SIRIS), an ultrasensitive analytical technique being developed commercially by Atom Sciences, has been demonstrated to have good sensitivity and to be interference free in silicon. Detection limits of 2 ppb ($1 \times 10^{14}/\text{cm}^3$) have been shown for gallium in bulk silicon. Prior to the work reported here, lateral and depth resolution of the SIRIS apparatus was not adequate for the present and future needs of the semiconductor industry. Lateral resolutions of 100 to 5 microns are now necessary, and resolutions into the submicron range will be required in the future. In the project reported here, the SIRIS instrument was modified to include a microbeam ion gun system, and the new instrument was demonstrated to have a lateral resolution less than 100 microns with a (continued on reverse)					
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detection limit in the few ppb range. An ion beam of about 5 microns diameter was demonstrated with a predicted detection limit of 0.1-1 ppm.

A layered Si doped sample of GaAs and AlGaAs was analyzed with SIRIS for Si concentration. The technique was shown to be capable of interference free, matrix independent measurements in the two materials and near the interfaces. Depth resolution of the SIRIS method was determined by monitoring the aluminum concentration in a sample as sputtering progressed from GaAs to an AlGaAs layer and then to another GaAs layer. Using 10 keV argon ions, a resolution of 140 A (Angstroms) was measured. Future measurements at lower sputtering energies should produce measurements with depth resolution well below the desired 100 A limit.

Most importantly, this work has outlined a procedure for extending the lateral resolution of the SIRIS technique and apparatus to 1000 A. This feasibility study has shown that even with lateral resolution of 1000 A, the SIRIS technique can maintain sensitivities of 1-100 ppm. We believe that the work reported here warrants further improvement of the SIRIS technique and apparatus for making useful submicron resolution measurements.

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PREFACE

The work reported here is the demonstration of the applicability of Sputter Initiated Resonance Ionization Spectroscopy to the analysis of III-V semiconducting compounds for trace amounts of various elements. This is a new technique possessing ultrahigh sensitivity and selectivity and excellent resolution both laterally and in depth. The results for the concentrations of trace elements are independent of the bulk material in which they are found. Because of these properties, this work is of great interest to manufacturers and users of semiconductors, and is therefore closely related to the Air Force work with semiconductors, particularly to the VLSI and VHSIC development programs.

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INTRODUCTION

The VLSI and VHISC development programs of the Air Force require characterization of III-V compounds with an interference-free, highly sensitive element analysis technique having good lateral and depth resolution. Sputter Initiated Resonance Ionization Spectroscopy (SIRIS), is a relatively new ultrasensitive analytical technique that combines ion beam sputtering for atomization of solid materials and lasers for the resonant ionization of sputtered neutrals. This technique developed by Atom Sciences has been demonstrated to have good sensitivity and to be interference free. Detection limits of 2 ppb ($1 \times 10^{14}/\text{cm}^3$) have been shown for gallium in bulk silicon. While having good sensitivity, the SIRIS apparatus as originally designed did not have adequate lateral and depth resolution for the present and future needs of the semiconductor industry. Lateral resolution of 100 to 5 microns is necessary, and resolution into the submicron range will be required in the future. The main thrust of this SBIR Phase I project was to determine the feasibility of using the SIRIS technique and apparatus developed at Atom Sciences for making ultrasensitive analyses of III-V compounds with high depth and lateral resolution, yet free from interferences and matrix effects.

The SIRIS apparatus was modified by installing a new commercial ion beam system, and it was tested for improved sensitivity and lateral resolution. A lateral resolution of 100 microns was readily achieved with the new system and measurements with this lateral resolution were made and are reported. The feasibility of improving lateral resolution to 5 microns was to be determined for a Phase II demonstration program, but in fact, a lateral resolution of 5 microns was achieved and is reported in this Phase I work. It would be highly desirable to achieve lateral resolutions of 1000 angstroms or less with SIRIS and yet maintain a useful sensitivity for analysis. The feasibility of achieving submicron resolutions was studied and it was determined that this would be feasible for SIRIS. With the early achievement of reaching a 5 micron resolution, it is now feasible to address submicron resolutions for SIRIS in a to-be-proposed Phase II project. A method for doing this is reported in the results and discussion.

SIRIS is a technique that measures the sputtered neutrals and as such should have minimal matrix effects. One of the problems facing the Air Force and the semiconductor industry in general is the change in useful yield in Secondary Ion Mass Spectrometry (SIMS) measurements when sputtering through an interface from one matrix material to another. A compound material made up of epitaxial layers of GaAs and AlGaAs, grown and doped with silicon by MBE was measured with SIRIS. The silicon, aluminum, and gallium concentrations were determined as the sample was sputtered through the layers and were found to agree with the concentrations determined in the MBE growth process. No discontinuities appeared in the data at the interface and the data indicated no significant matrix effect. Supporting data are reported and discussed in the results section.

The depth resolution of the SIRIS technique is determined mostly by the energy of the primary ion beam, as in the SIMS technique. In this work, it was determined that at 10 keV, depth resolution of approximately 140 angstroms was

possible. This is typical of SIMS measurements using primary ion beams with energies of this magnitude. In general, it is found in SIMS that lowering the energy of the ion beam to 2.5 keV will allow depth resolutions of 30 angstroms to be achieved. No measurements at lower energies were made in this project, but the new ion beam system installed for this Phase I work will allow ion beams of this energy.

Following in this report, we will review the significance of the problem, the technology of resonance ionization spectroscopy, the SIRIS technique, and the SIRIS apparatus. The objectives of the work will also be reviewed and discussed with the presentation of data and results. A final summary with recommendations will be presented.

REVIEW OF THE SIGNIFICANCE OF THE PROBLEM

The VLSI and VHSIC development programs of the Air Force and the semiconductor industry are requiring higher and higher lateral and depth resolution and sensitivity to specific elements in materials analysis. Thin layers of semiconducting material with very sharp interfaces have a number of properties that are interesting for semiconductor device development. Presently, molecular beam epitaxy (MBE) and metal organic chemical vapor deposition (MOCVD) are the primary methods used to grow the thin structures. There is interest in programs to improve these techniques and these programs will require new and improved measurement techniques to understand material properties that control device performance. The Air Force is interested in the development of experimental techniques to measure interface sharpness between layers of GaAs and AlGaAs or InGaAs to within a half a lattice constant. They are interested in developing ways to determine carrier concentration profiles within 100 angstroms of an interface. They are also interested in the development of measurement techniques that would give impurity and defect profiles close to interfaces for both residual and doping concentrations.

For example, an innovative device being developed for very high speed radar systems could use measurement methods with better lateral and depth resolution. In this example, a grid of tungsten is deposited on the surface of a GaAs substrate and covered with another layer of GaAs. This grid controls the device in a way reminiscent of a vacuum tube. Researchers are interested in determining the extent, if any, of tungsten diffusion into the GaAs. Both good lateral and depth resolution are required, as well as good sensitivity. Lateral resolution needs to be less than 100 microns, and depth resolution less than 100 angstroms. Detection limits need to be less than $1 \times 10^{15}/\text{cm}^3$ or 30 ppb in GaAs. It is known that tungsten levels in GaAs of $1 \times 10^{17}/\text{cm}^3$ will have an adverse effect of the performance of the device.

Secondary Ion Mass Spectrometry (SIMS) has long been the established analytical technique for measuring impurities in semiconductor materials. SIMS relies on the production of secondary ions by the sputtering process. The yield of secondary ions is highly dependent on the type of secondary ion, the host matrix, and the surface condition. A mass spectrometer is used for identification of the ionized species and is subject to interferences from

isobaric atoms and molecular ions. SIMS experiences difficulty in maintaining a balance between sensitivity and depth resolution for tungsten measurements in the device mentioned above. To have good sensitivity, SIMS must use an intense sputtering beam, but this causes sputtering to proceed too rapidly, thereby missing the thin, 300-400 angstrom tungsten layer. If the sputter rate is decreased to improve the depth resolution, then the sensitivity is compromised.

Isobaric interference is another limitation of the SIMS technique. If, for example, InP were being analyzed for Fe in the presence of Si, a mass spectrometer could not efficiently distinguish between $^{28}\text{Si}^+_2$ and $^{56}\text{Fe}^+$. Obviously, the situation is even worse if one were analyzing for Fe in pure Si. The largest limitation, though, is the dramatically differing ionization efficiency for the various elements. Some, like Te, Zn, and Ge do not ionize at all, while others only with very low efficiency. SIMS utilizes different ion species for their sputter beam in order to optimize the secondary ion yield.

Resonance Ionization Spectroscopy (RIS) is a new technique for ultra-trace element analysis. In this method, one or more laser wavelengths are tuned to specific transitions of an element. Because of the sharpness of atomic spectral lines, this ionization method is extremely selective as the laser wavelengths can be chosen to promote the electron from the ground state through several excited states that are unique to that particular element, and then, to the ionization continuum. The power available from modern pulsed dye lasers is sufficient to saturate most atomic transitions in a reasonable volume of space, making this method also extremely sensitive, as all selected atoms in that volume will be ionized. Once ionized, the selected atoms can be counted with conventional charged particle detectors. Unlike SIMS, the ionization efficiency of RIS should be constant for most elements. Atom Sciences, Inc. combined the ultra-sensitivity and selectivity of RIS with sputtering to provide a solid analysis technique (patent #4,442,354) which we call Sputter Initiated Resonance Ionization Spectroscopy (SIRIS). In this technique, an ion beam is used to remove (sputter) atoms from the surface of a solid sample. The expanding cloud, mostly neutral atoms with a few ions (SIMS ions, which are suppressed), is probed by the RIS laser beams that ionize all the atoms of the selected element within the laser beams. The technique eliminates interferences which are generated by non-resonant ionization processes such as thermal ionization and electron bombardment. SIRIS has demonstrated measurements of impurities in both silicon and gallium arsenide, matrices of interest to the semiconductor industry.

Detection limits as low as 2 ppb have been demonstrated at Atom Sciences using the present SIRIS instrument (1). The SIRIS technique, being a pulsed technique, does not sputter much material during the measurement process. In depth profiling measurements, ion milling to a specified depth, which can be controlled at a desired rate, is done with a dc ion beam. The SIRIS measurement is subsequently done with a pulsed beam. Therefore, the sputter rate and sensitivity are independent of each other. For example, with an ion current of 5 μA and pulse width of 1 μsec , only about 1×10^8 atoms will be sputtered. For an effective beam area of 1 mm^2 , the yield per unit area is $1 \times 10^{10}/\text{cm}^2$. As the GaAs surface density is $1.25 \times 10^{15}/\text{cm}^2$, it will require 10^5 ion pulses to remove an equivalent monolayer. It has been our experience that even at low ppb concentrations in the sample, 10^5 ion pulses (~ 50 minutes at 30 Hz) yield

sufficient signal-to-noise. Therefore, the combination of high sensitivity with low sputter rate suggests that the SIRIS technique could yield the best possible depth resolution.

Until the present project, very little attention was given to the depth and lateral resolution achievable with the SIRIS apparatus. The original SIRIS instrument was designed to deliver the highest current possible to the target in a reasonable spot size in order to achieve the highest sensitivity for bulk analysis. In practice this required high extraction potentials at the ion source, giving the sputtering particles high energies. The best depth resolutions are achieved with low sputtering energies. The SIRIS technique can be optimized for lower sputtering energies, yielding better depth resolutions while maintaining useful sensitivities for impurity or dopant analyses. Since the technique is less sensitive to matrix effects, SIRIS could provide a very useful method for studying the migration of dopant atoms across an interface separating two differing matrix materials. There is a strong interest in the semiconductor industry for having a reliable method of measuring an element while sputtering through the interface from one matrix material to another, while maintaining constant sensitivity to the element being analyzed and good depth and lateral resolution.

Phase I of this project was to determine and demonstrate where possible the feasibility of improving the lateral and depth resolutions of the SIRIS technique and apparatus of Atom Sciences.

REVIEW OF RESONANCE IONIZATION SPECTROSCOPY

Resonance Ionization Spectroscopy (RIS) (2) was introduced 11 years ago as a new technique for probing the atom and for providing a basis for new ultrasensitive analytical techniques. Introduced by G.S. Hurst and his associates at Oak Ridge National Laboratory, the RIS concept was conceived as a general technique of high selectivity and high efficiency applicable to all the elements in the periodic table, (3) although helium and neon are excluded for practical reasons. Applications for RIS are being developed at several universities and government laboratories including Penn State, (4-6) NBS (7-9) Los Alamos, (10) Argonne National Laboratory, (11,12) and Oak Ridge National Laboratory. (13-18) The RIS process is patented by the Department of Energy (19) and is licenced to Atom Sciences for commercialization and for development of commercial applications of the technology. In the resonance ionization process, a laser is tuned precisely to the wavelength required to excite an atom from its ground state to a particular excited state that is unique to the element being measured. In the simplest RIS scheme, a second photon of the same wavelength interacts with the atom in its excited state and causes an electron to be released from the atom; thus a positive ion and a free electron are produced. This RIS process, therefore, involves two steps: (1) excitation of the atom to a specific quantum state (resonance), and (2) removal of an electron from the selected excited state (ionization), as shown in Figure 1. The simplest RIS scheme, or a variation of it involving frequency doubling, can be used for many but not all elements.

A second process uses an additional laser (with or without frequency doubling) in combination with the first laser to excite two states sequentially prior to ionization, as also shown in the figure. A third RIS process involves a two-photon transition to an intermediate, virtual state prior to ionization. Once the atom is ionized, a charged particle detector is used to detect and count the ions or electrons produced in the RIS processes to achieve the ultrasensitive, ultrasensitive measurement.

The unique advantage of resonance ionization is that it is both extremely selective and extremely efficient. It is uniquely selective in that only atoms of a given element are ionized, since the selected intermediate excited states through which the process proceeds are unique to the selected element. It is uniquely efficient in that all atoms of the selected element that are present in the laser beam are ionized. In contrast, the more familiar methods of ionization (such as electron bombardment) are non-selective and inefficient.

The RIS process is completely selective as to element and completely selective as to isotope for elements of atomic number less than about 5. To identify and detect isotopes of the other elements, an appropriate mass analyser is incorporated into the RIS apparatus. This can be a magnetic sector, r-f quadrupole, or time-of-flight analyzer.

REVIEW OF SIRIS TECHNIQUE

Sputter Initiated Resonance Ionization Spectroscopy (SIRIS) is an ultrasensitive elemental analysis technique under commercial development at Atom Sciences (20-25). This technique, patented by Atom Sciences (26), combines RIS with ion beam sputtering to selectively ionize neutral atoms of a pre-selected element.

An inherent condition of almost all RIS techniques is that the sample must be in the gas phase. In a few cases, the sample is initially a gas, but most samples of interest are either solid or liquid. SIRIS uses sputtering for atomization. Sputtering is a well established technique for vaporizing solids and is very reproducible. The basic concept for SIRIS is illustrated in its simplest form in Figure 2. In SIRIS a pulsed ion beam (usually argon) is focused onto a solid sample, thus producing a cloud of vapor immediately above the sample. RIS lasers then selectively ionize atoms of the chosen element in the vapor cloud; these are subsequently accelerated through a mass filter for detection at an electron multiplier. Secondary ions, produced along with the neutral atoms by the impact of the ion beam, can be rejected by electrostatic fields, electrostatic energy analysis, the relative timing between the ion beam pulse and RIS laser pulse, or some combination of the above.

SIRIS has several important advantages over Secondary Ion Mass Spectrometry (SIMS) (27-29), a technique which also uses a primary ion beam to sputter a solid for mass analysis. The secondary ion yield (the quantity measured in SIMS) varies greatly depending on the composition of the surface of the sample. Also, the ratio of secondary ions to neutrals is usually on the order of 10^{-3} to 10^{-4} . This means that SIRIS should be more sensitive than SIMS by several orders of magnitude and should not be susceptible to variations in signal magnitude caused by matrix effects. SIRIS provides ultrasensitive analysis of solid

samples for all the elements except helium and neon. Sensitivities of a few parts per billion are possible in SIRIS analyses, and greater sensitivities are possible for special cases.

A schematic diagram of the original SIRIS apparatus that was designed and built by Atom Sciences is shown in Figure 3. The primary ion beam was generated with a duoplasmatron ion source and was mass analyzed by the analyzing magnet to insure beam purity. A set of deflection plates was used to sweep the beam across a pair of chopping slits. Focusing was achieved magnetically, with quadrupole magnets as well as by the analyzing magnet itself. A final 4° bending magnet was placed in the ion beam just prior to the target to prevent any beam contaminants or line-of-sight ions generated by scattering from the walls from hitting the target.

Typically this system would generate a 45 μ amp argon beam; ion pulses of about 0.7 microsecond duration are directed to the target sample. Generally, the frequency with which the ion pulses are directed to the target is equal to the frequency of the laser used as the RIS probe, typically 30 Hz. However, the duty cycle can be increased to 100% (direct current mode) to sputter away substantial amounts of material from the top surface. The ion beam intersects the sample plane at 60° with respect to the normal and is focused to an approximate 2.6 x 3.3 mm spot. By reducing the beam current to about 5 μ A with the use of apertures, the beam spot size can be reduced to about 0.5 mm.

Ions of the selected elemental species which are created by the RIS process are verified and their isotopic mass is measured with a mass spectrometer. The mass spectrometer used in SIRIS is a double focusing type consisting of an electrostatic sector which acts as the energy analyzer and a magnetic sector which determines the mass of the ion. Neutral atoms sputtered from a solid sample, and therefore the ions produced by RIS, have an energy distribution that extends to several hundred eV. Fortunately, the distribution is sharply peaked and most of the ions are centered about an average energy of a few eV. The electrostatic sector is adjusted to allow these ions to be transmitted, while rejecting the SIMS ions. Ions having higher energies are rejected, thus insuring that the input to the magnetic sector is such that mass analysis can be performed with the required high resolution and abundance sensitivity. The resolution of the mass spectrometer is designed to be 440 at 300 amu. More importantly, its abundance sensitivity is designed to be greater than 10^6 .

After the RIS selected ions are verified as to element and their mass is measured with the mass spectrometer, they are detected with a channel electron multiplier equipped with a special conversion electrode. Since SIRIS is a pulsed technique, background is reduced to a minimum by time gating the electronics of the data acquisition system. The ion counting system is set to accept pulses only during the time period when an ion is expected to arrive. Typically, noise factors for single ion detectors are reduced by more than a million using this technique.

The RIS process is achieved by using a neodymium YAG laser to pump one, two, or more tunable dye lasers. Each dye laser consists of three sections: an oscillator, a preamplifier, and an amplifier. These sections can be used in various combinations to optimize the production of light of the desired

wavelength and intensity. The dye laser produces tunable light of the desired wavelength throughout the visible spectrum. In addition to visible light, ultraviolet light down to about 2200 Å can be generated by additional non-linear processes consisting of second harmonic generation and mixing.

The target chamber of the SIRIS apparatus has been designed to have versatility in its sample handling capability. Sample sizes from about 5 x 5 mm to 20 x 20 mm can be accommodated. Samples can be manipulated in X, Y, and Z and can be loaded on a carousel. Other features of the SIRIS apparatus incorporate special ultrahigh vacuum techniques and computer control. The SIRIS apparatus is entirely microprocessor controlled and a minicomputer is programmed to control the microprocessor. The minicomputer also functions as a computer-based data acquisition system. The system is designed to collect data in a variety of ways, e.g., in a single ion counting mode or in an analog current mode.

Depth profiling is accomplished by first using the ion beam in the dc mode and rastering the beam over a rectangular area usually about 10 times the size of the ion beam. The raster pattern is repeated until the desired amount of material is removed and the desired depth is reached. When the desired depth is reached, the ion beam is shut off and then restarted in the pulsed mode. Data are then accumulated for a specified number of laser pulses and then the ion milling process is repeated to expose a new layer for analysis until the measurement is completed.

For this work, the original ion beam system with the analyzing magnet and magnetic beam optics was replaced with a commercial duoplasmatron microbeam ion gun system leased (with Atom Sciences' funds) from the Physical Electronics Division of Perkin Elmer. This system, designed for a dynamic SIMS measurement system, was designed to deliver approximately 5 µA of argon current in a spot less than 100 microns FWHM and can be apertured and refocused to spot less than 5 microns FWHM. Currents of 10 µamps of argon ions have been extracted from the new source. A schematic of the new, modified SIRIS system is shown in Figure . The system can be operated using either a magnetic sector or time-of-flight mass spectrometer.

Review of Technical Objectives

The technical objective of this Phase I SBIR project was to determine the feasibility of using the SIRIS technique and apparatus developed at Atom Sciences for making ultrasensitive analyses of III-V compounds with high depth and lateral resolution, yet free from interferences.

Specific objectives of the proposed work were stated as:

1. To demonstrate that there are no interferences in detecting either Si or Fe in GaAs while the other is present in test samples supplied by the Air Force.

2. To make measurements to detect Si in a compound material consisting of a layer of GaAs and a layer of AlGaAs, doped and grown by MBE, to determine whether there are significant matrix effects in detecting Si while sputtering through the interface from one matrix to another.
3. To utilize newly designed and constructed SIRIS equipment to demonstrate better lateral resolution with an ion beam diameter on target of 100 microns or less.
4. To determine the feasibility of achieving lateral resolutions as small as 5 microns for analyses using the SIRIS technique.
5. To determine the feasibility of achieving lateral resolutions as small as 0.1 microns for future analyses using the technique of Sputter Initiated Resonance Ionization Spectroscopy.

These objectives have been met in the Phase I project, and in fact, in some cases the Project was able to proceed beyond these objectives and accomplished some of the tasks that were to be demonstrated in the Phase II Project. We believe that the work performed in this Phase I Project demonstrates that SIRIS can make sensitive, interference free measurements with good lateral resolution. The technical objectives in a Phase II Project can be expanded and can include a set of demonstration measurements to prove the feasibility study reported here.

RESULTS AND DISCUSSION

Technical Objective 1.--To demonstrate that there are no interferences in detecting either Si or Fe in GaAs while the other is present in test samples supplied by the Air Force.

Objective 1 had to be met differently than was originally planned because a sample of GaAs doped with both Si and Fe could not be obtained. Instead an experiment was performed in which Si was analyzed in an iron matrix to determine if there were any interferences in detecting silicon while in the presence of iron. The results of that experiment are shown in Figure 5. This mass spectrum is interference free of other mass peaks even though the steel sample has many impurities in it. The matrix material of iron is nearly nonexistent in the spectrum.

Technical Objective 2.--To make measurements to detect Si in a compound material consisting of a layer of GaAs and a layer of AlGaAs, doped and grown by MBE, to determine whether there are significant matrix effects in detecting Si while sputtering through the interface from one matrix to another.

A layered sample of GaAs and AlGaAs was supplied to Atom Sciences by Dr. David Weyburne of Hanscom Air Force Base which he had obtained from Dr. Susan Palmateer of MIT/Lincoln Laboratory. The sample was identified as Layer 47 and had been grown by MBE and electrically characterized by Dr. Palmateer. The structure and make-up of the sample is illustrated in Figure 6. The layers were grown on a substrate of undoped LEC GaAs. Beginning with the substrate material, a buffer layer of GaAs was first grown which was approximately 1 μm thick. Then a layer of $\text{Al}_{.30}\text{GaAs}$ doped with silicon was grown on top of the buffer layer. This AlGaAs layer was approximately 350 $\text{\AA}$ thick and the concentration of the silicon was determined electrically to be approximately $7 \times 10^{17}/\text{cm}^3$. A final layer of GaAs also doped with Si was grown on top of the AlGaAs. This layer is approximately 500 $\text{\AA}$ thick and the silicon concentration was determined electrically to be approximately $2 \times 10^{18}/\text{cm}^3$. The physical concentrations and the electrical concentrations are different in that the electrical concentration is based on the sum of N and P type carriers. The physical concentration of the silicon in each of the layers is unknown; however, the ratio of the physical concentrations in the two layers is known from the parameters of the MBE growth process to be 0.77.

Sample Layer 47 was depth profiled with the silicon, aluminum, and gallium concentrations monitored. Since silicon was the dopant, it was first measured and profiled as a function of depth. The results are shown in Figure 7 with the indicated data points and a smooth line fit to guide the eye. The silicon concentration, based on electrical carrier measurements, in the top layer is in the $2 \times 10^{18}/\text{cm}^3$ range (40 ppm) and the SIRIS signal was high. The SIRIS signal for silicon in the GaAs layer decreased to 77% of the value found in the AlGaAs layer when the top layer was sputtered through and the measurements were made in the second layer. This agrees exactly with the expected ratio of silicon concentrations in the layers of GaAs and AlGaAs based on the MBE growth rate parameters. This exact agreement is fortuitous since the SIRIS measurements have a precision of approximately 10%. This ratio based on the MBE growth parameters is significantly different from the ratio determined by the electrical carrier measurements. The free silicon carrier concentration in the AlGaAs layer was determined electrically to be approximately $7 \times 10^{17}/\text{cm}^3$ so that the ratio based on electrical measurements is 35%. Assuming that the 77% ratio based on the MBE parameters is correct, then this result indicates that there is no significant matrix effect in detecting Si with SIRIS while sputtering through the interface from one matrix, GaAs, to another, AlGaAs.

The concentration level of silicon measured in the undoped buffer layer was found to be surprisingly high, about $4 \times 10^{17}/\text{cm}^3$ (when normalized to the $2 \times 10^{18}/\text{cm}^3$ electrical measurement of the top layer). Based on depth profile measurements of Si implanted in GaAs shown in Figure 8, the detection limit for Si in GaAs should have been less than $5 \times 10^{15}/\text{cm}^3$, well below the level in the buffer layer.

The depth scale of the depth profile data taken with SIRIS was calibrated by measuring the depth of the sputtered crater with a Dek Tak profilometer. According to the data supplied by Dr. Palmateer, the interfaces between the first and second layers (starting from the top) and between the second and third layers should occur at depths approximately 0.050 and 0.085 microns. It is clear from the data presented in Figure 7 that the measurements are in good agreement with these values.

The aluminum and gallium concentrations were also measured as a function of depth; the results are shown in Figures 9 and 10. The aluminum depth profile shown in Figure 9 is interesting and indicates the depth resolution of this measurement. The slope of the peak from the 10% value to the 90% value spans a depth of about 150 Å which is typical of measurements with argon ions with energies of 10 keV. This result is encouraging since depth resolutions of 30 Å can be achieved usually by reducing the primary beam energy down from 10 keV to 3 keV. Presently, the source of the SIRIS apparatus can be operated as low as 0.5 keV and depth resolutions of 30 Å or less are feasible for SIRIS measurements. The feasibility of achieving 30 Å or less depth resolutions suggests a set of demonstration measurements for a Phase II project.

Measurements of the gallium concentration are shown in Figure 10. Two separate determinations are shown. The gallium signal in AlGaAs should be expected to decrease to 71% of the value observed in GaAs. One measurement shows a value about 85% and the other shows a value of about 55%. Since these measurements were naturally made at different points on the sample, the most probable explanation for the difference in the ratios is sample inhomogeneity from point to point. It has been observed in isotope ratio measurements that the ion detector exhibited non-linearity when used with high intensities. It is unlikely that non-linearity in the measuring system contributed to this difference in signal because care was taken to keep the intensities below levels where non-linearities had been found to appear.

Technical Objective 3.--To utilize newly designed and constructed SIRIS equipment to demonstrate better lateral resolution with an ion beam diameter on target of 100 microns or less.

The original SIRIS apparatus was designed for a high-current primary ion beam capable of delivering as much as 10 mA in microsecond pulses to the target in a spot size 2.6 x 3.3 mm. A complicated magnetic system was designed to do this, but obtaining the right source and forming the correct magnetic fields to match ion beam optics design became a problem too great to solve consistent with the Company's resources and main goal of developing the SIRIS technology. Currents as large as 50 µA could be achieved with spot sizes of 2.6 x 3.3 mm. Spot sizes as small as 0.5 mm FWHM could be achieved with the sacrifice of beam current. Figure 11 shows a Dek Tak profile of a series of spot sizes and a rastered crater with a width 10 times the smallest spot size. The 0.5 mm spot size was produced with a beam current of a 1-2 µA. While this spot size was small enough to allow depth profiling the sample, the spot size could not meet the desired technical objectives of this project. Two approaches for achieving the small spot size were anticipated--one involving a new source and ion beam column designed and built by Atom Sciences as a part of a new SIRIS instrument and a second involving the installation of a commercial microbeam ion gun system to be installed on the original SIRIS system. Both approaches were taken and achieved ion beam diameters of less than 100 microns FWHM. But the completion of the new SIRIS instrument was delayed by fiscal constraints. Therefore, Technical Objective 3 was achieved through the lease of a commercial microbeam ion gun system.

A PHI Model 6050 ion gun system was leased by Atom Sciences from the Physical Electronics Division of Perkin-Elmer and was installed in the present SIRIS

apparatus in place of the original ion beam system. The new ion beam system was integrated into the SIRIS instrument and data acquisition system with a few modifications to make the pulsing of the source faster. Beam diameters from less than 5 microns to 100 microns are possible and beam currents as high as 10 μ A have been obtained from the source. The beam energy is adjustable from 0.5 to 10 keV.

Figure 12 shows a typical beam profile measured in a sputtered piece of silicon and measured with the Dek Tak profilometer. A beam current of 1.3 μ A was used for the sputtering and the width of the hole at FWHM is approximately 93 microns, thus meeting our objective of 100 microns or less.

Technical Objective 4.--To determine the feasibility of achieving lateral resolutions as small as 5 microns for analyses using the SIRIS technique.

Results obtained in this project with the new ion beam source indicate that it is feasible to use SIRIS for analyses in which lateral resolutions as small as 5 microns are achieved. The specifications for the new source include beam diameters on target of 5 microns or less so that the size of the beam should be no problem. The question that remains is whether or not useful sensitivities can be achieved with the currents available with the smaller spot sizes. Since the sensitivity of SIRIS depends directly on the primary beam current, the sensitivity can be calculated from the ratio of the beam current of the large spot size to that of the smaller spot size times the sensitivity obtained with the larger current. For example, if 1 ppb sensitivity is achieved with a spot size of 100 microns and a current of 1 μ A, then with a spot size of 5 microns and a current of 10 nA, a sensitivity of 100 ppb will be achieved. Since 1 ppb sensitivities have been shown to be possible with a current of 1 μ A, and a current of 10 nA is possible with the new source operated with a 5 micron beam size, then this example is realistic and sensitivities of 100 ppb with a 5 micron beam are feasible. Such sensitivities with a 5 micron lateral resolution would be quite useful to the semiconductor industry.

Figures 13 and 14 show photographs obtained with a scanning electron microscope of two small holes sputtered with the ion beam system tuned for a smaller size hole. The beam current delivered to the samples was 160 nA and the beam struck the samples at an angle of 60° with respect to the normal. Figure 13a shows a photograph of one of the samples with the sample aligned perpendicularly to the line of sight. The left edge is sharply defined while the right side is diffuse because of the grazing incidence. The magnifications for the photographs in Figure 13a and Figure 13b are 1.11k and 1.84k respectively. The scale is shown in the legend at the bottom of each photograph as the length of the white line which is given in microns. For example, in Figure 13a the white line is 9.01 microns and in Figure 13b it is 5.43 microns.

Figure 13b shows a view of the same hole shown in Figure 13a but with the sample tilted 60° so that the view is aligned with the hole and the direction of the ion beam. The profile of the hole is thought to be gaussian and the view shown in the photos are near the surface. Figure 13b shows that the hole at the surface is about 14 microns in diameter. If the profile is gaussian, then at FWHM the width would be very near 5 microns. Figures 14a and 14b show two

different magnifications of another hole, x1.14k and x2.28k. The diameter at the surface measures about 16 microns. Again at FWHM the diameter should be close to the desired 5 microns.

The beam profile of the ion beam is not easily measured nor is it easy to tune an ion beam to get the smallest, best focused beam. Normally, a Faraday Cup is used to collect the beam current and with the use of apertures the beam current can be maximized and optimumly focused for the smallest beam diameter. However, there are limits to using small apertures, particularly near 5 microns. Another procedure for measuring the spot size involves the sputtering of a sample and then profiling the hole with the Dek Tak profilometer. However, the stylus of our profilometer has a diameter of 25 microns and hence it does not have the resolution to measure a 5 micron hole. As indicated above, the scanning electron microscope gives no information about the profile of the hole; it only gives information near the base.

As an aid to focusing the ion beam and achieving the smallest size beam, an electron detector was installed in the target chamber. By feeding the signal output into the z-axis (intensity modulated) of an oscilloscope and synchronizing the signal with the rastering of the ion beam, a video image can be obtained. Figure 15a shows such a video image obtained with the secondary electrons emitted from a 300 mesh aluminum grid. The 300 mesh grid material is made up of lines 85 microns apart. Figure 15b shows the same grid with higher magnification. This imaging capability is important for achieving small spot sizes and high lateral resolution. It is also important for locating the points of interest on a sample for analysis.

The results reported here indicate that it is feasible for SIRIS to achieve lateral resolutions of 5 microns or less. In this work, beam diameters of 5 microns FWHM have been demonstrated. Based on currents measured with a 5 micron beam and the sensitivities measured with a 100 micron beam, it is feasible for SIRIS to be used for the analysis of materials with a lateral resolution of 5 microns or less with a sensitivity of 100 ppb ($5 \times 10^{15}/\text{cm}^3$). Technical Objective 4 therefore, has been achieved and the full demonstration of this objective would be an excellent task for the Phase II project.

Technical Objective 5.--To determine the feasibility of achieving lateral resolutions as small as 0.1 microns for future analyses using the technique of Sputter Initiated Resonance Ionization Spectroscopy.

Liquid metal field ion sources have been a subject of investigation for several years for the development of high contrast, high resolution scanning ion micrographs and for use in microcircuit fabrication. Seliger and his co-workers (30) at Hughes Research Laboratories have reported the development of a liquid-metal gallium-ion source capable of delivering 0.12 nA of gallium ions to the target in a 1000 Å diameter spot. The energy of the ions was reported to be 57 keV and the current density was reported as 1.5 A/cm^2 . Considering that presently a sensitivity of 1 ppb can be achieved with a 1 µA argon beam operated at 10 keV, then using this source, sensitivities better than 10 ppm are feasible.

FEI, Field Electron and Ion Sources Company, of Hillsboro, Oregon, presently have a commercial liquid metal field ion source which they offer for sale at a modest cost of \$43k. Their source is specified to deliver 0.025 nA of 25 keV gallium ions in a beam focused to 1000 Å. Using this source on the present SIRIS apparatus, it is anticipated that a sensitivity of 40 ppm would be possible. In addition, however, FEI is developing a new, improved liquid metal field ion source which they hope to offer for sale in the fall of 1987. Presently, they say, this new source is delivering 0.25 nA in a 1000 Å focused beam and they anticipate achieving 0.5 nA of gallium ions. A 0.25 nA current would suggest a SIRIS sensitivity of 4 ppm and a 0.5 nA current would yield a sensitivity of 2 ppm.

The FEI source comes complete with rastering and beam blanking to allow pulsing of the source. Such a source would be an excellent candidate to consider for use on a time-of-flight based SIRIS system which would have a greater transmission efficiency than the present SIRIS system. It is possible with a higher transmission efficiency that sensitivities of 1 ppm could be demonstrated.

If detection sensitivities of sub-parts to few-parts per million could be useful, then the use of SIRIS for achieving lateral resolutions of 1000 Å is certainly feasible and would be an excellent demonstration project for a Phase II project.

SUMMARY OF RESULTS AND RECOMMENDATIONS

The accomplishments of this Phase I SBIR contract may be itemized as follows:

1. SIRIS apparatus was modified by installing a commercial microbeam ion gun system.
2. Ion beams of up to 10 microamps of argon ions were produced with the new ion source.
3. Argon ion beams of up to 5 microamps were produced and focused to a beam diameter of less than 100 microns on samples.
4. Argon ion microbeams focused to a diameter approximately 5 microns were produced with currents of approximately 160 nanoamps.
5. A method of optimizing the focusing of the 5 micron ion beams was devised, whereby a video image is produced by synchronizing the rastering of the ion beam over a fine aluminum grid with an oscilloscope display in which the intensity was modulated by the detection of secondary electrons sputtered from the grid material.
6. An interference free measurement of silicon in steel was demonstrated.
7. An epitaxially grown sample consisting of layers of GaAs and AlGaAs doped with Si were depth profiled with SIRIS for the Si, Al, and Ga concentrations.
8. The SIRIS measurement of Si profiled through an interface between layers of GaAs and AlGaAs was shown to be matrix independent.
9. The SIRIS measurement of Al profiled through interfaces between GaAs and AlGaAs with 10 keV argon ions indicated a depth resolution of 140 Å.
10. Two samples of Si implanted GaAs with peak concentrations of $5 \times 10^{18}/\text{cm}^3$ and $5 \times 10^{17}/\text{cm}^3$ were depth profiled and shown to have detection limits less than $5 \times 10^{15}/\text{cm}^3$.
11. A feasible method of achieving a lateral resolution of 1000 Å with SIRIS having a detection limit of 100 ppm or less was determined and outlined.

We believe that the work reported here warrants further improvement of the SIRIS technique and apparatus for making useful submicron resolution measurements.

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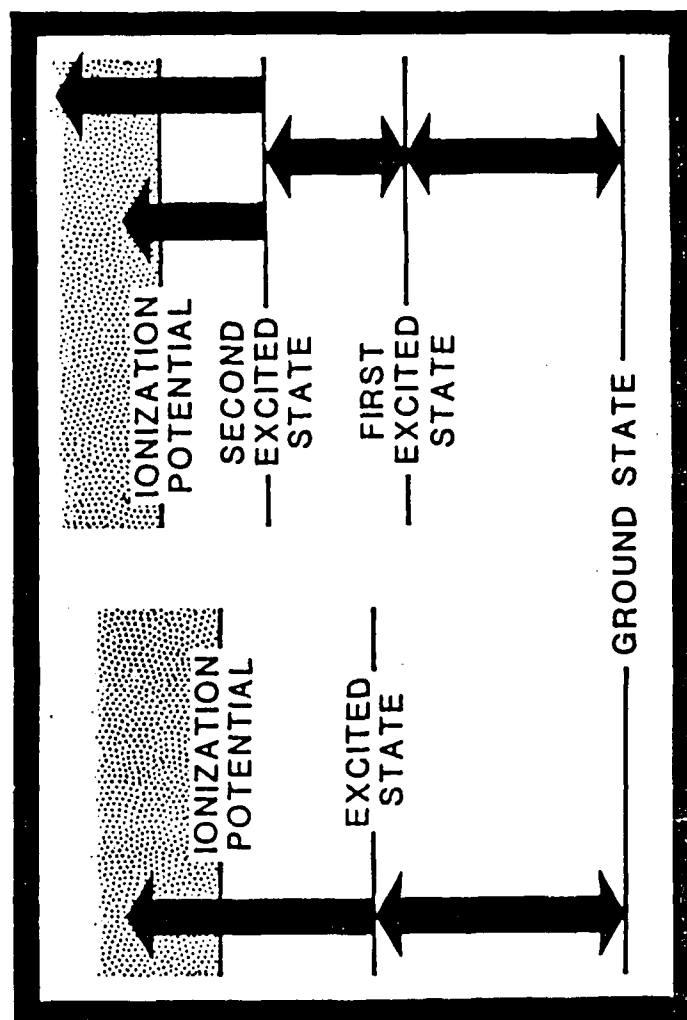


Figure 1. Two basic schemes for RIS showing one or two excitation steps followed by an ionization step.

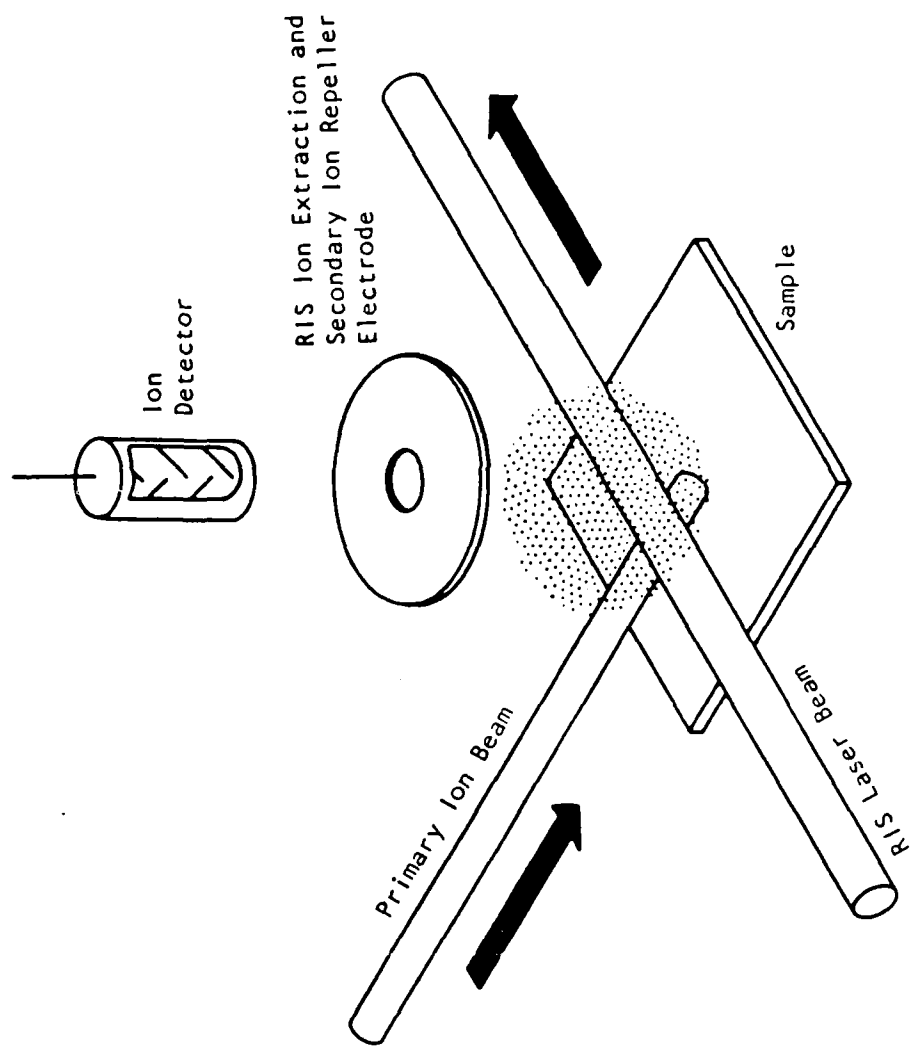


Figure 2. Basic concept of SIRIS.

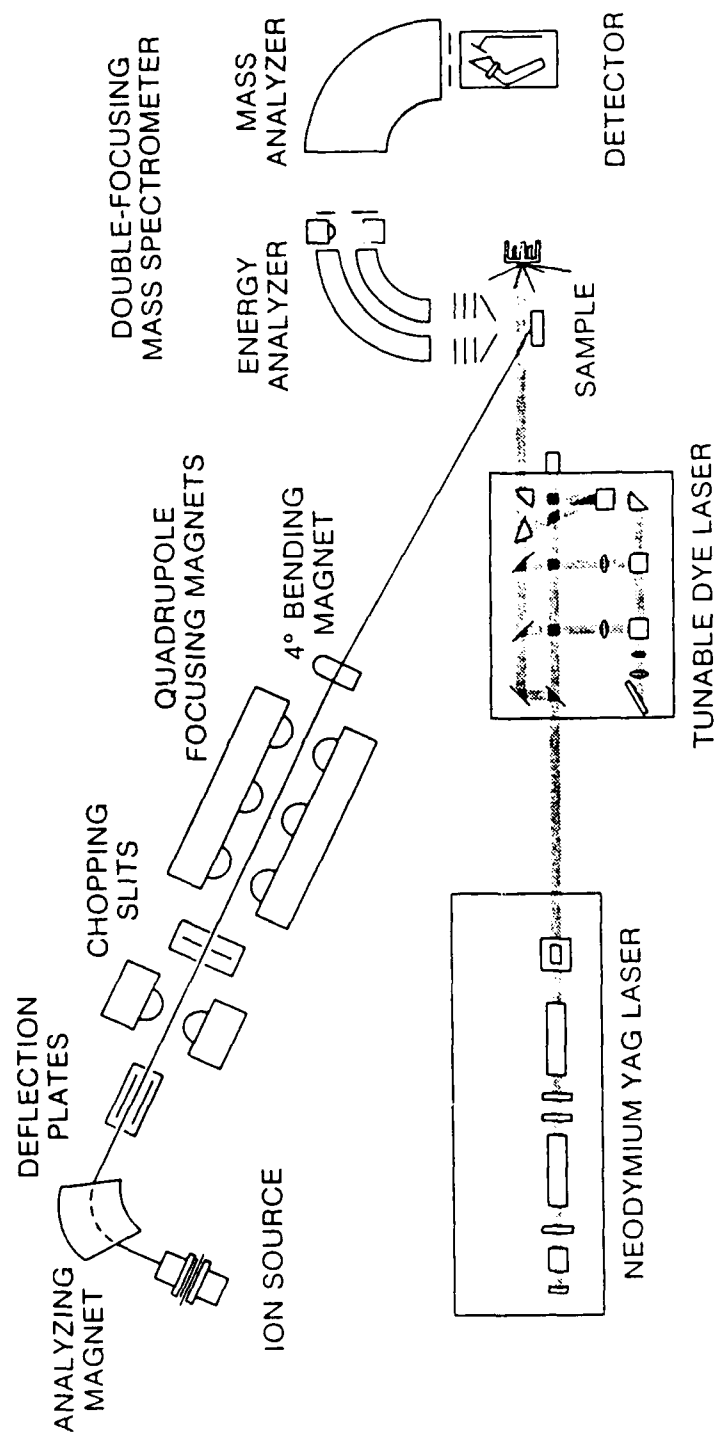


Figure 3. Schematic diagram of the original SIRIS apparatus.

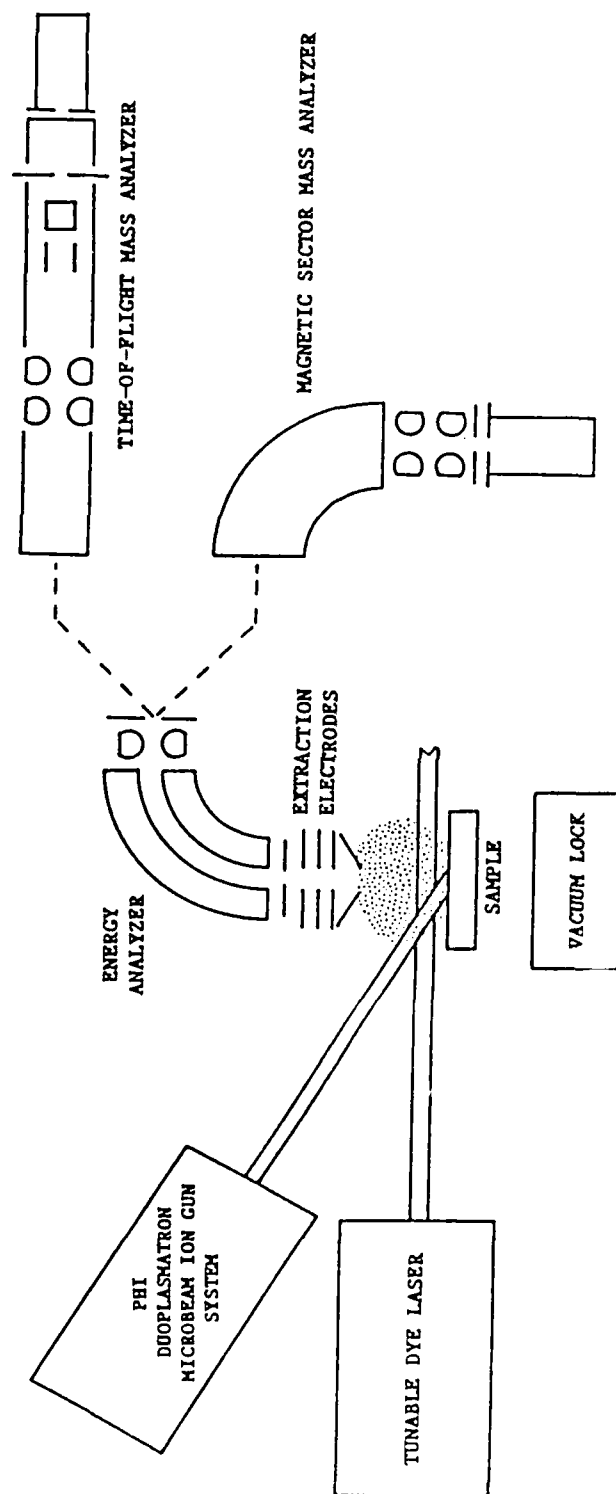


Figure 4. Schematic diagram of redesigned SIRIS apparatus using Perkin Elmer duoplasmatron microbeam ion gun system.

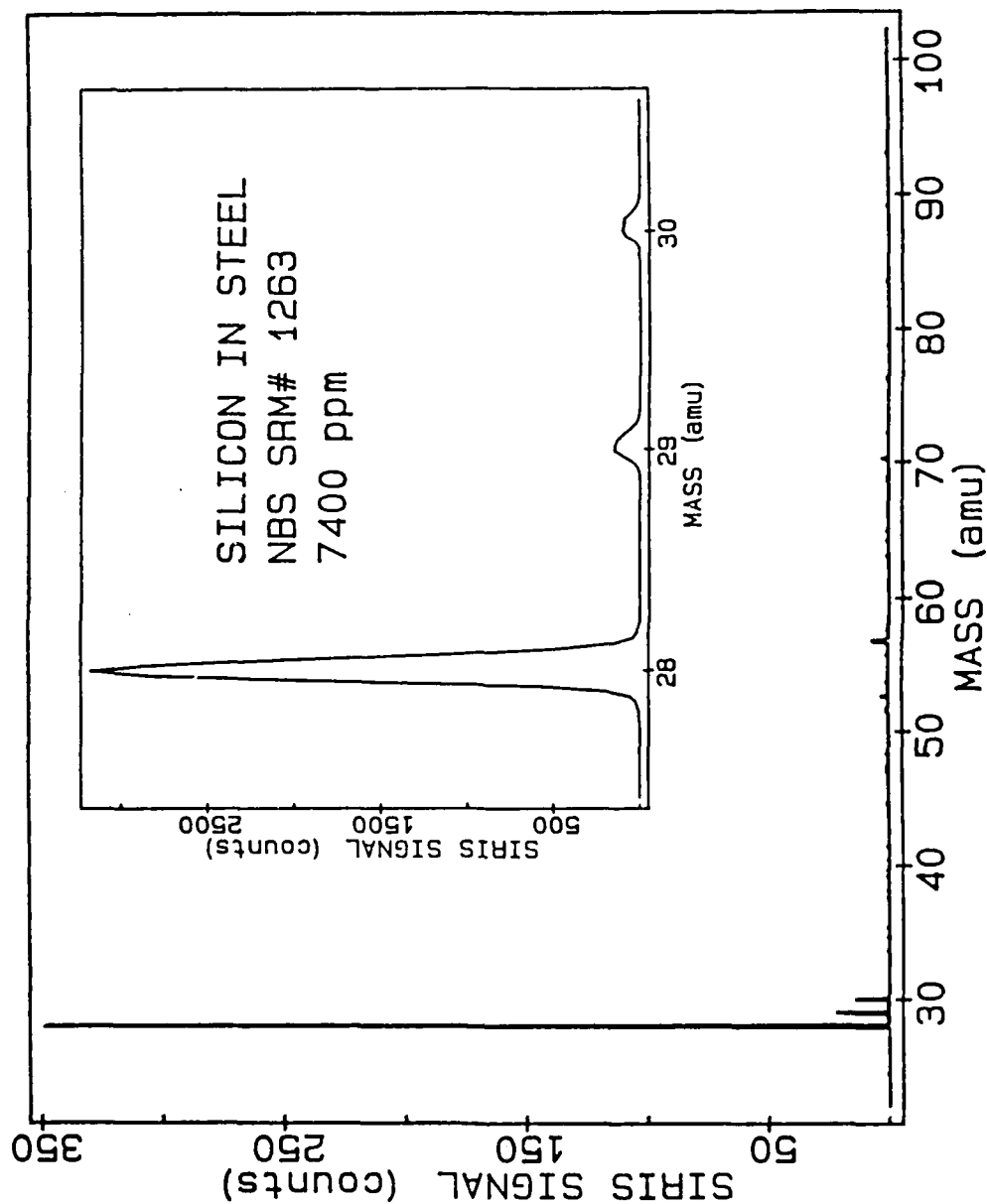
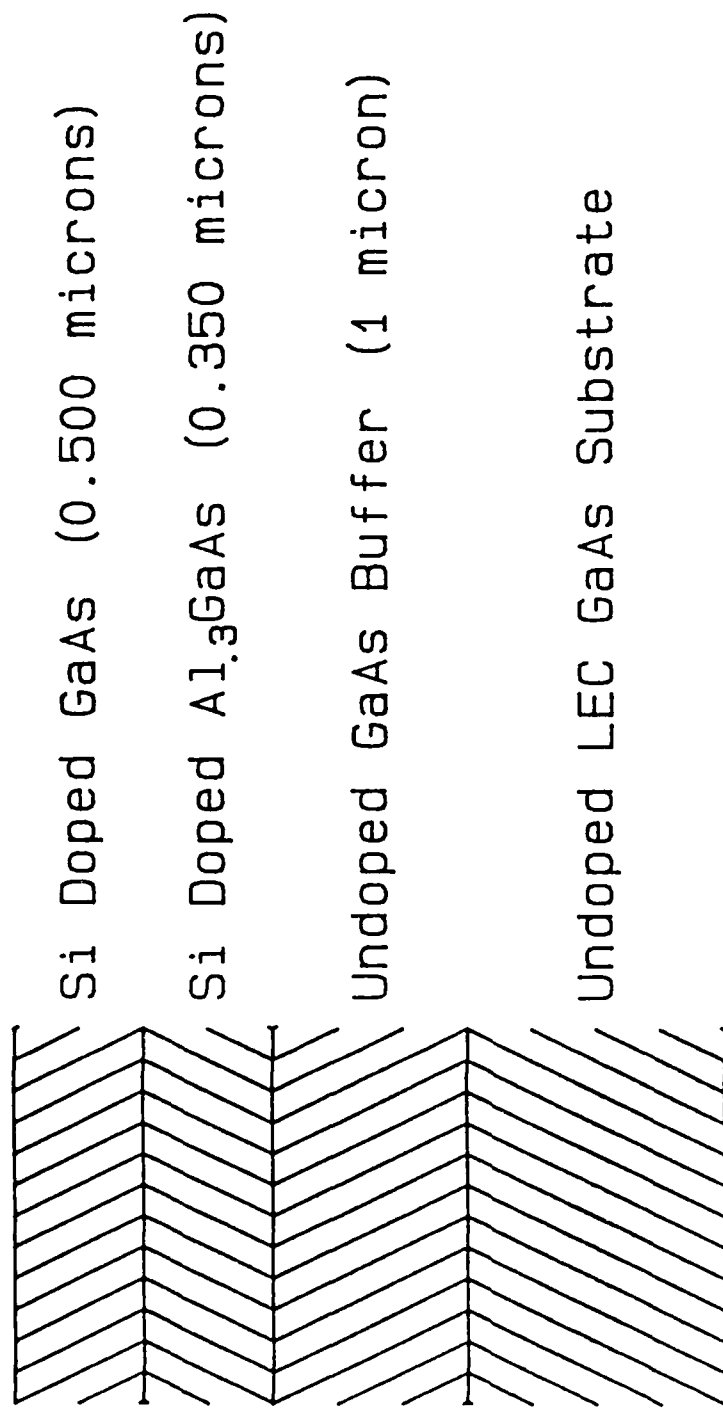


Figure 5. Mass scan of silicon measured in steel by SIRIS showing no interferences by the presence of iron and other impurities.



SAMPLE--LAYER #47

Figure 6. Structure of Layer #47 MBE sample of GaAs/AlGaAs/GaAs/GaAs.

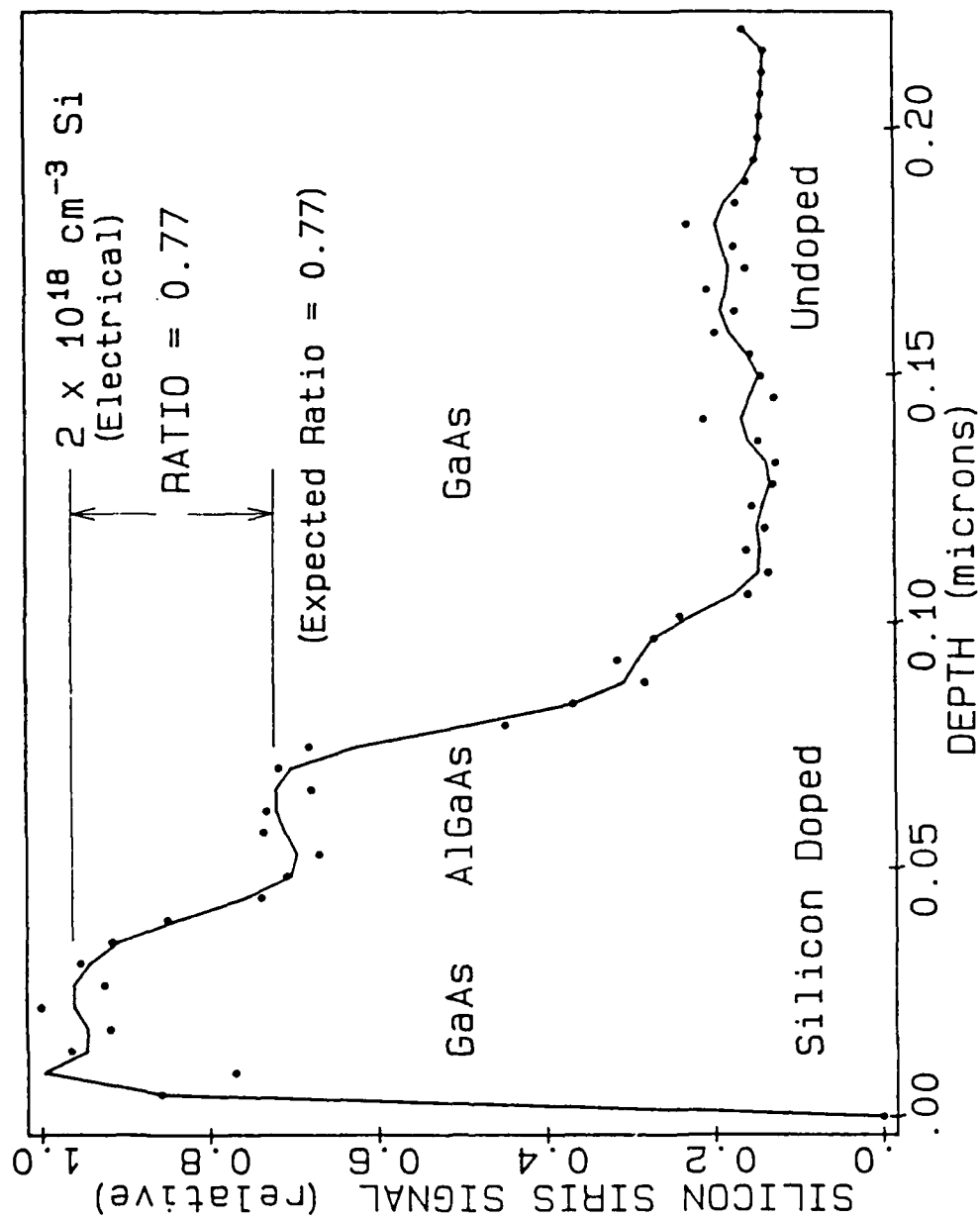


Figure 7. Silicon concentration profile in Layer #47.

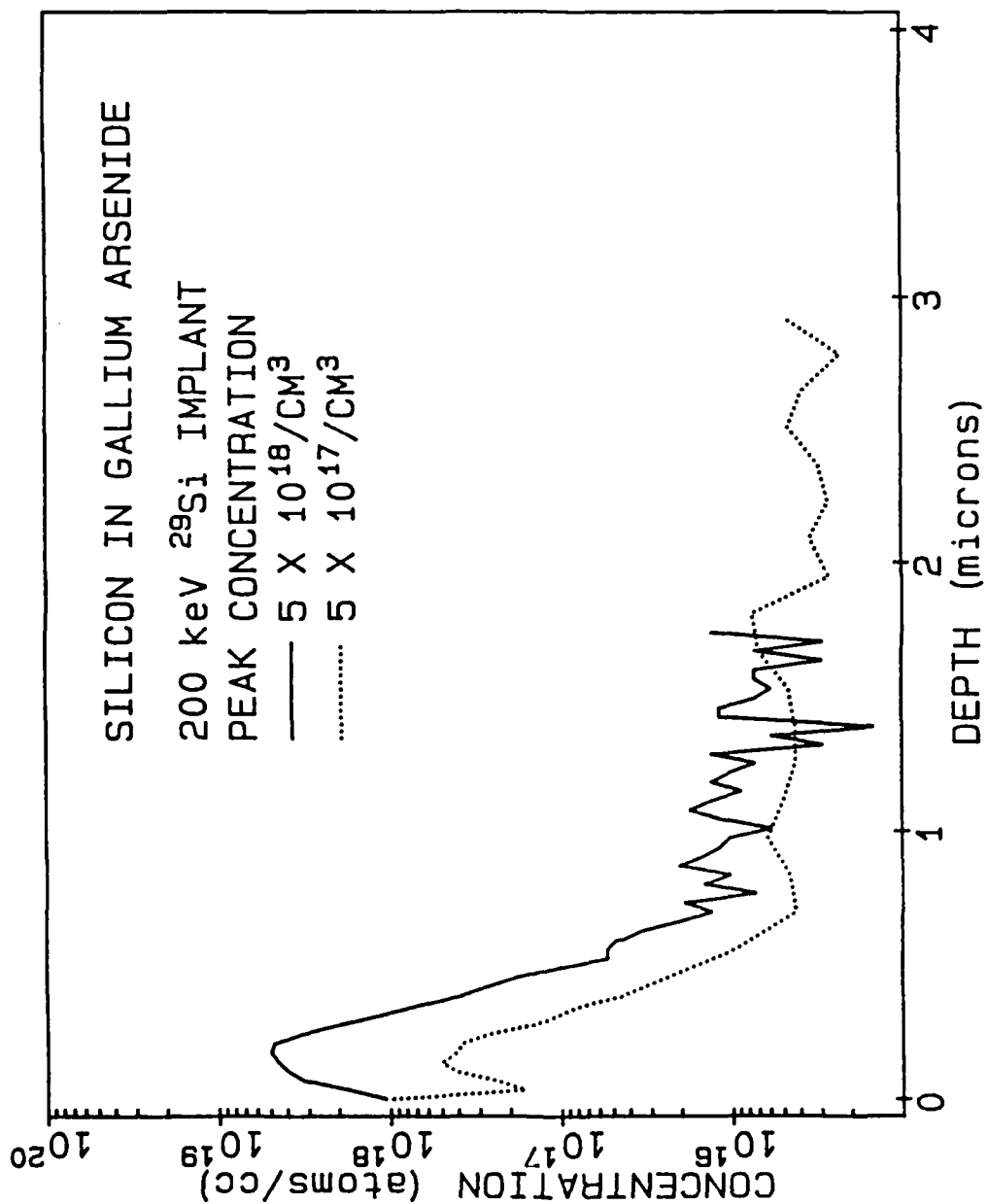


Figure 8. Depth profiles of two samples of silicon implanted in gallium arsenide with peak concentrations of $5 \times 10^{18}/\text{cm}^3$ and $5 \times 10^{17}/\text{cm}^3$.

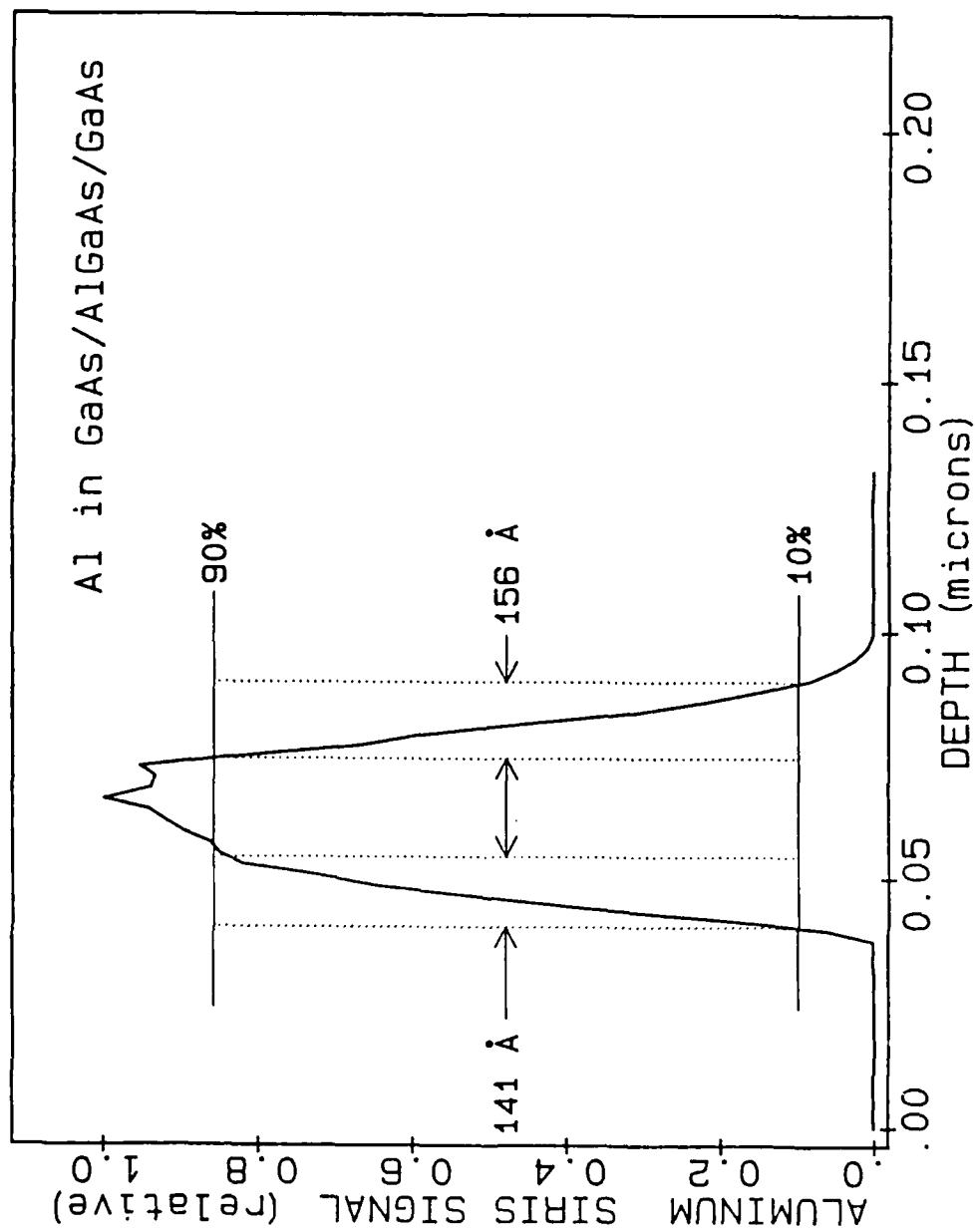


Figure 9. Aluminum concentration profile in Layer #47.

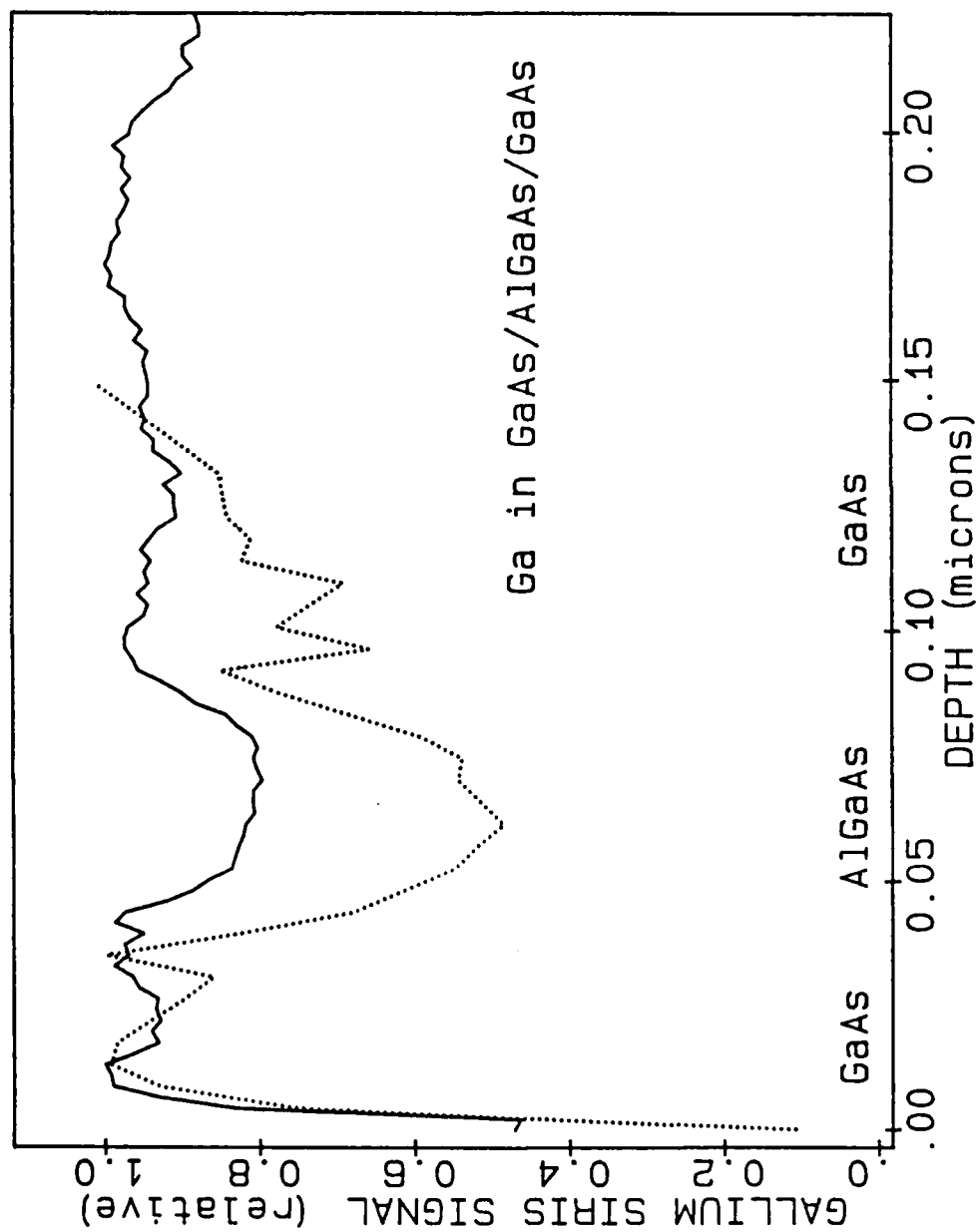


Figure 10. Gallium concentration profile in Layer #47.

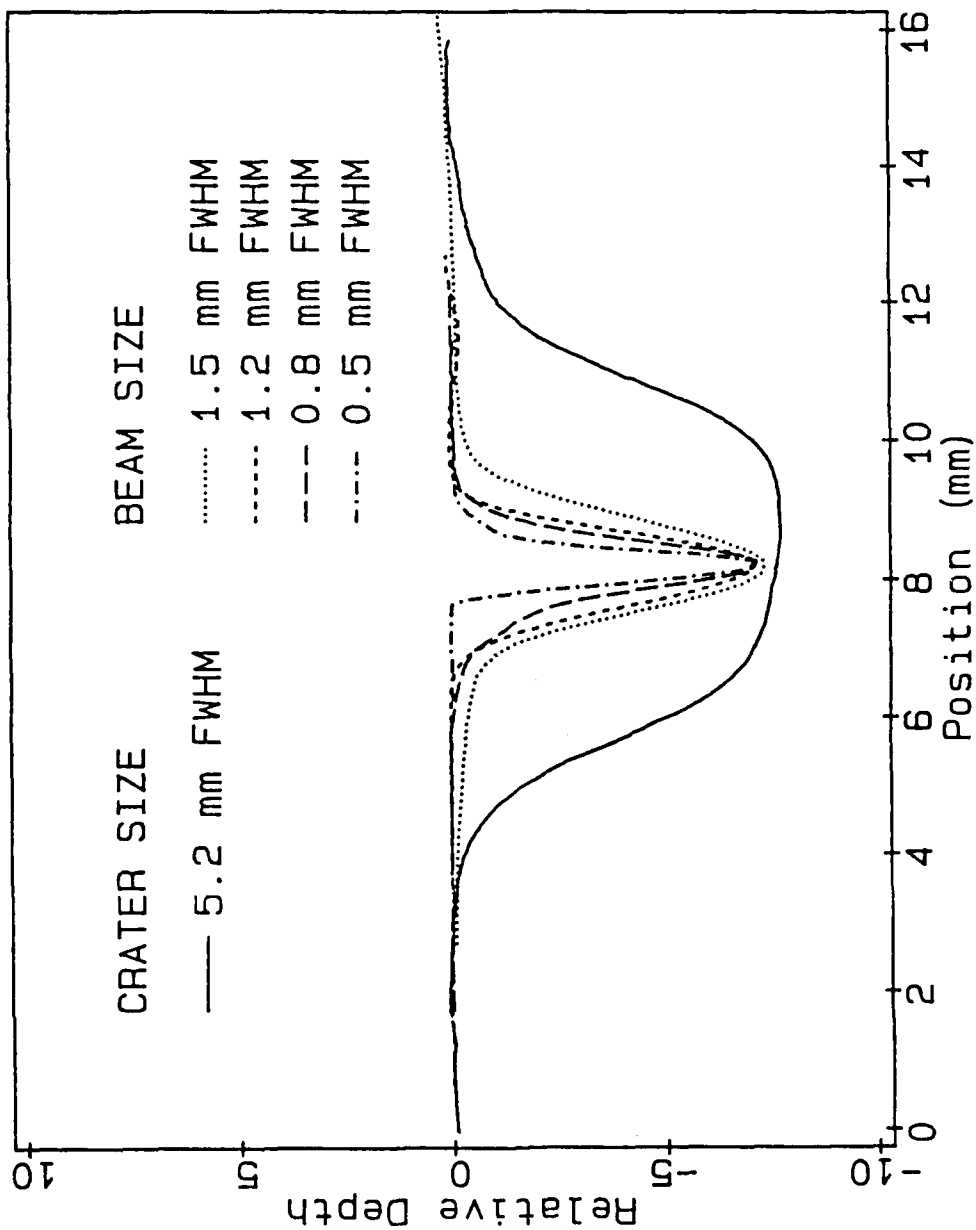


Figure 11. Dek Tak profiles of rastered crater and holes sputtered by ion beam using the original SIRIS ion beam system.

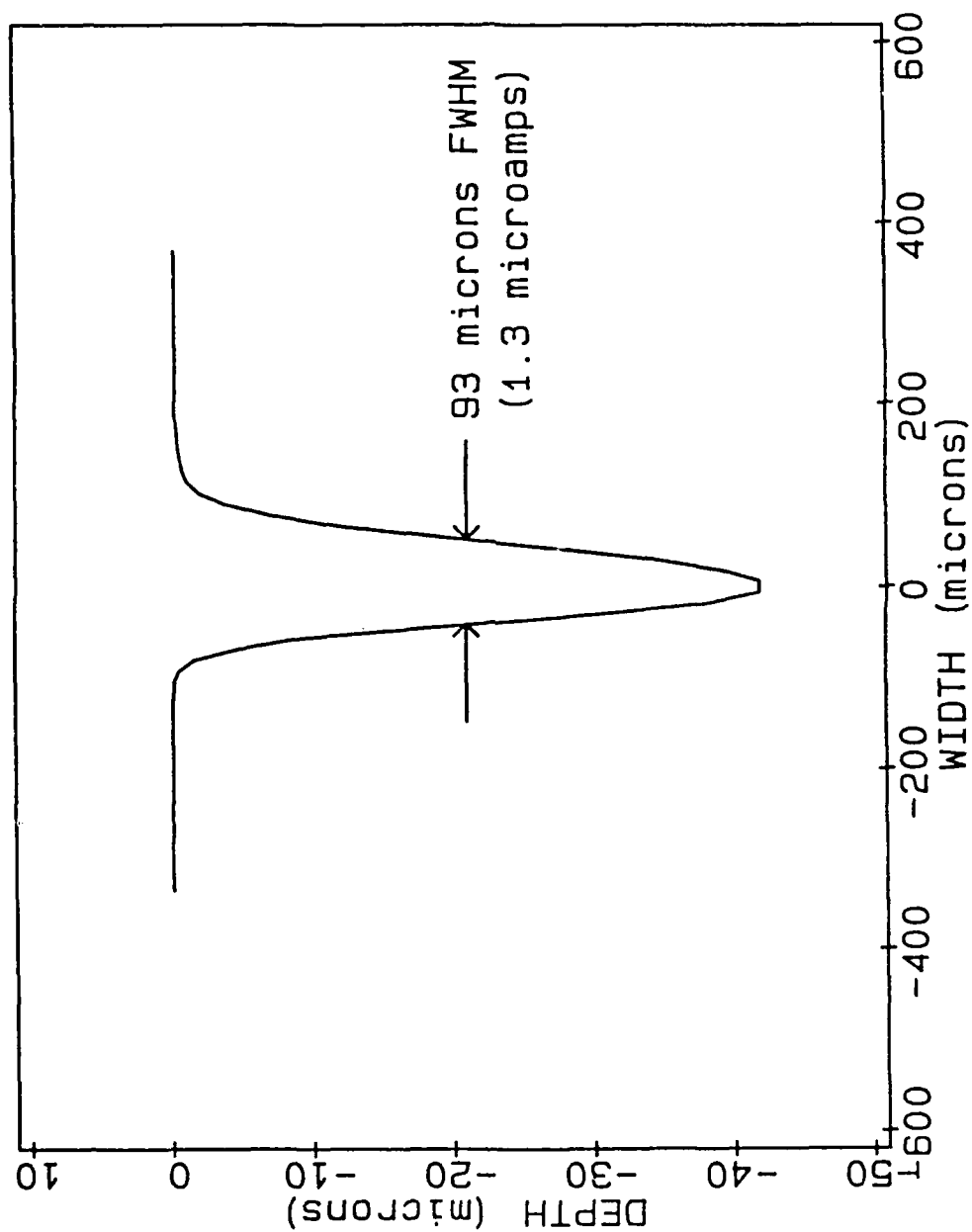
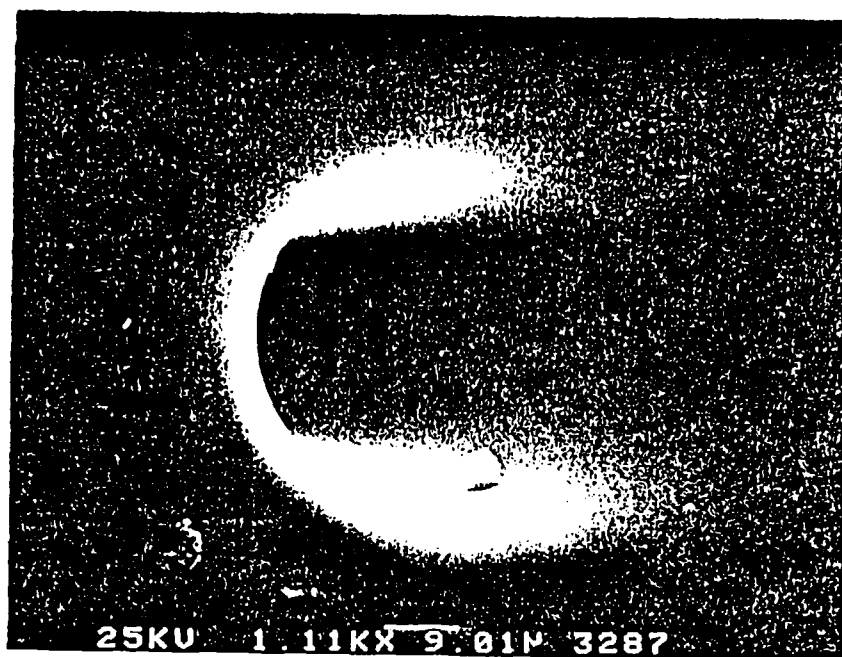
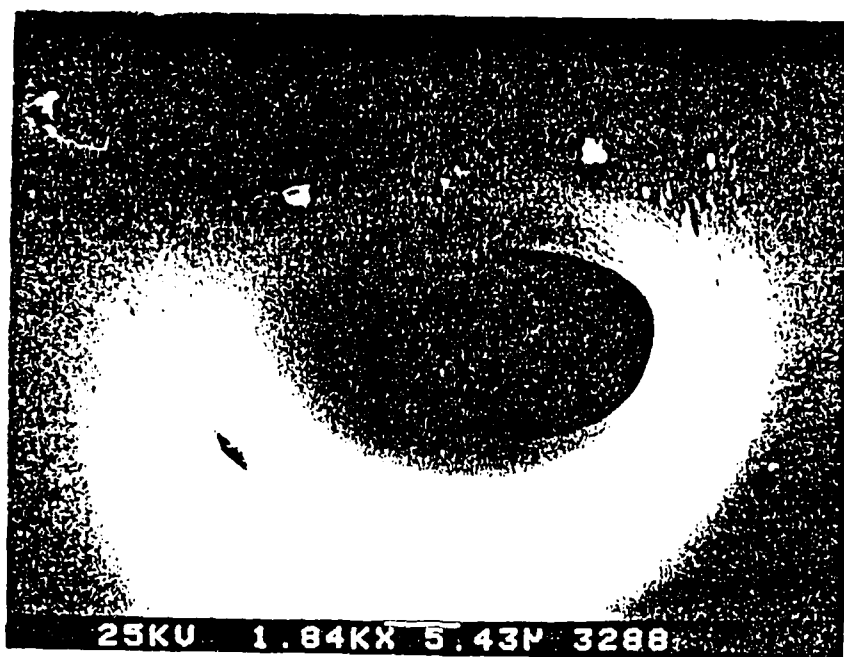


Figure 12. Typical beam profile measured in a piece of sputtered silicon with the Dek Tak profilometer which indicates a beam diameter of 93 microns FWHM.

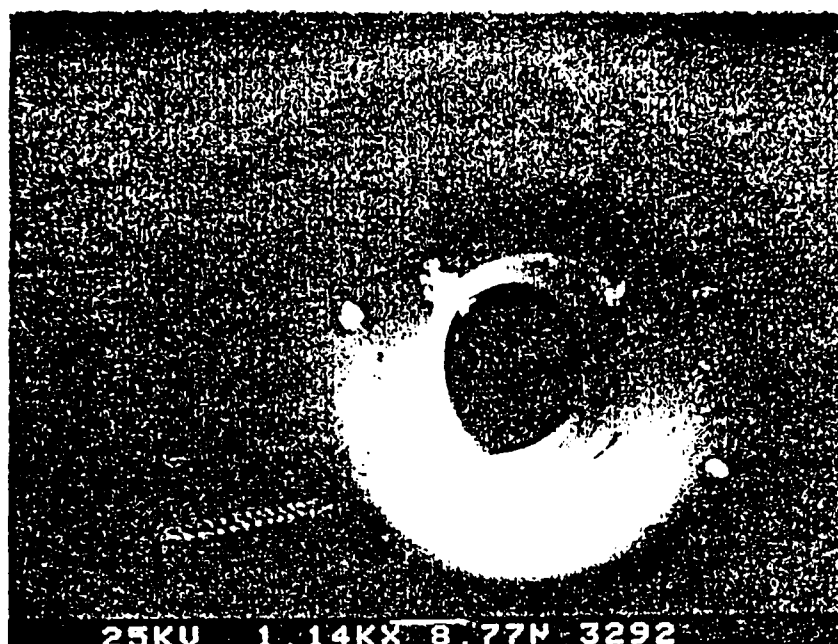


(a)



(b)

Figure 13. Photographs of holes sputtered in silicon with source focused to a diameter near 5 microns FWHM.

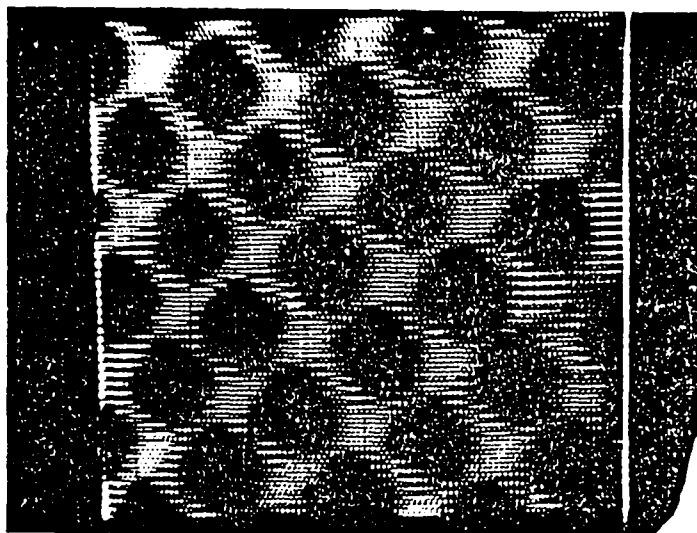


(a)

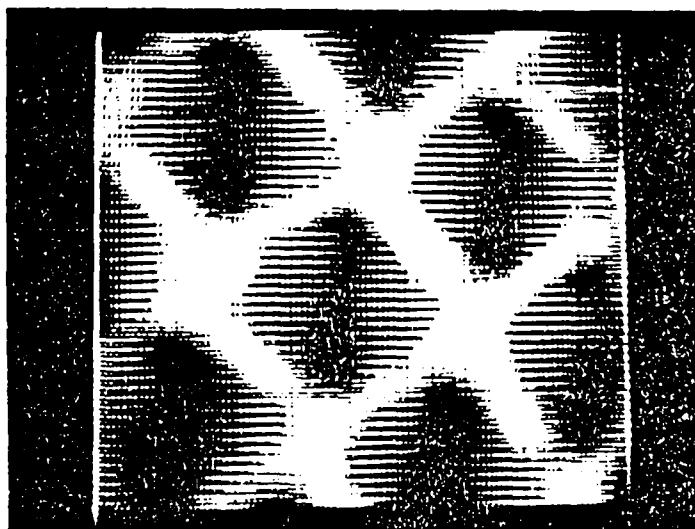


(b)

Figure 14. Photographs of holes sputtered in silicon with source focused to a diameter near 5 microns FWHM.



(a)



(b)

Figure 15. Photographs of video images of 300 mesh (85 micron spacing) aluminum grid material taken with different magnifications.