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Analysis Of Semi-Insulating Bulk GaAs Using Glow Discharge Mass Spectrometry

Andrew E. Souzis
National Research Council
and
Richard T. Lareau and Richard Wittstruck
Electronics and Power Sources Directorate

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Dr. Andrew E. Souzis*, Dr. Richard T. Lareau and Richard Wittstruck

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US Army Research Laboratory (ARL)
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ATTN: AMSRL-EP-EC-M
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The ability to perform sub-part per billion (ppb) analysis of semi-insulating (SI) GaAs substrate material is of critical importance for the development of electronic materials used in microelectronic device applications. Glow discharge mass spectrometry (GDMS) is commonly used to perform high sensitivity (part-per-trillion) measurements of metals and semiconductors. Since the sample is utilized as the cathode in a DC glow discharge, it must be conducting to some degree. Thus analysis of SI wafers presents major difficulties. Possible solutions include the use of a high purity mask or the mixing of pulverized sample material with high purity powder. Due to the finite purity of available materials, these methods have limitations. This study has resulted in the development of a novel method to perform maskless analysis of SI wafers with sub-ppb sensitivity across virtually the entire periodic table. This method has been utilized in various studies related to electronic device applications.

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1. INTRODUCTION

Nominally undoped semi-insulating (SI) GaAs is widely used as a substrate material for fabricating monolithic microwave and digital integrated circuits.[1] As the electrical properties of SI GaAs are very sensitive to residual impurities and defects,[2] it is of crucial importance to detect these impurities even at the sub part-per-billion (ppb) elemental level. GDMS is widely used to perform sub-ppb analysis of both metals and semiconductors,[3] however SI samples have proven to be non-trivial. There are a wide variety of electronic device applications for which the ability to analyze trace impurities in SI GaAs would be critical. Performance of field effect transistors (FET's) where thin conducting layers are grown on SI substrates is governed in part by the transconductance, which is directly dependent on substrate purity.[4] Identification and quantitation of various deep traps and impurities in SI material are crucial in evaluating substrate suitability for microwave devices[5] and high-power, optically controlled semiconductor switches.[6]

GDMS provides capabilities for high mass resolution, semi-quantitative, sub-ppb analysis of bulk and thick film materials. Typical analyses involve placing the sample into a liquid nitrogen (LN_2) cooled DC discharge cell, which is biased so that the sample acts as the cathode and the surrounding cell as the anode. When high purity argon gas is admitted into the cell and a voltage is applied between the anode and cathode, a glow discharge is formed. Argon ions are accelerated across the plasma sheath and impact on the sample (cathode) surface, resulting in sample sputtering. These sputtered atoms drift into the glow region and are ionized either by Penning ionization (metastables), or electron impact ionization. These ions are extracted from the cell and analyzed with a high mass resolution, double-focussing mass spectrometer. Ionization cross-sections in the discharge vary by ≈ an order of magnitude across the entire periodic table, thus allowing semi-quantitative data to be acquired without the use of standards. By using relative sensitivity factors (RSF's) derived from steel standards, unknown materials can typically be quantified within a factor of three. The sub-ppb sensitivity of this instrument results from the large amount of sputtered material removed by the discharge, the use of a high transmission mass spectrometer and sensitive pulse counting electronics.

The above arrangement requires that the sample be conducting to some degree. If the sample does not conduct, it will charge up immediately upon voltage application between the anode and cathode, thus making it impossible to maintain a stable DC discharge. Doped semiconductors are typically conductive enough to be analyzed directly without any difficulties, even at very low doping levels. A key step which is often necessary with semi-conductor analysis is to start the discharge while the sample is warm. If the sample is at LN_2 temperature, a large fraction of the carriers are frozen out, making it effectively insulating and impossible to analyze. If the discharge is struck while the sample is at room temperature, the discharge current keeps the sample warm enough to perform the analysis even after starting the LN_2. When the sample is intrinsic, and truly semi-insulating (resistivity > 10^7 Ω·cm), it is virtually impossible to either start the discharge or maintain it in a stable fashion, even if the analysis is attempted with no cooling whatsoever.

Two commonly used methods for GDMS analysis of insulating materials are the use of a conducting mask and the mixing of pulverized sample material with conducting powder. In the first case a high purity conducting mask with a hole slightly smaller than the anode hole is placed in direct contact with the sample. This setup allows the discharge to maintain itself even though most of the cathode area is insulating. The conducting edge of the mask is sufficient to conduct current away and reduce charging. A problem with this method is that the mask is being sputtered in addition to the sample, thus impurities in the mask will show up as being in the sample. Even 99.9999% pure mask material contains 1 ppm total impurities. Through judicious selection of various mask materials, it is possible to perform high sensitivity (ppb) analysis as long as the mask does not contain the impurity desired. In general however, sub-ppb analysis becomes very problematic and in the best case is useful only for a limited number of elements. The second method involves grinding the sample material, mixing it with high purity conducting powder and pressing the combination into a pin or flat shape for analysis. As with the mask material, the limited purity of conducting powder is a significant problem. In addition, this method introduces impurities into the sample during the grinding process. Neither of these methods is truly applicable for routine sub-ppb analysis of semi-insulating samples.

We have developed a method that permits direct, maskless analysis of semi-insulating wafers. The method involves plasma sputter deposition of a thin (≈ 100 Å) layer of high purity (99.995%) gold on all sides of the wafer. Sample analysis is simple, routine, and does not contribute detectable amounts of any additional impurities other than ≈ 15-20 ppbw (ppb by weight) of gold. The limits of detection (LOD) are sub-ppb across virtually the entire periodic table. This new method and the resulting experimental data will be discussed fully in the following sections.

2. EXPERIMENTAL DETAILS

2.1. INSTRUMENTAL

The analyses were performed on a VG9000 GDMS, a double-focusing magnetic sector instrument with typical mass resolution of 4000 and sub-ppb elemental sensitivity. The sample and discharge cell can be in either of two basic configurations, flat or pin. Both cells are capable of LN_2 cooling as well as heating with a power resistor. As this work involved semi-insulating wafers, the flat cell was primarily used. A basic schematic of the flat discharge cell is shown in figure 1. The analysis area is defined by the area of the aperture in the front anode plate. The apertures range from 2 mm to 15 mm in diameter. The sample is isolated from the cathode by two teflon insulators stacked on top of each other. These insulators are in the form of annular disks. The aperture in the first disk is smaller than the sample diameter and is used to make a gas seal against the sample. The second has a larger aperture, which increases the insulating path length (along the teflon surface) in order to maximize the analysis time before anode-cathode shorts occur. Contact to the cathode is made from the backside through a spring-loaded aluminum pressure plate that holds the sample against the teflon insulators. The anode is held at 8 kV and the cathode is biased at 1 kV negative with respect to the anode. The Ar gas used to create the discharge is initially 99.9999% pure, and is further purified prior to admittance into the discharge cell by passage through a getter pump. Chamber pressure is typically in the 10^{-5} torr range during analysis. System base pressure is 5×10^{-9} torr. Normal discharge operating conditions are 1 kV and 3 mA.

2.2. ANALYSIS METHOD

The samples consist of square pieces ≈ 2.3 cm on a side cut from bulk SI GaAs wafers. They are then sputter coated with a thin (≈ 100 Å) layer of high purity gold on both sides and the edges. The films are deposited using an Anatech Hummer VI sputter coater. The coating process takes one minute for each side, plus 3 to 4 minutes for pump

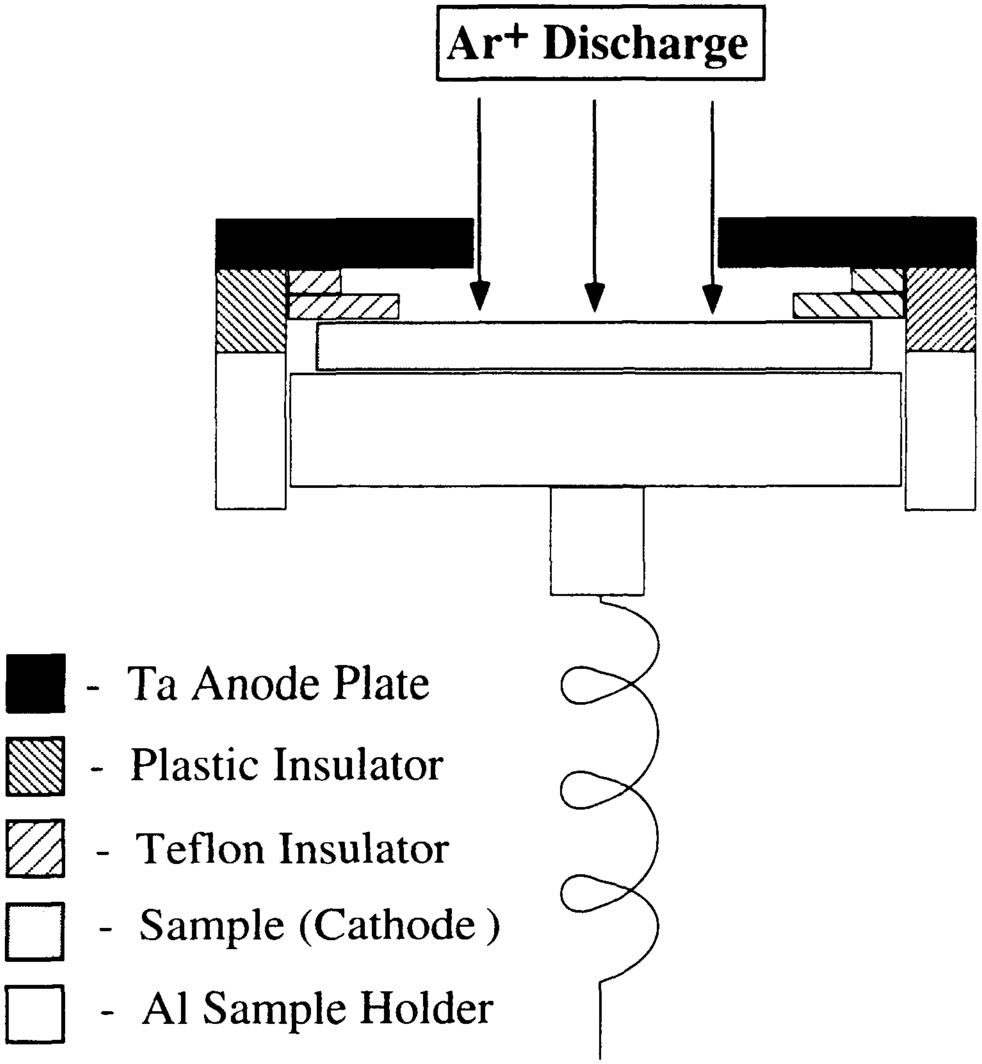


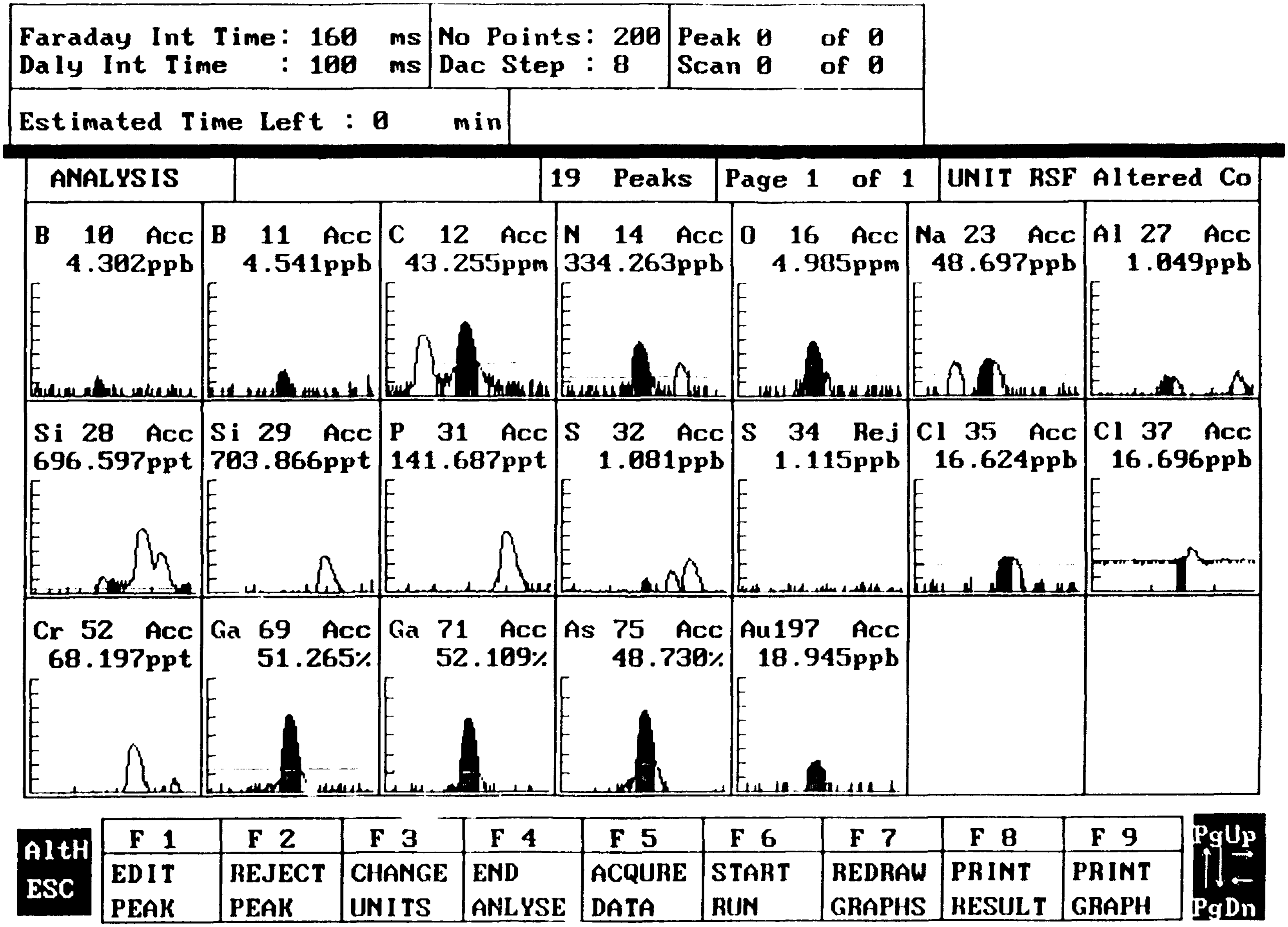
Figure 1. Schematic diagram of the GDMS flat cell arrangement.

This schematic shows the position of the discharge on the sample (cathode) and the arrangement of the teflon and plastic insulators separating the anode from the cathode. The sample is held in place by a spring loaded Al plate.

down for a total of only 5 to 7 minutes. Once the samples are coated they are loaded into the flat cell using the anode with a 15 mm diameter aperture. The discharge is then started and run for 30-45 minutes, allowing the sample to become sputtered over the area of the 15 mm anode aperture. During this period the cell is maintained at room temperature by using the power resistor to heat the sample holder, while simultaneously allowing enough LN_2 to flow through the cooling block to balance the heat input from both the resistor and the discharge. If the sample holder is allowed to cool appreciably, water condensation will occur upon subsequent removal. After 45 minutes, the sample is removed and the 15 mm anode plate is replaced with the 10 mm anode plate for analysis. This is done to minimize any gold contribution to the analysis. If the 15 mm anode is left in during analysis, gold levels of as high as ≈ 0.02 % are seen. By using the 10 mm anode we will only sputter and acquire data on the clean central area, where no gold is present. The cell is reintroduced into the analysis chamber and the discharge and LN_2 cooling are then started. The discharge starts immediately and is stable even though it is sputtering on a non-coated area. After approximately 15 minutes, the cell is cooled down to ≈ -185°C and sample analysis can be performed.

3. RESULTS

After completing the initial procedure and inserting the sample using the 10 mm anode as described previously, data is then collected. An example of experimental spectra as acquired is shown in figure 2. Each window shows a small mass region around the peak of interest. The signal intensities are shown on log scale and the concentrations use standard steel RSF's. Experimental results show that only small residual amounts of gold are detected in the analysis, approximately 15-20 ppbw. At this percentage, even an impurity in the gold at a level of 0.1% would only appear in the analysis at a level of 20



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Daly Int Time : 100 ms
No Points: 200
Dac Step : 8
Peak 0 of 0
Scan 0 of 0
Estimated Time Left : 0 min

ANALYSIS | 19 Peaks | Page 1 of 1 | UNIT RSF Altered Co

B 10 Acc 4.302ppb	B 11 Acc 4.541ppb	C 12 Acc 43.255ppm	N 14 Acc 334.263ppb	O 16 Acc 4.985ppm	Na 23 Acc 48.697ppb	Al 27 Acc 1.049ppb
Si 28 Acc 696.597ppt	Si 29 Acc 703.866ppt	P 31 Acc 141.687ppt	S 32 Acc 1.081ppb	S 34 Rej 1.115ppb	Cl 35 Acc 16.624ppb	Cl 37 Acc 16.696ppb
Cr 52 Acc 68.197ppt	Ga 69 Acc 51.265%	Ga 71 Acc 52.109%	As 75 Acc 48.730%	Au197 Acc 18.945ppb		

Figure 2. Typical mass spectra on GaAs as taken by GDMS.

Intensities are shown on log scale and the concentrations are calculated using standard steel RSF's. The mass range in each window varies from ≈ 0.03 AMU at the low mass end to ≈ 0.75 AMU at the high mass end.

Table 1. Typical survey analysis of semi-insulating GaAs.

Values listed as < are to be taken as the instrumental background detection limits. These data illustrate the excellent detection limits possible with this technique over a wide range of elements

Element	Atomic Concentration (#/cm^3)	Weight Concentration (ppbw)
Li	$< 1.51x10^{14}$	< 0.33
Be	$< 6.82x10^{13}$	< 0.19
B	$1.51x10^{15}$	4.42
Na	$4.86x10^{14}$	3.49
Mg	$< 1.32x10^{13}$	< 0.10
Al	$< 1.35x10^{13}$	< 0.11
Si	$6.27x10^{13}$	0.55
P	$9.67x10^{13}$	0.93
S	$6.30x10^{13}$	0.63
Ti	$< 8.73x10^{12}$	< 0.13
V	$< 6.39x10^{12}$	< 0.10
Cr	$< 6.14x10^{12}$	< 0.10
Mn	$< 4.19x10^{12}$	< 0.07
Fe	$< 1.45x10^{12}$	< 0.03
Ni	$< 5.38x10^{13}$	< 0.99
Co	$< 9.40x10^{12}$	< 0.17
Cu	$< 4.30x10^{13}$	< 0.85
Zn	$< 6.19x10^{13}$	< 1.26
In	$< 2.56x10^{14}$	< 9.19
Sn	$< 7.50x10^{13}$	< 2.78
Au	$2.65x10^{14}$	16.28

pptw (part per trillion by weight). Data are acquired for those elements considered to be important or likely, and the results are shown in table 1. These data illustrate the excellent detection limits possible with this method. Those values listed as < (less than) should be considered upper limits for the elements in question. The LOD for these elements range from 10^{12} to 10^{14} #/cc. These LOD's were achieved using moderate signal integration times (1-3 sec) in order to make the total run time reasonable (≈ 1 hr). They can be improved further by increasing the integration time and acquiring fewer elements. Raw data is reported in concentration by weight. In table 1 are shown both the weight concentration values and the atomic concentrations calculated from those data.

As part of an ongoing program with industrial partners to improve the quality of GaAs substrates, we have analyzed wafers from a number of different manufacturers. In table 2 are shown the data for impurities monitored and detected above the instrumental background. Note the variations in B content, ranging from 10^{15} to 10^{17} /cm^3. The B comes from the B_2O_3 used as an encapsulant in the liquid encapsulated Czochralski (LEC) growth process. The wide disparity in B concentration may be caused by variations in the processing details such as temperature profile, crystal pulling speed and initial starting material. Some vendors obviously use processing steps which are more effective at reducing boron incorporation into the bulk material. Using Vendor #4 as a baseline, it is seen that differences between vendors for other elements vary by up to a factor of 100. These data are being used by the manufacturers to fine tune their processes.

As part of another program looking at substrate material for use in optical waveguides, data have been acquired comparing n+ and SI wafers supplied from the same manufacturer. Significant and unexplained differences are observed when comparing

Table 2. Multi-vendor analysis of semi-insulating GaAs substrates.
Data illustrate the wide variety of impurity concentrations among the various vendors.

Element	Vendor #1 (#/cm^3)	Vendor #2 (#/cm^3)	Vendor #3 (#/cm^3)	Vendor #4 (#/cm^3)	Vendor #5 (#/cm^3)
B	2.78×10^{15}	3.00×10^{17}	6.45×10^{16}	1.31×10^{15}	1.56×10^{17}
Na	6.20×10^{15}	4.24×10^{14}	2.25×10^{15}	4.86×10^{14}	2.36×10^{14}
Mg	$< 4.77\times10^{13}$	1.23×10^{15}	$< 1.86\times10^{13}$	$< 1.32\times10^{13}$	$< 4.09\times10^{13}$
Al	4.92×10^{14}	1.94×10^{15}	9.95×10^{13}	$< 1.35\times10^{13}$	$< 2.84\times10^{13}$
Si	5.77×10^{13}	2.46×10^{14}	1.21×10^{15}	6.27×10^{13}	6.83×10^{14}
P	7.70×10^{14}	8.43×10^{14}	2.83×10^{14}	9.67×10^{13}	2.82×10^{15}
S	1.38×10^{14}	4.60×10^{14}	7.16×10^{14}	6.30×10^{13}	7.94×10^{14}
Cr	8.28×10^{12}	9.51×10^{13}	$< 9.49\times10^{12}$	$< 6.14\times10^{12}$	$< 9.51\times10^{12}$
Mn	$< 1.75\times10^{13}$	2.70×10^{14}	$< 8.20\times10^{12}$	$< 4.19\times10^{12}$	$< 1.57\times10^{13}$
Fe	1.31×10^{13}	5.61×10^{14}	$< 4.94\times10^{12}$	$< 1.45\times10^{12}$	$< 1.31\times10^{13}$
Cu	$< 5.91\times10^{13}$	$< 3.21\times10^{13}$	9.45×10^{13}	$< 4.30\times10^{13}$	$< 3.72\times10^{13}$
Zn	1.26×10^{14}	2.48×10^{14}	$< 4.17\times10^{13}$	$< 6.19\times10^{13}$	$< 7.20\times10^{13}$
Au	3.03×10^{14}	9.41×10^{14}	6.95×10^{14}	2.65×10^{14}	5.71×10^{14}

waveguides grown on these substrates. The n+ wafer is doped with silicon to a level of 1.2 to 1.7 x 10^{18} Si/cc, according to the manufacturer specification sheet. The value determined by GDMS without the use of standards was 1.81 x 10^{18} Si/cc. This illustrates the semi-quantitative nature of GDMS analysis. The n+ wafer was coated with gold to maintain consistency with the analysis of the SI sample. In table 3, data are shown comparing the two wafers. The most glaring difference between wafers is the boron concentration. The n+ wafer has ≈ 70 times the boron concentration of the SI wafer. It is possible that boron is diffusing out into the active layers of the device, causing the observed performance changes. This possibility is presently being investigated.

4. DISCUSSION

Maintenance of a stable discharge requires that positive argon ion current impinging on the sample surface have some conduction path to the back of the sample where the cathode contact is made. The fact that the discharge runs as stably on SI substrates as with conductors implies that a conductive path exists. When using the 15 mm anode, the gold film initially supporting the discharge is sputtered away in less than a minute. It is possible during this initial period that the gold film outside the sputtered area is close enough to the discharge to allow excess charge to leak away. This cannot be the case however, when performing analysis with the 10 mm anode. The discharge is not near the gold film in this case. Thus the only way to complete the circuit is for the current to move through the GaAs itself. The proposed mechanism for this effect is related to defect-induced conductivity. When the discharge is initiated, the current is conducted away through the gold film. As the film is sputtered away, the GaAs substrate begins to be sputtered as well. As it sputters, physical defects are created in a thin surface region with a thickness governed by the depth range of the impinging argon ions. These defects can increase the

TABLE 3. Comparison of n+ and SI GaAs substrate material from the same vendor. In particular note the unexpected increase in boron concentration.

Element	n+ Wafer ($\#/cm^3$)	SI Wafer ($\#/cm^3$)
B	4.69×10^{18}	6.45×10^{16}
Na	8.72×10^{14}	2.25×10^{15}
Mg	2.06×10^{14}	$< 1.86 \times 10^{13}$
Al	1.15×10^{15}	9.95×10^{13}
Si	1.81×10^{18}	1.21×10^{15}
P	2.95×10^{13}	2.83×10^{14}
S	4.10×10^{14}	7.16×10^{14}
Cr	$< 6.59 \times 10^{12}$	$< 9.49 \times 10^{12}$
Fe	$< 2.84 \times 10^{12}$	$< 4.94 \times 10^{12}$
Cu	$< 3.61 \times 10^{13}$	9.45×10^{13}
Zn	$< 4.90 \times 10^{13}$	$< 4.17 \times 10^{13}$
Au	2.50×10^{14}	6.95×10^{14}

density of states within the GaAs to make the sample no longer SI, but semiconducting instead. Measurements of the surface conductivity were taken using a four-point-probe technique. Results showed the σ of the sputtered region to be more than an order of magnitude greater than that of the unsputtered region. Thus, when performing analyses with the 10 mm anode, current is carried through the thin, conductive surface region which was sputtered previously with the 15 mm anode. It then reaches the unsputtered region still coated with gold and travels directly to the back cathode contact to complete the circuit. This arrangement is shown schematically in figure 3.

5. SUMMARY

We have developed a new analytic procedure for GDMS which enables direct analysis of bulk semi-insulating GaAs wafers. This method is simple, routine and does not contribute any detectable impurities into the analysis other than ≈ 15-20 ppbw of gold. Measurements verify sub-ppb limits of detection over a wide range of elements across the periodic table. Future studies will involve testing this method on other semi-insulating materials, as well as on pin-shaped samples, and further investigation into the mechanisms involved.

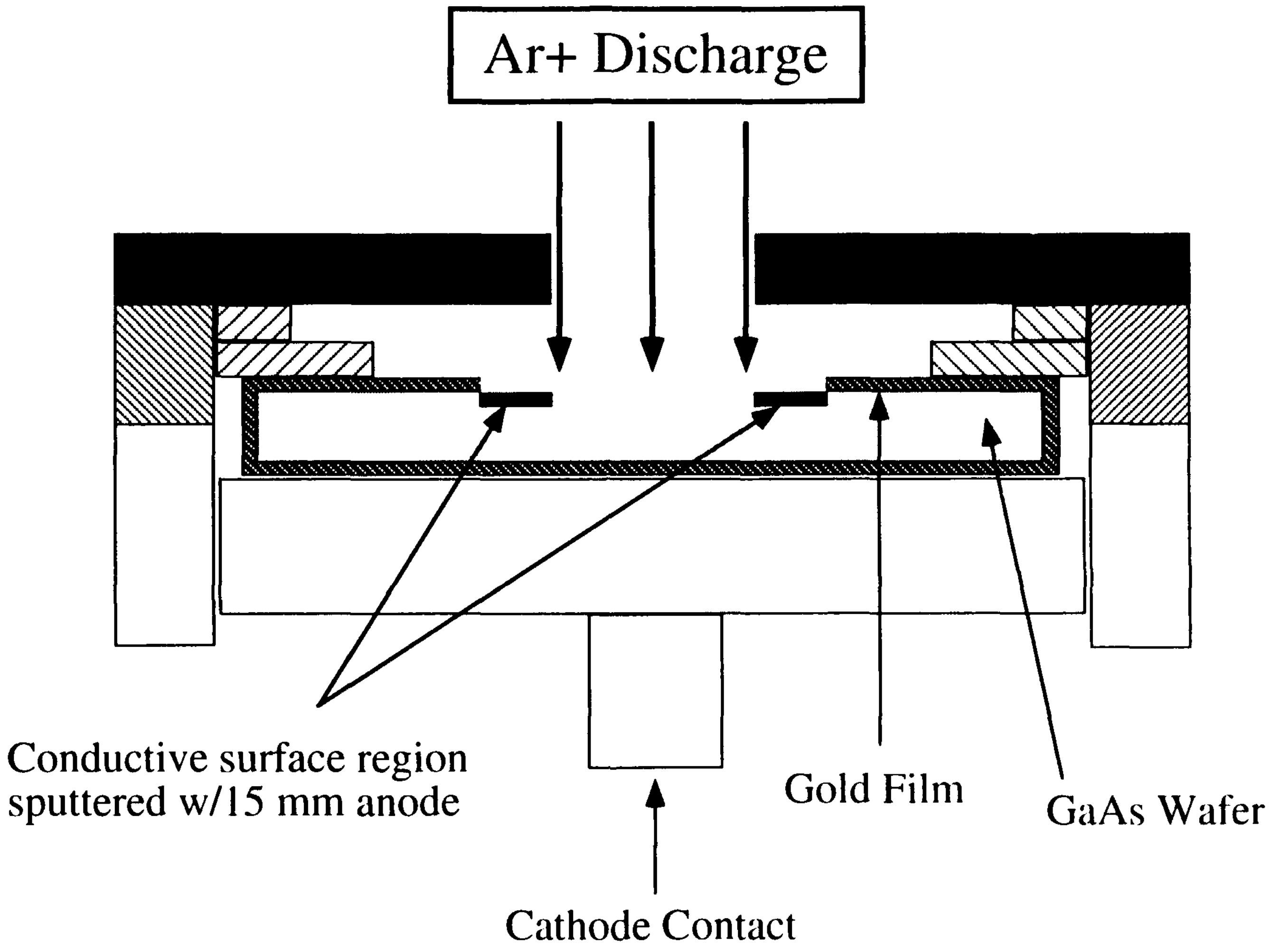

Figure 3: Schematic showing conduction path to cathode contact.

The black area on the sample corresponds to the area sputtered with the 15 mm anode. This area is slightly conductive (leaky). Current is carried through this surface layer to the gold film and around to the back cathode contact.

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