

Final Report on Project “Collaborative Research: Fundamental Studies of Plasma Control Using Surface Embedded Electronic Devices”.

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### 3. Executive Summary:

The research program was collaborative between the researchers at the University of Texas at Dallas and the University of Texas at Austin. The primary subject of this program was to investigate the possibility of active control of secondary electron emission (SEE) from surfaces in contact with plasmas and thereby actively control plasmas.

**How the research adds to the understanding of the area investigated:** Very few studies of ion induced electron emission (IIEE) from semiconductors exist and those that do exist primarily used high energy ion-beams in the experiments. Furthermore, those few studies took extreme measures to ensure that the measurements were performed on atomically clean surfaces because of the surface sensitivity of the IIEE process. Even a small exposure to air can change the IIEE yield significantly. In addition, much of the existing data for IIEE from semiconductors was obtained in the 1950s and '60s when semiconductor materials were first being refined. As a result, nearly all of that data is for p-type Ge and Si. Before this investigation, experimental data on n-type materials was virtually non-existent. While the basic theory assumed that IIEE yields ought to be substantially independent of doping type and concentration, recent measurements of near atmospheric pressure plasmas and of breakdown suggested otherwise. These indirect measurements were made on surfaces that were not atomically clean and seemed to indicate that deep sub-surface changes to the bulk conduction band electron density could lead to substantial variations in the IIEE yield. Exactly in contradiction to the generally accepted theory. Insufficient direct data existed to settle the matter.

We performed both experimental measurements and theoretical calculations of IIEE yields from both Si and Ge in order to help clarify whether or not conduction band electrons substantially change the IIEE yield. We used three wafers of each material to carry out the investigation: a heavily doped p-type, an intrinsic and a heavily doped n-type wafer. There was approximately a factor of  $10^{15}$  difference in the conduction band electron densities of the p-type and n-type Si wafers and a factor of  $10^{10}$  for Ge. We investigated semiconductor surfaces that were both chemically cleaned (not atomically clean) and sputter cleaned (much closer to atomically clean) since such measurements are more relevant to recent indirect measurements. In addition to IIEE measurements, X-ray and ultraviolet photoelectron spectroscopies (XPS and UPS) were utilized to better understand the results.

No measureable difference in the IIEE yield could be found between p-type and n-type wafers despite the orders of magnitude variation in the conduction band electron densities. This result held for both sputter cleaned and chemically cleaned surfaces. We also found that while some Fermi level pinning occurs for both Si and Ge, there was no correlation between the location of the measured surface Fermi levels and the IIEE yields. In addition, we were able to use UPS to confirm that the surface density of allowed energy states does correlate well with the IIEE yields. These results both confirmed the basic theory and appeared to contradict recent indirect measurements.

**The technical effectiveness and economic feasibility of the methods or techniques investigated or demonstrated:**

The project has demonstrated a need to better understand whether an imposed electric field on a semiconductor surface can enhance IIEE yield. There is reason to believe that a synergistic relationship exists between incoming ions and an electric field at the surface which can substantially increase the IIEE yield. It can be characterized as either an ion enhanced field emission process or a field enhanced Auger neutralization process. No experimental measurements of such synergy exist to our knowledge. Conventional IIEE measurement systems would not have the ability to make such measurements because of the large voltages required to impose such electric fields on the macro scale. (The electric field strengths must be in the range of 1 - 10V/ $\mu\text{m}$  to be consistent with those found in the sheath of a typical microplasma.) A possible solution to this problem is to fabricate a MEMS device for making the measurements. By shrinking down the electron detection system, it is possible to create the 10V/ $\mu\text{m}$  electric fields by applying a small 10V across a 1  $\mu\text{m}$  gap. (As compared to 10kV across a 1 cm gap.) We spent some effort trying to fabricate such a MEMS device and have some preliminary designs, but have been unsuccessful in completing a device to date.

**How the project is otherwise of benefit to the public:** This project has supported the graduate education of four (4) students (D. Urrabazo – PhD 2015, K. Hernandez presently writing his MS Thesis, A. Press and Z. Wang.) In addition, it supported travel to conferences and equipment maintenance. One of the students (D. Urrabazo) obtained his Ph.D. as a direct result of his work on this program. Another student (K. Hernandez) is writing his MS Thesis on the device fabrication efforts this fall. The third and fourth students (A. Press and Z. Wang) are continuing their Ph.D. research in plasma science. Three of the 4 students are US citizens. The fourth is a citizen of China.

4. Comparison of the actual accomplishments with the goals and objectives of the project:

The major goals enumerated for the UT Dallas team were: “Provide experimental data on the variations in SEE which embedded electronic devices actually achieve. This includes detailed experimental examination of both the capability to control SEE and the limitations to that control” and “detailed measurements and modeling of plasma characteristics during active SEE control as part of exploring how sub-surface devices can exert control over a plasma discharge.” Please note that “Secondary Electron Emission” (SEE) and “Ion-Induced Electron Emission” (IIEE) can be used interchangeably. These final goals were not fully accomplished during the time of the project for two reasons: There were unanticipated major difficulties in device fabrication and our IIEE measurements convincingly indicated that the conduction band electron density (sub-surface) did not independently affect IIEE yields. The latter had not been measured previously and is a major result of this project. In addition, theoretical calculations developed at the same time reinforced this conclusion. On the other hand, there was reason to theorize that an increase in the sub-surface conduction band electron density could affect IIEE if the electric field intensity at the surface were made sufficiently large. No known measurements of IIEE as a function of surface electric field intensity exist since the MEMS technologies needed to make such measurements have only become available relatively recently.

Back tracking slightly. Sub-surface electronic devices can primarily affect the conduction band electron density at the device surface and have little to no effect on the electric field intensity at the surface. Our initial measurements of IIEE from differently doped Si and Ge samples indicated that the sub-surface conduction band electron density alone produced little to no effect on IIEE yields. Thus, the first goal was fundamentally achieved without the fabrication of a sub-surface electronic device. It was found that a sub-surface electronic device will not exert control over IIEE from semiconductor surfaces (when used alone). Control of IIEE by such a device requires the imposition of an electric field on the surface as well. That electric field must be set externally and is imposed by the plasma sheath in the case of atmospheric pressure

plasma sources. As a result, it became crucial to make measurements of IIEE from semiconductor surfaces in combination with an imposed electric field. Thus, the second goal of the original project was delayed to allow us to begin development of a novel IIEE measurement technique as a function of surface electric field intensity. This is where we encountered major difficulties in the MEMS device fabrication. To date, we have been unsuccessful in fabricating the needed MEMS device. The achievements and difficulties will be described in detail below.

## 5. Summary of project activities:

### **Development of a Mass Spectrometer based IIEE measurement tool.**

At the initiation of the project, we spent extensive time modifying, automating and calibrating an Extrel Corporation mass spectrometer to act as an ion beam source as well as Ion Induced Electron Emission (IIEE) detector. In addition, we formulated our sample preparation processes for the initial measurements. The sample preparation processes produced ohmic contacts for biasing the sample in-situ as well as maintained hydrogen-terminated surfaces (i.e. minimized surface oxidation).

Experimental measurements of IIEE yields from surfaces are known to be difficult because they require extensive equipment and detailed calibration.<sup>1</sup> Achieving acceptable accuracy and control with our mass spectrometer for absolute yield measurements was also a difficult task, but was been successfully accomplished for so-called “dirty” (chemically cleaned) surfaces. We used the ion source of our mass spectrometer to create the ion beam, the quadrupole mass filter to select the ions desired for testing and the conversion dynode (CD) to accelerate those ions into the surface of interest. The resulting yield of secondary electrons is directed to a channeltron multiplier directly opposite the CD where they were counted and determine the IIEE Yield. We modified the CD and channeltron detector region substantially to enable us to obtain absolute IIEE yields for ion energies between approximately 25 and 300 eV. It is more typical in the literature to find IIEE yields measured for ions having keV energies since this is easier and the yields are substantially larger.

Several complexities were overcome in the course of this portion of the research including difficulties in setting up the mass spectrometer calibration as well as sample preparation. In addition to extensive testing, we used COMSOL 3.5a, a finite element - multiphysics software, to investigate how the channeltron biasing influences the ion trajectory and affects the IIEE measurements. The numerical simulation results provided insights which caused us to add a 40% open area grounded mesh at the front face of the channeltron. The mesh acts to electrostatically isolate the channeltron from the CD while still allowing secondary electrons from the CD to enter the channeltron and be detected. This greatly improved our ability to measure IIEE yields of low energy ions.

The experimental configuration used for the tests performed in our modified mass spectrometer is shown in Fig. 1. Ions from the mass spectrometer are accelerated onto the surface being tested (“sample”) which sits at the location of the conversion dynode (CD). The emitted electrons are collected by a channeltron electron multiplier and the ratio of the electron counts (current) to the current at the sample defines the IIEE measurement. There is a grounded mesh screen that sits between the sample and the channeltron which minimizes the effect of the channeltron bias on the ion trajectory. In order to get accurate IIEE yield

measurements we had to quantify the electron loss to this mesh (as well as other factors). To do this accurately, we made electron yield measurements on our p-type Si sample and normalized the results to published data<sup>3</sup>. In addition, we made measurements of Pt and found the results to match reasonably well with those in the literature. This allowed us to algebraically determine the transmission efficiency through the mesh which was then used in estimating the yield values for all the remaining samples.

Information on the semiconductor samples we have studied is listed in Table I and primary results from our experiments are shown in Figs. 2 and 3. All of the samples had back-side ohmic contacts. This allowed us to accurately control the surface potential and measure the ion current. In addition, the samples were carefully chemically cleaned before each measurement using an acetone/ipa solution to remove organics followed by a 100:1 HF dip to remove oxides and form a hydrogen terminated surface. The ions used to bombard the samples and induce electron emission were  $\text{Ar}^+$ ,  $\text{Ar}^{++}$ ,  $\text{Ne}^+$ , and  $\text{He}^+$ . Many other ions could be tested, but we limited our tests to these four. The ion kinetic energy was varied from 25 eV up to 300 eV.

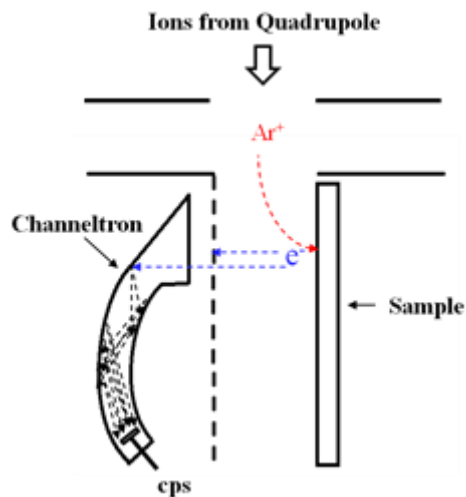


Figure 1. Ion impact and electron collection schematic of detection system.

Table I. Properties of Semiconductor Samples

Material	Type	Resistivity [ $\Omega\text{-cm}$ ]	Dopant	Dopant Density [ $\text{cm}^{-3}$ ]
Silicon (100)	P-type	1-5	B	$0.3\text{-}1 \times 10^{16}$
	Intrinsic	>1000	P	$<4 \times 10^{12}$
	N-type	0.002-0.004	As	$1\text{-}3 \times 10^{19}$
Germanium (100)	P-type	0.01-0.04	Sb	$0.2\text{-}1 \times 10^{18}$
	Intrinsic	>40		$<1 \times 10^{13}$
	N-type	0.005-0.02	Ga	$0.2\text{-}2 \times 10^{18}$

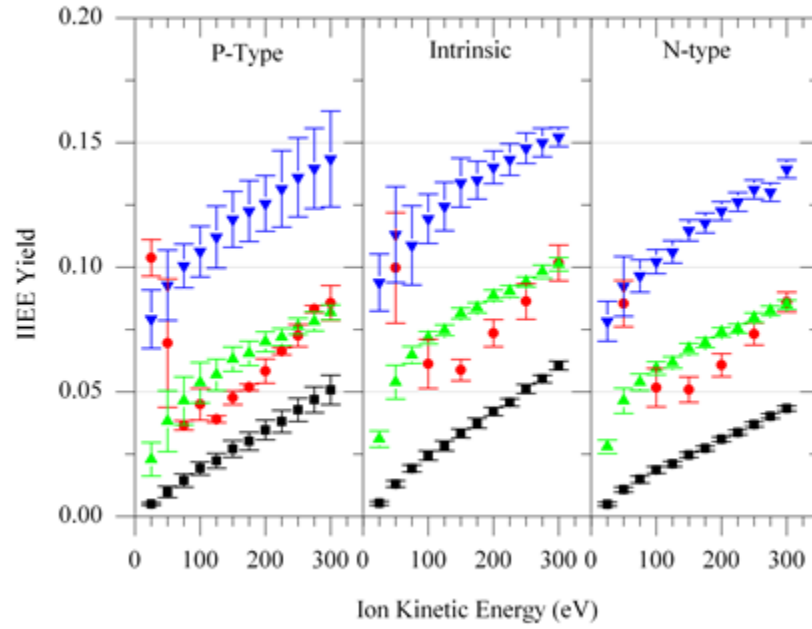


Figure 2. Calibrated IIEE yields for  $\text{Ar}^+(\blacksquare)$ ,  $\text{Ar}^{++}(\bullet)$ ,  $\text{Ne}^+(\blacktriangle)$  and  $\text{He}^+(\blacktriangledown)$  on p-type, intrinsic and n-type Si.

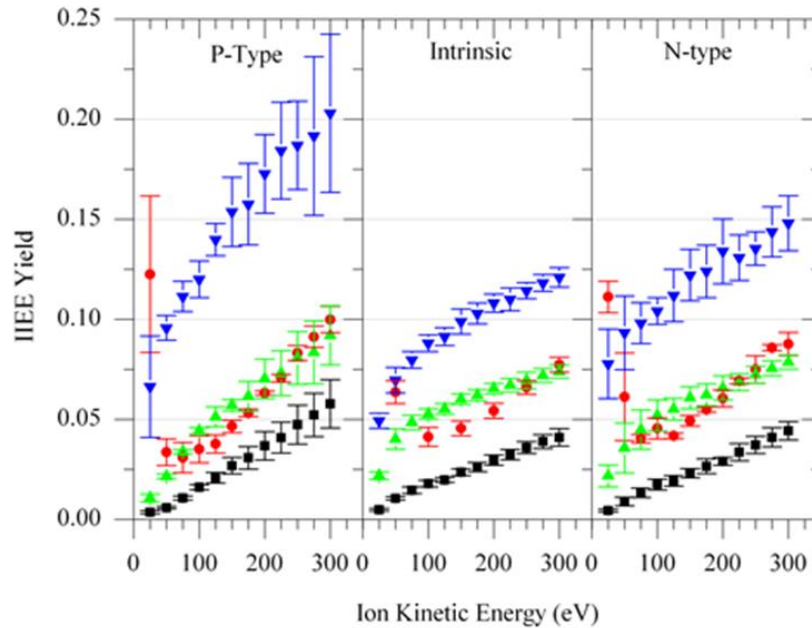


Figure 3. Calibrated IIEE yields for  $\text{Ar}^+(\blacksquare)$ ,  $\text{Ar}^{++}(\bullet)$ ,  $\text{Ne}^+(\blacktriangle)$  and  $\text{He}^+(\blacktriangledown)$  on p-type, intrinsic and n-type Ge.

The primary observation to be made from Fig. 2 is that **the IIEE yields from p-type and n-type Si are essentially equal (within the experimental error bars)**. This is despite the fact that the conduction band electron concentration changes by approximately 15 orders of magnitude. This observation agrees well with

the underlying assumption in Hagstrum's models<sup>2</sup> that the IIEE yield is independent of semiconductor properties, i.e. bulk dopant density. Another item of note is the variation in the  $\text{Ar}^{++}$  yields at low ion kinetic energies. The  $\text{Ar}^{++}$  yield appears to rise below 100 eV. We do not believe this rise to be real, but rather speculate that it is an artifact of the double charge on the ion causing a variation in its trajectory as it exits the quadrupole. This changed trajectory affects the calibration and thus the estimated yield. This deviation in the calibration is diminished at the higher ion energies as expected. A third observation is that the yields for the intrinsic Si sample are slightly larger than for n-type or p-type. This might arise from a number of sources. Despite the slightly larger yields from the intrinsic Si sample, one can clearly conclude that the IIEE yield is not influenced by the number of electrons in the bulk conduction band for a chemically cleaned Si surface.

IIEE yield measurements were also made for Ge using the same calibration and are shown in Fig. 3. The Ge IIEE yields show similar trends to those of Si. For example, the IIEE yield dependencies on ion kinetic energy as well on ion specie remain consistent. Like Si, the Ge measurements indicate that IIEE is not dependent on the bulk material conduction band electron density. In fact, for all ions at kinetic energies above 150 eV, p-type Ge is found to have larger IIEE yields than n-type Ge. The difference between p-type and n-type Ge reduces as the ion kinetic energy decreases. At the lowest ion kinetic energy (25 eV), the difference between the different doping types is within the error bars for  $\text{Ar}^+$ ,  $\text{Ne}^+$  and  $\text{He}^+$ . These findings were repeatable between different samples and wafers of the same doping type and level. In addition, the data was taken in random order to minimize the effects of progressive changes to the surface morphology from ion sputtering. Thus we conclude that the larger IIEE yields from p-type Ge are real and repeatable.

#### **Use of an XPS tool to measure SEE.**

The first journal paper review on the results presented above was very critical of the fact that we had made measurements on "dirty" surfaces (only chemically cleaned and not atomically cleaned surfaces) and that our results were taken at pressures  $\sim 10^{-7}$  Torr. Therefore, we were forced to find an alternative means for measuring the IIEE yield at much lower pressures and at better defined surface conditions for comparison to our results. We found that this was at least partially possible by using a Versa Probe II X-Ray Photoelectron Spectroscopy (XPS) system which operates at a pressure of  $\sim 10^{-10}$  Torr. XPS was used to verify the removal of the adventitious carbon and oxygen that contaminate the surface and thereby define the surface conditions much more fully. In addition, the Versa Probe II system has an argon ion beam that can impact the substrate under test at an angle of 45 degrees. The ion beam consists primarily of  $\text{Ar}^+$  but has a small and unavoidable admixture of  $\text{Ar}^{++}$ . This beam is ordinarily used to sputter clean the surface; but we found that it could also be used to make relative IIEE yield measurements under some circumstances. So we did. The ion impact energy was set to 34 eV by floating the gun to 25V and having the sample biased to -9V. Emitted secondary electrons enter a grounded hemispherical energy analyzer positioned normal to the surface. The energy analyzer allows the user to accurately and consistently measure the electron energy distribution curves (EEDCs) of the secondary electrons. Integrating the EEDCs and dividing by the Ar ion beam current allows one to obtain a relative yield. The results are shown in Fig. 4.

Figure 4 shows the measured relative IIEE yield values for chemically cleaned and sputter cleaned Si and Ge. Independent of the cleanliness of the samples, the IIEE yield does not change significantly when comparing p-type to n-type samples of both Si and Ge. In fact, any change is within the error bars. From these results, it is concluded that the conduction band electron density, in the bulk, does not influence the IIEE yield. A second

observation is that the IIEE yield independence from bulk doping is found for both “dirty” (chemically clean) and sputter cleaned (nearly atomically clean) samples even though they have different surface chemistries (i.e. oxygen and carbon content). We note that the variation between the IIEE yield values is typically attributed to the removal of the adventitious carbon and oxygen. Another observation is that the difference between the chemically clean and sputter clean samples is almost a consistent factor of 1.5-1.9 for both Si and Ge. This result indicates that the atmospheric contamination does not change the IIEE yields dramatically. Additional measurements were obtained using this XPS/UPS tool and other conclusions made which are detailed in the dissertation by D. Urrabazo.

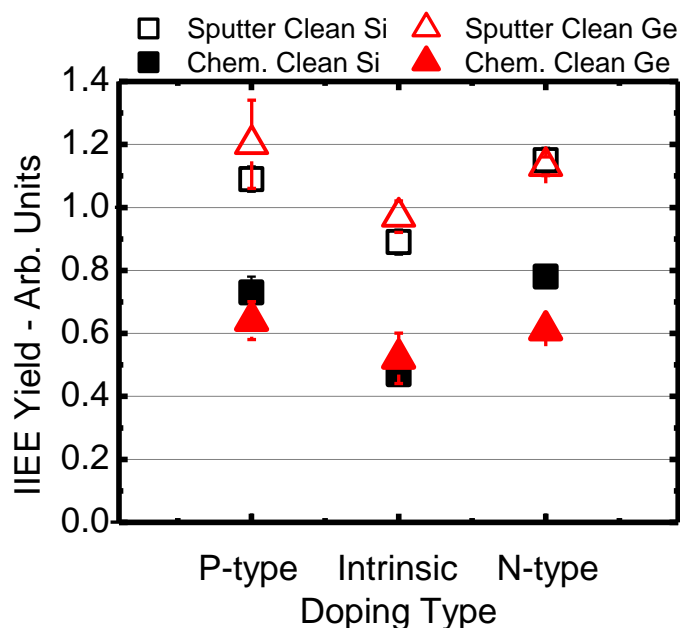


Figure 4: IIEE Yield from chemically and sputter cleaned Si and Ge.

**Overall, the results for Si and Ge indicate that changing the bulk dopant concentration (and thereby changing the electron concentration in the conduction band) does not influence the IIEE yield.** This result appears to contradict a common conception today that injecting electrons into a p-type surface layer (by a sub-surface pn-junction) can enhance IIEE and thereby lead to IIEE control. On the other hand, convincing measurements in microplasma and breakdown experiments indicate otherwise. Those indirect measurements indicate that IIEE can, in fact, be enhanced significantly by injecting conduction band electrons toward the surface. The authors of those articles have rightfully noted that there is a large electric field at the surface of the semiconductor under the conditions of their experiments. We did not have the ability to impose such an electric field for our measurements. Thus, it has become clear that we need to impose an electric field on the surface of the semiconductor while injecting electrons sub-surface. The lack of a surface electric field may be the reason we have not found any dependence in the IIEE yield upon conduction band electron density.

**Development of a MEMS device to accurately measure IIEE in the presence of an electric field.**

The results outlined above caused us to modify our goals. In our annual progress reports we noted:

*Our research thus far has indicated that achieving active control of SEE cannot be achieved simply by varying the conduction band electron density. Therefore, testing a subsurface device (like a pn-junction) appears to be less valuable now. In order to move forward we needed to re-evaluate the possible mechanisms that drive the functionality of the “plasma transistor.” In the latest publication on the plasma transistor<sup>4</sup>, it was observed that increasing the electron concentration in the conduction band, in the presence of a microplasma, increased the apparent electron yield by 360%. The exact mechanism driving this apparent yield enhancement remained speculative. Some have assumed it to be driven by some form of synergistic effect between ion species, external sheath electric fields and the conduction band electron density. Thus, in order to progress towards active control of emission yield we have decided to investigate the synergy between ion bombardment and a surface electric field.*

To our knowledge such an experimental investigation (for semiconductors) had not yet been tried and still has not been tried. This is because the electric field strengths involved in such tests require a micron scale device to allow for testing low energy ions. As a result, we have put extensive effort into combining an intense electric field (as high as  $10 \text{ V}/\mu\text{m}$  – covering the range expected for microplasma sheaths) with low energy ion bombardment. It is our aim to provide experimental insight into whether or not an electric field can enhance ion induced electron emission from semiconductors. This work is intended to lead to a more complete understanding of how one can achieve active control of electron emission using sub-surface devices.

We have designed, numerically modeled and tried diligently to fabricate a micro-scale device that allows largely independent control over both the ion impact energy and the surface electric field. If we are able to get past the fabrication challenges, this device could be fabricated on top of flat surfaces able to withstand  $300^\circ\text{C}$ . The goal was to have a MEMS device which enabled IIEE measurements in the presence of strong electric fields on any semiconductor and many other materials as well. The hope has been to examine the synergy between ion bombardment and a surface electric field in the secondary electron emission process.

The envisioned device is a multi-layered, perforated structure. A schematic view of a cross section is shown in Fig. 5. An array of holes allows a substantial fraction of incoming ions ( $\text{Ar}^+$  in Fig. 5) to enter the structure and bombard the surface of interest (“Si” in Fig. 5). The local potential structure is set by the bias applied to the electron collector (“ $V_{\text{coll}}$ ”) and the electron suppressor (“ $V_{\text{supp}}$ ”) while the ion energy is controlled by the sample potential (“ $V_{\text{sample}}$ ”) with respect to ground. The suppressor voltage forces the IIEE electrons to be collected at the electron collector. The potential difference between the sample and the electron collector sets the surface electric field. A thin conducting film (Al) between the suppressor and collector prevents the electron current measurement from being overwhelmed by current flowing between these two levels.

We performed extensive modeling of the device operation using COMSOL and had settled on a set of design rules for fabricating the first devices on Si substrates. We identified a set of 5 main concerns/limitations to address in designing the micro-device. These are:

- (1) High efficiency in collecting the emitted electrons (greater than 90%),
- (2) High uniformity in the electric field intensity at the surface (less than 10% variation).
- (3) As large as possible ion transmission efficiency (greater than 40%),



- (4) Electrostatic deflection limitations (less than a 5% reduction in distance between the collector electrode and the substrate due to electrostatic attraction) and finally
- (5) Limitations imposed on the fabrication by both the clean room (allowed materials and processes) and substrates for test (thermal budget, etc.).

Addressing these limitations and concerns has required extensive effort in developing the fabrication processes: low temperature deposition steps for the high dielectric-strength, thin-film layers; etch steps for forming both the holes and electrical connections to the various layers; as well as photo-lithography steps and masks for the complete structure. On several occasions we were convinced that we were close to completing a device only to have an unexpected failure/challenge arise. At present, we are able to deposit metallic layers (with sufficiently large conductivity) and insulating layers (with sufficiently small conductivity). Unfortunately, thick metal-layers cause unacceptable damage to the insulating layers due to either morphology (pinholes and surface protrusions) and/or the addition of film stress inducing cracks/delaminations. In what follows, we will briefly note the various challenges and solutions investigated to date.

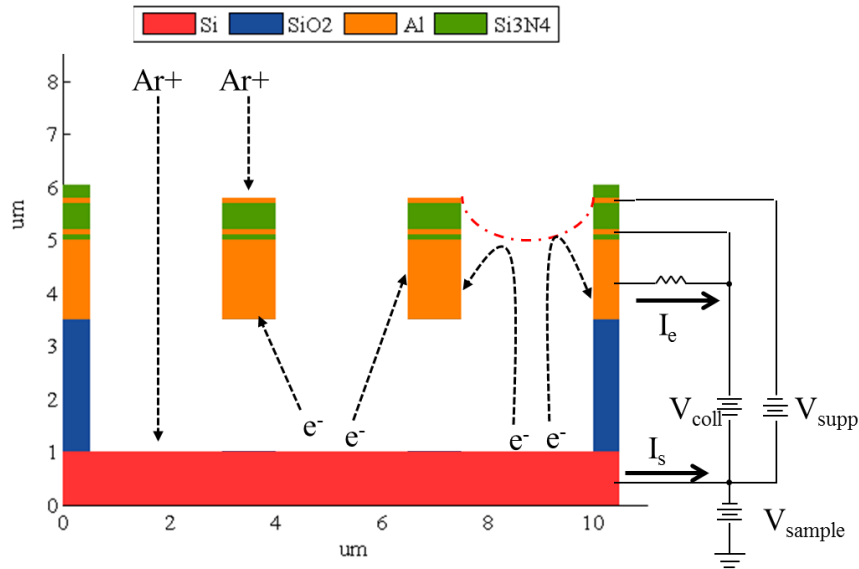


Figure 5: A cross-sectional view (not to scale) of a micro-device for imposing large electric-fields on a surface while making IIEE yield measurements of low-energy ions.<sup>5</sup>

To create the IIEE measurement device, lithography, deposition, etch and ball-bonding processes had to be developed in the UTD cleanroom. Dielectric deposition was first performed using PECVD for both  $\text{SiO}_2$  and  $\text{Si}_3\text{N}_4$ . Dielectric etch was performed in a Plasma-Therm ICP reactor using  $\text{SF}_6$  for  $\text{Si}_3\text{N}_4$  and  $\text{CHF}_3$  for  $\text{SiO}_2$ . Metal layer deposition was performed in either a sputter or e-beam evaporator while metal etch was performed in another Plasma-Therm ICP reactor having  $\text{BCl}_3$  chemistry. Lithography was performed using a Karl Suss MA6B Contact Printer. Four lithography masks were used to create contact pads at the three different layers as well as etch through-holes to the surface under test. Finally, the semiconductor substrates were cleaned using acetone, IPA and DI water followed by an HF acid dip. Each step has presented challenges, many of them relatively unexpected since the standard processes had been expected to function well for us. For examples:

1. Metal depositions: Aluminum deposition was initially performed in a CHA Mark 50 (e-beam evaporator). Unfortunately, the deposited aluminum layers were not uniform and bright flashes were observed during the evaporation. The bright flashes coincided with the ejection of macroscopic agglomerations of atoms (“clusters”) from the crucible. We were unable to eliminate either the flashes or the Al “clusters” and therefore had to switch to a different deposition tool. A Temescal 1800 (also an e-beam evaporator) did not have the same uniformity issues and was more successfully used for both Al and Cr deposition. The metal layers were found to still have sufficient surface roughness and stress to affect subsequent dielectric depositions. A literature search indicated that reduced stress Al layers could be deposited by heating the substrate to 165°C or higher during the depositions. The Temescal does not have this capability; therefore selected metal depositions were performed in an AJA 1500 (sputter deposition tool). Unfortunately, the Al layers deposited in this tool had an overly white coloration. The electrical properties were acceptable but the films appeared to have Al<sub>2</sub>O<sub>3</sub> incorporation.
2. Dielectric depositions: Initially, SiO<sub>2</sub> and Si<sub>3</sub>N<sub>4</sub> dielectric depositions were performed using a Plasma-Therm 790 PECVD tool. Unfortunately, the film stress caused complete delamination when the structures were etched. We attempted to deposit lower stress films but those were found to be too conductive. The conductivity of the insulators became an issue of significant concern and pinholes were identified as a significant component of through insulator conductivity. This led to using Al<sub>2</sub>O<sub>3</sub> films deposited using atomic layer deposition (ALD) process. A Metal-Insulator-Metal structure consisting of Al electrodes (having a thin Cr interfacial layer) and 100 nm ALD Al<sub>2</sub>O<sub>3</sub> plus 500 nm of sputtered Al<sub>2</sub>O<sub>3</sub> was found to have appropriate conductivity and dielectric strength. See Fig. 6. Making the Al layers thicker was found to require using thicker Cr layers to retain a good dielectric layer due to increased surface roughness.
3. Lithography: We first fabricated the various masks (4) required for the device in the UTD cleanroom. Unfortunately, this quickly led to the realization that the laser-writer used to create the Cr masks could not print our critical dimensions. While the tool claims 1 micron resolution, the repeated patterns required for our device caused unacceptably large deviations. As a result, that critical mask was purchased from Photronics. Immediately, a second lithography issue was encountered. Reflection of the UV light off of the underlying metallic surface induced interference patterns that manifested in the developed photoresist. The use of anti-reflective coating under the photoresist removed this.
4. Etch: Once the lithography was ready, etch step development began. The timing and plasma conditions for each etch step were determined from device cross sections measured using a SEM. After plasma etching through the top layers of the device as well as partially into the bottom SiO<sub>2</sub> layer, an acid dip was used to remove the remaining sacrificial SiO<sub>2</sub> layer at the bottom of the structure. Unfortunately, this step caused unexpected and destructive chemical reactions. For examples: Hydrofluoric (HF) acid was found to etch the Si<sub>3</sub>N<sub>4</sub> and Al layers nearly as fast as the SiO<sub>2</sub> layer. Further, the use of the HF liquid caused the remaining structure to delaminate. In addition, compressive stress in the thicker Al layer was released once the bottom SiO<sub>2</sub> layer was removed and no longer anchoring the top layers to the substrate. As a result, the remaining layers bowed up. (See Fig. 7.) An etch process using HF vapor was also tried, but unfortunately induced other significant challenges.

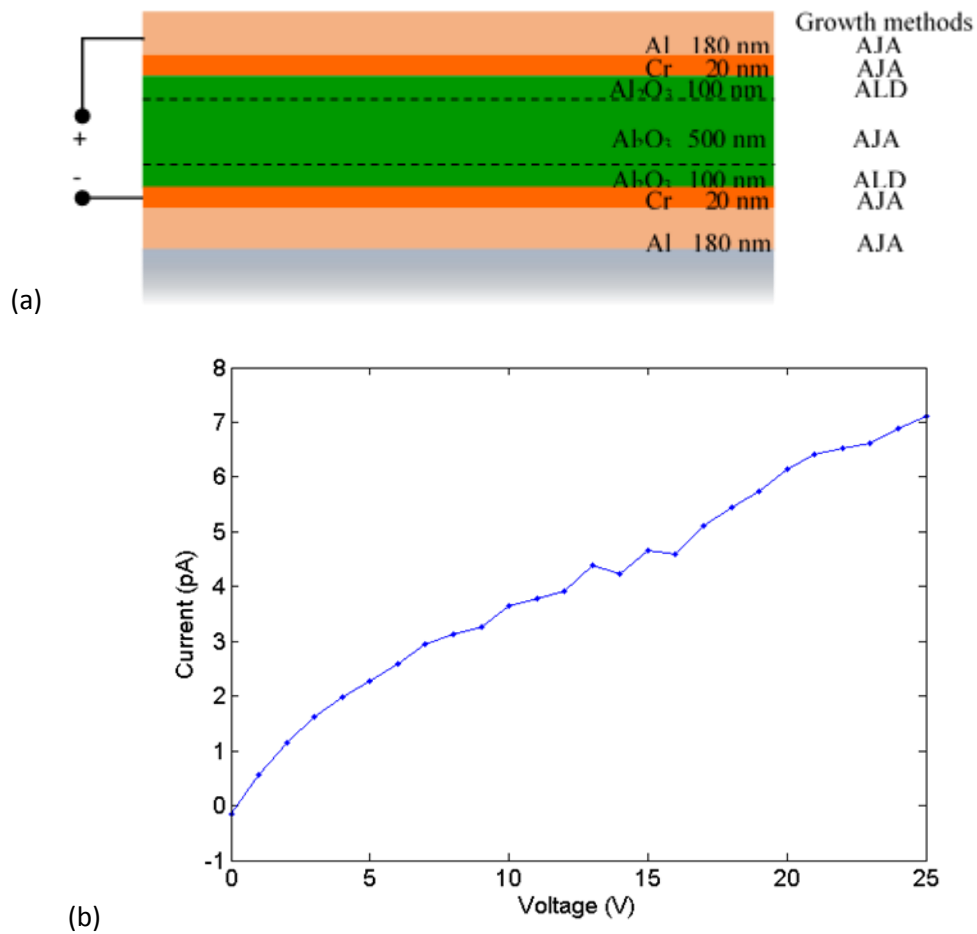


Fig.6. (a) The MIM structure used for testing the ALD and sputtered (AJA) Al<sub>2</sub>O<sub>3</sub> film. (b) The I-V curve obtained from the sample showing the small leakage current level.

5. Electrical Connections (Wire Bonding to Printed Circuit Boards): We also took steps to prepare for device insertion into the mass spectrometer. We designed and machined an aluminum Printed Circuit Board (PCB) holder shown in Fig. 8 (Left – with PCB attached) which places the PCB at the correct location for the measurements. In addition, we designed a PCB which will hold the device of Fig. 5 and allow electrical connections to be formed to it (Fig. 8, Right). This PCB allows electrical connections to be made to the device from the 4 electrical contacts. A wire bonding process window was found for the aluminum contact pads on the device but none could be found for the PCB that the chip sat on. It was discovered that a coating on the PCBs, had no known process window for wire bonding. As a result, new PCBs were built in house having bare-copper contacts. A process window for these boards was found.

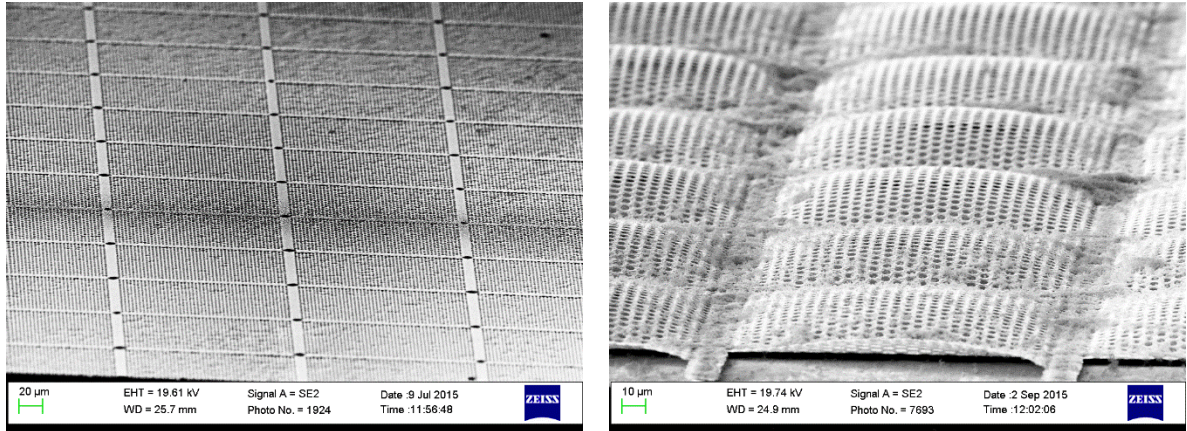


Figure 7: The device of Fig. 5 before (left) and after (right) an HF acid dip and drying process. One can see that the HF acid dip has successfully removed the bottom SiO<sub>2</sub> layer under the hole pattern while retaining it under the ridge lines. Residual stress in the thick Al layer caused the uppermost film layers (both insulating and conducting) to disintegrate and the thick Al layer to bow upwards.

As a result of these challenges, we have not yet been able to fabricate a MEMS device enabling us to investigate the interaction of an electric field with low energy ion bombardment on the IIEE yield.

## 6. Identify products developed:

### Publication summary

2012

1. Conference Presentation: "Active Control of the Secondary Electron Emission Coefficient: A New Plasma Control Paradigm?" D. Urrabazo, C. Liu, L. Overzet (UTD), L. Raja, D. Breden, H. Hariswaran, and P. Paneerchelvam (UT Austin), 65<sup>th</sup> Gaseous Electronics Conference, Austin TX, 10/2012. Bull. Amer. Phys. Soc. 57 (2012) <http://meetings.aps.org/Meeting/GEC12/Session/NW1.73>

2013

1. Conference Presentation: "Secondary electron emission yield dependence on the Fermi level in Silicon" D. Urrabazo, M. Goeckner, L. Overzet (UTD), 66<sup>th</sup> Gaseous Electronics Conference, Princeton, JY, 10/2013. Bull. Amer. Phys. Soc. 58 (2013) <http://meetings.aps.org/Meeting/GEC13/Session/NR2.4>



Figure 8: (Left) The aluminum Printed Circuit Board (PCB) holder mounts into the mass spectrometer and holds the PCB in the correct location. (Right) A top view of the PCB. The device will be adhered to the large pad at the top center of the PCB (forming a backside contact). Wire bonding will be used to make the remaining three electrical connections.

2014

2. Conference Presentation: "Ion Induced Electron Emission from Semiconductors." D. Urrabazo Jr., M. Goeckner and L. Overzet, AVS 61st Int'l Symposium and Exhibition, Baltimore, MD 11/2014.
3. Conference Presentation: "Ion induced electron emission from semiconductors: The effect of conduction band electrons and surface electric fields." D. Urrabazo Jr., M. Goeckner and L. Overzet, 67th Annual Gaseous Electronics Conference, Raleigh, NC, 11/2014. Bull. Amer. Phys. Soc. 59(69) (2014). (<http://meetings.aps.org/link/BAPS.2014.GEC.FT1.3>)
4. Conference Presentation: "Impact of the Fermi level and a surface electric field on ion induced electron emission from semiconductors." D. Urrabazo Jr., L Overzet and M. Goeckner, AVS Texas Chapter Conference 2014, Richardson, TX 8/2014.
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