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Novel Thin Film Field Emission Electron Source Laboratory Directed Research and Development Final Report

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**Novel Thin Film Field Emission Electron Source
Laboratory Directed Research and Development
Final Report**

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Abstract

The objective of this project was to demonstrate proof of concept of a thin film field emission electron source based on electron tunneling between discrete metal islands on an insulating substrate. An electron source of this type should be more easily fabricated permitting the use of a wider range of materials, and be less prone to damage and erratic behavior than the patterned field emitter arrays currently under development for flat panel displays and other vacuum microelectronic applications. This report describes the results of our studies of electron and light emission from such structures, and the subsequent discovery of a source of light emission from conductive paths across thin insulating gaps of our semiconductor - insulator - semiconductor (SIS) and metal - insulator - semiconductor (MIS) structures. The substrates consisted of silicon nitride and silicon dioxide on silicon wafers, Kapton®, quartz, and cut slabs of silica aerogels. The conductive film samples were prepared by chemical vapor deposition (CVD) and sputtering, while the MIS and SIS samples were prepared by CVD followed by cleaving, grinding, mechanical indentation, erosion by a sputter Auger beam, electrical arcing and chemical etching. Electron emission measurements were conducted in high and ultra high vacuum systems at SNL,NM as well as at SNL,CA. Optical emission measurements were made in air under an optical microscope as well as in the above vacuum environments. Sample morphology was investigated using both scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

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I. Introduction

1.1 Motivation - Flat Panel Displays, Vacuum Microelectronics

Electronic displays, and flat panel displays (FPDs) in particular, are an important technology in the modern world. Practically, they represent the most flexible interface between humans and a host of electronic devices ranging from computers, avionics and telecommunication products, to televisions, portable games and other sources of entertainment. Currently, the leading FPD technology is the full color active matrix liquid crystal display ((AM)LCD). Although this technology was invented in the US, Japan made the considerable capital investment needed to develop it into the commercial product it is today, and currently own ~98% of the flat panel display market. This market dominance is an important issue, not only because of the number of high-tech jobs involved, but because the lack of domestic sources for critical military applications. The latter situation could force the military to either change their long-standing policy on limiting foreign procurement of non-critical items, or face the consequences of using obsolete technology.

1.2 Liquid Crystal Displays

Despite its commercial success, the AMLCD is not without its limitations. Liquid crystal devices do not work well in extreme hot or cold environments. An excellent example of this problem exists in some military aircraft where AMLCDs are replacing mechanical or cathode ray tube (CRT) avionic displays. Console temperatures can approach 80C, and extreme measures have to be taken to keep liquid crystal displays cool enough to operate. They also have a narrow viewing angle, which can be a problem in some instances. LCDs are inherently low voltage (1-5 volts), low power (~50 nW/pixel) devices. However, since they act only as light valves by changing their polarization in response to an applied field, they require a backlight in low ambient light conditions. This backlight must operate at full power whether all or only one pixel is activated, and represents a constant significant power drain which limits battery life in portable applications. Full color AMLCDs typically transmit only 5% of this light straining the power reserve even more. Finally, AMLCDs are touted as expensive, primarily (it is thought) due to the high capital investment necessary to implement this complicated technology in larger screens. Although it has been reported that it could be manufactured at a profit for ~\$150, a typical 10 inch diagonal AMLCD currently costs ~\$350. This significant cost disparity may in fact be due to the absence of any other competing technology.

1.3 The Field Emitter Display

A possible emerging competitor to the AMLCD is the field emission display (FED). This is an emissive display (generates its own light) related to the CRT which uses a matrix addressable

array of field emission sources instead of a single scanned electron beam to activate each pixel. A schematic of one type using a Spindt type array (named after its inventor [1]), is shown in Figure 1. Like the CRT, the FED is not especially temperature sensitive, and does not suffer from a narrow viewing angle. Since field emission is a quantum mechanical process [2], it can be highly efficient, requiring no filament or standby- power. The electron emission is a highly field dependent process, requiring no additional transistors or switches (necessary in the AMLCD) to sharpen up the pixel response to increase its contrast. The emission is also immediately available - limited only by the rise time of the field generating voltage. As an emissive display, the power consumed will depend only on the total number of pixels activated, and should not represent a large continuous drain on the available power. Microelectronic fabrication technology allows such close spacing between an individual field emitter and its extraction (gate) electrode that significant electron currents can be drawn with gate voltages of 20-100 volts. The emitted electrons are then post-accelerated to the phosphor screen by maintaining a larger positive voltage on the screen. Finally, an FED should be easier to build than an AMLCD. Thus, the lower capital investment and manufacturing cost should make FEDs competitive with AMLCDs.

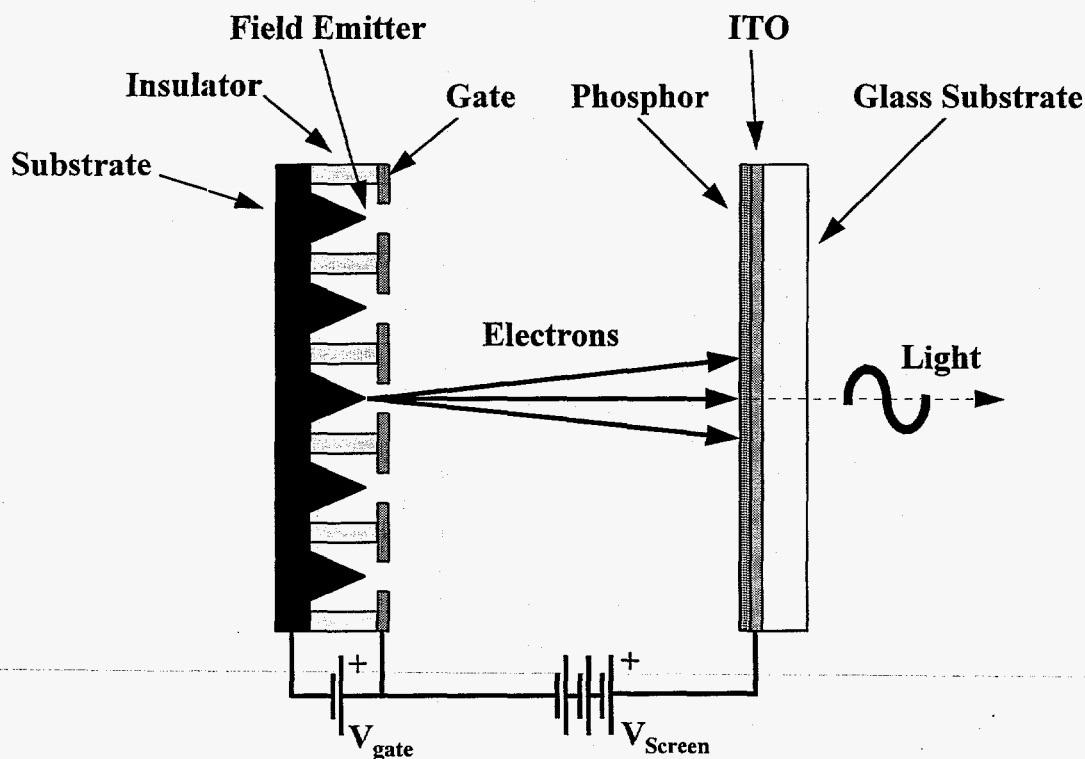


Figure 1. Schematic of a field emission display based on a Spindt design field emitter array.

Despite these advantages, FEDs also have limitations which could be show stoppers. In particular, field emitters are notorious for their sensitivity to the level of the residual gas pressure and surface contamination. Background vacuums of 10^{-9} Torr or less are required to prevent explosive destruction induced by residual gas sputtering with high voltage field emitters [3]. Small changes in the field emitter work function due to adsorption/desorption of even sub-

monolayer levels of contaminants can change the emitted current density by orders of magnitude [2]. In theory, the emitter structures as designed should direct all of the extracted electrons to the phosphor screen. However, tip-to-tip variations in the emitter surface morphology can result in variations in the emission direction. When combined with manufacturing tolerance limits, this can significantly change the electron trajectories, resulting in appreciable gate current and reduced emission efficiency.

In the standard FED configuration, shown in Figure 1, a negative field is applied to the emitter, and the electrons are collected by the anode. Ions generated in the space between the gate and screen have a clear line of sight to, and can directly bombard, the emission sites. For ion energies > 200 eV, this can result in catastrophic destruction of emitters.

1.4 Novel Thin Film Field Emitter

Discontinuous thin films of conductive islands on insulating substrates can exhibit electrical conductivity aside from that due to simple substrate leakage. One of several mechanisms proposed for this process is field emission [4-9]. A field emitter concept which might alleviate some of the above difficulties is based on extracting some of the electrons which tunnel laterally between these conductive islands. This is shown schematically in Figure 2. If this lateral current were high enough, an electron space charge cloud should form along the surface, much as it does along a heated cathode, due to electron scattering and tunneling electrons emerging with their full Fermi energy and their velocity directed tangential to the metal surface. Such films might thus serve as a low voltage (< 100 volts) electron source if a reasonable fraction of this lateral current could be extracted by an electric field normal to its surface produced by a secondary anode.

As the size of the islands decrease to the nanometer regime, quantum effects can separate the bulk band structure into more discrete levels approaching those of individual atoms. Normally, one should expect an increase in the apparent work function as the bands separate and narrow to discrete atomic levels. However, it has been reported that the work function of gold and copper decrease significantly for islands containing < 15 atoms (> 0.1 nm). It appears that this reduction represents a local minimum between the bulk value and the atomic ionization potential for gold and copper, but is probably not a universal effect in all metals (conductors) because of the specific electron orbitals involved [10]. In any case, if this work function reduction does occur, it would significantly increase the laterally emitted current permitting a much lower applied voltage. This would be especially important in (semi-) conductors associated with field emitters and/or micro-electronics since higher currents at lower voltages are a highly desired feature.

Although ionized residual gas atoms could still bombard the film, they would not be ion-optically aimed at the emission sites as they tend to be in the standard configuration. This increased immunity could be further improved by considering a configuration shown in Figure 3. Here, laterally flowing electrons which overshoot the positive edge of the film could be extracted, and the substrate could be tilted at a slight negative angle to further hide the emitting edges of the

islands from bombarding ions. While changes in work function may or may not be affected (depending upon the chemical nature of the emitter and residual gas), sputter damage would be avoided and reliable, albeit higher work function emitters, could be designed. The two-sided version of 3 could easily be extrapolated to a cylinder or cone with the base of the cone to the left of the figure.

Finally, it would be useful if the emission efficiency of this emitter were at least comparable to that of standard field emitter arrays, ~50%. However, since most of the power is expected to be in the high voltage / phosphor screen side of the display (estimated at ~3W), the total emitter section power could easily be as much as 0.3 W or 10% of the high voltage section. Assuming the screen operated at 5 kV, the total screen current would be 600 microamps. If it required 100 volts to achieve the required lateral current of 3 milliamps, the extraction efficiency could be as low as 20% and still be considered a viable option. This would especially be true if the reliability or lifetime could be improved over standard field emitters, or if the operating background residual gas pressure in the display could be relaxed by several orders of magnitude from the stringent 10^{-9} Torr currently required.

There are numerous publications in the open literature, both theoretical and experimental in nature, regarding electron conductivity through such "island" films under the generic title of "Discontinuous Films" described in the references. Some of these papers discuss the possibility of such films serving as electron sources. However, in virtually all of these previous works, ill defined and often irreproducible processes involving the application of "forming" voltages and conditioning procedures are used. These procedures typically result in stepwise and irrevocable changes in the emitter voltage / current profiles, possibly caused by electromigration or micro-arcs at insulator interfaces. In our study, we purposely chose to avoid those conditions given the level of irreproducibility and variability of those sources. In addition, we limited our applied voltage to less than 100 volts, since this is probably the practical limit for solid state drivers being considered for the more traditional field emitter display technologies. If this technology is to compete in this arena, it will have to be stable, reproducible, and operate in this voltage range.

A unique aspect about SNL's capability in this area is the selective tungsten deposition process. In this process, WF_6 can be made to react with silicon resulting in volatile SiF_4 and a deposited tungsten film. The morphology of the tungsten film can be controlled via temperature, silicon thickness, WF_6 flow rate and time yielding films ranging from dense continuous coatings to sparse tungsten islands. In particular, the spacing between non-contacting islands can be accurately selected since the volume of silicon removed by the chemical reaction is greater than the volume of tungsten deposited. Since the process does not distinguish between single crystal silicon and polysilicon films, tungsten can be deposited on a variety of insulating films of arbitrary thickness as long as the insulator is compatible with the deposition of polysilicon. This property yields a wide range of material combination possibilities which can be easily fabricated.

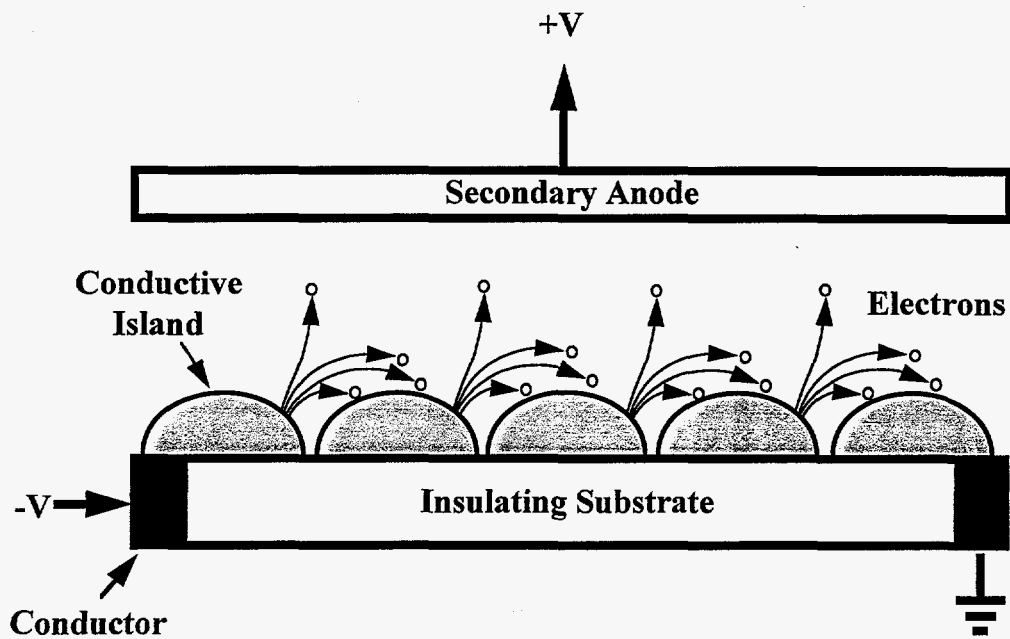


Figure 2. Lateral field electron emission resulting in transverse current.

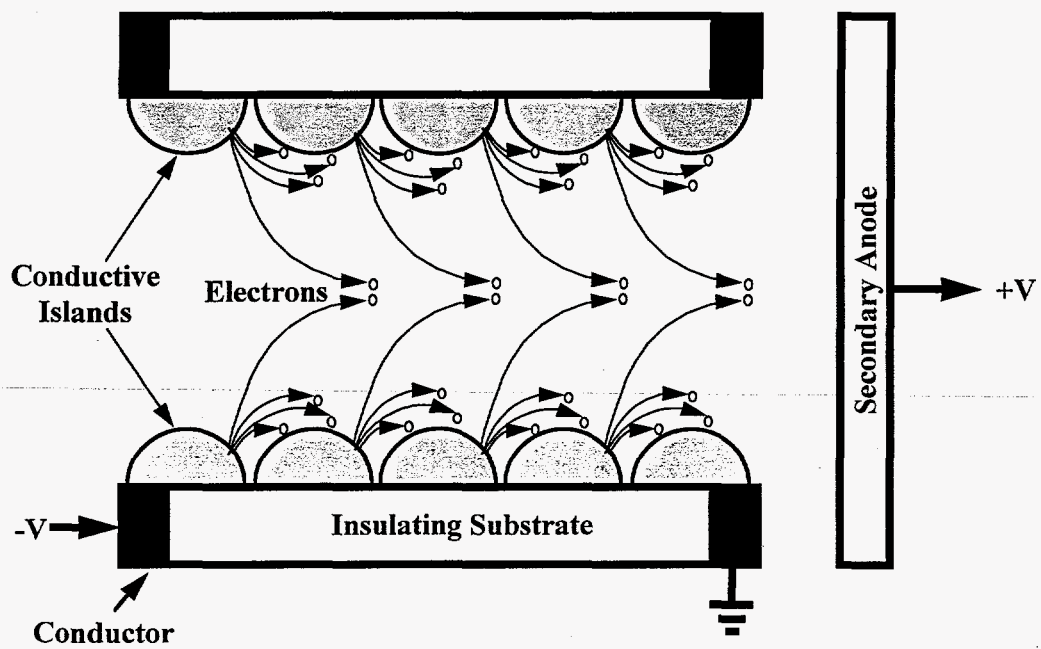


Figure 3. Two sided lateral thin film field emitter resulting in end current.

II. Experimental Results

II.1 Sample Preparation

A number of different samples were prepared to provide comparisons between different material combinations. These are listed below.

- 1.) Samples were prepared by organization 1323 consisting of sequential layers of tungsten deposited on the sacrificial n-type poly silicon (0.15 microns); an insulating layer of silicon nitride (0.12 microns); an n-type silicon substrate (650 microns) using the selective WF_6 CVD process. Variations on this sample structure were also prepared with variations in material and thickness consisting of: polysilicon | silicon nitride | silicon substrate; aluminum | silicon dioxide | silicon substrate; and titanium nitride | silicon nitride | silicon substrate either by CVD or sputter / evaporation after light emission was discovered on the original samples (discussed below).
- 2.) Evaporated platinum on Kapton ® (Kapton ® is a registered trademark of the I. E. Dupont de Nemours Company) was also prepared by organization 1323. The platinum aerial density was varied by physically grading the deposition to allow for a range of island sizes and spacings on a single continuous substrate.
- 3.) Thin gold films were sputter deposited on quartz by organization 1471 using a moving shutter to control the amount of deposition. These films were masked to form gaps of 125 and 400 microns to test and compare their emission properties.
- 4.) Gold was also sputter deposited on silica aerogel by organizations. 1492 and 1471. These were similar to the gold on quartz samples above, but because of the porous nature of the silica aerogels, they also had the potential for achieving a 3-D distribution of particles extending into the aerogel volume.
- 5.) Free standing diamond/diamond-like-carbon films were prepared by organization 1411. Properly prepared diamond and diamond-like carbon films are known to exhibit good field emission properties. Given a report of electron emission from discontinuous carbon films [5], we felt that if the resistivity of the film was in the right range, a lateral current might result in significant electron emission.

II.2 Experimental Apparatus

Electron emission measurements were performed using a sample holder shown schematically in Figure 4. Isolated electrical contacts were made to the top and bottom of the samples which were mounted below an electrically isolated collector electrode. The holder could be used in air or in

vacuum. Voltage could either be applied across the top of the sample through contacts A and B if the film were on an insulating substrate, or through the sample through contacts A+B and C if the substrate was conducting. Figure 5 shows a typical setup using the HP4140B Picoammeter/Voltage Sources.

Photos of some samples were taken using a Leitz microscope fitted with a Polaroid camera. Other analyses were performed using SEM, TEM, Sputter Auger, hardness testing and chemical processing capabilities within organization 1800, the field ion / field emission equipment in organization 1114, and the Emission Characterization Facility (ECF) at SNL/CA in organization 8715.

II.3 Electron / Light Emission Measurements

1. CVD Tungsten | Poly-Silicon/Silicon Nitride | Silicon Substrate Samples

Two 6 inch silicon sample wafers having two different tungsten film thickness were fabricated for test. The stacked layers consisted of : the tungsten CVD film | 120 nm of polysilicon | 150 nm layer of silicon nitride | 650 micron n-silicon substrate. The sample with the thicker tungsten had a surface conductivity of 40-60 ohms per square, whereas that of the other sample measured infinity using a standard thin film conductivity probe. However, both samples were found to be very conductive when the current flowing across the tungsten films was measured using 100 volts between isolated probes spaced from 1 to 50 mm apart. Since our estimates indicated we should not expect any conductivity until the probe spacing was reduced to 125 microns or less, we suspected the conductivity was probably due to leakage either laterally through the insulator, or through pinholes through the insulator to the substrate. Subsequent measurements confirmed a significant leakage path from the tungsten film through the insulator to the silicon substrate on both the as-supplied wafer and on 1 square cm cleaved samples. Apparently, the samples were behaving like MIS Schottky diodes, but with only weak rectification properties dependent on the polarity of the applied voltage.

Because of this leakage path, we were not able to measure electron emission from these samples with current flowing laterally through the tungsten film. However, weak electron emission was observed from cleaved tungsten | polysilicon | silicon nitride | silicon wafer samples when voltage was applied across the thickness of the wafer. Emission of this type has been seen in similar metal - insulator - metal sandwich structures reported in the literature [4, 10-16]. Measured emission current is shown for two samples in Figures 7 and 8. Only one value of the emission current for an applied voltage of 80 volts and collector voltage of 10 volts is shown in Figure 7. When the collector voltage was raised to 20 volts, the sample stopped emitting, and could not be revived.

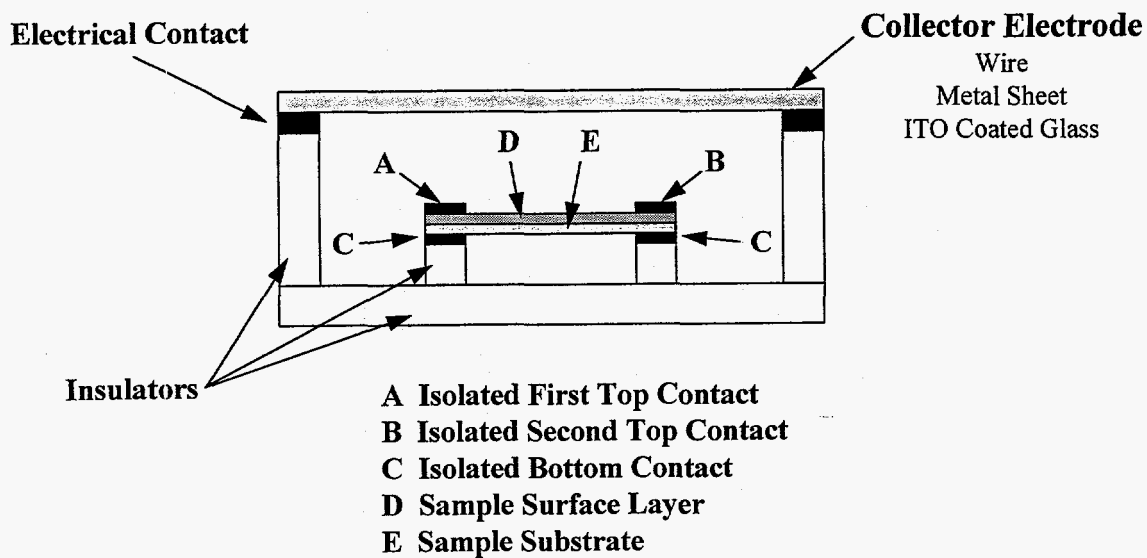


Figure 4. Schematic of sample holder.

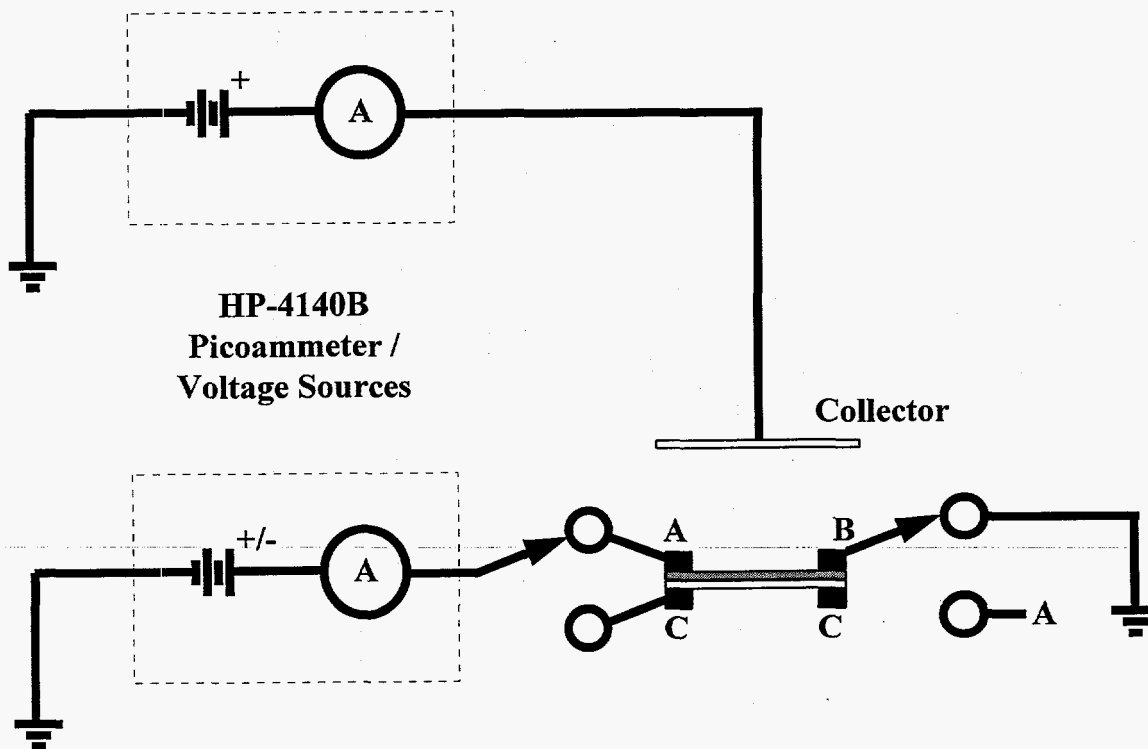


Figure 5. Electrical current measurement setup.

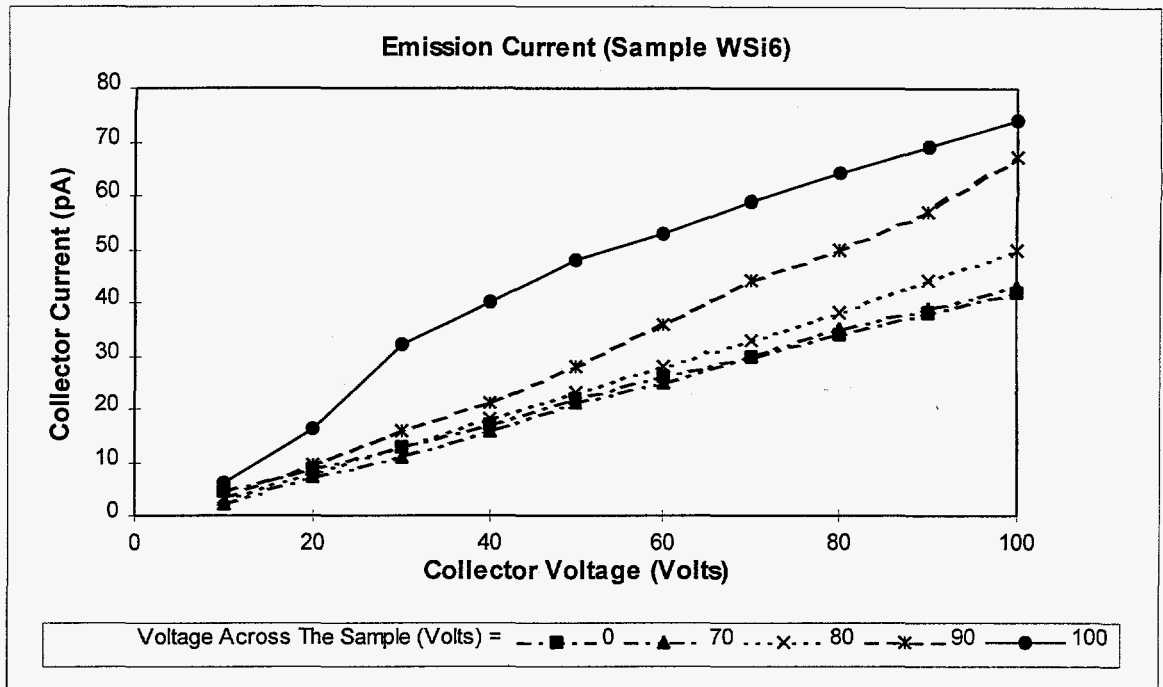


Figure 6. Emission current as a function of collector voltage and voltage applied across the sample.

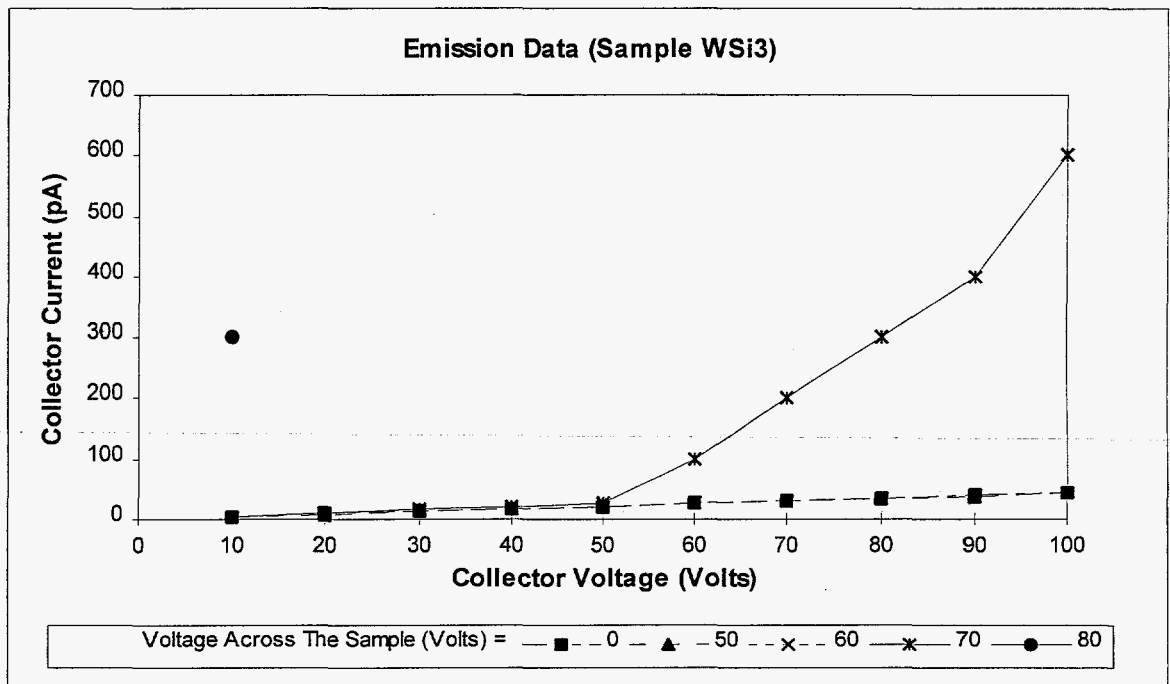


Figure 7. Emission current as a function of collector voltage and voltage applied across a different sample from that of 6.

This emission was later confirmed by Felter (organization 8715) with similar samples. Felter's measurements also showed the electron emission originated from spots along the cleaved edges, and although an emission current measurement was not made, it was high enough to excite a phosphor screen at +1000 volts to visible luminescence.

During the course of investigating the source of the leakage current, we discovered light emission from the edges of some cleaved samples in air. Examination under an optical microscope revealed the light originated from intense discrete sites whose size was below the resolution limit of the microscope. The light sources appeared mainly red-white in color although isolated blue, green, orange and red spots could occasionally be seen. We attempted to obtain an emission spectrum from these sources, but were unsuccessful.

The presence of these discrete lights on the sample edges, and the observation that the electron emission also appeared to originate from discrete spots on the edges, led us to speculate whether the source of the lights might also be the source of the electrons that we and Felter had observed. We therefore decided to further investigate the lights with the intent of understanding their source, determining if they did emit electrons, and finding ways to easily, but reproducibly, increase their number and density.

We found that the lights could easily be obtained along cleaved edges, the edges of holes chipped out of the wafer with a sharp stylus or drilled using diamond or boron carbide bits, and along cracks formed by hardness indentation styli. The number of lights could also easily be increased by mechanical abrasion using diamond dust or by filing the edges with an alumina rod. They could also be produced whether water, oil, or no lubricant was used. However, the use of a lubricant, especially water, appeared to increase their number on some samples. Samples obtained from near the center of the wafer produced more lights than those near the edge of the wafer. This correlated with an observation that the insulating film was slightly thicker near the center of the wafer.

A silicon wafer was also prepared having the same dimensional structure as those above, but which did not have the tungsten deposited on top of the polysilicon film. Samples from this wafer also showed an abundance of lights when cleaved and drilled, implying that the tungsten was not responsible for the lights.

Although the lights appeared as very intense point sources when viewed through the microscope (100-400 X), it was difficult to photograph them using Polaroid film. Exposure times of up to 20 minutes were required, probably due to reciprocity failure of the film. An example, shown in Figure 8, was obtained by drilling a 750 micron hole into the wafer using a diamond drill bit. Both an overall view of the hole and lights, and a magnified section are shown.

Although some lights appeared to flicker, most did not and were visible for hours to days in air and vacuum. However, a slow degradation in intensity was observed after repeated use in air. We measured the light output as a function of time for one sample using a photocell inserted into the eyepiece of the optical microscope. The results are shown in Figure 9.

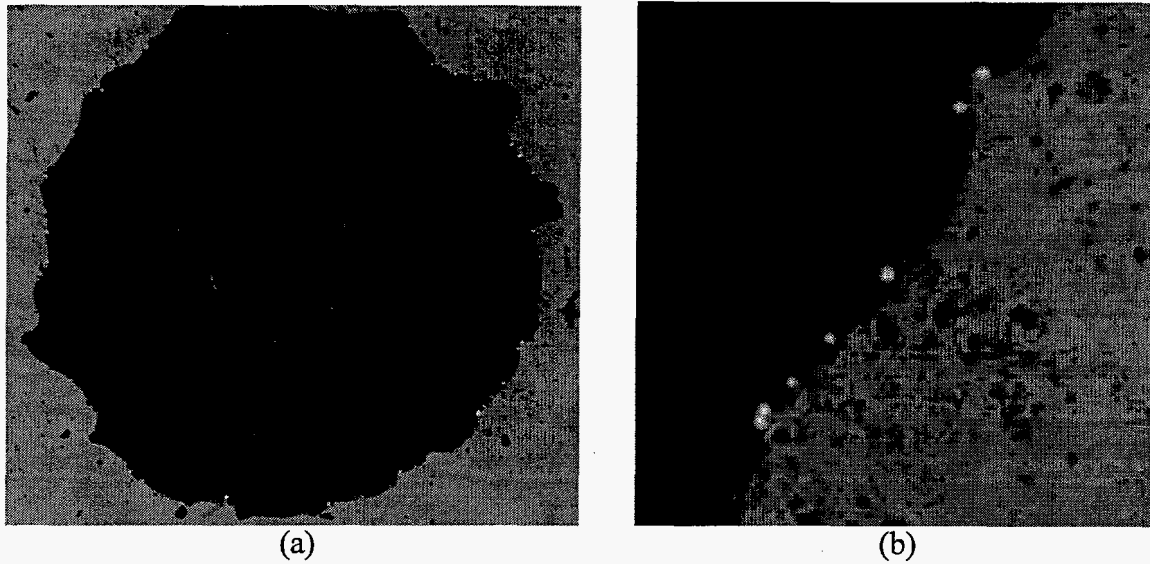


Figure 8. Optical micrographs of lights around the edge of a hole drilled into a sample. (a) Overall view. The hole diameter is ~750 microns. (b) Magnified view of the lights in the lower right hand corner.

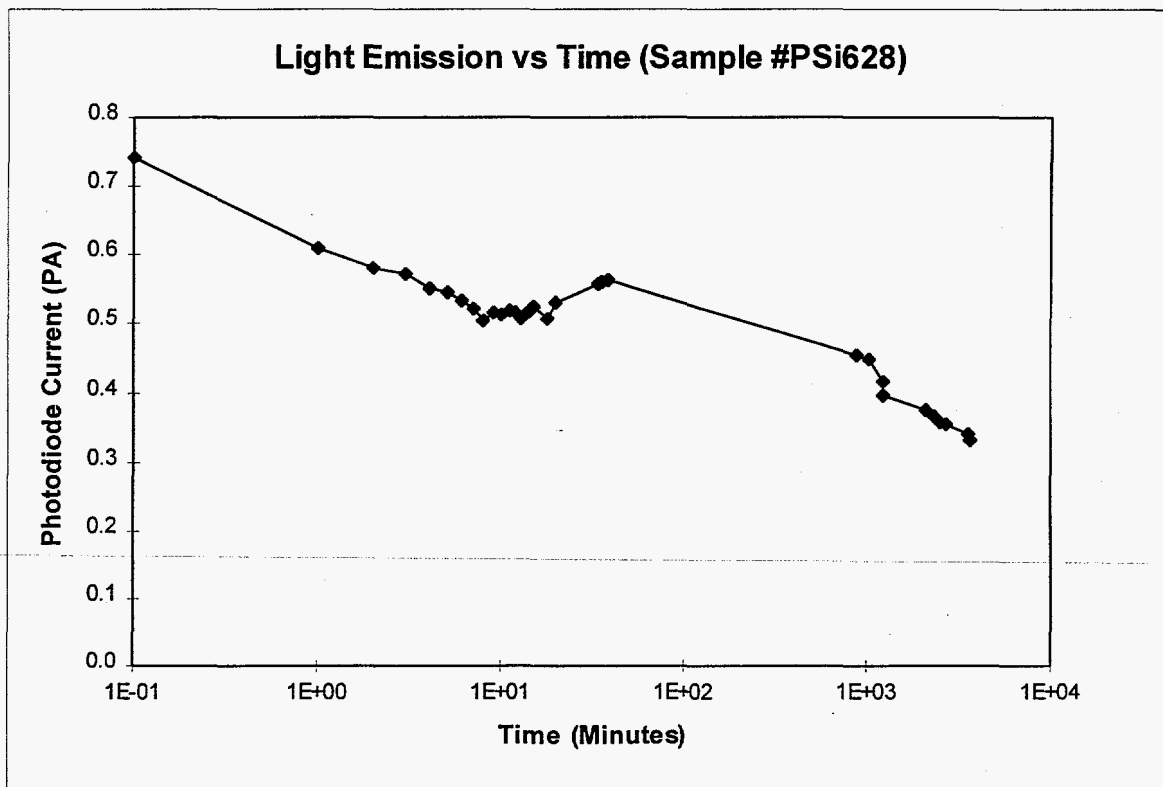


Figure 9. Light emission as a function of time for a polysilicon | silicon nitride | silicon substrate sample as a function of time.

We also observed that the number and intensity of the lights, and sometimes the appearance of lights at different locations, depended on the polarity of the applied voltage on any specific sample. With most of the tungsten | polysilicon | silicon nitride | silicon substrate and polysilicon | silicon nitride | silicon substrate samples, more lights were visible with the top film positive relative to the substrate. However, this was not true in every case. Since the HP-4140B power supplies could only source a maximum of 10 mA, the magnitude of the leakage current determined the maximum voltage drop across the sample.

Figure 10 shows a plot of the current through one sample and the corresponding light intensity as a function of applied voltage using the photocell / microscope arrangement discussed above. Light emission was visible with as little as 8 volts applied to this and other samples.

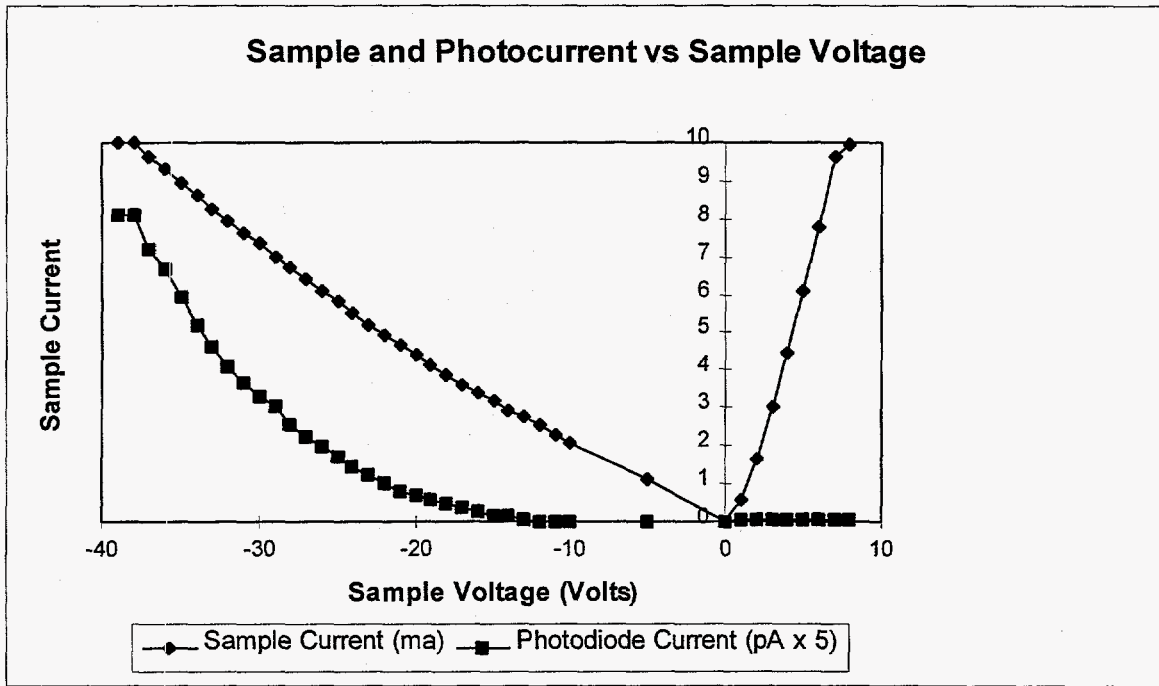


Figure 10. Light emission and current through the samples as a function of voltage polarity across the sample for a polysilicon | silicon nitride | silicon substrate sample.

Several tests were performed to determine the nature of the lights. The lights could easily be moved (or removed) mechanically with a cotton swab or small artist brush. This implied they might be sub-micron particles bridging the silicon nitride insulating gap between the top conducting layer and the silicon substrate. Surprisingly, however, they were visible even while immersed under water, oil, and melted wax. Most even remained after treatment in an ultrasonic cleaner with water and LIQUI-NOX[®] (LIQUI-NOX[®] is a registered trademark of Alconox, Inc.) cleanser.

To try to determine the nature of the light sources, a sample was prepared with many lights on the edge of a drilled hole. This sample was subsequently photographed through the microscope.

with the lights activated so it could be used as map in later SEM microscopy. The objective was to try to correlate specific surface features with the emission sites. These SEM micrographs revealed many agglomerations of sub-micron particles bridging the insulating gap. Not every agglomeration had a corresponding emission site, but there were no emission sites without at least one particle bridging the insulator gap. An example of one such mapping involving a polysilicon | silicon nitride | silicon substrate sample is shown in the SEM micrographs of Figure 11 below. Note the presence of particles bridging the insulator gap which appear to be associated with the lights.

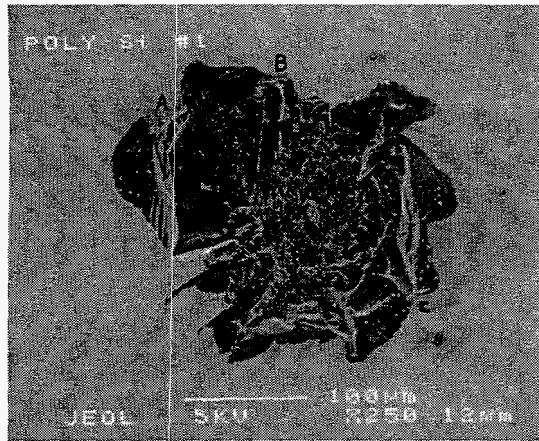
Another sample was prepared with many lights both on the edges of a drilled hole and the sample edges. The hole was chemically etched using several drops of 1M KOH at 90° C for several minutes. Afterward, it was found that the lights on the edges of the hole had disappeared, but those on the perimeter of the sample remained. Subsequent SEM micrographs of the hole edge showed virtually no particles present. A representative SEM micrograph is shown in Figure 12. Other samples also showed the presence of light emission sites.

Cathodoluminescence (CL) scans were also performed using the SEM and it was found that many of the particles at the emission sites exhibited CL. Examples are shown in Figures 12 and 13. An attempt was made to identify the nature of the particles at the emission sites using energy dispersive x-ray analysis, but the particles were too small to isolate their signature from the background material they were on.

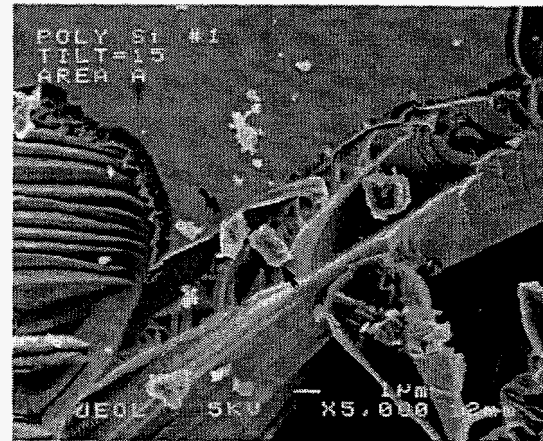
Another sample was prepared using sputter Auger to erode through the top and insulating layer to the substrate on the assumption that this process would not produce particles, and if light emission occurred, it might be evidence that the emission also had a non-particle origin. Subsequent testing revealed the original lights along the sample's perimeter remained, but none were produced anywhere near the sputter eroded areas. It should be noted, however, that the erosion produced a large extended region of exposed insulator in contrast to the short distance between the top layer and substrate produced on the cleaved edges or on those of the fractured or drilled holes. While the test produced no lights, it may simply have been due to the lower electric field in the region of the erosion.

We (personnel in organization 1231) then attempted to measure electron emission on samples prepared with many lights on the supposition discussed above that the light sources might also be electron sources. However, while the lights could clearly be seen in the vacuum system, no electron emission was detected. Kellogg (organization 1114) installed two samples with lights in his field ion microscope which was equipped with a microchannel plate (MCP) electron multiplier.

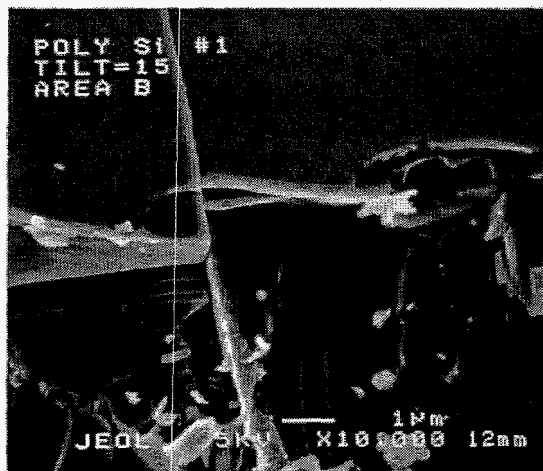
The MCP responded when one sample was activated, implying the emission of electrons, but no primary emission current was measured. The MCP response occurred for an applied voltage as low as 20 volts. Similar samples were sent to organization 8715 at SNL/CA, but while they saw and recorded the lights, they also did not detect electron emission. An example of the lights recorded by organization 8715 is shown in Figure 15.



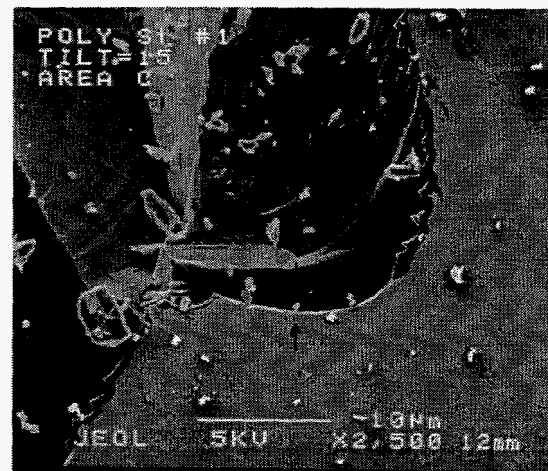
(a)



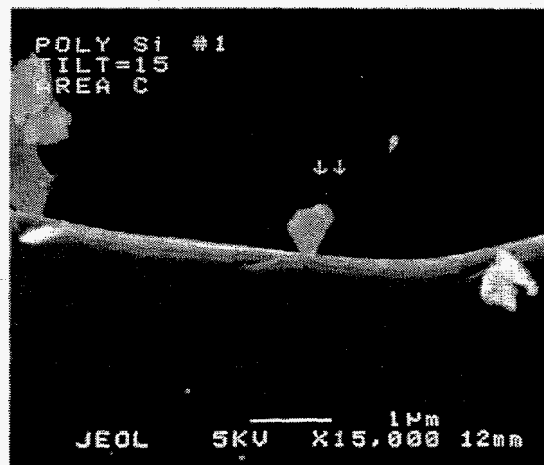
(b)



(c)



(d)



(e)

Figure 11. SEM micrographs of a polysilicon | silicon nitride | silicon substrate sample chipped out with a scribe, and exhibiting lights in regions A, B and C. (a) is the overall view; (b) is a close-up of region A; (c) is a close-up of region B; and (d) and (e) are close-ups of region C.

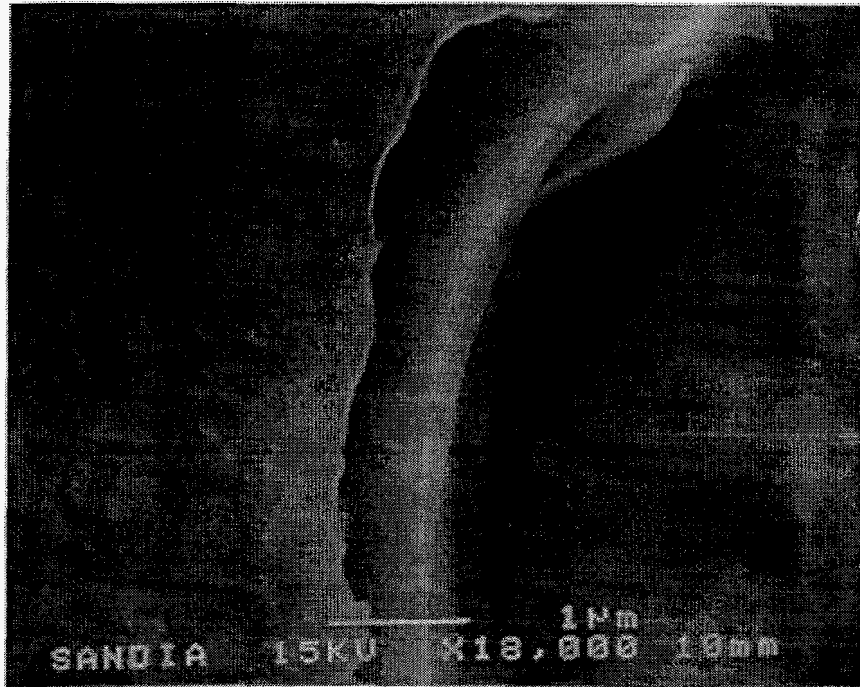
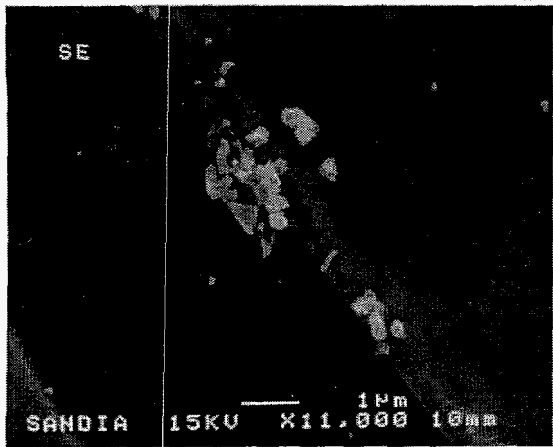


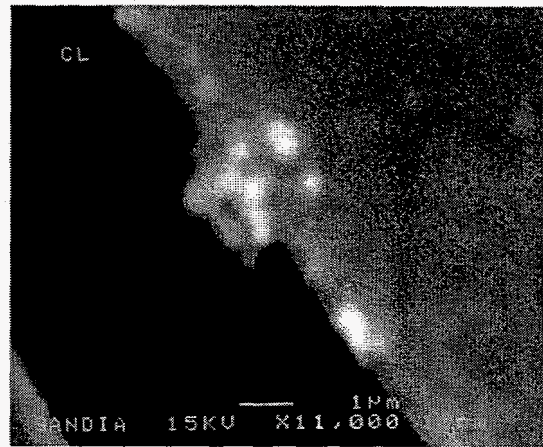
Figure 12. A polysilicon | silicon nitride | silicon substrate sample in which the edge of a hole with many lights was etched in 1M KOH at 90 C. This view is representative of those scanned around the rest of the hole. Virtually no particles were seen bridging the insulator gap, and the original lights had disappeared after the etch.

Similar lights were produced with the other material combinations listed in section 1.3. We found identical behavior with the polysilicon | silicon nitride | silicon substrate samples. The alumina | silicon dioxide | silicon substrate samples yielded slightly brighter and whiter lights, whereas the titanium nitride | silicon nitride | silicon substrate samples yielded only a few deep red to orange colored lights. Unfortunately we were unsuccessful in obtaining optical emission spectra from any of the light sources.

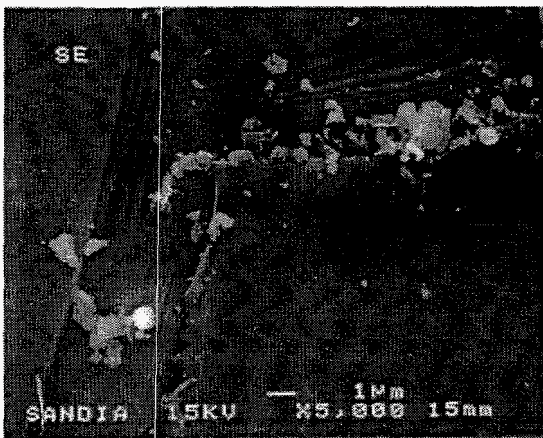
A subsequent literature search and discussions with individuals in organization 1300 revealed that both light and electron emission from silicon derived materials and microelectronic devices was not a totally new observation. Photo-, electro- and cathodoluminescence has been observed in porous silicon [17-24]. Light emission has also been seen in silicon/silicon dioxide nanostructures [25-28], and electroluminescence in silicon/silicon oxides or nitrides has been seen and even used by the microelectronic industry as an indicator of defects [29-38]. In addition, electron emission has even been observed for silicon MIS devices [39]. What did appear to be novel was that this may have been the first definitive study directly linking at least the light emission to cathodoluminescent particulates in high field regions, where the particles bridge an insulator between two conductive layers.



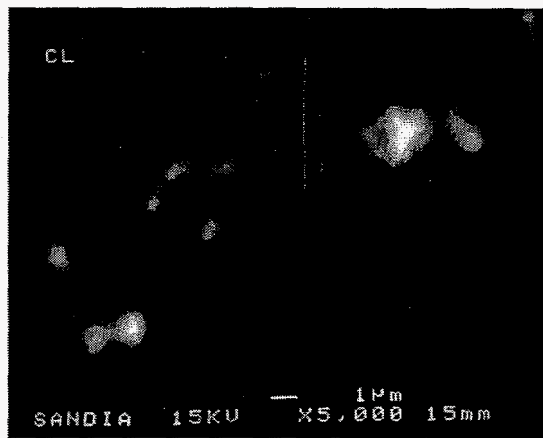
(a)



(b)



(c)

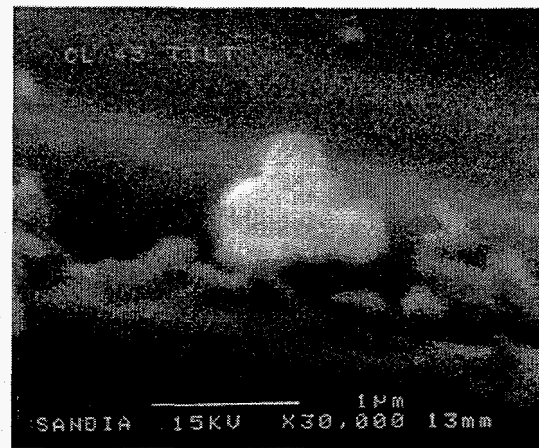


(d)

Figure 13. Examples of cathodoluminescence from some of the particles seen at the edge of holes and fractures with the SEM. Figs. a and c are secondary electron images, and Figs. b and d are the corresponding cathodoluminescent images.



(a)



(b)



(c)

Figure 14. Another example of a particle exhibiting cathodoluminescence. (a) The secondary electron image of the particle. (b) The corresponding cathodoluminescence image of the same particle. Also shown is the particle from two sides ~180 degrees apart - compare (a) and (c). In Fig. (c) it appears that the particle has "dripped" over the edge of the insulator gap, and was making contact with both the top layer and the substrate. Also note the melted appearance of the upper spherical "lobes". This might be an indication of high temperature as this particle was observed in an area which had an exceptionally bright light in the optical microscope.



Figure 15. Image taken from video tape of the test of a sample sent to SNL,CA for electron emission measurements at the ECF. Lights are clearly recorded with a CCD camera along the periphery of the sample as well as along the edges of three holes drilled through the top and insulator layers and part way into the substrate. However, no electron emission was detected.

2. Platinum on Kapton ® Samples

Platinum on Kapton ® samples were mounted 75 microns below the collector using the apparatus discussed above. Currents through the platinum film in air ranged from 2 - 6 nA for applied voltages of up to 100 volts. However, this current proved to be due to surface leakage. When the sample was placed in the vacuum system, the leakage current fell to the pA regime, and no electron or light emission was observed. The leakage was probably due to adsorbed water from the atmosphere, a well known property of polyimide material like Kapton ®.

3. Gold on Quartz Samples

Organization 1471 prepared samples of thin gold films which were sputtered onto quartz substrates through a rotating aperture/shutter. The shutter was needed to allow the sputter system to start up and stabilize before deposition to avoid possible contamination, and the rotating aperture limited the amount of deposition per pass over the substrate to obtain the dispersed island structure we wanted. TEM carbon grids were co-sputtered with the quartz substrates to

verify that we obtained the required deposition rate to produce island structures. TEM micrographs of the carbon grids are shown in Figure 16.

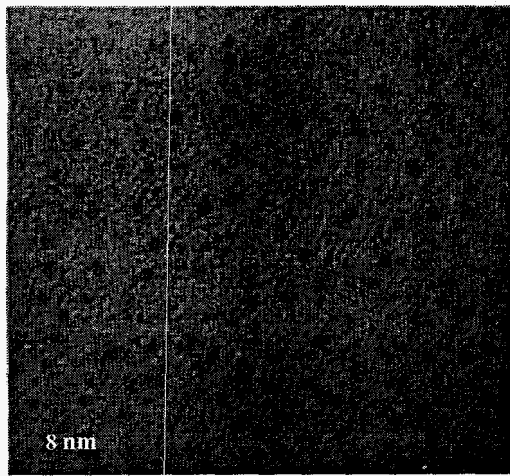
After the island deposition was completed, 125 and 500 micron diameter wires were mounted directly over the samples to serve as sputter masks in order to form narrow gaps. Thicker gold was then sputtered over the samples to form electrical contacts on either side of the gaps containing the island structures. These samples were then tested in the emission apparatus described above.

For the 1 pass through 50 pass samples, no electron or light emission was observed. The current through both gap sizes measured nearly a constant 10 pA (leakage levels) for applied voltages up to the maximum of 100 volts. In contrast, the samples with 100 passes were nearly a direct short. It appears that obtaining the optimum island structure (size and spacing) is a very sensitive function of the deposition process parameters.

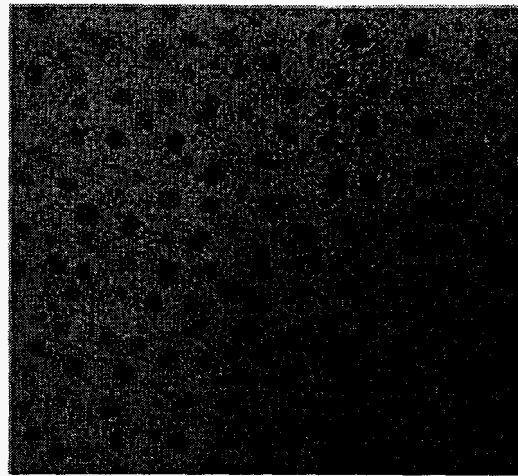
4. Gold/Aerogel Samples

Gold was also sputter deposited onto silica aerogel samples by organization 1471. The samples were 10mm wide, 2 mm thick and 50 mm long and were made using the same technique developed for the deposition of gold onto silica substrates described above. The 250 micron gaps were prepared using a 250 micron wire shadow mask placed on top of the samples after the thin gold depositions were complete. Samples for 20 and 100 deposition passes, as described above, were prepared.

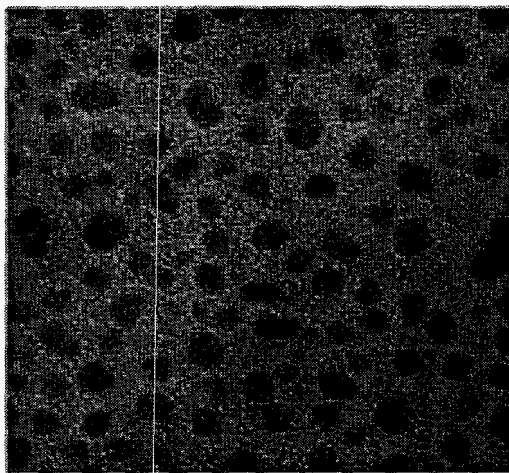
For an applied voltage of 100 volts, leakage currents of <1 pA in air were detected flowing through the gap for the 20 pass samples. Under comparable conditions, the current in the 100 pass samples ranged from <1 pA to 1.2μ A. One sample was tested under vacuum. At 1 atmosphere in air the leakage current through the sample gap was 23 nA @ 100 volts. However, during pump-down, that leakage current fell to 1 nA when the sample was immersed in pure nitrogen gas, 20 pA at 100 mTorr, and 1 pA at 5×10^{-6} Torr. The leakage values nearly repeated themselves when the pressure increased as the vacuum chamber was backfilled with dry nitrogen gas, and after the sample was removed from the vacuum chamber. This indicates that the leakage current was most likely caused by atmospheric gas absorption, probably water. Because the vacuum resistance was so high, no electron emission measurements were attempted with this or any other aerogel sample. It should be noted that the thick gold layer exhibited the typical gold color on the surface that was sputtered onto, but appeared black on the underside (through the aerogel). This was taken as evidence that the gold penetrated into the aerogel forming a highly light absorbing layer, possibly as a (nano)particulate dispersion within the aerogel pore structure.



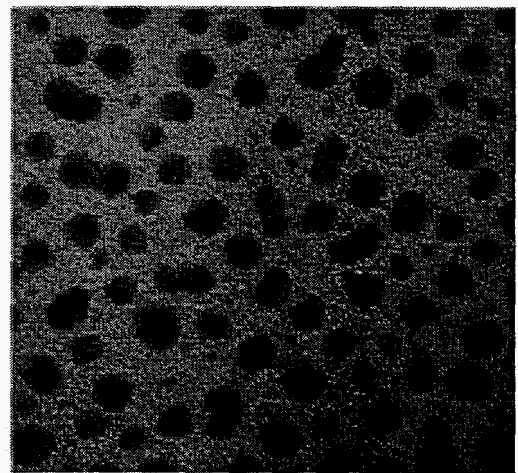
(a) 1 Pass



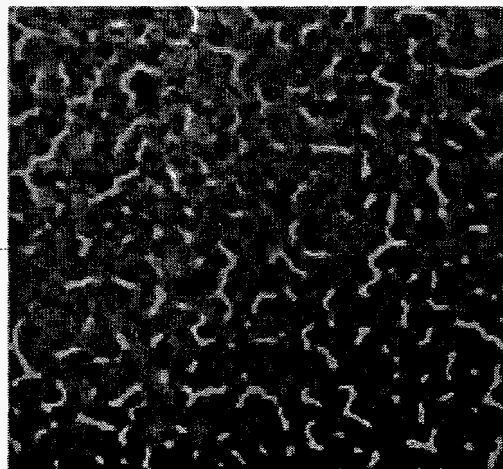
(b) 4 Passes



(c) 10 Passes



(d) 50 Passes



(e) 100 Passes

Figure 16. Gold sputtered onto carbon TEM grids. The scale on (a) applies to all micrographs in this figure.

5. Diamond-Like Carbon Films

Several free standing diamond like carbon films were obtained from organization 1411. Since properly prepared diamond and diamond-like films are known to be good field emitter materials, it was felt that they might be even better candidates as lateral emitters similar to that of plain carbon films reported in the literature [5]. We chose to try free standing films to avoid the substrate shunt problems we encountered with the tungsten films on silicon described above. Unfortunately, the lateral conductivity of the films was too high to be useful, and we did not attempt to extract electrons from them.

III. General Discussion

We did not achieve our goal of electron emission from a thin discontinuous conductive film on an insulating substrate. In the case of the tungsten films, there was significant leakage through the insulating silicon nitride layer to the conductive silicon substrate below. This limited the actual voltage which could be applied across the surface, and hence the field which could be developed between individual islands. For the cases of gold on silica aerogel substrates and platinum on Kapton® substrates, the surface leakage of the substrates in air made determining the desired film resistivity for electron emission in vacuum impossible in the limited time available for this project. In the case of the available diamond/diamond-like carbon, the conductivity of the films were simply much too high.

The gold films deposited on quartz had the greatest chance of success based on the fact that the conductivity was very low, and TEM micrographs indicated the presence of the desired island structure, at least on carbon TEM grid witness substrates shown in Figure 15 above. However, this approach also failed, probably for the following reasons. First, although the island size and intra-island spacing was ~ 8 nm for the samples made with 50 shutter passes (Figure 15 (d)), this was an order of magnitude smaller than for samples reported in the literature [9], in which emission was observed at 50 volts for a gap of 30 microns. In our case, the thinnest gap width was 125 microns. Since to first order, the fields should scale with gap width, the minimum voltage needed for our sample should have been ~250 volts instead of the 100 volts we used. Unfortunately, in going from 50 passes to 100 passes, the film morphology changed radically, resulting in the nearly continuous film seen in Figure 15 (e). Also, unfortunately, no samples were fabricated with a number of passes between 50 and 100.

Second, there was no guarantee that the island structure seen on the carbon grids would be replicated on the quartz substrate. In hindsight, we might have been able to verify this with direct high resolution SEM images of the gold films. The density of the gold should have been high enough to distinguish it in the SEM from the quartz substrate. Alternatively, we might have been able to sputter (or CVD deposit) thin silica films on the carbon TEM grids to see if the gold islands formed on them as well as they did on the carbon grids. Of course, this assumes the silica could be deposited uniformly and thin enough to do TEM, and that the silica film surface would allow the gold morphology to be the same as that on the quartz substrate.

Although we did not achieve our goal of a lateral emitting electron source, these efforts resulted in the discovery of visible light emitting sites at cleaved or fractured edges of the silicon samples. The source of the lights was correlated to the presence of sub-micron particles bridging the insulating gap at these sites. It is believed the light is due to electroluminescent excitation of some of those particles. Although the color of most emission sites appeared red-white to the eye, we occasionally observed red, green and blue emitters. These emitters may have resulted from silicon - silicon oxide (or nitride) nano-particle dispersions within the more macroscopic particles similar to that described in the literature [25-28]. The red-white emitters may perhaps be even more interesting if this emission is comparable to some of those reported in the literature. In one of several similar reports [31], the emission spectrum was highly peaked at a wavelength of ~1.1 microns. Given that the visible portion of these emitters appeared very intense, this would mean that the near IR emission would have been considerably more so. And, since the photo-sensitivity of silicon detectors also peaks at ~1 micron, this effect might be useful in optoelectronic applications if the emitter is efficient enough.

Unfortunately, the lights did not unequivocally prove to be sources of electrons. Although electrons appeared to be emitted in Kellogg's system, it must be noted that his channel plate electron multipliers would have amplified even a minuscule current by at least 10^8 . Alternatively, the channel plates may have been responding as photodetectors to the light generated by the particles, especially if the emission spectrum extended into the near ultraviolet.

IV. Summary

Our objective was to obtain efficient electron emission by passing a lateral current through a discontinuous conductive film on an insulating substrate thereby. Although we were not successful in this attempt, we did detect electron emission on several occasions with the voltage applied between the top film and the conductive substrate. Due to time constraints, we were not able to test as many samples as we wished to either better correlate the light emitters with electron emitters, or identify the electron emission sites seen earlier. The light sources we discovered may warrant further investigation for potential optoelectronic applications, given the variety colors seen. These observations may be the first to correlate silicon based particulates with light emitters which can be easily generated. Suggestions for future work include: 1) determining the chemical nature of the light emitting particles; and 2) identification of the true sources of the electrons seen in the early trials, perhaps by scanning with a scanning tunneling microscope. If the electron emitters are in fact a separate entity, but could be fabricated as easily as the light sources, it could have a major impact on vacuum microelectronic technology.

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