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Fabrication of Nanometer Features on Conductive Surfaces Using Electrostatic Spark Discharges

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Abstract

This study demonstrates the feasibility of using a localized plasma discharge from an atomically sharp tip to produce nanometer-scale craters, holes, and mounds on conducting surfaces. It further discusses the potential application of such an approach for nanolithography, nanoimprinting, production of masks for nanostenciling, and the fabrication of membranes.

Key Words: Nano-features; Electrostatic; Discharge; Inchworm

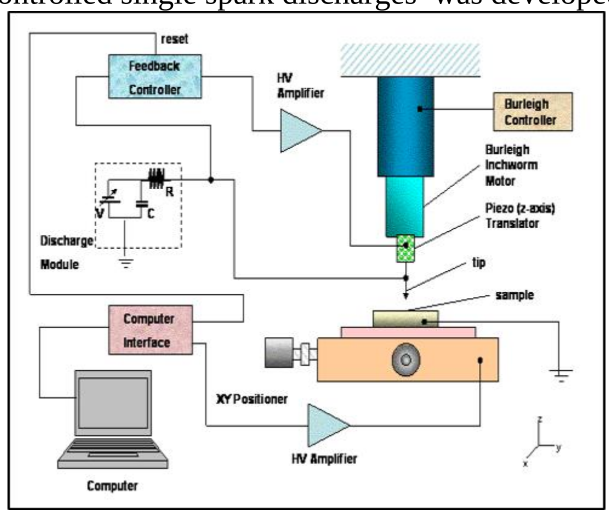
1. Introduction

Fundamentally a capacitor, charged to a certain potential , is discharged by bringing together two electrodes setting up an electric current. When a spark occurs between the electrodes before the circuit is closed, almost all of the energy is absorbed by the positive electrode. A pit or crater is formed on the anode as the result of the melting and boiling of metal caused by electron bombardment before the electrodes touch [1]. Field emission of electrons enhanced by positive space charges has been thought to be the primary producer of electrons for electrodes separated by gaps of 100 um or less in the subsequent initiation of a spark discharge [2], [3], [4].

Craters on the anode reported thus far has been irregularly shaped with varying sizes [5]. In an effort to develop a new technique for nanofabrication using electrostatic discharges, the present workers have observed two types of discharges that create radically different features on the anode surface. It was found that one of these spark discharges can be harnessed to produce deliberate patterns in the nanometer dimension.

2. Equipment Set-Up and Operation

As a starting point, a system that can deliver controlled single spark discharges was developed.



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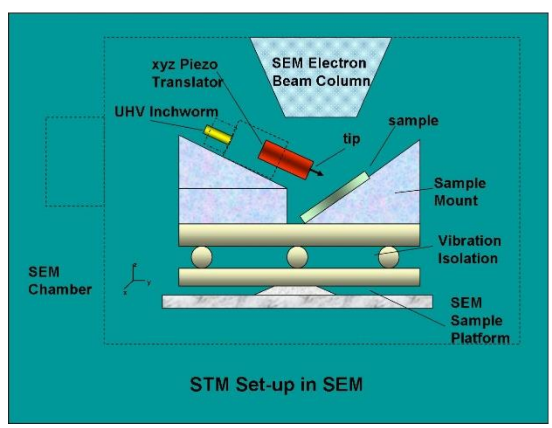

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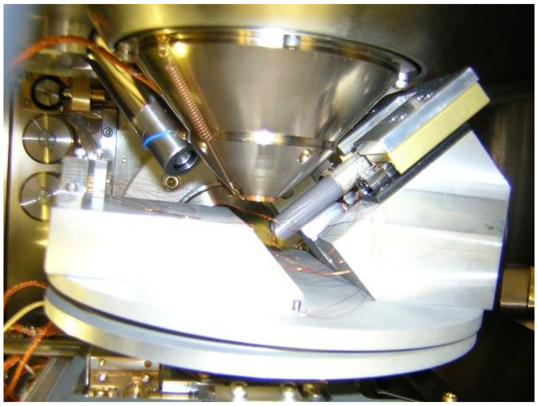

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Figure 1. Nano Electro Discharge Machine Set-up. (a) CNC diagram (b) SEM-integrated diagram (c) actual SEM-integrated system.

It is comprised of the following :

1. A piezo cube transducer and an inchworm for nanometer resolution movement of the cathode tip.
2. Tungsten tip electrochemically etched to a fine point of around 100 nm as the cathode shown in figure 2. Etching procedures has been reported elsewhere.[6]
3. A positively charged gold thin film sample as the anode.
4. Closed loop feedback discharge control circuitry.
5. A capacitive circuit as the voltage source.
6. Gross and piezo driven fine positioning.
7. Computer interface, ancillary recording instruments and software control

The process is readily done in standard ambient conditions. A bias voltage is first applied between the tip and the sample in the range of around 15 V to 20 V depending on the desired size of the crater. The tip is then moved towards the sample by the inchworm to just about the right distance so that the piezo cube can reach the field emitting distance when it extends the tip to the sample surface. Afterwards power is applied to the piezo cube to move the tip to the sample. When a spark discharge occurs between the tip and sample, the corresponding voltage drop is detected by the feedback controller which retracts the tip accordingly.

3. Fabrication of Tungsten Tips

Tungsten wire of 99.98% purity and a diameter of 0.25 mm available from Johnson Matthey Electronics was used as the raw material for the tip. The process involved electro etching of the wire in a solution of potassium hydroxide (KOH). Around seven flakes of technical grade KOH (from J.T. Baker) was dissolved in 100 ml of distilled water. About 200 mm of the wire was cut and one end of it was dipped in the KOH solution without touching the bottom of the beaker. The dipped end was covered by about 50mm of heat shrinked wire insulation. About 10 mm from the tip, 3 – 4 mm of the wire was exposed to the solution where the etching occurred. In the solution, the wire was surrounded by a coil of copper wire about 5 mm in diameter. The tungsten wire was attached to one terminal and the copper coil to the other terminal of a Variac Autotransformer with an output of 5 amps. About 60 volts was applied to the system until the bottom portion of the exposed wire dropped. The upper part of the exposed wire was recovered and cut into about 15 mm in length for use in the discharge operation. Tips were cleaned in a 100 ml beaker of acetone in an ultrasonic bath for about 10 seconds to remove detritus clinging to the tip.

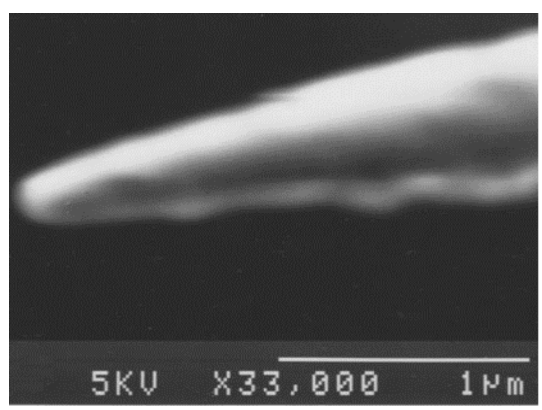

Figure 2. Tungsten tip with apex diameter of around 100 nm.

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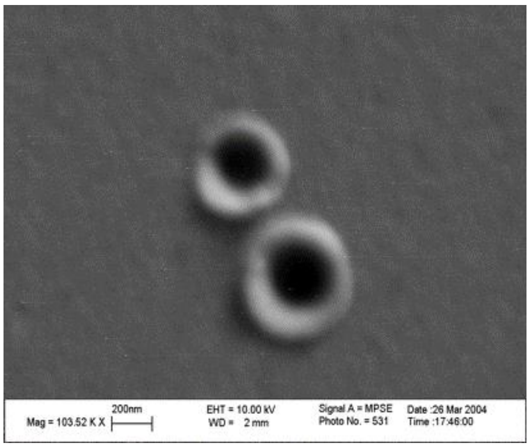
4. Initial Objective and Results

The first goal of the study was to find the optimum power level to be applied to the gap between the tip and the sample so as to produce a spark that can create submicron craters or pits. The minimum size that has been achieved so far is around 50 nm as shown in figure 3b.



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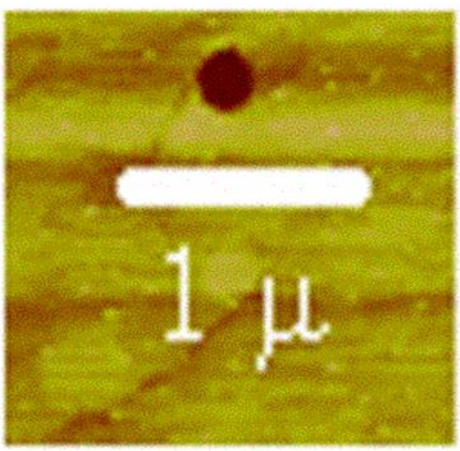

(b)

Figure 3. Discharge crater with diameter (a) 200 nm and 300 nm diameter craters and (b) 50 nm.

The melt region was eliminated by optimizing the pulsing parameters so that the right amount of energy was delivered to the surface to just generate a symmetrical depression without affecting the integrity of the periphery. The discharges were done on Au thin film.

In one of the experimental runs, the workers came upon results that exhibit multiple features per one discharge.

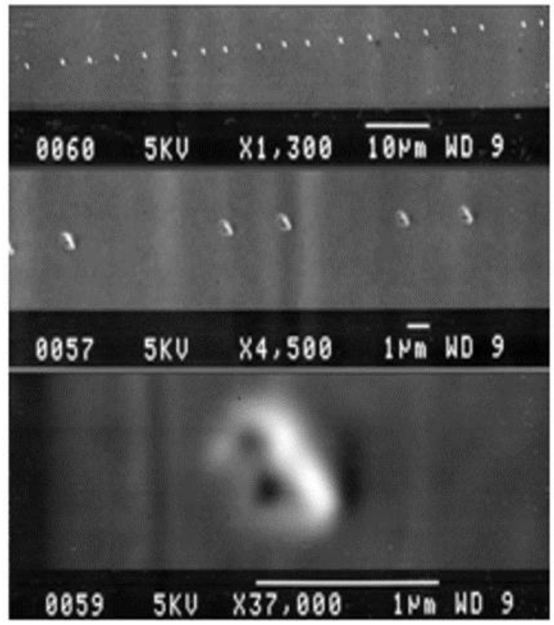

Figure 4. Series of craters exhibiting dual pits per discharge.

Figure 4 shows a portion of a 100 or so series of craters. It exhibits the consistency of the morphology of the features and indicates the retention of the shape and surface characteristics of the tip even after more than a 100 discharges. A higher magnification of the image of the pits is also shown. It is manifested here that they are actually two craters surrounded by the attendant heat zone. It is proposed that this pattern is a 'facsimile' of the surface of the cathode tip produced by the spark discharge. This leads to an interesting way of stamping or printing whole patterns onto surfaces. Figure 5 shows an example of such an imprint pattern.

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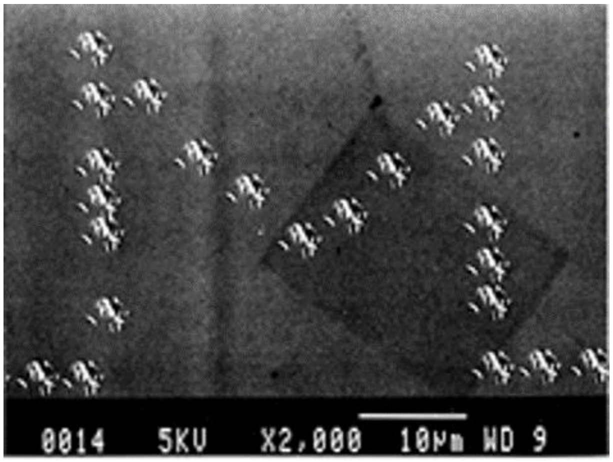

Figure 5. Series of uniform patterns formed as the letter 'M' exhibiting a composition of pits and mounds.

Combined with the movement of the xy-stage positioner, it is established here in this series of discharges producing the letter 'M' that highly regulated marks or features can be created on any area of the sample. Applying relatively high voltages, the marks were generated by design to be easily observable under the SEM. As it happens, the features are made up of an intricate pattern of craters and mounds with the smallest resolvable shape of around 50 nm. Each pattern is exactly identical to one another and it is assumed that they are a projection of the cathode surface created by the interaction of the spark discharge and the thermo mechanical properties of the anode surface. The general uniformity of the marks again suggests that there is minimal damage to the tip during the discharges.

5. Discharge Characterization

Because of the above results, the research study has sought to determine the type of discharge that can produce such a pattern. Two kinds of discharges were observed. Type 1 produces a bluish white spark and creates irregular craters with varying sizes. This type was observed to be relatively more energetic and leads to partial melting of the tip.

Figure 6. Bluish white discharge

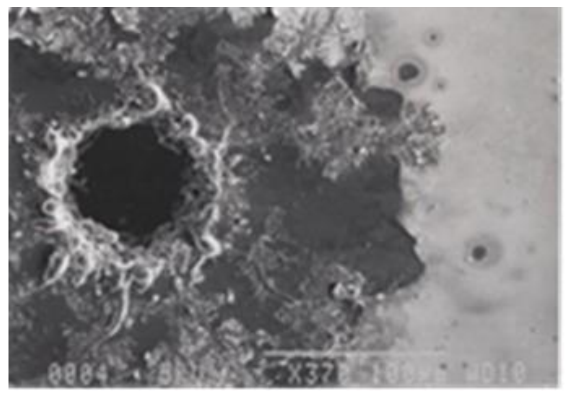

Figure 7. Crater produced from a bluish white discharge

Type 2 produces a faint yellowish appearance which creates fairly uniform features on the surface. The energy delivered by this type is just at the right level to produce the image of the cathode surface onto the anode while at the same time leaving the cathode tip intact.

Figure 8. Faint yellowish glow discharge

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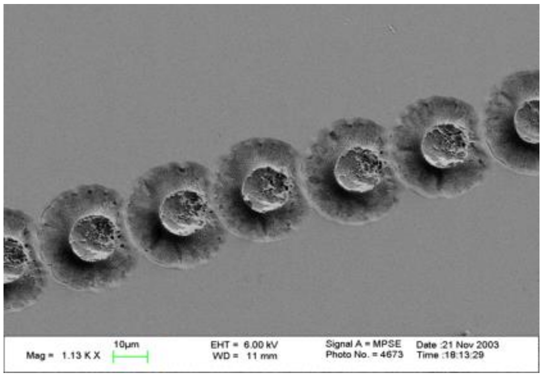


Figure 9. Series of highly regular features created by a faint yellowish glow discharg**e**

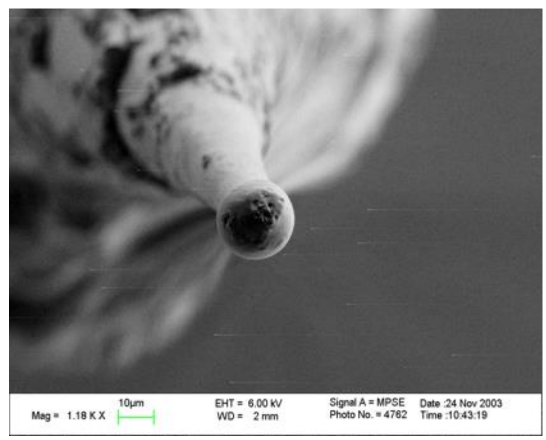
Previews workers have also observed two types of discharges with different physical properties [7] [8]. It all depends on the rate of injection of field emitted electrons from the cathode into the weakly ionized plasma. At the initial stages, there will always be a region where charged species exist within the electric field of the gap. These may be electrons or ionized gas molecules. For a given gap voltage, the sharpest point will generate the highest electric field introducing the electrons at a fast rate into the weakly ionized plasma. The plasma now rapidly becomes strongly charged and more susceptible to interacting with extraneous signals like vibrations, sounds, and electromagnetic radiation . The extra energy from the outside signals creates a shock wave in the plasma that generates a bright, loud, filamentary spark characterized by a much faster deposition of energy into the gap. This is considered as a nonlinear process.

If we now lower the electric field by, say, lowering the applied voltage or by using a blunt tip, the velocity of the electrons emitted from the cathode tip will be reduced. Instead of being introduced into the weakly ionized plasma at a rapid rate, the electrons now diffuse into it at a much slower pace. This leads to a much smoother and linear transition into a strongly ionized plasma which is less affected by other forms of energy . This gives rise to a spark with a luminous diffuse divergent cone with an apex at the cathode.

It has been established for the first time in this present work that the diffuse spark discharge can be used to create regular, repetitive nanometer size imprints on the anode surface reproducing the cathode tip surface. Examples of these are presented in the next section

6. Spark Patterns

The following figures exhibit two examples of imprints created by a spark discharge from two kinds of tip surfaces. The first tip surface is shown in figure 10. It has a half-spherical shape with a rising ridge running in the middle. This unique feature is recreated onto the anode surface shown in figure 11.



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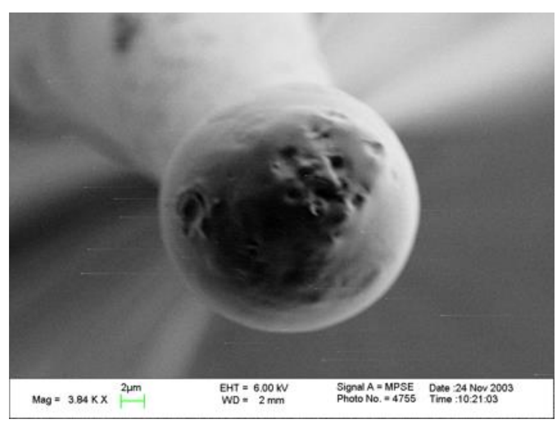

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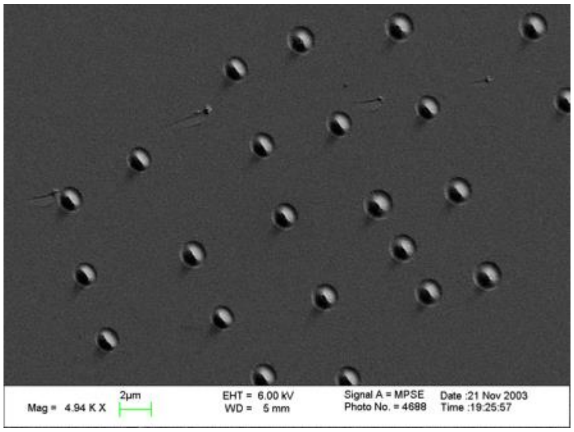
Figure 10. Tip shaped as a half-sphere with a slight ridge in the middle.(a) Wide field of view. (b) Large magnification image



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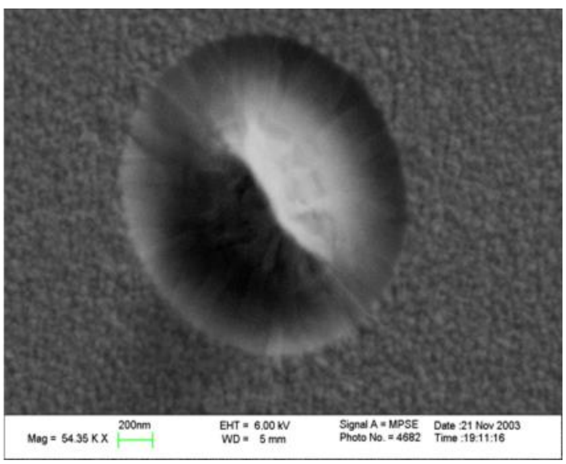

(b)

Figure 11. (a) Highly regular and uniform features created by the spark using this particular tip geometry. (b) High resolution image of the features reveals patterns that reflect the surface character of the tip. The ridge is around a 100 nm.

The second tip surface has a curving trench that runs in the middle (figure 12). Again this distinctive feature is reproduced by the spark discharge onto the sample presented in figure 12.

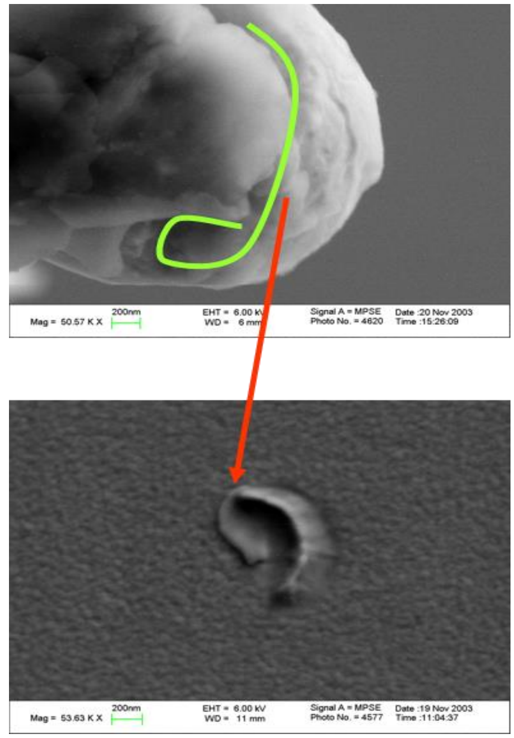

Figure 12. Tip surface with a curving trench in the middle and the pattern created by the spark discharge reproducing the surface features of the tip. The curving ridge is around 100 nm.

A computer control system was also developed in this project that allows the workers to create desired patterns on surfaces with the use of the plasma discharge and a nanopositioning stage. An example of this is shown in figure 13 with the nanocraters arranged in the shape of a turtle 100 µm across.

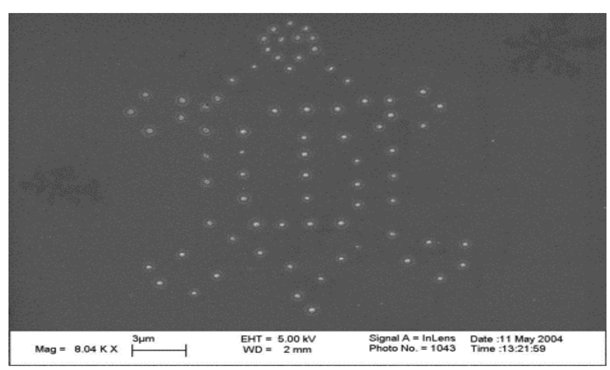

Figure 13. Nanocraters arranged in a turtle pattern.

7. Fabrication of Holes in SiN Membranes

The Silicon Nitride membranes used (from SPI Supplies) were 30 nm thick with areas of 0.5 mm^2. These were suspended on windows etched in 2mm x 2mm SiO_2 wafers about 0.5 mm thick. On one side the membranes were flushed to the surface of the wafers and on the other side the widows were beveled. On the flat side, a coating of around 40 nm thick Au was deposited using a benchscale plasma deposition reactor commonly used for coating of SEM samples. The membranes were mounted onto the discharge set-up for the hole fabrication. The power applied was equivalent to around 6 V and about 4 – 6 nF.

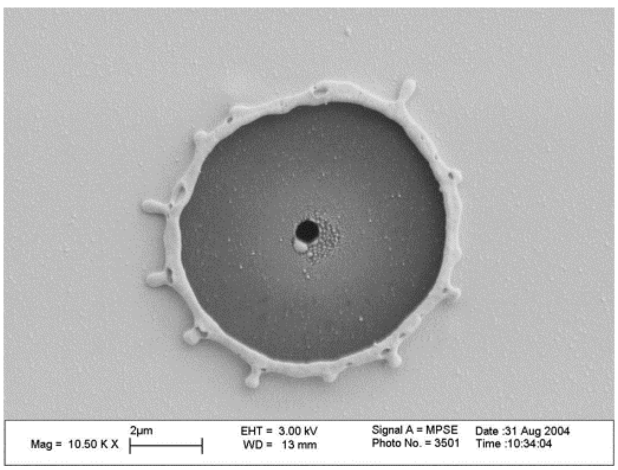

Figure 14. Hole created in SiN membrane

Figure 14. shows a hole created in a SiN membrane. It consists of the hole in the middle of the membrane, and Au deposit layer on top forming a crater from the plasma discharge. Figure 15 shows a hole in SiN being closed by exposure to an electron beam under a scanning electron microscope. Complete closure is done within 1 to 2 minutes. Further development of this process can be used to create patterns on SiN for use as masks in nano-stenciling of patterns on surfaces [9].

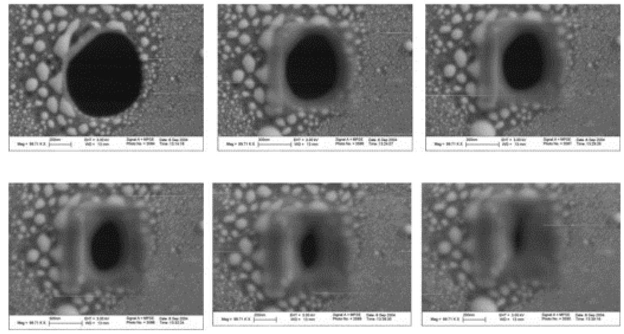

Figure 15. Hole created in SiN membrane being closed by an electron beam.

8. Fabrication of Holes in Al Foil

Ordinary Reynolds aluminum foil was used for this experiment. Small strips of it was stretched over a metal holder and mounted onto the set-up. For this material the power applied was equivalent around 25 to 30 volts and about 0.01 amps for the plasma to blast through the thickness of the foil. The sharpness of the tip controls the diameter of the hole created. The bigger the diameter of the tip the bigger the hole created.

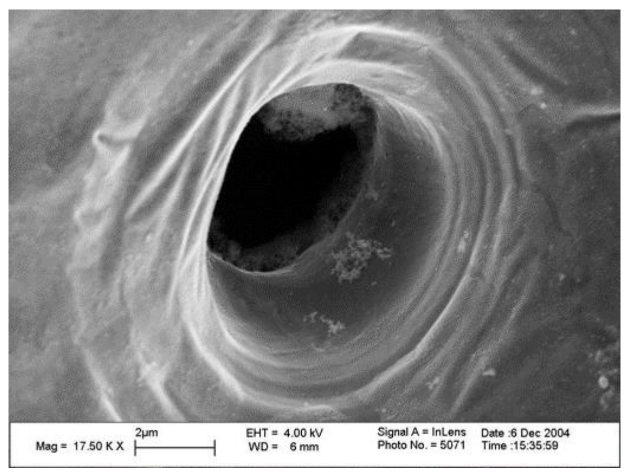

Figure 16. Hole created in aluminum foil

Figure 16 shows a hole created in aluminum foil. The foils were tested in a custom made 'micro'-coulter counter. Two 1 ml syringes were attached together head to head and a foil with a hole was placed between them to act as a membrane. Au wires were introduced into both syringes which act as electrodes.

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At first lab grade ultrapure water was placed inside the syringes and a constant voltage of around 5 V was applied to the electrodes to cause ions to pass through the hole carrying the current. This measurement is shown in figure 17.

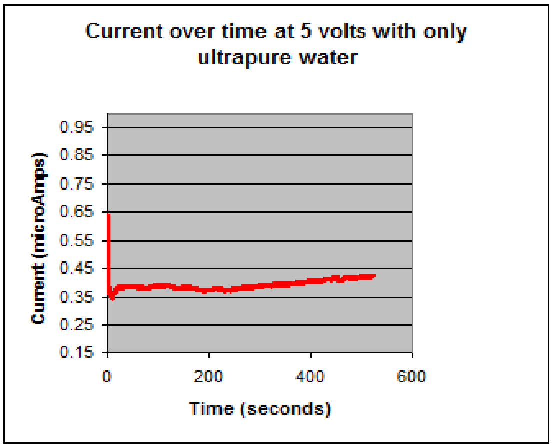

Figure 17. Measurement of ionic current through the foil membrane

Next an amount of micro-spheres (from Magsphere Inc.) was placed inside one of the syringes and the measurement was repeated. We can see in figure 18 that curve of the current versus time now has a lot of fluctuations which is due to the microspheres passing through and blocking the hole temporarily stopping the ionic current.

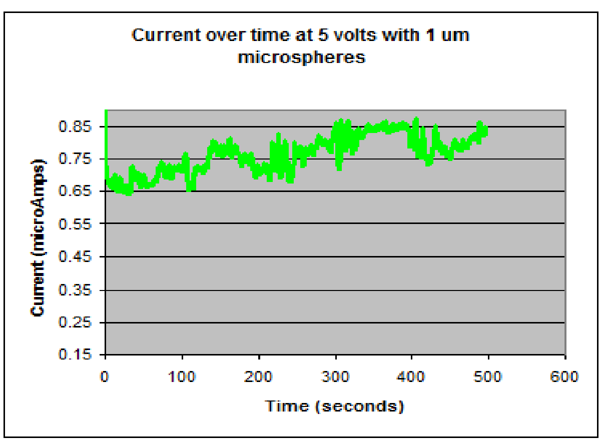

Figure 18. Measurement of ionic current through the foil membrane intermittently blocked by microspheres.

9. Significance of the Project

This technique takes advantage of the field emission of electrons at short distances so that lower voltages (power) can be applied to the electrodes. This results to craters and patterns in the order of nanometers. Also the deterioration of the tip by the plasma discharge is reduced considerably since the cathode surface is protected by the positive space charge. This results in features with great uniformity and cathodes with longer lifetimes.

Because it is based on the use of a sharp tip commonly employed in SPM techniques, the process is readily adaptable to any STM or AFM set-up and can fill the gap between processing features in the micron range and the actual manipulation of atoms.

The process can also be likened to a sort of ‘xerographic process’ wherein any design that is fabricated on the cathode surface can be recreated onto the anode surface in one discharge. This will greatly reduce the time it takes to generate patterns on surfaces leading to higher productivity. This will further enlarge the potential of nanoimprint lithography as a key nanolithography process in future integrated circuits and integrated optics [10].

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