Surface & Coatings Technology 203 (2009) 2436–2441

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Fabrication of nano structures in thin membranes with focused ion beam technology

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ARTICLE INFO

Available online 3 March 2009

Keywords:
Nano pore
Dual beam FIB
Nano slit
Nano wire

ABSTRACT

In recent years, Focused Ion Beam (FIB) technology has emerged as an important tool for nanotechnology [V.J. Gadgil, F. Morrissey, Encyclopaedia of Nanoscience and Nanotechnology, vol. 1, American Science Publishers, ISBN: 1-58883-057-8, 2004, p101.]. In this paper, applications of focused ion beam technology to fabrication of nanostructures are presented. The structures are fabricated on free standing silicon nitride membranes. Nanopores are nanometer diameter holes used in bio medical research for high speed DNA sequencing [D.K. Stewart, L.A. Stern, G. Foss, G. Hughes and P. Govil, Proc. SPIE 21, 1990, 1263.]. FIB was used to mill nanopores in the membrane. The pores were further reduced using epitaxial deposition using electron beam, at a controlled rate. A STEM detector was used to monitor the pore in situ. Nanowires can be fabricated using shadow mask technique. The shadow mask for nanowires was fabricated using FIB. The mask was used to produce nanowires. Fabrication method and FIB process parameters for the fabrication are reported. Results of the nanopore fabrication are presented with STEM images. Results of the nanowire fabrication are presented. Various strategies employed to achieve the desired nanostructures are discussed.

1. Introduction

The focused ion beam (FIB) systems are widely used in semiconductor industry. The main applications are process control and failure analysis. In recent years specialised dedicated systems are commercially available for research in other disciplines. The technology makes it possible to carry out localized milling of almost any type of material, and deposition of conductors and insulators with high precision [1–6]. The possibility to image at high magnification and milling at a very specific site has made possible preparation of specimens for transmission electron microscopy (TEM). Recently a number of reports have appeared in the literature about the use of FIB in micromachining applications for MEMS [7–9]. This powerful tool shows a promising future in nanotechnology and nano-fabrication.

Sequencing of DNA with conventional methods is a slow time consuming and expensive process. Recently several groups have been working on an alternate approach of sequencing using a nanopore. A DNA molecule is electrically not neutral and carries a charge. The principle of the method is to use a thin membrane with a nanopore between two electrochemical cells. When an electric potential is applied between the cells, it can drive the DNA molecule through the nanopore. The electrochemical signal as the molecule passes though the nanopore can serve as the signature for identifying the molecule. Since this was demonstrated by Kasianowicz et al. [10], both biological and solid state nanopores have been used successfully as single molecule detectors to characterise nanoscale molecules [11]. Recent experiments also showed that solid state nanopores could have certain advantages over biological nanopores. The solid state nanopores offer flexibility of diameter. They are mechanically robust and chemically stable. They can operate over long period of time unlike the biological nanopores. A nanopore is most effective when the diameter of the pore is close to the diameter of the molecule to be detected which is typically 2–10 nm. Fabricating a solid state nanopore is difficult. For instance it is almost impossible to mill pores below 30 nm diameter consistently reproducibly in terms of size and shape using commercially available FIB systems. The quality, consistency and thickness of the substrate are critical in producing the nanopores.

Nanowires are devices limited to a few nanometers laterally while extending in length to several thousand nanometers. Nanoscale devices have been realized for applications ranging from electronics and optics, to biosensing [12]. Many of these reports demonstrate that nanoscale devices such as nanowires, carbon nanotubes, nanoparticles, and nanocantilever beams are ultrasensitive sensors due to their one-dimensional (1D) structure. The 1D structures, such as nanowires (NWs), are particularly compelling due to their potential for biosensing applications and suitability for large-scale high-density integration. With many unique properties and promising functions, nanowires and nanowire-based devices are being extensively studied around the world.

A major difference in the form of the materials as a nanowire is that there is an ultrahigh surface-to-volume ratio. The limitation in lateral dimension leads to phenomena like the quantum confinement effect inside the nanowires. The high surface-to-volume ratio makes surface process like adsorption and desorption etc. to easily take place. As a result, these new shapes of materials — the nanowires — exhibit a variety of interesting and fascinating properties, and may function as the building blocks for nanoscale electronics, nano-optics, and especially for

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doi:10.1016/j.surfcoat.2009.02.036

nanosensing technology. Semi conducting oxide nanowire gas sensors can be operated at room temperature and are fast in detecting various gases at concentrations of parts per million (ppm), or even parts per billion (ppb). Another example is the single crystalline silicon nanowire sensor. Very recently, this nanowire sensor has been used to detect (bio-) chemical species at very low concentrations, even at the molecular level. Single viruses and bacteria, DNA and DNA sequence variations, small molecule protein interactions etc., can now be detected. In addition, the sensor allows real-time and online detection with a quick response time. These new capabilities are a great development in the sensor field, and are beyond what conventional (bio) sensors can do. The just developed sensor has thus opened up many new research possibilities and practical applications in many fields, including important and quickly developed sectors like health care, life sciences, pharmacy, and biotechnology. Generally speaking, all these capabilities of nanowire sensors are beyond that of conventional sensors.

Although nanowire-based devices possess a variety of interesting properties and promising functions, the advantage in the utilization, and especially commercialization, of nanowires had been relatively slow, probably due to the difficulties associated with the synthesis of such nanostructures with well-controlled size, phase purity, crystallinity, chemical composition, and an integration of individual nanowires into a complete device. Nanowires can be fabricated using several techniques. Here we present a top down approach using a shadow mask milled with FIB. The shadow-mask technique has been a common low-cost fabrication technique to pattern materials on a substrate without the direct use of photolithography. In addition, the patterning resolution of conventional shadow masks is typically low due to the large distance between the mask and target substrate. The shadow masks used for fabricating the nanowires are manufactured from silicon substrates with nanoscale deposition windows, or nanoslits, created using focused ion beam machining. The nanoslit shadow masks have been fabricated from free standing low-stress (LS) silicon nitride (SiN) membranes using FIB machining.

2. Fabrication of thin film

Both for the shadow mask and the nanopore, free standing silicon nitride substrates were fabricated. As mentioned earlier, the quality of the membrane is critical to the successful fabrication of the nanopore as well as the shadow mask. When the thickness of the membrane is too large, the profile of the nanopore is no longer cylindrical. At the same time the nanopore becomes a nano channel. This creates further problems in the sensing of the biomolecules. The details are beyond the scope of this paper. The thickness and consistency of the membrane also directly affect the process parameters of milling the

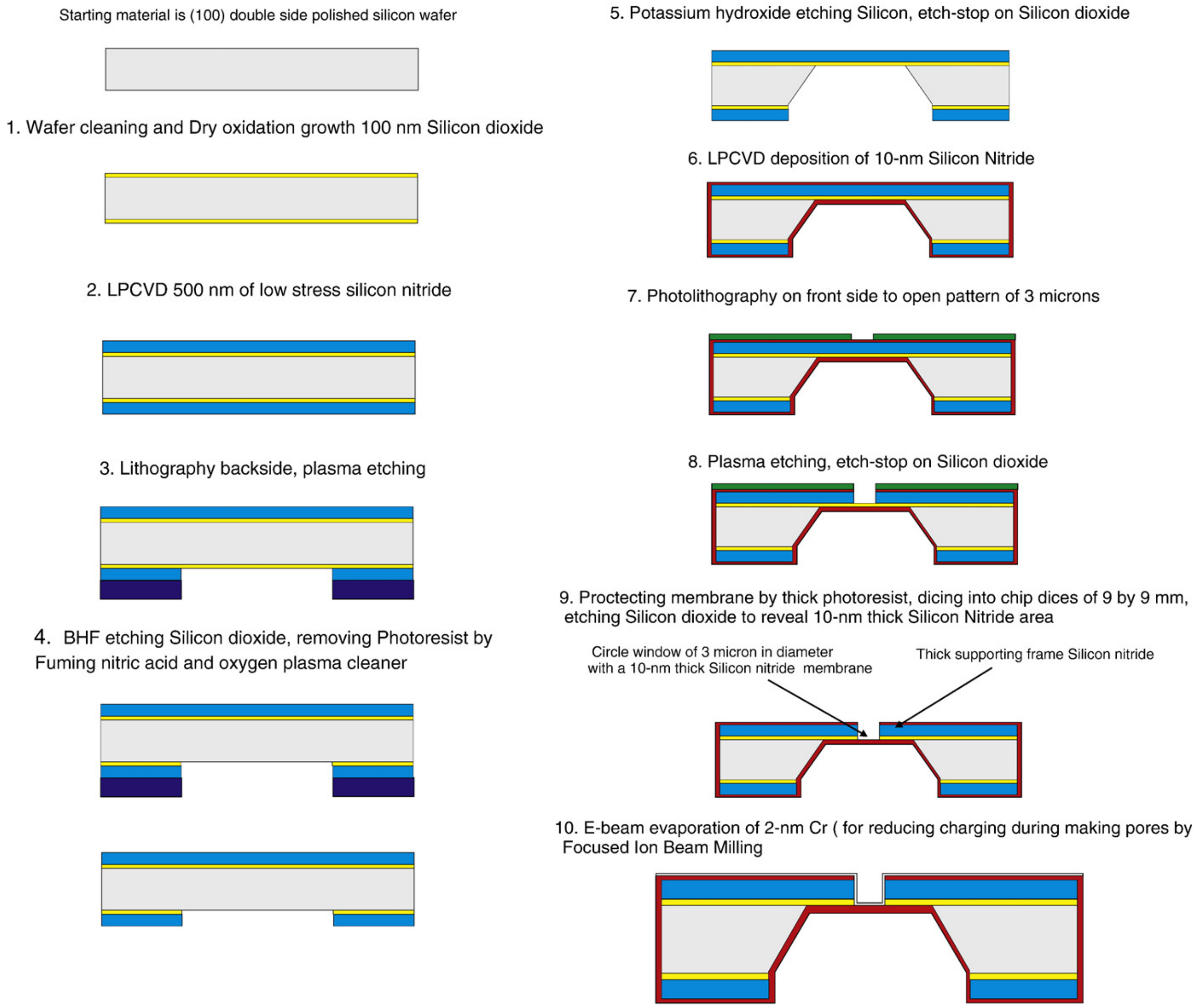

Fig. 1. Process for low-stress silicon nitride membranes used for nanopore and shadow mask.

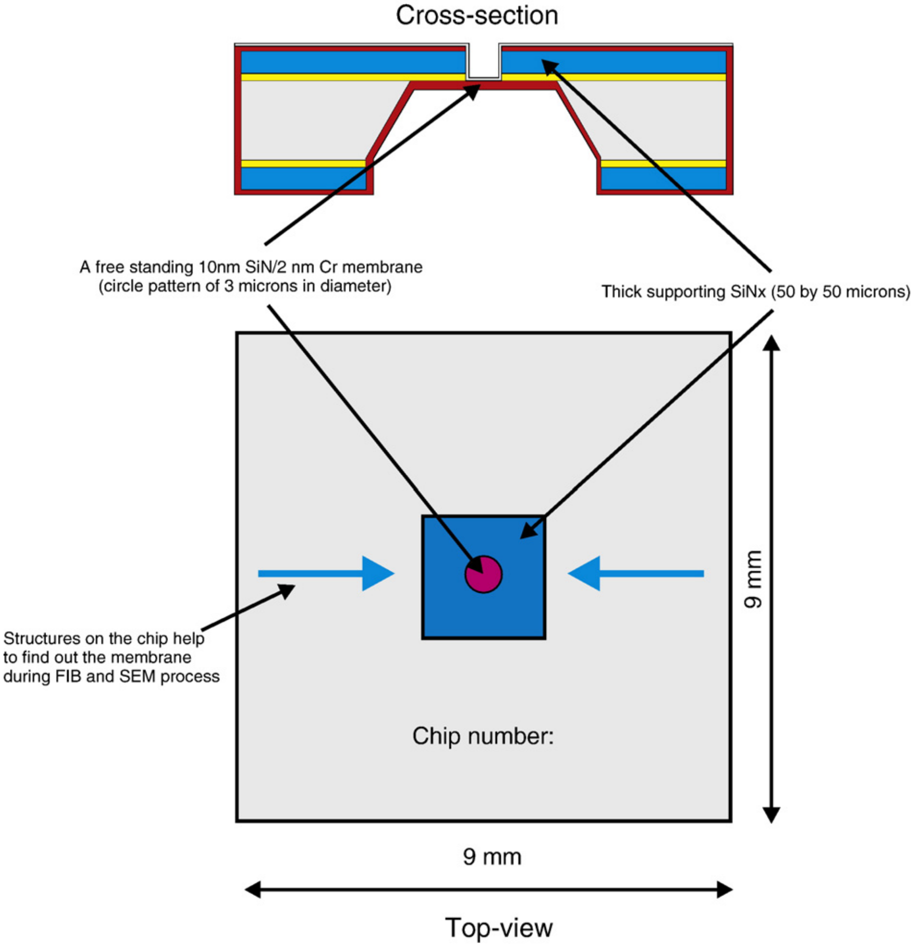

Fig. 1 (*continued*).

nanopore. The time of milling with FIB is determined by the amount of material that has to be milled. The fabrication process of the membranes is given in Fig. 1.

3. Interaction of the ion beam with surfaces

Acceleration voltage used typically in the commercially available FIB systems is around 30–50 keV. The incident ion loses its energy when it strikes the surface of the specimen. This energy is partially lost to the electrons and partially to the atoms itself. Due to Coulomb interaction the ion excites the electrons in the solid to bound states or to continuum states. This is a predictable process. The loss of energy to the atom is more complicated. This is because of finite number of random collisions with significant energy loss. When an ion strikes the surface of a solid, the ion is deflected from its path and the atom is displaces from its original position. The ions penetrate the solid to a depth R_p called penetration depth or range. If a Gaussian curve is fitted to the distribution of ions at a depth Z it has a form $\exp[-(Z-R_p)^2/2(\Delta R_p)^2]$ where ΔR_p is called range straggle. In addition to this the collisions also produce uncertainty in the transverse position of the ion in the solid. This is described as ΔR_t or transverse straggle.

The ions cause various effects when they are incident on the specimen surface.

1. The ions striking the surface can cause sputtering. This is removal of atoms from the substrate. This occurs at low energies of 50–1000 eV. The removal of atoms from the substrate is dependent on the energy of the incident ions. The yield or the number of atoms removed increases with energy up to about 100 keV and then decreases. Typical yield is about 1 to 10 atoms per ion. The sputtered ions leave the surface with a few eV of energy.
2. When the ion strikes the surface, electrons are emitted. About 1–10 electrons are emitted per ion with typical energies of a few eV. In a FIB system these electrons called secondary electrons are used to image the surface.
3. Chemical effects are also produced by ions. These are because of the ion electron interactions as well as ion atom interaction. When a photoresist is exposed to the ions, the molecules are changed because of the ions. They can either make the resist soluble or insoluble in developer. Ions can also break molecules into parts. A typical example is an organic gas molecule carrying a metal atom. The ions can break the molecule causing deposition of the metal on the surface. Another example is ion assisted etching where a gas is used in combination with the ion source.

4. Nano fabrication with focused ion beam

4.1. Milling

The main purpose of the FIB equipment is milling of materials. The energetic ions striking the surface of the specimen eject the atoms on the surface. This results in milling of the specimen surface. The yield or the number of atoms removed increases slowly with energy, then flattens out and then decreases at about 50 keV [13]. If the material has a density ρ (atoms/cm^3) and an ion dose Δ (ions/cm^2) mills the material to a depth d (cm), then the yield can be defined as

$$Y = (\rho d/\Delta)\ (\text{atoms/ion}).$$

Since in focused ion beams, milling ions can be buried in the material, and other phenomenon as re-deposition can also take place, the actual yield may not correspond exactly to the atoms removed per ion. The sputtering yield is also dependent on the angle of incidence. It roughly increases with 1 / cos(□), with □ the angle between the surface normal and the ion beam direction.

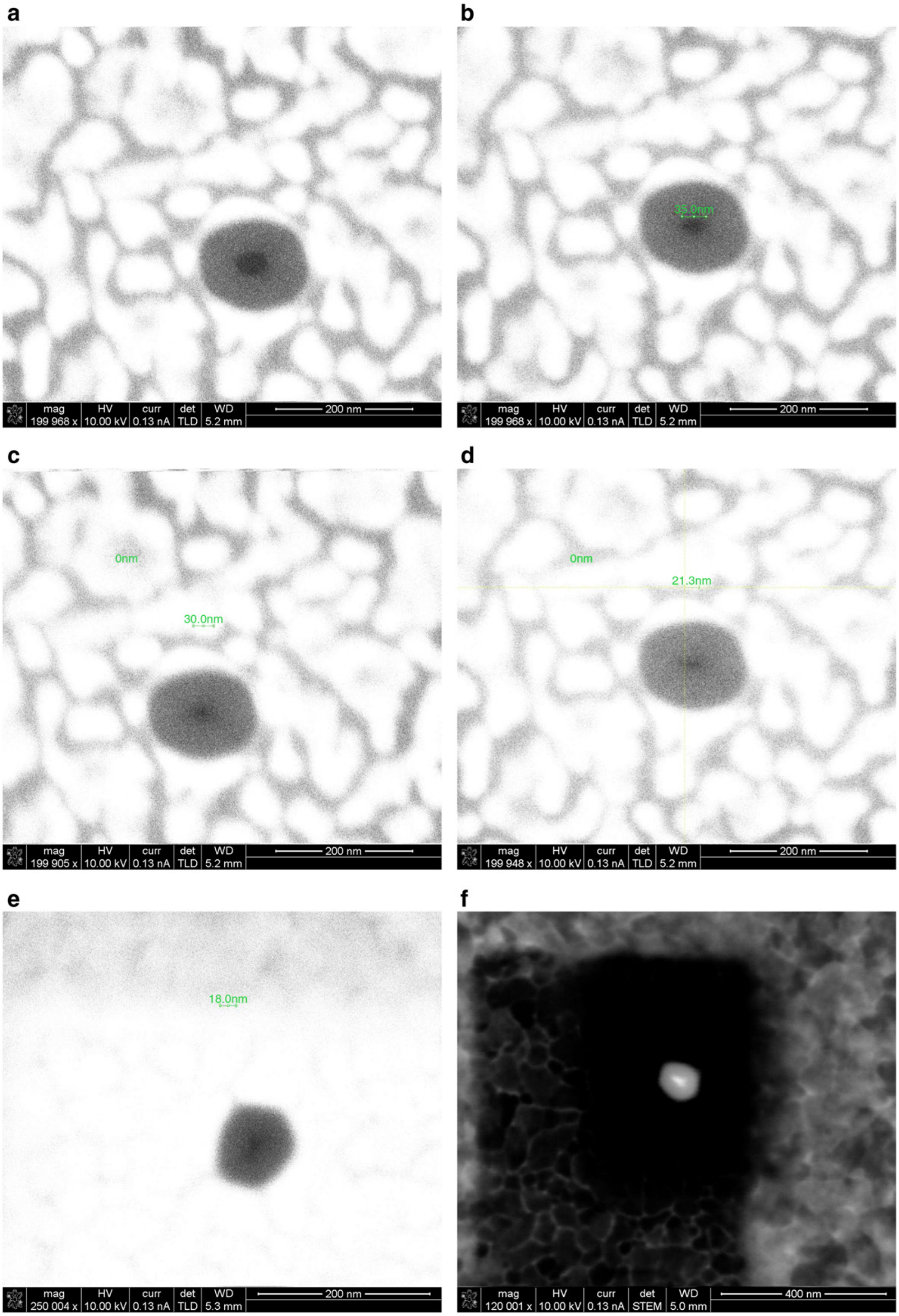

Fig. 2. a) Initial pore milled with FIB. b) Pore after 10 scans with electron beam. c) Pore after 30 scans with electron beam. d) Pore after 40 scans with electron beam. e) Pore after 60 scans with electron beam. f) STEM detector image of the pore.

4.2. Deposition

For many years people have used FIB systems for doing beam induced deposition of metals and insulators onto device surfaces. The deposition of materials is accomplished by introducing metal or insulating atoms as a part of a carrier molecule, very close to the sample surface where they collide with gallium ions from the primary ion beam and are forced onto the sample surface. The principle is chemical vapour deposition (CVD). As the ion beam can be focused to a very small spot size, the deposition is very localized. The beam can be made to follow a preprogrammed pattern and this results in deposition of the metal in the desired form. The carrier molecule is in vapour form and is introduced through a needle which is brought in close proximity of the sample surface. The gas is adsorbed on the surface of the specimen. The ion beam dissociates the carrier molecule as it collides with it. The dissociated products are volatile and are removed by the vacuum system while the desired reaction products carrying the metal atoms remain attached to the surface. The deposited metal is not pure as it also contains Ga and parts of the carrier organic molecule. The most common metals deposited in commercially available machines are Pt and W. Insulating material like SiO_2 can also be deposited. In this case tetraethylorthosilicate (Si $(OC_2H_5)_4$ and oxygen or water vapour is used as a precursor. The gas is injected through a needle which is inserted to about 100 mm from the surface of the sample. Electron beam or the ion beam interacts with the gas depositing SiO_2 on the surface of the specimen. A wide range of metals has also been deposited for research purpose such as Ta, Au, C, Fe and Al. The smallest feature that can be deposited by conventional FIB is of the order of <100 nm. Traditionally these depositions are mainly used to connect or isolate wires when doing device edit operations. Apart from the ion beam, the electron beam can also be used to deposit material.

5. Results

The FIB system used in this investigation was a dual beam FEI NOVA 600 nano lab. This equipment has two columns. It has a high resolution SEM column and a Ga ion column.

An initial pore was drilled in the thin free standing membrane with FIB. Imaging the membrane was kept to the minimum as when imaging with the ion beam, the surface is also milled. The initial pore was milled with 9.2 pA current and can be seen in Fig. 2a . After milling the pore, it was imaged with the SEM. The SEM was set at 10 kV with 130 pA current. The needle which introduces TEOS gas was left in retracted position. The valve was opened so that the gas entered the specimen chamber. As the needle was retracted, the concentration of the gas was much lower at the sample surface than when the gas is introduced just at the surface of the sample. Because of this, very low quantity of gas was available at the sample surface for deposition and the deposition rate was very low. The SEM was used to scan the nanopore and with each scan, silicon dioxide was deposited on the membrane because of the electron beam. After every 10 scans an image was captured. The images after 10 scans, 30 scans, 40 scans and 60 scans can be seen in Fig. 2 b,c,d and e. As can be seen the pore starts to shrink with subsequent scans. As the pore shrinks, it becomes more and more difficult to image with the SEM. The FIB also has a scanning transmission electron microscopy detector (STEM). With this detector, an image can be made using the transmitted electrons. A STEM detector image of nanopore after shrinking can be seen in Fig. 2 f. The diameter of the nanopore with subsequent scans was measured and it was found that the reduction in diameter is linear. Shrinking of two such holes can be seen graphically in Fig. 3.

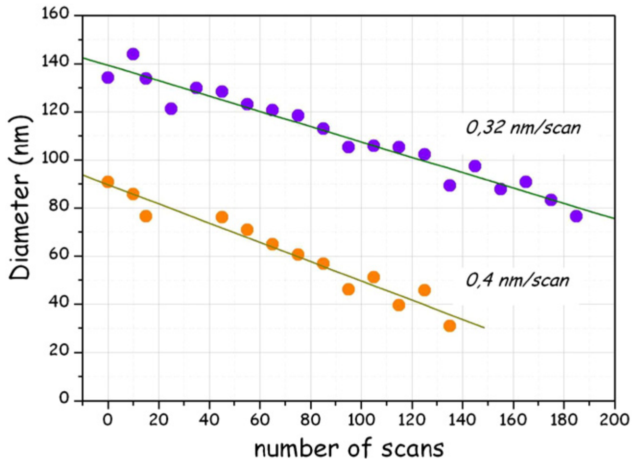

Fig. 3. Pore diameter versus number of scans for two different pores.

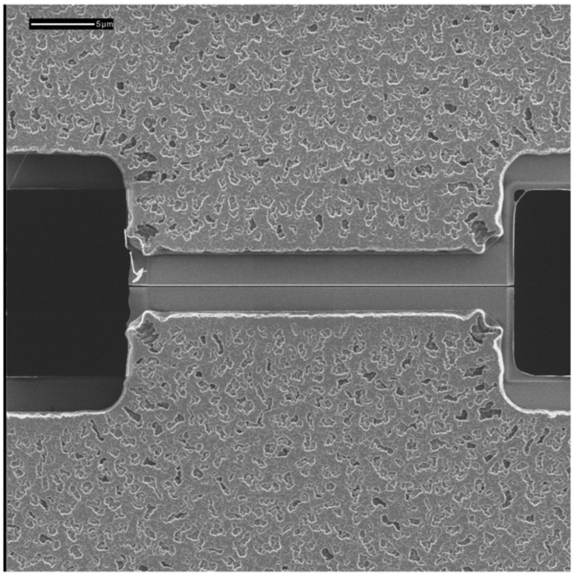

Fig. 4. Shadow mask fabricated with FIB.

The fabrication process for a thin film for the shadow mask was identical to the process for the nanopore. Instead of a round hole, a dumbbell shape hole was made. This was done so that the contact pads were integral to the deposition of the nanowire. The contact pads were first milled with FIB using 93 pA current. After that, using a lower current of 9.2 pA, a thin line was milled between the contact pads. This can be seen in Fig. 4. Using the shadow mask, a gold nanowire was deposited with electron beam sputtering. The nanowire can be seen in

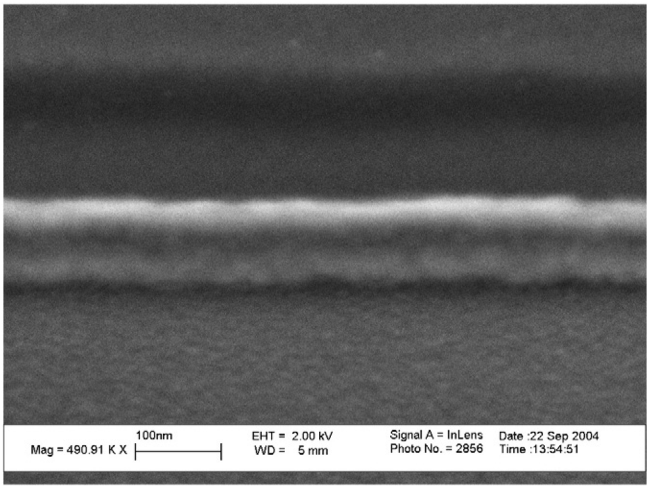

Fig. 5. Nanowire deposited using shadow mask.

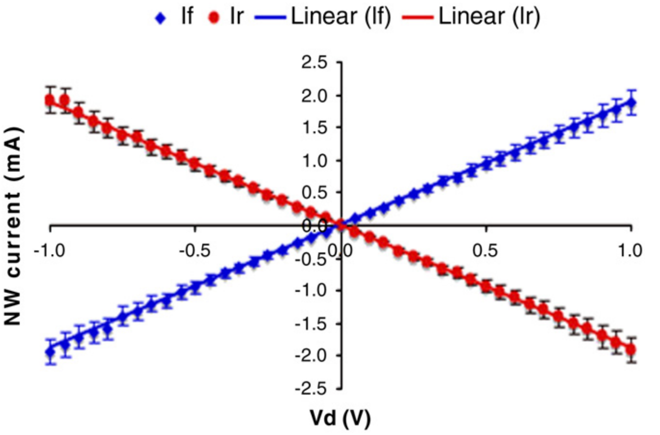

Fig. 6. Electrical characteristics of the nanowire.

Fig. 5. The nanowire was approximately 50 nm in width. Electrical measurements of currant versus voltage can be seen in Fig. 6. This was found to be linear.

6. Summary

We have seen that there are ever increasing applications of FIB in semiconductor industry, materials science and nanotechnology. The developments have led to smaller beam diameters, better positioning hardware and software and more number of materials that can be deposited. More number of gases for GAE and selective etching are also being made available. Over the years FIB has become a powerful tool in mask repair, defect analysis, and TEM specimen preparations. FIB remains to be the only tool that can be used for device modification and prototyping. Further developments in detectors have made it possible to use FIB also as an analytical tool. In the field of nanotechnology FIB has very important applications in making near field optical probes, sensors, photonic devices, and fabricating nanostructures by the deposition of material. It is however not suitable for mass throughput fabrications. It remains a relatively slow process. Despite the drawbacks the applications show a great promise in path breaking technologies in nanotechnology. It is clear that FIB technology will play a central role in the coming years.

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