TWO DIMENSIONAL (2D) NANOFABRICATION PROCESS ON QUARTZ SUBSTRATE

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by

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LIST OF ABBREVIATIONS

2D	Two dimensional
3D	Three dimensional
EBL	Electron beam lithography
SEM	Scanning electron microscope
SPL	Single pass line
C	Coulomb (Ampere.second)
H_2O	Deionised water
H_2O_2	Hydrogen peroxide
NH_4OH	Ammonium hydroxide
HF	Hydrofluoric acid
HCl	Hydrochloric acid
PMMA	Polymethyl methacrylate
LMW	Low molecular weight
HMW	High molecular weight
wt.%	Weight percentage
C_6H_5Cl	Chlorobenzene (C)
C_7H_8O	Anisole (A)
PEDOT/PSS	Poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate)
IPA	Isopropanol
MIBK	Methyl isobutyl ketone
Ni	Nickel
NiCr	Nichrome
NMP	N-methyl-2-pyrrolidone

CHF_3	Trifluoromethane / Fluoroform
Ar	Argon
O_2	Oxygen
RIE	Reactive ion etching
ICP	Inductively coupled plasma
RF	Radio frequency
OM	Optical microscopy
AFM	Atomic force microscopy
FESEM	Field emission scanning electron microscopy
EDX	Energy dispersive X-ray spectroscopy

PROSES NANOFABRIKASI DUA DIMENSI (2D) PADA SUBSTRAT KUARTZ

ABSTRAK

Fabrikasi struktur nano pada substrat kuartz telah menarik perhatian dan minat penyelidik sejak kebelakangan ini kerana pelbagai aplikasinya yang berpotensi seperti untuk NEMS / MEMS termasuk BioMEMS / BioNEMS, sensor dan elektrod sel bahan api. Ia akan membuka beberapa potensi baru kerana sifat kuartz yang keras, mampu bertahan pada suhu yang tinggi, mempunyai aliran UV yang tinggi dan sifat piezoelektrik- iaitu kebolehan menghasilkan elektrik apabila dikenakan tekanan mekanikal. Walaubagaimanapun, hanya sedikit maklumat mengenai proses fabrikasi 2D pada bahan penebat seperti substrat kuartz. Pelbagai jenis kaedah litografi moden telah diterokai sejak litografi optik tidak mampu mengfabrikasi struktur nano. Litografi alur elektron (EBL) adalah salah satu teknik yang sering digunakan untuk pencorakan langsung pada skala nanometer yang membolehkan nanofabrikasi struktur dan peranti dalam penyelidikan tentang nanosains dan nanoteknologi. Kaedah punaran ion reaktif (RIE) biasanya diguna dalam proses pemindahan corak 2D atau 3D kerana kebolehan punaran terarah. Gabungan plasma tambahan induktif (ICP) dengan punaran ion reaktif (RIE) boleh meningkatkan ketumpatan plasma dan kadar punaran semasa proses pemindahan corak. Kajian asas yang di jalankan dalam kajian ini boleh menyumbang kepada pengetahuan dan kefahaman proses teknologi nanofabrikasi. Dalam kajian ini, teknik fabrikasi atas-bawah telah digunakan. Dalam teknik ini, proses definisi corak menggunakan EBL

dan proses pemindahan corak dengan plasma tambahan induktif-punaran ion reaktif (ICP-RIE) sistem. Kaedah pemindahan corak aditif diperlukan untuk punaran kuartz, di mana bahan topeng yang tahan lasak telah digunakan semasa proses punaran. Faktor-faktor yang telah mempengaruhi proses definisi corak, termasuk pemilihan fotoresist, parameter alur elektron seperti arus eletrik, dos dan masa menghasilkan corak telah dioptimumkan. Manakala, kesan parameter punaran yang berbeza seperti kuasa ICP-RIE dan kadar aliran gas bahan punar telah disiasat ketika proses pemindahan corak. Reka bentuk garis tunggal telah ditakrifkan pada dua lapisan polymethyl methacrylate (PMMA). Dos alur elektron pada 900 pC/cm sesuai untuk menghasilkan corak pada 200 nm PMMA dan berjaya didepositkan dengan filem nipis nichrome (NiCr). Dengan itu, corak 2D telah berjaya dipindahkan ke atas substrat kuarzt menggunakan gas trifluorometana (CHF_3) dan argon (Ar) dengan kadar punaran pada 14 nm/min. Mikroskop optik, mikroskop daya atom (AFM), mikroskop pelepasan bidang elektron imbasan (FESEM) dan tenaga serakan sinar-X (EDX) telah digunakan untuk mencirikan profil struktur yang dipunarkan.

TWO DIMENSIONAL (2D) NANOFABRICATION PROCESS ON QUARTZ SUBSTRATES

ABSTRACT

The fabrication of nanostructures on a quartz substrate has attracted the attention and interest of researchers in recent years due to its wide range of potential applications such as for NEMS/MEMS including BioMEMS/BioNEMS, sensor and fuel cell electrodes. It would create several new potential due to the excellent properties of quartz in hardness, ability to withstand high temperatures, having high UV transmission and piezoelectric properties- which is a capability to develop an electric potential upon the application of mechanical stress. However, there is limited information of 2D fabricating process on insulating materials such as quartz substrate. Various types of modern lithography methods have been explored because optical lithography physical limitations have hindered the fabrication of nanostructure. Electron beam lithography (EBL) is one of the most commonly used techniques for direct patterning at the nanoscale meter range that enables the nanofabrication of structures and devices in the research field about nanoscience and nanotechnology. The reactive ion etching (RIE) method is usually utilized in process transfer of 2D or 3D structures due to the ability of directional etching. Combination of inductively coupled plasma (ICP) with reactive ion etching (RIE) could boost the densities of the plasma and enhance etch rate during pattern transfer process. The fundamental study conducted in this research can contributes to the existing knowledge and understanding on this crucial process in nanofabrication technology.

In this study, a top-down fabrication approach was applied. In this approach, pattern definition process uses the EBL tool and pattern transfer process by inductively coupled plasma-reactive ion etching (ICP-RIE) system. The additive pattern transfer method required for quartz etching, where high resistant mask material was used during etching process. The factors that influenced the pattern definition process, including the selection of photoresist, e-beam exposure parameters such as current, dosage and the developing time were optimised. Meanwhile, the effect of etching parameters ICP-RIE power and flow rate of etchant gases were investigated during the pattern transfer process. A single pass line design was defined on bi-layer polymethyl methacrylate (PMMA). An e-beam dosage of 900 pC/cm was found suitable for pattern definition on a 200 nm PMMA and was successfully deposited with nichrome (NiCr) thin film. With that, a 2D pattern was successfully transferred onto quartz substrate using trifluoromethane (CHF_3) and argon (Ar) gases with etching rate of 14 nm/min. The optical microscopy, atomic force microscope (AFM), field emission scanning electron microscopy (FESEM) and energy-dispersive X-ray (EDX) were used to characterize the etched structure profiles.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Nano-science, engineering and technology are research fields that deal with the manipulation of new materials and the design and fabrication of structures, devices and systems at the atomic, molecular or macromolecular scales. The main focus in this research area is to improved the properties but at a lowest cost possible. Among natural substances, deoxyribonucleic acid (DNA) is one of the examples of things that are several nanometers in diameter. Current handmade objects even reach the nanometer level (1-100 nm) in one, two (2D) or three dimensions (3D), as shown in Figure 1.1 (National Research Council, 2002). The potential of nanostructures opens a wide range of opportunities in terms of fundamental and applied sciences.

There are numerous applications of nanostructures, including solar photovoltaics and photonics (Back *et al.*, 2014, Hauser *et al.*, 2012, Landis *et al.*, 2013), microfluidics (Catalano *et al.*, 2014, Mogi *et al.*, 2014), fuel cell electrodes (Arthur *et al.*, 2011, Huang *et al.*, 2009) and micro- or nano- electromechanical systems (MEMS/NEMS) such as BioMEMS/BioNEMS (Bhushan, 2007, Shklovsky *et al.*, 2012). As so, many nanofabrication techniques have been developed and investigated such as electron beam lithography (EBL), focused ion beam (FIB), electron deposition, self-organization and chemical synthesis (Jiang *et al.*, 2001). However, fabrication becomes challenging when the feature size is beyond 100 nanometers small and the structures are very close to each other.

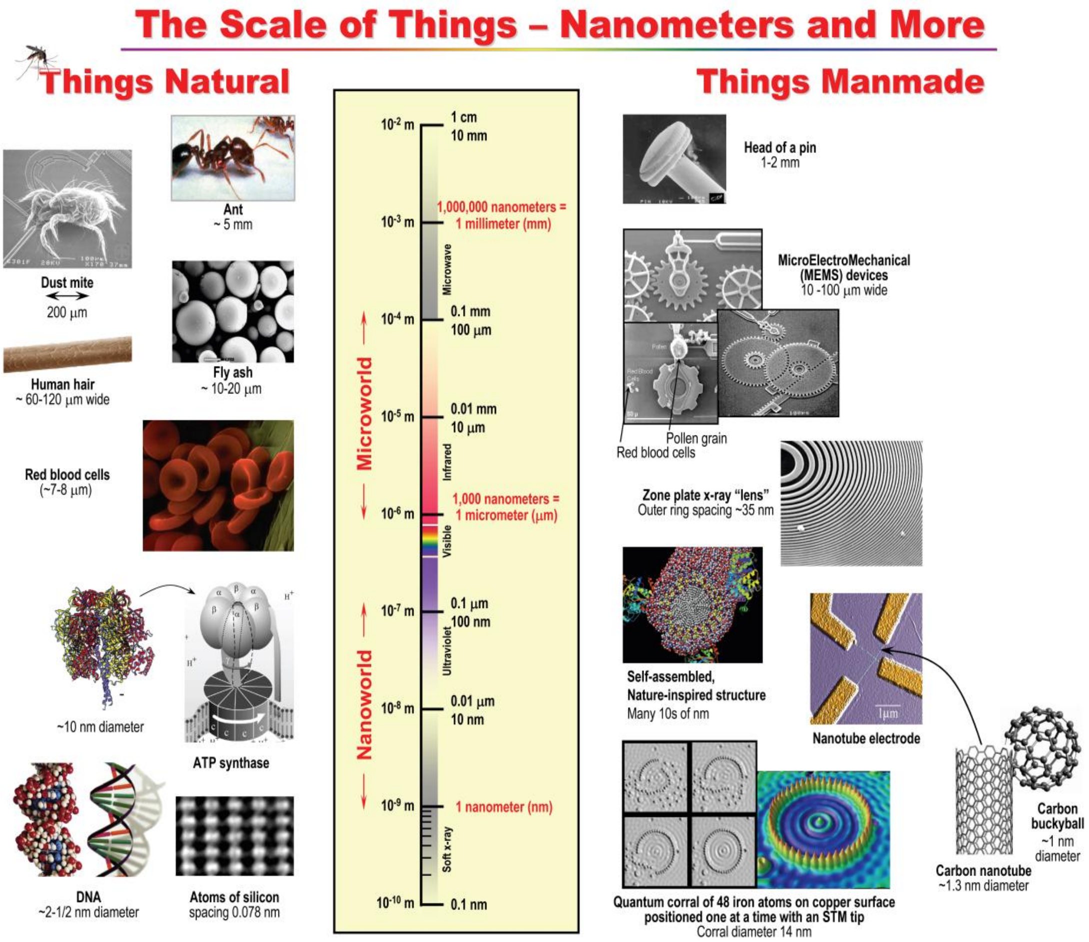

Figure 1.1 Nanometer scale chart- Natural thing are compared to man made things in term of dimensions (National Research Council, 2002).

The fabrication process of nanostructures on insulating materials such as quartz substrate has attracted the attention and interest of researchers in recent years due to its wide range of potential applications. Quartz has superior properties in hardness (7.0 Moh's), high thermal (melting point 1610 °C) and chemical stability. Quartz or silicon dioxide (SiO_2) is an abundant mineral made up of continuous framework of SiO_4 tetrahedra have piezoelectric properties, which is the ability to develop an electric potential upon the application of mechanical stress. EBL is one of the main direct write techniques available for high resolution pattern definition of nanostructure by exposing a radiation sensitive resist with electron beam.

Pattern transfer is a process of creating a functional pattern structure onto a substrate material and accomplished by an etching process that selectively removes unmasked portions of a layer (Cui, 2008). Plasma etching becomes an important technique for the pattern transfer process of nanostructures for years to come. Plasma is a partially ionized gas with a combination of free electrons, ions, radicals and natural species. The plasma is typically generated by a RF electric field. The principle behind plasma is the reactive species, which are formed by the dissociation of gases in plasma and then are adsorbed on the substrate surface where it react with the substrate atoms to form volatile compounds that are desorbed and pumped away. The most important step involved in the plasma etching processes is as illustrated in Figure 1.2. The passivation layer at the vertical surface technique is the most familiar mechanism to obtain an anisotropic etching process. Therefore, the ions bombardment mainly occurs on the horizontal surfaces.

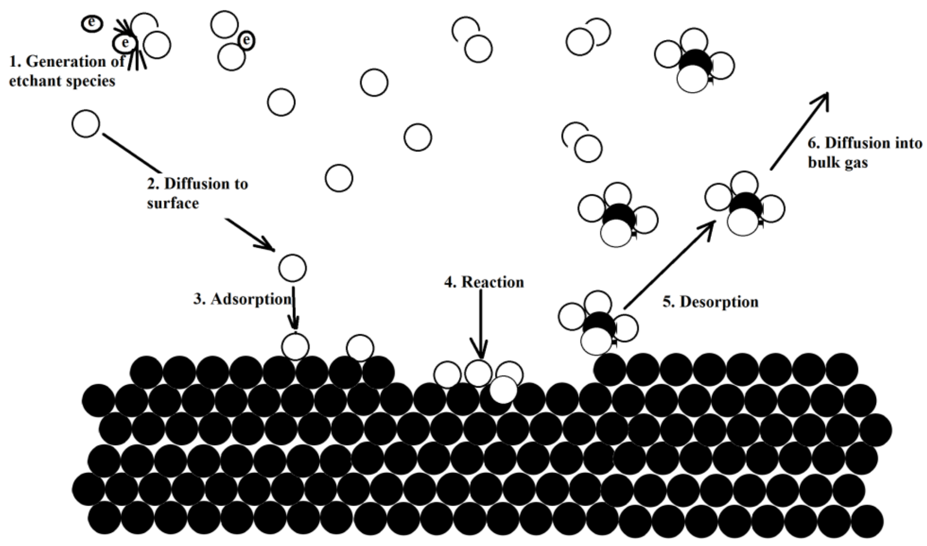

Figure 1.2 The primary process involved in a plasma etching (Madou, 2002).

One of the common methods in etching is the Bosch process. This process which based on alternating multiple steps of etching and sidewall passivation, is normally employed to achieve high aspect ratio nanostructures on silicon wafer. Figure 1.3 illustrates the Bosch process mechanism where the passivation layer is deposited onto the structures and the etching driven deeper into the trenches (Laermer *et al.*, 2010). Another technique is the cryogenic technique, where liquid nitrogen are used to cooling the substrate to a cryogenic temperature in order to achieve vertical sidewall of etched profiles. There are three basic etch requirements in order to achieve high aspect ratio etches, which are high etch rate, anisotropic etching and high etch selectivity on substrate than mask. Low etch selectivity limits the maximum etch depth and pattern transfer fails if etching occurs laterally. However, these processes are not suitable for fabrication on quartz substrate due to different chemical composition of quartz materials which leads to low etching rate and selectivity of etch mask.

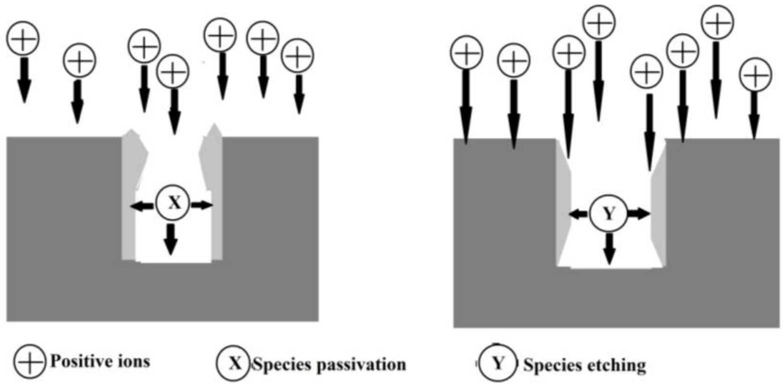

Figure 1.3 The basic mechanism of the Bosch etching process (Laermer *et al.*, 2010).

1.2 Problem Statement

There are numerous publications that have been reported for nanostructure fabrication on silicon substrates, but there are limited amount of information on fabricating insulating materials such as quartz and glass substrates. Mohamed (2009) developed a fabrication techniques for fabricating 2D and 3D nanostructures on quartz substrate. The patterns were directly written on the positive and negative tone photoresists using EBL tools, followed by the pattern transfer process using RIE technique. Surface charging is the most challenging part for pattern definition on top of insulating substrates surface using an EBL tools. The e-beam exposure traps and builds up charges on the surface of insulator substrates, which may deflect or distort the e-beam positioning, and may eventually create undesired effects. Therefore, a conductive polymer layer is employed to ground the trapped charges for minimizing the surface charging effects. Another suggested solution draining the charges to the ground, through thin metallic sputtering layer on top the substrate surface or a metallic coating on the photoresist layer to suppress the charging effects.

The reactive ion etching (RIE) method is usually utilized in process transfer of 2D or 3D structures due to the ability of directional etching. However, the results of the nanostructures pattern transfer process using the conventional RIE method are unpredictable. The theoretical understanding of the different mechanisms involved in plasma etching is also still very poor, although plasma etching is widely used in the industry. Without further research, the phenomena of chemical and physical reactions in plasma etching, the electrical interaction between the different particles, between electrically charged particles and electromagnetic fields are difficult to understand. ICP-RIE consists separate RF and ICP generators which provide separate control over ion energy and ion density. Therefore, combination of ICP with RIE