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# Charge dissipation layer optimisation for nano-scale electron-beam lithography pattern definition onto diamond

A.I.M. Greer *, D.A.J. Moran

School of Engineering, Rankine Building, University of Glasgow, Glasgow, G12 8QQ, United Kingdom

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ABSTRACT

This paper demonstrates that the pattern feature size achieved for electron beam lithography (EBL) on diamond substrates can be minimised through optimisation of the thickness of a surface deposited metallic discharge layer. The purpose and benefits of a charge dissipation layer are presented and the subsequent trade-off with feature size examined. 5 nm of Al is demonstrated to be the optimum thickness of charge dissipation layer for polymethyl methacrylate (PMMA) resist on polycrystalline diamond as the feature size retains a similar variance to thicker layers, has good reproducibility and ultimately produces the smallest feature sizes. PMMA can be used as either a metal deposition mask, or an etch mask for $SiO_2$ which in turn can be used as an etch mask for diamond. Using this process we have demonstrated pattern transfer and metallisation of features onto diamond and $SiO_2$ coated diamond down to a dimension of 20 nm.


## 1. Introduction

Diamond is a material which is finding an ever growing variety of applications within nano-technology for applications such as nano-imprint lithography [1], biological implant coatings and interfaces [2], particle sensing [3] and electronics [4]. Electron-beam lithography (EBL) is an essential step in producing the required feature size for many of these applications. In its intrinsic form, diamond is essentially an insulator due to its large band-gap of 5.45 eV [5], so is unable to effectively dissipate introduced charge. During EBL utilising diamond substrates, charge may build up on the surface, leading to deflection of the electron beam (e-beam), causing the pattern written in the resist to differ from the design [6]. A common method of alleviating this problem is to deposit a layer of electrically conductive material known as a charge dissipation layer (CDL) on top of the electron-sensitive resist [7], shallow enough for electrons to penetrate and pattern the resist below but thick enough to easily dissipate charge. Due to radial beam divergence, focusing an electron-beam on the surface of a sample with a CDL results in the thickness and composition of the CDL distorting the effective spot size experienced by the resist and modifying the desired pattern feature size and shape [8]. Little evidence has been reported on the impact of this process on ultra-nano feature pattern potential on diamond. The objective of this work was to determine the optimum thickness of CDL for ultra-nano pattern transfer using polymethyl methacrylate (PMMA) resist masks on polycrystalline diamond.

Pattern transfer onto a substrate by EBL is typically performed by one of two processes following resist development; etching, whereby the resist acts as a mask to protect the substrate surface from an etch chemistry [9], and deposition and lift-off of material onto the surface of the substrate [10]. Metals are deposited in this manor for a range of applications on diamond and can also be used as a mask [11] or catalyst [12] for etching. PMMA is readily attacked by traditional diamond dry etch recipes [13], so is not typically used as a dry etch mask itself. For this work PMMA is instead utilised as a deposition and lift-off mask for metal features onto the diamond surface. In this instance the metal features act purely as an indicator of the dimensions and shape of the resist profile at the interface with the diamond substrate, allowing for inspection of potential distortion of or deviation from the designed pattern. In addition to pattern transfer via etching, the ability to produce nano-scale metallic contacts onto diamond is vital for devices such as high frequency field effect transistors [14] and quantum probes [15].

## 2. Material and method

A 580 μm thick, 10 mm × 10 mm, polycrystalline diamond substrate with ~50 μm grain size sourced from Element Six was used as the substrate material for this series of experiments. The sample was initially subjected to acid (HF) and solvent cleaning. Thereafter the roughness was confirmed to be 0.5 nm Ra with an atomic force microscope (AFM). PMMA was then spin-coated on top of the sample as a bi-layer with undercut to aid lift-off of deposited material onto the diamond surface.

Al is often used as a CDL for electron-beam patterning of insulating substrates because of its good electrical conductivity, its relative low

* Corresponding author.
E-mail address: a.greer.1@research.gla.ac.uk (A.I.M. Greer).

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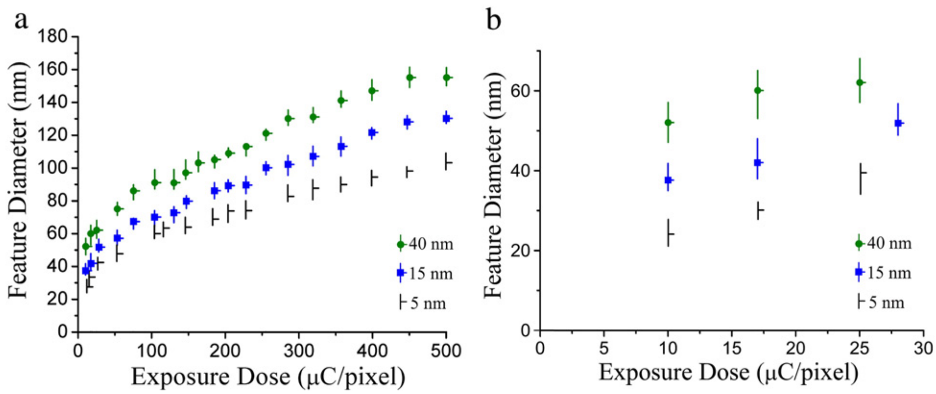


**Fig. 1.** (a) A comparison plot of feature diameter (nm) against dose (μC/pixel) for three samples containing an Al CDL on PMMA on diamond. The bottom contour is the 5 nm thick version, the middle one 15 nm and the top contour 40 nm. (b) An expansion of the first 30 μC/pixel results. The vertical lines for all plots represent error bars covering 8 measurements per inspected dose.

cost and its availability in many fabrication facilities. Previous work has reported the use of a 40 nm thick Al layer for the purpose of a CDL [16]. Therefore 40 nm of Al was adopted as an initial thickness for experimentation. This was then reduced to investigate the resulting impact on EBL defined pattern feature size and shape, and the point at which the Al layer ceases to operate efficiently as a CDL. This data was also compared with a control run utilising a sample without any CDL. The Al was deposited on top of the PMMA using an electron-beam metal evaporator.

Pattern transfer into the PMMA/Al stack was achieved using a Vistec Gaussian Vector Beam 6 (VB6) EBL tool with 12 nm beam spot size and operating at 100 kV accelerating voltage. The pattern design consisted of a square matrix of size 150 μm of single point exposures at 305 nm pitch. Since features were created with single point exposures, proximity effects were not a concern. The design was repeated on the sample for a range of doses from 10 to 500 μC/pixel. Doses below 10 μC/pixel were not examined as this level approached the underexposure region and feature sizes were reaching a scale where the processing environment needs to be kept meticulously consistent for accurate statistical comparison. Pixels were square with length 305 nm. After patterning the resist, the Al CDL was removed with Tetramethylammonium hydroxide. The PMMA was then developed in MIBK:IPA solution. Following development, an oxygen barrel asher was used to remove ~10 nm of residual PMMA. It was important to ensure that the developed patterns in the PMMA penetrated all the way through the resist to the surface of the substrate. In order to confirm the shape and depth of the exposed features at the PMMA/substrate interface, 60 nm of gold was evaporated using a metal evaporator. The gold coated PMMA was then lifted-off with acetone, leaving 60 nm high Au features. The gold features were then inspected using a Scanning Electron Microscope (SEM). To resolve the resultant metalized features and minimise charging of the diamond substrate within the SEM, a 5 nm layer of Au was evaporated over the sample surface. Analyses of the features involved capturing images for a range of doses across the sample after each run and processing them with IMAGEJ software [16]. For each dose, multiple sites were examined and the circularity and diameter of the point exposures measured. The feature diameter was recorded on a maximum, minimum, average graph against exposure dose (Fig. 1). The average diameter was calculated using Eq. (1) (used to calculate average feature size, where '*n*' is the total number of sites, '*i*' refers to a site and '*a*' is the value of a site).

$$Ave = \frac{1}{n}\sum_{i=1}^{n}(a_i) \qquad (1)$$

An alternative approach for nano-patterning diamond with PMMA is to first use the PMMA as an etch mask to transfer a pattern into a $SiO_2$ coating deposited on the diamond surface, which then itself acts as a mask for reactive ion etching [17]. Unlike metal mask layers, $SiO_2$ is not a good electrical conductor; diamond with a $SiO_2$ coating also requires the incorporation of a CDL during EBL. To investigate the impact of the presence of such a dielectric layer between the diamond and electron sensitive resist, the experiment was repeated with ~120 nm of chemical vapour deposited $SiO_2$ beneath the PMMA.

## 3. Results and discussion

### *3.1. Effect of Al CDL thickness on PMMA above diamond*

As an initial reference point, 40 nm of Al proved to be an effective CDL with average feature size plotted against dose following a clear contour. The maximum variance in feature size was no greater than 13 nm for all inspected sites. This data is presented in Fig. 1. In addition, the feature geometry remained circular across the range of doses tested. At the lowest dose examined (10 μC/pixel) the average feature size was 52 nm (minimum size = 47 nm), and at the largest dose (500 μC/pixel) the average feature size was 155 nm.

The CDL thickness was then decreased to 15 nm and the resultant feature dimensions compared with the 40 nm Al CDL results. It can be seen from Fig. 1 and Table 1 that the feature sizes obtained by reducing the CDL thickness are smaller for all tested doses. The smallest features had an average diameter of 38 nm, compared to 52 nm for the thicker CDL. The maximum deviation was also smaller at 12 nm and the feature shape maintained good circularity across the range of doses tested.

The CDL was then thinned down from 15 nm to 5 nm and again the resultant features were inspected at varied exposure dose sites. The results followed a steady contour and was similar to that of the 15 nm layer; however a shallower gradient for the average feature size versus exposure dose slope was observed. For exposures below 20 μC/pixel the features begin to lose true circularity because even small defects around the perimeter become more relative as the feature size approaches 20 nm. Although the reproducibility of the exact

**Table 1**
Summary of key feature size statistics for the three effective CDL thicknesses tested (40 nm, 15 nm and 5 nm) on diamond.

| CDL thickness | Average feature size at 500 μC/pixel | Average feature size at 10 μC/pixel | Minimum feature size at 10 μC/pixel | Maximum deviation of feature size at any dose |
|---|---|---|---|---|
| 40 nm | 155 nm | 52 nm | 47 nm | 13 nm |
| 15 nm | 130 nm | 38 nm | 35 nm | 12 nm |
| 5 nm | 103 nm | 24 nm | 21 nm | 12 nm |

CDL = charge dissipation layer, PMMA = polymethyl methacrylate, IPA = isopropyl alcohol, EBL = electron-beam lithography, MIBK = methyl isobutyl ketone, HF = hydrofluoric.