Deterministic Fabrication of Micro- and Nano-Structures
by Focused Ion Beam

Jining SUN

Submitted for the degree of Doctor of Philosophy

Heriot-Watt University
School of Engineering and Physical Sciences
January 2012

The copyright in this thesis is owned by the author. Any quotation from
the thesis or use of any of the information contained in it must
acknowledge this thesis as the source of the quotation or information.
Abstract

The micro- and nano-structures of material are some of the most important microscopic characteristics that govern macroscopic behaviour in terms of physical properties. To best use and fully benefit from these structures, it is important to properly develop deterministic fabrication techniques that can achieve these structures with high accuracy. Recent developments in the technology of focused ion beam (FIB) has led to beam sizes ranging from 400nm to 3.5nm, which makes FIB machining an idea tool for both micro and nano fabrication. This work aims at developing a deterministic fabrication approach to accurately obtain predicable micro- and nano-structures and develop proper solutions to overcome the challenges in FIB machining, including dimensional, geometrical material and machining efficiency challenge.

The deterministic approach is implemented through the development of a surface topography model which predicts the surface generated during FIB machining process and a divergence compensation approach which is used to obtain fabrication parameters to compensate for the predicted machining error. Ion beam machining process is characterized by an ion-solid sputtering model. In this work, the sputter yield and sputtered atoms’ distribution are evaluated using a Monte Carlo simulation with the simulation of the dynamic surface generation realised by the level set method.

The effectiveness of the developed fabrication approaches has been fully demonstrated through a number of case studies which show that the surface topography of the machined material can be precisely predicted; the divergence compensation method can reduce the machining error and the machined surface form accuracy can be dramatically improved. Subsequent measurement of the actual fabricated micro- and nano-structures using the settings provided by the model helps to overcome the existing dimensional, geometrical machining efficiency and machinability challenges in FIB machining.
Acknowledgements

I am indebted to many for their support during both the research and the writing of this thesis. First and foremost, I am deeply indebted to my supervisor Dr. Xichun Luo for his guidance, attention, encouragement and patience throughout the years. I appreciate Prof. James Ritchie for his valuable suggestions on my work.

I would also like to thank Dr. Tomas Hrncir from Tescan s.r.o for his great help with the demonstration of FIB system and his suggestions on my research. I thank Dr. Chengge Jiao, Mr. David Beamer, Mr. John Martin and Mr. Alan Shore from FEI (UK) Ltd. for their training and continuing maintenance of the FIB system. I am grateful to Mrs. Marian Miller from Heriot-Watt University, who helped me throughout the research with SEM and AFM. I am also grateful to Mr. Mark Leonard who helped me throughout the electron beam evaporation and white light interferometer. I would also acknowledge Mr. Jun Li for the testing work of the optical fibres.

Many thanks to Mr. Andrew Cox from Contour Fine Tooling Ltd. and Mr. Ruiting Jian from MIRDC in Taiwan who generously supplied single crystal diamond tools for this research.

I also acknowledge contributions from Dr. Will Shu from Heriot-Watt University, Prof. Tieshuan Fan from Peking University in China, Mr. Jiacheng Hu from National Institute of Metrology in China, Dr. Xiaoye Du from Cambridge University, Mr. Lin Chen from King’s College London and Dr. Jiaran Qi from Aalto University for their support and encouragement. Without your continuing support the completion of the project would not have been possible.

I would also like to thank Heriot-Watt University and the School of Engineering and Physical Sciences for the financial support which made this research possible.

I am very grateful to my family, especially to my parents, my wife Mrs. Dongxue Li and my lovely daughter Tongtong.

Last but not least, I also appreciate all my friends in Heriot-Watt University. Thank you all for a pleasant time. You are the treasure in my life.
---

**Declaration Statement**

**ACADEMIC REGISTRY**

**Research Thesis Submission**

<table>
<thead>
<tr>
<th>Name:</th>
<th>Jining Sun</th>
</tr>
</thead>
<tbody>
<tr>
<td>School/PGI:</td>
<td>School of Engineering and Physical Sciences</td>
</tr>
<tr>
<td>Version:</td>
<td>Final</td>
</tr>
<tr>
<td>Degree Sought</td>
<td>PhD Mechanical Engineering</td>
</tr>
<tr>
<td>(i.e. First, Resubmission, Final)</td>
<td>Degree and Subject area</td>
</tr>
</tbody>
</table>

**Declaration**

In accordance with the appropriate regulations I hereby submit my thesis and I declare that:

1) the thesis embodies the results of my own work and has been composed by myself
2) where appropriate, I have made acknowledgement of the work of others and have made reference to work carried out in collaboration with other persons
3) the thesis is the correct version of the thesis for submission and is the same version as any electronic versions submitted*
4) my thesis for the award referred to, deposited in the Heriot-Watt University Library, should be made available for loan or photocopying and be available via the Institutional Repository, subject to such conditions as the Librarian may require
5) I understand that as a student of the University I am required to abide by the Regulations of the University and to conform to its discipline.

* Please note that it is the responsibility of the candidate to ensure that the correct version of the thesis is submitted.

**Signature of Candidate:**

**Date:**

---

**Submission**

**Submitted By (name in capitals):** JINING SUN

**Signature of Individual Submitting:**

**Date Submitted:**

---

**For Completion in the Student Service Centre (SSC)**

**Received in the SSC by (name in capitals):**

**Method of Submission**

(Handed in to SSC; posted through internal/external mail):

**E-thesis Submitted (mandatory for final theses)**

**Signature:**

**Date:**

---

III
Table of Contents

Chapter 1 Introduction ................................................................................................... 1
  1.1 Background .......................................................................................................... 1
  1.2 Aim and objectives .............................................................................................. 4
  1.3 Structure of the thesis .......................................................................................... 5

Chapter 2 Fundamentals of FIB Machining ..................................................................... 7
  2.1 The FIB instrument .............................................................................................. 7
    2.1.1 Liquid metal ion source ................................................................................. 8
    2.1.2 Ion column ..................................................................................................... 9
  2.2 Applications of FIB technology ......................................................................... 12
    2.2.1 Imaging ....................................................................................................... 13
    2.2.2 Micro- and nano-machining ....................................................................... 15
    2.2.3 Deposition ................................................................................................... 15
  2.3 The FIB machining mechanism ......................................................................... 17
    2.3.1 Scattering .................................................................................................... 18
    2.3.2 Binary collision approximation .................................................................. 21
    2.3.3 Sputtering and redeposition ........................................................................ 23
  2.4 Key fabrication parameters in FIB machining ................................................... 25
    2.4.1 Sputter yield ................................................................................................ 25
    2.4.2 Scanning strategy ........................................................................................ 31
    2.4.3 Beam diameter ............................................................................................ 32
    2.4.4 Pixel spacing ............................................................................................... 33
    2.4.5 Scanning passes .......................................................................................... 35
  2.5 Processing methods in FIB micro machining .................................................... 36
    2.5.1 Surface topography prediction .................................................................... 37
    2.5.2 Fabrication parameters prediction ............................................................. 39
  2.6 Challenges in FIB machining ............................................................................ 42
  2.7 Summary ............................................................................................................ 44

Chapter 3 The Development of a Deterministic Three-Dimensional FIB Machining Approach ............................................................................................................. 45
  3.1 The prediction of surface topography evolution in FIB machining ............ 45
Table of Contents

3.1.1 The surface generation mechanism in FIB milling process .................. 45
3.1.2 Surface topography simulation by level set method .......................... 47
3.2 The determination of machining parameters for obtaining high precision micro- and nano-structures .......................................................................................................................... 54
  3.2.1 The divergence compensation method ................................................. 54
  3.2.2 The implementation of the divergence compensation method in FIB machining ................................................................................................................. 58
3.3 Summary ............................................................................................................ 61

Chapter 4 Deterministic Fabrication of Nanodot Arrays ................................. 63
— A case study to overcome the dimensional challenge in FIB machining ....... 63
  4.1 Introduction ........................................................................................................ 63
  4.2 Deterministic fabrication of silicon mould for nanodot arrays .................. 64
    4.2.1 Determination of sputter yield ................................................................ 64
    4.2.2 Determination of angular distribution of sputtered atoms ..................... 66
    4.2.3 Determination of sticking coefficient .................................................... 70
    4.2.4 Simulation of surface topography evolution ....................................... 70
  4.3 Experimental validation ..................................................................................... 74
  4.4 Deterministic fabrication of a silicon mould ................................................. 75
  4.5 Investigation of the redeposition effect .......................................................... 76
  4.6 Summary ............................................................................................................ 78

Chapter 5 The Deterministic Fabrication of Three-Dimensional Structures ...... 79
— Case studies to overcome the geometric challenge in FIB machining ............ 79
  5.1 Introduction ........................................................................................................ 79
  5.2 Deterministic fabrication of three-dimensional structures ........................ 79
    5.2.1 Determination of fabrication parameters ............................................ 80
    5.2.2 Investigation of scanning strategy ....................................................... 81
    5.2.3 Experimental results and discussions .................................................. 83
    5.2.4 Optimization of the divergence compensation method ....................... 89
  5.3 Deterministic fabrication of optical sensors ................................................. 91
    5.3.1 Fibre-top cantilever and fibre-side cantilever ...................................... 91
    5.3.2 Fabrication of fibre cantilevers by FIB ............................................... 92
  5.4 Summary ............................................................................................................ 95
Chapter 6 Deterministic Fabrication of Nanoscale Diamond tools
— A case study to overcome the material and machining efficiency challenge in FIB machining

6.1 Introduction

6.2 Challenges in fabrication of nanoscale SCD tools by FIB

6.3 Solutions

   6.3.1 Measures to form a sharp cutting edge
   6.3.2 Solution to reduce Ion beam drifting
   6.3.3 Solution to reduce ripples generated on the diamond surface
   6.3.4 Solution to overcome the redeposition effect
   6.3.5 Evaluation of the beam tail effect

6.4 Fabrication of nanoscale diamond tool

   6.4.1 Sample preparation
   6.4.2 Three-dimensional deterministic fabrication of a diamond tool

6.5 Results and discussion

6.6 Summary

Chapter 7 Conclusions and Recommendations for Future Work

7.1 Research Assessment

7.2 Conclusions

7.3 Recommendations for future work

References

Appendices

Appendix I

Essential specification of FEI Quanta 3D FEG FIB system

Appendix II

Gallium ion sputter yield on silicon substrate

Gallium ion sputter yield on diamond substrate
Nomenclature

$A_{cs}$ the cross sectional area on the target surface ($\mu m^2$)

$A_i$ the actual area on the target surface ($\mu m^2$)

$C$ the coefficient matrix

$d$ the distance travelled between collisions ($\mu m$)

$d_f$ the beam diameter (nm)

$d\Omega$ the infinitesimal emission solid angle of the sputtered atoms

$dA$, $dA'$ the infinitesimal areas on the target surface

$D^{+x}$ right-hand derivative $D^{+x}\phi^n$

$D^{-x}$ left-hand derivative $D^{-x}\phi^n$

$D^{+y}$ right-hand derivative $D^{+y}\phi^n$

$D^{-y}$ left-hand derivative $D^{-y}\phi^n$

$e$ the charge of a single electron ($1.602 \times 10^{-19}$ C)

$E$ the kinetic energy of the incident ion in the laboratory system (J)

$E_c$ the kinetic energy of the incident ion in the centre-of-mass system (J)

$\Delta E_e$ the electronic losses of the incident ions (eV)

$f$ ion beam current distribution

$f(\alpha)$ the angular distribution of sputtered atoms

$f_{mr}$ a function of the mass ratio of the incident ion and the target atom

$F_{direct}$ the flux of sputtered atoms caused by the incident ion beam directly (atoms/$\mu m^2$/s)

$F_{indirect}$ the flux of redeposition atoms (atoms/$\mu m^2$/s)

$F_{total}$ the total sputtering flux (atoms/$\mu m^2$/s)

$h$ the overlap of the two adjacent beam spots (m)

$H$ the number of simulation histories

$I$ the ion beam current (nA)

$i, j, k, l$ the indicators of the pixel position

$J(x, y)$ the ion flux density at a point $(x, y)$

$L$ the total length of beam scanning route in each scanning pass (m)

$m_1$, $m_2$ the mass of the incident ion (kg)

$m_2$ the mass of the target particle (kg)

$M$ the pixel number along the X and the Y directions in the bitmap
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>MRR</td>
<td>the material removal rate ($\mu m^3/s$)</td>
</tr>
<tr>
<td>$n$</td>
<td>the atoms density of the substrate (atoms/$\mu m^3$)</td>
</tr>
<tr>
<td>$_n$</td>
<td>the normal direction of the moving front</td>
</tr>
<tr>
<td>$N$</td>
<td>the pixel number along the X and the Y directions in the bitmap</td>
</tr>
<tr>
<td>$p$</td>
<td>the impact parameter in ion-solid interaction which represents the effective interaction distance for collision between two atoms</td>
</tr>
<tr>
<td>$p_x$</td>
<td>the pitch along the X direction (nm)</td>
</tr>
<tr>
<td>$p_y$</td>
<td>the pitch along the Y direction (nm)</td>
</tr>
<tr>
<td>$p_x/df$</td>
<td>the normalized pixel spacing</td>
</tr>
<tr>
<td>$r_{min}$</td>
<td>the closest approach during the sputtered atoms collision (m)</td>
</tr>
<tr>
<td>$sp$</td>
<td>the number of scanning pass</td>
</tr>
<tr>
<td>$S_c$</td>
<td>the sticking coefficient of the sputtered atoms</td>
</tr>
<tr>
<td>$S_e$</td>
<td>the electronic stopping cross section ($m^2$)</td>
</tr>
<tr>
<td>$S_{cs}$</td>
<td>the cross section area of each nano-structure ($\mu m^2$)</td>
</tr>
<tr>
<td>$S_n(E)$</td>
<td>the elastic stopping power (eV/m)</td>
</tr>
<tr>
<td>$t$</td>
<td>the dwell time (ms)</td>
</tr>
<tr>
<td>$t_{total}$</td>
<td>the total machining time (ms)</td>
</tr>
<tr>
<td>$\Delta t$</td>
<td>the time step in level set method (ms)</td>
</tr>
<tr>
<td>$T$</td>
<td>the kinetic energy of the target particle</td>
</tr>
<tr>
<td>$U_s$</td>
<td>the atom binding energy (eV)</td>
</tr>
<tr>
<td>$v_{ext}$</td>
<td>the extension velocity in the whole domain of level set function ($\mu m/s$)</td>
</tr>
<tr>
<td>$v_c$</td>
<td>the cutting speed of nano-SCD tool ($\mu m/s$)</td>
</tr>
<tr>
<td>$v_s$</td>
<td>the beam scanning speed (m/s)</td>
</tr>
<tr>
<td>$v_{\perp}$</td>
<td>the velocity which is perpendicular to the moving front ($\mu m/s$)</td>
</tr>
<tr>
<td>$V$</td>
<td>the volume of the sputtered cavity ($\mu m^3$)</td>
</tr>
<tr>
<td>$V(r)$</td>
<td>the inter-atomic potential (J)</td>
</tr>
<tr>
<td>$Y$</td>
<td>the sputter yield (atoms/ion)</td>
</tr>
<tr>
<td>$Z_1$</td>
<td>the atomic number of the incident ion</td>
</tr>
<tr>
<td>$Z_2$</td>
<td>the atomic number of the target atom</td>
</tr>
<tr>
<td>$\Delta Z$</td>
<td>the milling depth (nm)</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>the factor that depends only on the efficiency of energy transfer</td>
</tr>
<tr>
<td>$\phi$</td>
<td>level set function</td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
</tr>
<tr>
<td>--------</td>
<td>-------------</td>
</tr>
<tr>
<td>$\Phi$</td>
<td>the ion flux density (ions/s/nm$^2$)</td>
</tr>
<tr>
<td>$\Gamma$</td>
<td>the initial level set function</td>
</tr>
<tr>
<td>$\eta$</td>
<td>the atom density of silicon (atoms/nm$^3$)</td>
</tr>
<tr>
<td>$\theta$</td>
<td>the ion incident angle (degree)</td>
</tr>
<tr>
<td>$\vartheta$</td>
<td>the scatter angle in the centre-of-mass system (degree)</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>the standard deviation of the Gaussian distribution</td>
</tr>
<tr>
<td>$(x_p, y_p)$</td>
<td>the centre coordinate of the incident ion beam</td>
</tr>
<tr>
<td>$\Omega$</td>
<td>the emission solid angle of the sputtered atoms (sr)</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Full Form</td>
</tr>
<tr>
<td>--------------</td>
<td>-----------------------------------------------</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic Force Microscope</td>
</tr>
<tr>
<td>ASE</td>
<td>Amplified-Spontaneous-Emission</td>
</tr>
<tr>
<td>BCA</td>
<td>Binary Collision Approximation</td>
</tr>
<tr>
<td>CAD</td>
<td>Computer Aided Design</td>
</tr>
<tr>
<td>CAM</td>
<td>Computer Aided Manufacturing</td>
</tr>
<tr>
<td>DAC</td>
<td>Digital-to-Analog Converter</td>
</tr>
<tr>
<td>FEG</td>
<td>Field Emission Gun</td>
</tr>
<tr>
<td>FIB</td>
<td>Focused Ion Beam</td>
</tr>
<tr>
<td>FWHM</td>
<td>Full Width at Half Maximum</td>
</tr>
<tr>
<td>GDSII</td>
<td>Graphic Database System II</td>
</tr>
<tr>
<td>GIS</td>
<td>Gas Injection System</td>
</tr>
<tr>
<td>HFW</td>
<td>Horizontal Field Width</td>
</tr>
<tr>
<td>IC</td>
<td>Integrated Circuit</td>
</tr>
<tr>
<td>LMIS</td>
<td>Liquid Metal ion Source</td>
</tr>
<tr>
<td>MCP</td>
<td>Multichannel Plate</td>
</tr>
<tr>
<td>MEMS</td>
<td>Micro Electro Mechanical System</td>
</tr>
<tr>
<td>NIL</td>
<td>Nanoimprint Lithography</td>
</tr>
<tr>
<td>PDMS</td>
<td>Polydimethylsiloxane</td>
</tr>
<tr>
<td>SCD</td>
<td>Single Crystal Diamond</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>SIMS</td>
<td>Secondary Ion Mass Spectrometry</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission Electron Microscope</td>
</tr>
<tr>
<td>µCP</td>
<td>Micro Contact Printing</td>
</tr>
</tbody>
</table>
List of Publications from this work

Journal Papers:


Conference Papers:


List of Publications from this work


Chapter 1 Introduction

1.1 Background
Nowadays, micro- and nano-technology are undoubtedly at the frontier of both scientific and engineering research. Great endeavours on the design and fabrication of micro- and nano-structures on functional materials are being made for innovative applications. Micro- and nano-structures on the surface can affect material mechanical properties such as strength, toughness, ductility, hardness, corrosion resistance, adhesion and abrasive wear resistance [1-8]. It has also been found that micro- and nano-structures can affect the energy absorption on the surface of materials [9]. In the science of photonics, micro- and nano-structures in lens, gratings and photonic crystals are vital to obtain unique optical phenomena and enhance optical performance, and are therefore necessary for emerging applications. A typical representative application is the plasmonic waveguide which is a key component in the development of sub-wavelength resolution optical systems [10-11]; the size and shape of the nano structures on the waveguide are essential to excite the surface plasmon resonance. Indeed, the poor form accuracy of such micro- and nano-structures in these applications is a performance bottleneck.

Due to the broad applications of micro- and nano-structures in multiple disciplines, the capability of fabricating highly accurate repeatable micro- and nano-structures on different materials is essential for modern science and engineering research. Innovative techniques and processing methods are continually developed to meet the demand of current research and industrial progress. Some dominant micro- and nano-fabrication techniques that are currently used to obtain micro- and nano-structures are listed in Table 1-1. Optical lithography and electron beam lithography are enabling technologies for fabricating structures over a large area but only two-dimensional structures can be obtained. Scanning probe lithography is a machining approach in which a stylus is moved mechanically across a substrate surface to form a pattern but this method is limited by low throughput and only materials that are softer than the stylus can be machined. High throughput machining technologies such as single point diamond turning are adopted in micro- and nano-fabrication but the minimum line width that can be obtained is limited by the dimension of the cutting tools. Femtosecond laser machining is another state-of-the-art technique in micro- and nano-fabrication but the
surface roughness is about 10 times higher than the above mentioned techniques. A three-dimensional fabrication technique which can overcome the limitations of both lithography fabrication technologies and high throughput machining technologies is in required. One important tool that has successfully met such a demand and has the potential to continue to meet future demands in both micro- and nano-manufacture is focused ion beam (FIB) machining technology. Fundamentally, an FIB system produces and directs a beam of high energy ions, focusing them onto a sample for the purpose of both machining and imaging. Comparing with electron beam lithography, FIB is much easier to remove surface atoms from their positions due to the larger ions’ mass which also yields greater material removal rates. It is a direct writing technique that requires no mask during the fabrication process. Even more important, FIB machining enables fabricating both two-dimensional and three-dimensional micro- and nano-structures.
Table 1-1 Comparison of different micro- and nano-fabrication technologies

<table>
<thead>
<tr>
<th>Micro/nano machining technologies</th>
<th>Machining capability</th>
<th>Resolution (nm)</th>
<th>Minimum line width (nm)</th>
<th>Machinable material</th>
<th>Material removal rate</th>
<th>Minimum surface roughness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scanning probe lithography</td>
<td>3D</td>
<td>10 [17]</td>
<td>~ 10 [17]</td>
<td>Soft solid material</td>
<td>$10^3 \sim 10 \mu$m$^3$/s</td>
<td>~ 0.1</td>
</tr>
<tr>
<td>Focused ion beam machining</td>
<td>3D</td>
<td>5 [18]</td>
<td>~ 10 [18]</td>
<td>Almost any solid material</td>
<td>$10^{-2} \sim 10^3 \mu$m$^3$/s</td>
<td>0.1 [19]</td>
</tr>
<tr>
<td>Femtosecond laser machining</td>
<td>3D</td>
<td>~ 10 [20]</td>
<td>~ 10 [20]</td>
<td>Almost any solid material</td>
<td>$10 \sim 10^7$ nm/pulse</td>
<td>1.5 [22]</td>
</tr>
<tr>
<td>Single point diamond machining</td>
<td>3D</td>
<td>0.1 [23]</td>
<td>~ $10^4$ [24]</td>
<td>Materials that are chemically stable with diamond</td>
<td>$10^8 \sim 10^9 \mu$m$^3$/s</td>
<td>0.1 [25]</td>
</tr>
</tbody>
</table>
Chapter 1     Introduction

The primary use of FIB is in micro- and nano-machining for fabrication, analysis or repair on a sample at the micro and nano scale. It is capable of building two-dimensional and three-dimensional structures on almost any kind of solid material. Combined with other techniques such as nanoimprint lithography (NIL) and micro-contact printing (µCP), these structures can be duplicated in a cost effective way. However, in the FIB machining process the shape of the machined structures is difficult to precisely control. The major causes of these difficulties are the redeposition effect, the variation of sputter yield and the limitation of ion beam spot size. Theoretical and technical measures need to be developed to overcome the complications of FIB machining and the achievement of this will provide this whole area of research a totally new perspective.

1.2    Aim and objectives

The overall aim of this project is to develop a deterministic fabrication approach for obtaining micro- and nano-structures using FIB machining. This approach will overcome the challenges of FIB machining to achieve high form accuracy and high machining efficiency for the machined structures.

This work has four main research objectives:

- To study the influence of fabrication parameters on the accuracy of the machined structures.

- To develop a modelling and simulation approach to accurately predict the generation of surface topography in FIB machining.

- To develop a divergence compensation method to predict and obtain fabrication parameters to compensate machining error for given structures.

- To implement the deterministic fabrication approach, based on the surface topography model and divergence compensation approach, to overcome the existing FIB machining challenges including dimension, geometric, material, and machining efficiency.
Chapter 1  Introduction

1.3 Structure of the thesis

This thesis is presented in seven chapters.

Chapter 1 presents the background of this research project and explains the motivation and objectives of the work.

Chapter 2 summarizes the fundamentals of FIB technology and the current fabrication methods developed by pioneer researchers. It also reveals several remaining challenges in FIB machining, including dimension, geometry, material, and machining efficiency.

Chapter 3 introduces the modelling and simulation methods which provide a foundation for the proposed deterministic fabrication process. It includes the modelling approach for generation of surface topography of FIB machined structures based on the input fabrication parameters. The approach for determination of FIB fabrication parameters from given structures is also presented in this chapter.

Chapter 4 to chapter 6 present a series of research case studies which overcome the challenges in FIB machining using simulation methods.

Chapter 4 provides a case study which applies the surface topography simulation to overcome the dimensional challenge in FIB machining. Surface topography simulation based on level set method is used to predict the surface generation in the FIB machining of silicon moulds used for NIL. The redeposition effect is considered in the simulation model as is its contribution to this.

Chapter 5 addresses the geometric challenge in FIB machining. Three dimensional structures are fabricated precisely on silicon substrate. Guidance for optimizing the fabrication parameters is introduced with this method further applied in a case study demonstrating the fabrication of optical fibre sensors.

Chapter 6 proposes a novel method to overcome the machining efficiency challenge. Single crystal diamond tools with nano-structures sculpted on the tool tip are made through FIB machining. The surface topography simulation method and the divergence compensation method are both applied to optimize the fabrication parameters.
Chapter 7 draws conclusions resulting from this research and recommendations are made for future work.
Chapter 2  Fundamentals of FIB Machining

Over the past decades, the enhanced resolution of single ion beam systems and the introduction of dual-beam FIB systems have enabled the wide application of FIB for the fabrication of micro- and nano-structures. In order to further improve FIB machining accuracy, significant efforts have been made to understand the FIB material removal process and its relevant phenomenon. Subsequently, sophisticated processing methods have been developed. This chapter reviews the fundamentals of the FIB machining technique with regard to hardware, applications, mechanisms and methods for the fabrication of three-dimensional structures. The current challenges remaining in FIB machining are also identified.

2.1  The FIB instrument

The world first FIB instrument based on field emission technology was developed by Levi-Setti [26] and Orloff and Swanson [27]. Fundamentally, an FIB instrument produces and directs a focused beam of ions which strike onto a sample surface for the purposes of etching or milling; this can also be used to apply sample surface imaging. The FIB instrument takes the form of a stand-alone single beam system. Alternatively, the ion beam column can be incorporated into other analytical or measurement systems such as a scanning electron microscope (SEM), a transmission electron microscope (TEM) or a secondary ion mass spectrometry (SIMS). Nowadays, the most widely used FIB instrument is the FIB/SEM dual beam system. As illustrated schematically in Figure 2-1, an FIB/SEM dual beam system consists of a vacuum system, a chamber, an ion source, an ion column, an electron column, a sample stage, a gas injection system (GIS) and a computer to control the whole instrument. Such a system allows the electron and ion beams to work together to achieve tasks beyond the limitations of either individual system. The ion source and ion column are the most critical components in the FIB instrument since the ion beam are generated, focused and directed in these parts.
2.1.1 Liquid metal ion source

Nowadays most FIB instruments are equipped with a liquid metal ion source (LMIS) which was developed in 1975 [28]. LMIS is a field ion emission source. Comparing this with other types of ion source such as an electron bombardment ion source, a gas discharge ion sources and a field ionisation sources, LMIS is superior in brightness and small spot size. The basic structure of a LMIS is illustrated in Figure 2-2. A tungsten wire is electrochemically etched into a needle with tip radius of 5 - 10µm. The needle is wetted with a small amount of liquid metal which is stored in a reservoir attached to the emitter. When applied to a high voltage, the liquid metal film on the needle tip deforms as a result of surface tension. With the increase in voltage, the effect of the electric field becomes more prominent. When the electrostatic force exerts a similar force on the droplet as the surface tension, a cone shape emerges comprising with convex sides and a rounded tip as illustrated in Figure 2-2(b) which is called the Taylor cone [29]. Under this situation, the electrical field at the liquid apex can reach $10^{10}$ V/m so that metal atoms can be ionized which will help breakthrough the constraint at the liquid metal surface in a form of field evaporation. To achieve a stable ion emission, the electrical field must be higher than a certain threshold voltage depending on the source material. Meanwhile, the emission surface must have a certain shape in order to establish the
surface electrical field. It is found that the shape of a Taylor cone with a whole angle of 98.6° approaches the theoretical shape just before the ion emission [29].

![Image of Taylor cone and liquid metal ion emission](image)

**Figure 2-2** The basic design for a LMIS. (a) SEM image of the LMIS; (b) Schematic of liquid metal ion emission [30].

There are several LMIS which offer a variety of metal species; the most commonly used ion source on today’s FIB systems is gallium. Comparing this with other materials, gallium has a low melting point (29.8°C) and liquid gallium can adhere to a tungsten surface very well. Both of these characteristics make gallium LMIS a primary source in contemporary FIB systems. Non-gallium ion sources such as Ar⁺, O₂⁺, P⁺ and B⁺ can also be used in different applications [31].

### 2.1.2 Ion column

As shown in Figure 2-3, the ion column is an electrostatic lens system to accelerate and focus the gallium ions extracted from the LMIS onto the sample surface.
Figure 2-3 Schematic diagram of an FIB ion column [32].

The emission ions from the LMIS are regulated by both an extractor and a suppressor. The suppressor uses an applied electric field of up to 2kV to work alongside the extractor in maintaining a constant beam current. By adjusting the voltage applied on the suppressor, the emission ion current can be changed. That is to say the ion current can be adjusted without changing the voltage on the extractor. This is very important for the stability of the FIB system. Since any voltage change on the extractor will result in a spatial displacement of the Taylor cone and apparently cause a beam drift on the sample. Therefore, the ability of the suppressor to adjust ion current without changing the source tip is essential for the stability of the FIB system.

The emanative ions emitted through the extractor are then refined by a spray aperture where the beam spot size is greatly reduced. The spray aperture only allows ions that are travelling more or less parallel with, and close to, the central axis at the column to
pass. After the spray aperture, the ion beam is then condensed in the first electrostatic lens, collimated into parallel beams and made ready for further adjustment. However, not all the ions entering the first lens are on parallel paths or evenly distributed in the ion column. Therefore, the focusing effect of the first lens is not uniformly applied and the ions do not become uniformly convergent to a circular spot. Astigmatism can be expected to develop during this process. An upper octopole electrode, also known as the stigmator, is needed to introduce a correction for ion beam astigmatism. A set of apertures are assembled below the upper octopole helping define the spot size and provide a range of ion currents that may be used for different applications. Unlike the suppressor electrode that influences the ion current by applying a different voltage, the variable aperture spatially constrains the ion current. A set of apertures that are arranged in two parallel lines on a horizontal sliding bar can be brought into position by a mechanical drive mechanism and both the ion current and the beam diameter will be refined at the point of interception. The relationship between the ion current and the beam diameter on a FEI Quanta 3D FEG (field emission gun) FIB system is shown in Figure 2-4. Under different acceleration voltages ranging from 2kV to 30kV, the beam diameter is reduced proportionally in relation to the ion current. A small beam diameter results in a high fabrication resolution and this constraint relationship reveals that in order to fabricate structures with a high resolution, only small current can be used in the FIB machining process.

![Figure 2-4 The relationship between the ion current and the beam diameter on a FEI Quanta 3D FEG FIB system.](image)
A beam blanker consists of blanking deflectors and a blanking aperture is used to switch off the ion beam when the column is powered up but is not in actual use. Given the amount of electrical power that is required to produce the ion emission, the ion beam cannot be rapidly switched on and off at the source. The combination of the blanking deflectors and the blanking aperture is a feasible method to achieve this switching function. When voltages of opposite polarities are applied to the blanking deflectors, the incident ions are repelled by the positive deflector and attracted by the negative deflector which results in a net effect, i.e. the ion beam misses the orifice and is absorbed by the aperture element. Although this blanking process is very rapid it still delivers a finite ion dose along the scanning path. Depending upon the application, this may result in a cumulative effect known as blanking tails, which is conspicuous when different scanning strategies are applied.

The lower octopole is used for directing beam scanning over the sample in a user-defined pattern. Similar to the upper octopole, four pairs of deflectors are assembled together to adjust the beam trajectories. In the deflection assembly, opposing pairs of deflectors are used to exert lateral forces to deflect the beam from the central axis. Thus, the beam can be placed at any point within the range of deflection. The deflected ion beam then enters the second lens where the beam is focused to a fine spot, enabling a best resolution in the sub 10nm range. The multichannel plate (MCP) is located near the bottom of the ion column and it is used to collect secondary particles emitted by the sample in response to ion radiation for imaging purpose.

The superior performance of the LMIS and the ion column enables the FIB technology to be applied in various applications.

2.2 Applications of FIB technology

When energetic ions bombard a solid sample, the energy is transferred to the target atoms as well as the electrons. During this process, the most pronounced physical effects of the incident ions on the substrate are: ion sputtering, secondary electron emission, target atom displacement, and emission of photons. Depending on the level of injected ion beam, the FIB can be used for various applications. The sputtering of neutral and ionized substrate atoms enables:

- Substrate milling;
Secondary electron emission enables ion imaging;
The reaction with deposit material enables ion beam assisted deposition.

2.2.1 Imaging

Ion beam scanning across the sample surface results in the ejection of electrons and ions, both of which can be collected for an imaging purpose. In this process the secondary electron yield is much higher than the secondary ion yield. Therefore, the FIB is mostly used in the secondary electron mode. However, as the beam current increases, more secondary ions can be ejected which results in a better imaging quality. FIB imaging has tremendous potential for a variety of investigations in material science and biology. Early work completed by former researchers gives an indication of the variety of information that can be obtained using FIB-based tomography, such as secondary electron images [33] and X-ray images [34-35] of three-dimensional structures, three-dimensional mass spectral images using planar FIB etching [36] and a three-dimensional reconstruction technique based on secondary electron images [37-40]. This prior work has also shown that the FIB imaging method is able to reach high resolutions down to the nanometre scale.

In terms of imaging principles, there are three primary FIB contrast mechanisms in FIB imaging including topographic contrast, orientation contrast and material contrast. Topography contrast produces shadows and highlights readily interpreted by the observer. It applies the same principles used by a SEM. Topography contrast is the key mechanism in forming images when observing an amorphous simple substance. However, this is not an imaging superiority of FIB over the SEM since the SEM is more competent to image topography contrast but with smaller radiation damage. The superiority of FIB imaging embodies the following two mechanisms: orientation contrast caused by ion beam channelling effect, which is the most striking feature of FIB microscopy of crystalline specimens, and the channelling effect used to image crystal grains, such as Cu [41-43], GaAlAs/GaAs [44], polycrystalline aluminium film [45], and nickel foam [46], revealing different crystal orientations. The latter is a process where the incident ions collide with the target atoms if the incident ions enter the sample in the direction of a low indexed axis. Some of the incident ions and scatter ions, due to the open channel of the crystal lattice in the sample, will result in several times the maximum range in amorphous materials. The result of channelling is the
generation of the secondary ions and electrons with respect to random-incidence condition, which gives rise to the orientation contrast in scanning ion microscopy imaging. An example of channelling contrast is showed in Figure 2-5. The dark areas occur where ions penetrate deep into the target and where secondary electrons produced cannot reach the surface to be collected to form an image. The mechanism behind the material contrast in scanning ion microscope images was studied by Monte Carlo simulation [47]. Material contrast arises from the difference in the yield of secondary electrons and/or ions and can be described as a function of the specimen element. This characteristic is especially useful in corrosion studies, as the secondary ion yields of metals can increase by three orders of magnitude in the presence of oxygen [48]. The enhanced secondary ion yield can therefore increase the brightness of this region and create a chemical contrast effect.

Figure 2-5 FIB secondary electron mode image of a nickel foam ‘strut’ which forms a component of nickel-hydroxide-based batteries intended for automotive applications [46].

A drawback of FIB imaging is that it inevitably induces some damage to the sample. The energy of the gallium ions extracted from the LMIS is above 2keV [49]. Ion implantation and sputtering will occur during the imaging process. Low current and
low energy ions are helpful in reducing the radiation damage [50] but this will in turn degrade the imaging resolution. A compromise method to reduce the radiation damage is by the applying a protective layer on the surface exposed to the ion beam and by the selection of a suitable operation protocol [51].

2.2.2 Micro- and nano-machining
Recent developments in FIB technology have led to beam sizes ranging from 400nm to 3.5nm, which makes it an ideal tool for micro- and nano-fabrication. FIB micro- and nano-machining has been demonstrated in several applications, such as FIB nano writer for photonic devices [52-53], micromachining of micro electro mechanical system (MEMS) [54-57], TEM sample preparation [58-59], semiconductor IC (integrated circuit) manufacturing and diagnostic [60-62]. The virtually stress-free FIB cross-sectioning capability is ideal for materials characterization [46] and the investigation of subsurface damage [63]. Ion beam sputtering combined with chemically active gases can be used to greatly enhance the sputter yield in machining process. When a small amount of chemical gas is introduced into the target surface, the incident ions ionise these gas atoms and result in a chemical reaction with the target surface material. Such a chemical reaction will convert the material on the target specimen into a volatile compound. Halogen gases, such as Br₂ [64], Cl₂ [65], or XeF₂ [66], can be directed onto the area of interest. In the milling process, the chemical gases used for assisted sputtering must match the target material. A study of these combinations and the corresponding enhancement factors in sputter yield¹ has been listed in Table 2-1 [67].

<table>
<thead>
<tr>
<th>Gases</th>
<th>Al</th>
<th>Si</th>
<th>SiO₂</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>Br₂</td>
<td>8~16</td>
<td>5~6</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Cl₂</td>
<td>7~10</td>
<td>1</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>XeF₂</td>
<td>0</td>
<td>7~12</td>
<td>7~10</td>
<td>7~10</td>
</tr>
</tbody>
</table>

2.2.3 Deposition
Deposition, also known as ion beam assisted chemical vapour deposition, is another feature that is used extensively in FIB applications. Selected materials may be
deposited onto a target surface by the introduction of appropriate gases into the system. The deposition process was first reported by Gamo et al. [68] who studied the process for the deposition of Al from Al(CH)\textsubscript{3} and W from WF\textsubscript{6}. This technique provides the ability to deposit functional materials onto almost any solid substrate with nanometre spatial resolution. A nozzle is brought into the chamber, hovering above the area of interest within 100 ~ 200\(\mu\)m. The gas is introduced by the nozzle and then adsorbed on the surface of the material. When the FIB hits the target surface, the secondary electrons break the chemical bonds of the precursor gas molecules which separate into different components: some of which remain volatile, others are deposited onto the surface. Tight beam control is critical to a successful FIB-assisted deposition since the precursor gas can easily be depleted and then cause the net deposition rate to be negative resulting in material sputtering instead. In order to maintain a constant positive deposition rate, the beam cannot stay at the same time point for a long time – it must move to another location and then come back to the same point. Once some new molecules are adsorbed on the surface the beam will revisit the same point and then add more deposited material.

The deposition of metal is used extensively for silicon semiconductor device modification and mask repair. It has also been applied for TEM sample preparation techniques to protect the top surface of interest from spurious sputtering. The areas of application of FIB deposition are listed in Table 2-2. Most of these deposition applications are two-dimensional structures with uniform height over the deposition area. By accumulating the two-dimensional structures layer by layer, one can fabricate three-dimensional and overhanging structures [55, 57, 69]. Further combination with computer aided design (CAD) enables the capability to fabricate more complex structures [70]. An interesting application of this technique is for the deposition of pyramidal roof-like structures [69]. As illustrated schematically in Figure 2-6, such structure can be built up by a series of rectangles with steadily decreasing dimensions. The encapsulation deposition is typically performed at a pressure below \(5 \times 10^{-6}\) mbar at which the deposition of SiO\textsubscript{2} is carried out with the low pressure maintained at the same level as that inside the encapsulation. The gas pressure sensor can be sealed inside the vacuum cavity which serves as an absolute pressure reference.

\textsuperscript{1} Sputter yield enhancement factor is the ratio between the chemically active gases assisted sputter yield and the original sputter yield.
2.3 The FIB machining mechanism

The applications of the FIB system in milling, imaging and deposition depend heavily on the nature of ion – solid interactions. As illustrated in Figure 2-7, when a beam of ions is incident on a solid material, several kinds of interactions take place on and beneath the target surface, such as scattering, sputtering and redeposition.
2.3.1 Scattering

Elastic and inelastic scattering are the most pronounced interactions during the ion bombardment process.

For elastic scattering, kinetic energy and momentum are conserved during the collision process. In the centre of mass coordinate system illustrated in Figure 2-8(b), the energy transferred from incident ion to the target particle can be described as [83]:

\[
\text{Energy transferred} = E - E_c
\]
where $m_1$ and $m_2$ are the masses of the incident ion and the target particle, respectively; $E$ and $T$ are the kinetic energies of the incident ion and the target particle; $\theta$ is the scatter angle in the centre of mass coordinate system. $\theta$ may be expressed in terms of the initial centre of mass energy $E_c$, the inter-atomic potential $V(r)$, and the impact parameter $p$ as shown in the following equation [84]:

$$
\theta = \int_{r_{min}}^{r_{max}} \frac{p}{\sqrt{r^2 - \frac{E_c}{V(r) - \frac{p^2}{r^2}}} \, dr
$$

where

$$
E_c = \frac{m_2}{m_1 + m_2} E
$$

and $r_{min}$ is the distance of the closest approach during the collision.

Figure 2-9 shows the relationship between the energy transfer ratio and mass ratio of the binary collision system. It indicates that the main parameters that govern the energy transfer rate from the incident ion to the target particle are dependent on the ratio of incident ion to the target particle mass and the scattering angle.

The ion-solid collision cascades can be divided into three regimes as it is shown in Figure 2-10. The first regime is the single knock-on regime where the ion mass is a lot less than the mass of the particle ($m_1 \ll m_2$). In this regime, the recoil particle does not receive enough energy to generate a cascade and the energy transfer from the incident ion to the target particle is minimal. The second regime is the linear cascade regime where the masses of the incident ion and the target particle are around the same ($m_1 \approx m_2$). This is the regime where the FIB sputtering process generally operates. In this regime, if the transferred energy is sufficiently large then the primary recoil particles will move along a trajectory similar to that of the incident ion and may undergo further collisions, thus creating further generations of recoils and a collision cascade. This is particularly the case when the target particles are near the surface of the material and the transferred energy is sufficient to overcome the surface binding energy causing ion sputtering. A portion of the sputtered particles may be ionized and can be collected to either form an image or be mass analyzed. The third regime is the
spike regime where \( m_1 \gg m_2 \). Under this condition, the majority of particles within the spike volume move during the collision cascade. This phenomenon occurs when an ion collides with electrons. The kinetic energy of the incident ions barely changes in this process. When the gallium ions are incident on a substrate, the mass ratio of the gallium ion to a single electron is in the order of \( 10^6 \). According to equation (2.1) the energy transfer ratio is only 0.001%. Therefore, the collisions between the ions and the atoms can be treated separately from that of the ions and the electrons in order to simplify the collision cascade process.

![Figure 2-9](image_url)  
**Figure 2-9** Kinetic energy transfer rate from the incident ions to the target particles in an elastic scattering process.

![Figure 2-10](image_url)  
**Figure 2-10** Schematic cascade regimes: (a) Single collision regime; (b) Linear cascade regime; (c) Thermal spike regime.
Inelastic scattering also occurs as a result of ion-solid collision cascades where secondary electrons can be produced during this process. The incident ion transfers part of its energy to the electrons surrounding an atomic nucleus. These electrons can either be excited to produce secondary electron emission from the solid surface, or be stripped off from the atom resulting in the ionisation of atom and the secondary ion emission. Electronic losses of the incident ions can be calculated by the follow equation [85]:

$$\Delta E_e = dnS_e(E_0)$$  \hspace{1cm} (2.4)

where

$$S_e(E_0) = K_d \sqrt{E_0}$$  \hspace{1cm} (2.5)

and

$$K_d = 1.211 \frac{Z_1^{7/6}Z_2^{2/3}}{(Z_1^{2/3} + Z_2^{2/3})^{1/2}} \sqrt{m_1}$$  \hspace{1cm} (2.6)

In equation (2.6), $Z_1$ and $Z_2$ are the atomic numbers of the incident ions and the target atoms respectively.

2.3.2 Binary collision approximation

The scattering theory for the calculation of ion-solid interactions in the linear cascade regime can provide valuable analytical expressions of the important physical mechanisms and dependencies. However, the solutions are often complicated and require simplifying assumptions [86]. The first assumption is that the particles do not move in any preferred directions, which exist in e.g. a crystalline target. The second assumption is that only elastic collisions occur between the incident ions and the target atoms. The third assumption is that the scattering is made up of hard-sphere collisions, that is to say the particles are assumed to be classical perfect spheres doing the collisions.

Binary collision approximation (BCA) is a simplified model used to calculate the paths of the particles in the cascade based on the above assumptions. In this model, the interaction of an incident ion with atomic nucleus in the target sample is regarded as an elastic collision process, while the interaction with electrons is regarded as an inelastic process without any scattering effects. Only two charged particles are involved in one scattering process, the incident ion and the target atom.
A schematic diagram of BCA model is illustrated in Figure 2-11. The ion is approximated to travel through a material by experiencing a sequence of independent elastic binary collisions with the target atoms. Between the collisions, the ion is assumed to travel in a straight path, experiencing an electronic stopping process. During this process, if the energy a particle received in a collision is larger than some threshold value then the particle is included in the cascade to induce a further cascade. When the energy of a particle falls below some threshold value, or, say when it escapes from the target, the particle is excluded from the cascade.

The BCA model is fundamental for investigating complex situations that depend on individual ion-solid combinations. This model has been widely applied in many simulation programmes based on the Monte Carlo method. The trajectories of each incident ion and all associated recoil atoms are traced until the kinetic energy has fallen below the cut-off energy or the traced particles have escaped from the defined region. Figure 2-12 shows the spatial distributions of the ions and the recoil trajectories for 30keV gallium ions in silicon as obtained from simulations using a Monte Carlo simulation code called TRIM. The left column of the figure (a, c) indicates the ion trajectories in the substrate and the range of implanted ions is predicted from this simulation. The right column (b, d) indicates the moving and stopped recoiling atoms of the target substrate. In Figure 2-12(a), it is evident that two of the ten ions are backscattered. Figure 2-12(b) indicates that, for the present ion-target combination, an individual ion creates several smaller sub-cascades with little overlap. The overlap of
many incident ions (Figure 2-12(d)) forms a cascade region which is similar to the region of the ion tracks (Figure 2-12(c)).

Figure 2-12 Ion only (a, c) and ion plus recoil (b, d) trajectories for 30keV gallium ions incident on silicon, for 10 (a, b) and 100 (c, d) incident ions, as obtained from TRIM.

2.3.3 Sputtering and redeposition

FIB can be used for direct and maskless patterning on a substrate. In this process, ion milling takes place as a result of physical sputtering of the substrate atoms. The ion beam sputtering is a very special scattering process which takes place near the surface of the target material. It is the most pronounced ion-solid interaction that occurs in the FIB milling process. A recoil atom near the surface can obtain enough energy in the collision cascade induced by the ion bombardment to be knocked out as a sputtered particle if it receives sufficient kinetic energy to overcome the surface binding energy. Figure 2-13 shows the energy distribution of recoil atoms in a silicon substrate under
30keV gallium ion beam radiation calculated by TRIM. Since the surface binding energy for silicon is 4.7eV, any recoil atoms with kinetic energy below this threshold cannot escape from the surface of the silicon substrate.

![Simulation result of the energy distribution of the recoil atoms in a silicon substrate under 30keV gallium ion beam radiation](image)

**Figure 2-13** Simulation result of the energy distribution of the recoil atoms in a silicon substrate under 30keV gallium ion beam radiation.

These sputtered atoms generally possess a kinetic energy in several eV. The emission angle of the sputtered atoms generally follow a cosine distribution for normal incidence ion bombardment [87-88]. For different ion incident angles, the angular distribution of the sputtered atoms is also slightly different. The redeposition effect takes place accompanied with an ion sputtering process. Some of the sputtered atoms induced by the incident ion beam will deposit at the site of interest, reducing the net removal rate and creating unwanted features. As it is illustrated in Figure 2-14, when the sputtered atom leaves the target material, it is ejected with a finite kinetic energy. Therefore, it can be considered as a projectile that can exert interactions on its trajectory. Depending on the sticking coefficient of the material, the sputtered atom may attach to the sidewall of the structure. The sticking coefficient is defined as the ratio of atoms that properly react and attach on the surface to the total sputtered atoms. It is a statistical measure of the material’s affinity to adhere to a surface. It depends on the chemistry of the material being deposited, and on the substrate temperature. For FIB sputtered atoms, the sticking coefficient is assumed to be constant for different materials [89].
The area that can be affected by the redeposited atoms depends on the surface topography, the angular distribution of the sputtered atoms and the surface local pressure. Due to the cosine rule of the angular distribution of the sputtered atoms the redeposition effect is more pronounced in structures with a high aspect ratio, in which case, the projectile is more liable to collide with the sidewall and therefore be trapped into the structure. The surface local pressure also has a profound effect on the degree of redeposition [90-91]. Due to the confining geometry restricting the escape of the sputtered material, the local pressure at the sputtering point is much higher than in other parts in the vacuum chamber; therefore, the collision mean free path is decreased, which leads to a further increase in the probability of the sputtered atom collisions and redeposition rate.

2.4 **Key fabrication parameters in FIB machining**

The FIB machining process is a combination of physical sputtering and material redeposition. The material removal process depends on many processing parameters including the sputter yield, beam scanning strategy, scanning passes, dwell time, and beam overlap. Understanding these processing parameters can lead to an enhancement of the sputtering rate and further improvement of machining accuracy.

2.4.1 **Sputter yield**

The sputter yield is an elementary parameter which characterizes the basic sputtering phenomena essential to the milling process in making microstructures from simplified
two-dimensional or three-dimensional structures. The accurate determination of the sputter yield is an essential requirement in FIB machining.

The sputter yield is defined as the average number of target atoms ejected from the sample per incident ion. Correspondingly, the sputter rate (or material removal rate) is the number of atoms being sputtered from the target per unit time. A reasonable estimation of the sputter rate by sputter yield is:

\[ Y_I = Y \times \frac{I}{e} \]  

(2.7)

where \( Y_I \) and \( Y \) are the sputter rate and the sputter yield, respectively; \( I \) is the ion beam current and \( e \) is the elementary charge.

Experimentally, the sputter yield can be measured at specific sputtering conditions. Through measuring the volume of the ion caved cavity, the average sputter yield can be evaluated using the following equation:

\[ Y = \frac{V \times n \times e}{I \times t_{total}} \]  

(2.8)

where \( V \) is the cavity volume (\( \mu m^3 \)); \( n \) is the atoms density (atoms/\( \mu m^3 \)) of the substrate; \( e \) is the charge of a single electron (1.602\( \times 10^{-19} \) C); \( I \) and \( t_{total} \) are the ion current and total machining time respectively.

Theoretically, the sputter yield can be calculated by Sigmund’s linear collision cascade model [92]:

\[ Y(E, \theta) = \frac{0.042}{U_s} \alpha S_n(E) \cos^{-f_{mr}} (\theta) \]  

(2.9)

where \( U_s \) is the atom binding energy; \( \alpha \) is a factor that depends only on the efficiency of energy transfer; \( S_n(E) \) is the elastic stopping power; \( f_{mr} \) is a function of the mass ratio of the incident ion and the target atom; and \( \theta \) is the incident angle. This indicates
that the sputter yield depends not only on the substrate material but also on many processing parameters, including the ion energy, the angle of incidence and the milling conditions.

Alternatively, Yamamura proposed an semi-empirical formula that can analytically describe the angle dependency of the sputter yield over a wide range of the incident angles [93].

\[
Y(\theta) = Y(0)\cos^{-\alpha}(\theta)\exp(-\beta(\cos^{-1}(\theta) - 1))
\] (2.10)

where \(\alpha\) and \(\beta\) are adjustable parameters.

The theoretical results of sputter yield angular dependence are compared with the experimental results [94] in Figure 2-15. This illustrates a beam of gallium ions with an energy level of 20keV bombarded on a silicon substrate with different incident angles. It indicates that the Sigmund theory coincides with the experimental data only for incident angles smaller than 65°, while Yamamura’s semi-empirical expression is consistent with the experimental data for all incident angles. The sputter yield reaches a maximum at about 80° and then decreases very rapidly to zero as the incident angle approaches 90°.

![Figure 2-15 Sputter yield of gallium ion incidents on a silicon substrate at 20 keV.](image-url)
The mechanism of this angular dependence can be explained by Figure 2-16, which shows the simulations of the collision cascades under different incident angles. From 0° to 80° the ion trajectories and recoiling atoms generated by the higher incident angle are observed to be closer to the surface. From a statistical perspective the more collisions occur proximal to the surface, the higher the sputter yield. Therefore, the sputter yield keeps increasing with the ion incident angle. For large incident angles, ion reflection plays an important role at glancing angles and results in a rapid drop in the sputter yield.

\[
\begin{align*}
\text{(a)} & \\
\text{(b)} & \\
\text{(c)} & \\
\text{(d)} & 
\end{align*}
\]

Figure 2-16 20keV gallium ion sputtering trajectories under (a) 0 degree, (b) 30 degree, (c) 60 degree, and (d) 80 degree.

Sputter yield also depends on the energy of the incident ions but high energy ions do not necessarily generate a high sputter yield. Figure 2-17 shows the sputter yield of gallium ions incident on a silicon substrate and a copper substrate. For both materials the

28
sputter yield grows as the ion energy increases but the rate of increase tapers off. In most cases the sputter yield either levels off or decreases when ion energies are higher than 100 keV, where the implantation becomes dominate as ions penetrate into the substrate and are trapped in the lattices.

Figure 2-17 Sputter yield of gallium ions incident on a silicon substrate and a copper substrate. Solid lines are the sputter yield calculated based on empirical equations at normal incidence [95].

Although there are numerous theories to estimate the sputter yield, none of them can explain all sputtering phenomena completely. Alternatively, the sputter yield can also be calculated by simulation methods. Computer simulation has long been an important tool in studying the complex ion-solid interactions. As computing power has increased enormously, the development of simulation methods has boomed in the last few decades. Amongst these Monte Carlo simulation and Molecular Dynamics simulation have been shown to be effective methods for solving complex systems. Monte Carlo simulation can calculate the stopping range of ions and provides a three-dimensional distribution of ions to predict the sputter yield. Based on the BCA model, the Monte Carlo method is widely used to simulate time-independent cascade processes. Various codes exist and have their own specialities: for crystalline target materials, the MARLOWE [96], crystal-TRIM [97], COSIPO [98], IMSIL [99], and XPTOPS [100] codes were developed. Yamamura [101] developed a Monte Carlo code taking the surface roughness into account; Ishitani [102] developed a Monte Carlo code which includes thermal dissipation; Boxleitner [103] developed FIBSIM Monte Carlo code to simulate
the compositional and topography changes caused by FIB milling. Besides, the simulation of ion sputtering by Molecular Dynamics methods were developed by Urbassek [104], Samela [105] and Satake [106].

The most widely used Monte Carlo simulation code for FIB machining is SRIM/TRIM developed by James F. Ziegler [107]. This code is in fact a collection of software packages which calculate many features of the transport of ions in matter. The main applications include ion stopping and range in targets, ion implantation, ion sputtering, ion transmission and ion beam therapy. It can deal with a complex substrate made of compound materials with up to eight layers but SRIM/TRIM is known to have limited accuracy, i.e. 10% ~ 30% error for the sputter yield calculation, which is partly caused by the simplification of surface composition changes due to implantation [108-109]. However, such an effect has been taken into account in another Monte Carlo code TRIDYN [110-111]. TRIDYN simulates the dynamic change of the thickness and/or the composition of multi-component targets during high-dose ion implantation or ion-beam-assisted deposition. In the dynamic system, sputtering parameters are kept constantly updated during primary ion fluence giving access to the system composition. The composition and thickness of the sample are dynamically modified to accommodate the transportation of the particles inside the target workpiece during the ion bombardment process. Therefore, the influences of high-fluence implantation, ion mixing and preferential sputtering caused by the atomic collisions have been taken into consideration. Compared with an experimental measurement, it is found that the sputter yield calculated by TRIDYN is more accurate than that obtained by SRIM/TRIM. As shown in Figure 2-18, the sputter yields of Si bombarded with Ga\(^+\) calculated by TRIDYN after a steady-state implantation profile has developed is more accurate than the results calculated by TRIM neglecting the implanted Ga ions [108]. The star in the figure corresponds to an experimental result [94].
2.4.2 Scanning strategy

In a typical FIB system, the machining process is performed by placing the beam sequentially at pre-defined positions to form an area of quadrilateral or more general shape. The scanning strategy produces the trajectories of the scanning beam in the ion beam machining process. The trajectories of the scanning beam spot are the beam scanning lines. Each of the scanning lines constituting the scanning path is in fact a linear array of discrete points to which the beam is rapidly deflected and at which it pauses for a predetermined period (dwell time) to remove the required amount of material. There are two kinds of scanning path that are commonly used in FIB machining: raster scan and serpentine scan both of which have been illustrated in Figure 2-19. In the raster scan, the beam proceeds along each scanning line in the same direction. The scanning beam is blanked at the end of each scanning line and retraced to the opposite end of the next scanning line. In serpentine scan, the beam moves in opposite directions between each pair of adjacent scanning lines. Therefore, the retrace is eliminated and the beam does not need to be blanked during the scanning process. Apart from the raster and serpentine scan, self-defined scanning paths are also available. This kind of scanning path is essential for machining complex structures where high precision is required. The “blanking tails” can also be minimized by optimizing the scanning path. In the beam scanning process, the pattern is represented by a series of milling points, the coordinates of which are stored sequentially in the computer memory. Whatever scanning mode is chosen, the beam is always blanked after the last scanning.

Figure 2-18 Sputtering yields of Si bombarded with Ga$^+$ as a function of energy [108].
line of each scanning pass while being deflected back to the start point on the first scanning line of the next pass.

![Scanning strategies in FIB machining process](image)

**Figure 2-19** Scanning strategies in FIB machining process with arrows indicating scanning direction: (a) raster scan; (b) serpentine scan.

### 2.4.3 Beam diameter

The analysis of beam profile is one of the fundamental issues in FIB machining. The beam profile is usually considered as a Gaussian distribution with a circular cross section, even if the beam had a wide beam skirt following a Holtsmark distribution [112-113]. The intensity distribution is Gaussian with the long ranging tails satisfying the following equation:

\[
J(x, y) = \frac{I}{2\pi\sigma^2} \exp\left[-\frac{(x-x_p)^2 + (y-y_p)^2}{2\sigma^2}\right]
\]  

(2.11)

where \(J(x, y)\) is the ion flux density at a point \((x, y)\); \(I\) is the ion current and \(\sigma\) is the standard deviation of the Gaussian distribution; the centre of the beam is located at point \((x_p, y_p)\).

The commonly used characteristic value to describe the beam spot size is the beam diameter which is the full width at half maximum (FWHM) of the intensity or \(e^{-1}(36.7\%)\) of the maximum intensity. The beam diameter \(d\) can be estimated from the Gaussian distribution:
Therefore, the ion flux density can be expressed as a function of the beam diameter:

\[ J(x, y) = \frac{4I \ln 2}{\pi d^2} \times 16 \frac{(x-x_p)^2 + (y-y_p)^2}{d^2} \]  

(2.13)

Generally the beam diameter grows as the ion current increases. An illustration of this relationship has already been shown in Figure 2-4.

### 2.4.4 Pixel spacing

As the ion beam scans across the surface, FIB milling is performed by a precise pixel-by-pixel movement. After scanning all the points on a particular line in the sputtering area the beam moves to the next line. After scanning the whole sputtering area, the beam re-starts scanning until it delivers the fully assigned ion dose. This is also known as a digital scan and is schematically shown in Figure 2-20. The distance between two adjacent pixels \( \delta \) is called the pixel distance. The overlap of the two adjacent beam spots is \( h \). To fabricate a smooth profile with a constant rate of material removal the pixel spacing must be small enough to allow a proper overlap between the adjacent pixels along both the \( x \) and \( y \) directions [114].

![Figure 2-20 Schematic diagram of digital scan. Each circle represents a beam spot.](image-url)
Figure 2-21 Normalized ion flux distribution in multiple scan-line FIB milling: (a) cross sectional view of the ion flux distribution at different pixel spacing. (b) and (c) are the three-dimensional flux field at $p = 1.5\sigma$ and $p = 2.4\sigma$, respectively.

As the intensity profile of the FIB can be defined as a Gaussian distribution [113], the ion flux distribution during the scanning process can be calculated as the sum of every beam profile:

$$J(x, y) = \frac{I}{2\pi\sigma^2} \sum_{n_x=0}^{N_x} \sum_{n_y=0}^{N_y} \exp \left[ -\frac{(x - x_p - n_x \delta_x)^2 + (y - y_p - n_y \delta_y)^2}{2\sigma^2} \right]$$ \hspace{1cm} (2.14)

where $N_x$ and $N_y$ are the total pixel numbers along the $x$ and $y$ directions, respectively.
In order to obtain a steady and unwavering ion flux, the threshold value for the minimum amount of the pixel spacing can be determined mathematically. Figure 2-21 shows the normalized cumulative ion flux distribution in a digital scanning process for a beam with 50nm diameter. The pixel spacing along the \( x \) direction changes from \( 8\sigma \) to \( 1.5\sigma \). It is found that when the pixel spacing is smaller than \( 1.5\sigma \), a constant flux field can be obtained. This uniformity condition of the ion flux with respect to the scanning direction can be directly extended to satisfy the uniformity condition between the scan lines.

2.4.5 Scanning passes

In FIB machining, the incident ions are to repetitively scan with the same scanning pattern on a sample many times. This is called the multi-pass scanning method. The iteration of each scanning pattern is called a scanning pass. The multi-pass scanning process is helpful in forming a uniform bottom surface [115]. Figure 2-22 shows a comparison of two micro-grooves which were fabricated on a silicon substrate (100) surface by FIB machining using a single scanning pass method and a multi-pass scanning method respectively. Under the same ion dose of \( 1.2 \times 10^{18} \) ions/cm\(^2\) the number of scanning passes was inversely proportional to the dwell time. Compared to using multiple scanning passes, the single scanning pass had a longer dwell time which led to a deeper and narrower scanning channel. Due to the cosine rule of the angular distribution of the sputtered atoms, the sputtered atoms generated in the single scanning pass have more difficulty escaping from the micro-channel which led to a higher redeposition rate as shown in Figure 2-22(a). The longer dwell time can also result in a higher local pressure at the sputtering point. This phenomenon will further lead to a reduction of the collision mean free path of the sputtered atoms; therefore, the probability of redeposition increases. However, when using the multi-pass scanning method some deposited atoms can subsequently be removed by the next scanning pass. The multi-pass scanning process is also helpful in forming a uniform bottom surface in the groove as shown in Figure 2-22(b). A similar phenomenon has also been observed by Yamaguchi et al. [115].
Figure 2-22 Cross sections of micro grooves on silicon (100) surface (coated with 500nm platinum) fabricated using: (a) a single pass scanning process; and (b) a multi-pass scanning process (20 passes in total).

2.5 Processing methods in FIB micro machining

FIB machining is grouped into two categories: sputtering and deposition, which can be used for removing or adding materials on the target surface respectively. Unlike the FIB assisted chemical deposition process, in which the material deposition rate can be maintained at a constant value, the ion-material sputter yield is varied during the ion sputtering process. Besides, the redeposition effect is more pronounced in ion sputtering process because the sputtered atoms are easier to trap in concave structures. Both the above facts indicate that FIB sputtering is much more unpredictable than the deposition. In the FIB sputtering process, major issues are to calculate sputtering parameters to achieve a specific geometry and associated surface finish. With the development of mathematical models for these two major issues the FIB sputtering technique becomes more reliable for micro- and nano-fabrication.

A concise review of the current modelling and simulation methods for FIB milling is introduced in this section. These simulation and modelling approaches can be classified into two categories:
• Surface topography prediction to predict the generation of surface topography based on the fabrication parameters as inputs. This approach is implemented based on two-dimensional and three-dimensional ion-solid interaction model followed by topography simulations.

• Fabrication parameters prediction to determine proper FIB fabrication parameters to obtain the required structures.

The experimental results obtained from these different fabrication methods are also compared and the advantages and disadvantages of each method analyzed.

2.5.1 Surface topography prediction
Surface topography prediction is essential for FIB manufactures to evaluate the sputtering result and provide references for FIB operators. The cornerstone of surface topography prediction is the evaluation of the sputter depth. The depth of microstructures with simple geometries fabricated by FIB machining is usually guided by the assumption of the existence of a proportional relationship with the local ion dose. However, this assumption becomes invalid for high aspect ratio structures or curved surfaces, where angular dependence of sputter yield as well as the redeposition effect cannot be ignored. The redeposition phenomenon depends on the local surface topography, material properties of the substrate and the ion-target interaction between the sample and the LMIS used. Besides, the distribution of the redeposited atoms highly depends on the local surface topography which can only be predicted by the use of adequate, theoretically motivated numerical methods. A sophisticated ion-solid interaction model combined with surface topography simulation may contribute to the understanding of these effects.

The theoretical description of the changes on surface topography under a given process has long been of great scientific interest. The earliest research on surface topography evolution under focused ion beam radiation was carried out by Mueller et al. [116] in 1986. Two-dimensional simulation program COMPOSITE (Complete Modelling Program of Silicon Technology) was modified for this process with the beam confined in one direction and given a Gaussian distribution. This work provided an insight into
the interplay of the angular dependent sputter yield and redeposition effect. Topography simulation was implemented by a string based algorithm in COMPOSITE.

In 1989, Katardjiev et al. presented a generalized kinematic theory of surface evolution to simulate the topography development of three-dimensional surfaces during growth and erosion based on the Huygens’ Principle [117]. A three-dimensional simulation code DINESE (direct numerical evaluation of surface evolution) [118-120] was developed to simulate the evolution of real three-dimensional structures during milling and deposition. The TRIDYN code was used to obtain angle-dependent sputter yields but the redeposition effect is not considered in DINESE code which means that it is only applicable to simulate the machining of low aspect ratio structures.

Biedermann and Platzgummer developed a software package called Ionshaper [121] which included first and second order sputtering as well as the first and second order redeposition of sputtered atoms. The natural erosion process was characterized by a surface velocity vector normal to the surface and a surface shape with a fully continuous derivative. A novel ion erosion model was presented in this work to produce a good simulation of sharp edges where the surface slope is ill-defined.

Boxleitner and Hobler developed a dynamic Monte Carlo simulation code FIBSIM [103] to evaluate the surface topography evolution and damage formation during TEM sample preparation [122]. FIBSIM combined the simulation of binary collision cascades with two-dimensional cell-based topography simulation model. Analogously, Kim et al. developed a two-dimensional [109] and three-dimensional [123] simulation code named AMADEUS 2D/3D (Advanced Modelling and Design Environment for Sputter Processes) to predict the surface topography evolution under ion beam radiation. The surface was represented by a structured or unstructured grid. Each node on the grid moved according to the calculated sputtered and redeposited fluxes. The node velocity along the surface normal direction is proportion to the fluxes received. Therefore, the surface evolution can be simulated by this string/cell-based tracking technique. This method works well for the simulation of machining low-aspect-ratio structures. For structures with a high aspect ratio, nonetheless the ion-solid interaction model is still applicable, but the string-based topography tracking technique would crash due to the sharp gradients and cusps. A more robust surface tracking technique needs to be
developed. Based on the same ion-solid interaction model, Kim et al. developed a two-dimensional topography simulation implemented using the level set method [124]. Introduced by Osher and Sethian [125], it is a highly robust and accurate computational technique for tracking moving interfaces. Via this method the surface topological merging and breaking, sharp gradients and cusps can form naturally with the help of an upwind difference scheme. The effects of curvature can also be easily incorporated, which makes it a more robust method.

A summary of the reviewed simulation codes is listed in Table 2-3. Both two-dimensional and three-dimensional models have been developed to predict the surface topography evolution in the FIB machining. The redeposition effect has been considered in most of the models. However, for structures with a high aspect ratio, more robust simulation techniques need to be developed. A three-dimensional topography simulation based on level set method may competent to give a solution for this issue.

<table>
<thead>
<tr>
<th>Simulation Code</th>
<th>Dimension</th>
<th>Topography model</th>
<th>Redeposition</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>COMPOSITE</td>
<td>2D</td>
<td>String-based</td>
<td>Yes</td>
<td>[116]</td>
</tr>
<tr>
<td>DINESE</td>
<td>3D</td>
<td>Huygens’ Principle</td>
<td>No</td>
<td>[120]</td>
</tr>
<tr>
<td>FIBSIM</td>
<td>2D</td>
<td>Cell-based</td>
<td>Yes</td>
<td>[103]</td>
</tr>
<tr>
<td>Ionshaper</td>
<td>2D, 3D</td>
<td>Huygens’ Principle</td>
<td>Yes</td>
<td>[121]</td>
</tr>
<tr>
<td>AMADEUS-2D</td>
<td>2D</td>
<td>String-based</td>
<td>Yes</td>
<td>[109]</td>
</tr>
<tr>
<td>AMADEUS-3D</td>
<td>3D</td>
<td>Cell-based</td>
<td>Yes</td>
<td>[123]</td>
</tr>
<tr>
<td>AMADEUS-Level set</td>
<td>2D</td>
<td>Level set method</td>
<td>Yes</td>
<td>[124]</td>
</tr>
</tbody>
</table>

### 2.5.2 Fabrication parameters prediction

The fabrication of customized three-dimensional structures is the reverse of the surface topography prediction. Several methods have been reported to realize three-dimensional structures using FIB machining. In the 1990s, Ishitani et al. first demonstrated the feasibility of FIB in two-dimensional fabrication by cutting a 45µm diameter micro-gear in stainless steel foil with the aid of a sample rotator [126]. Nellen et al. [127] introduced a two-dimensional simulation model to estimate the sputter
depth of simple geometric structures. Fabrication parameters, including ion fluence, beam diameter and scanning strategy were taken into consideration.

Several fabrication methods for the fabrication of three-dimensional structures were proposed by former researchers. Vasile et al. introduced a depth control method by controlling the dwell time on each milling pixel [128-129]. In this method, a general mathematical model was developed which directs the ion beam material removal process and has the capability of milling a three-dimensional cavity from a given initial geometry into a pre-specified final geometry. The factors that affect the ion-milling process, such as sputter yield and ion beam intensity distribution, were considered. Redeposition effect and the influence of the scanning path were ignored.

Fu et al. [130] employed a two-dimensional slice-by-slice method to fabricate three-dimensional structures by FIB. Sequential two-dimensional slices with small thicknesses were used to approach the desired three-dimensional structures. The number of the slices can be determined according to the maximum depth of the three-dimensional microstructures. The milling depth for each slice depends on the slice thickness. Analogously, Lalev et al. [131] developed a data preparation program for FIB machining of complex three-dimensional structures utilizing this slice-by-slice method. Complex surfaces can be easily designed in any three-dimensional CAD package and then converted into GDSII (Graphic Database System II) streams for FIB sputtering or deposition. Compared with the depth control method, this method has the advantages of a simple mathematical model, less memory space used and fast milling speed. The sputter yield and the dwell time are constant during the machining of flat slices. However, the fabricated structures have stair-step sidewalls due to the limitation of the number of finite slices.

Regarding the surface quality of the sidewall, Hopman et al. [132] proposed an “trial and error” fabrication method to optimize surface quality. It was found that the scanning routine of the ion beam can be used as a sidewall optimization parameter. For circular patterns, a spiral scanning routine can significantly reduce the amount of redeposition.
Based on Fu and Hopman’s methods, Kim et al. [133] chose a continuous slicing method which used spiral scanning as a part of the vector scan instead of using a raster scan to produce accurate circular patterns. Rather than using a discrete slice-by-slice process, the circular structure was fabricated in a contiguous way where the ion beam followed a spiral routine; therefore, stair-step structures on the sidewall were avoided. However, this is not a general method for all kinds of three-dimensional structures since only circular structures were studied in this work. The differences between the above fabrication methods are illustrated schematically in Figure 2-23.

![Figure 2-23](image)

**Figure 2-23** Three different methodologies for producing a 3D shape: (a) Depth control method: sputtering by changing the dwell time on the pixel, (b) Slice-by-slice method: sputtering the sequential 2D slice-by-slice, and (c) continuous slicing method: modified by repeating Fu’s method by continuously reducing the ion dose for each slice, whereas the total ion dose is the same as Fu’s method.

There are other methods developed for only fabricating specific structures. A quasi-direct writing method [134] and a self-organized formation [135] method were introduced to fabricate diffractive structures on silicon substrate. In these the dwell time was kept at a constant during the whole process. Diffractive structures with an amplitude of less than 1μm are generated by changing the pixel spacing along the direction of the cross scan with zero overlap and keeping the pixel spacing constant along the other scanning direction with a normal overlap of 50%–60%. The advantage of this method is that there is no need to program and it can be applied on almost any material. However, this is not a general method for micro-fabrication, only blazed-grating-like structures can be machined in this way.

Svintsov et al. developed their IonRevSim software [136] specifically for data preparation and the prediction of the shape of FIB machined structures. It demonstrated
a quantitative description of FIB machining for three-dimensional structures by means of an isotropic local etching model which assumes that the sputter yield is proportional to \( \frac{1}{\cos(\theta)} \), where \( \theta \) is the incident angle. This assumption works under a low incident angle; therefore, this model can be used for the prediction of FIB machined structures with a low aspect ratio and with an inclination not greater than 45º.

It can be concluded that FIB is a competent technology for fabricating three-dimensional micro structures by the aid of sophisticated data processing or data preparation. However, the limitations are also very obvious. The depth control method is able to fabricate complex structures precisely, but sophisticated data processing is needed before the fabrication. That is to say that user specified program is required and the calculation of the dwell time matrix is a time-consuming work especially for micro structures over large area. The slice-by-slice method is an efficient way for three-dimensional micro structure fabrication, but it will results in a stair-step structures on the sidewall which degrades the machining accuracy. The continuous slicing method and the quasi-direct writing method are only applicable to circular structures and blazed-grating-like structures, respectively. The IonRevSim software is only used for the prediction and fabrication of structures with low-aspect ratio. Redeposition effect is ignored in all of the above methods. Novel data processing and data preparation techniques that based on the current FIB control software need to be developed for the fabrication of complex structures with high accuracy and high usability.

2.6 Challenges in FIB machining

In recent years, the demand for machining materials by FIB has increased exponentially because of the emergence of many novel designed micro devices and novel applications. Such devices have become harder to fabricate as a result of growing complexity, decreased feature size and the introduction of new materials. To broaden the conventional machining abilities of FIB technology, key challenges and gaps in FIB machining confront the engineers can be classified as follows:

- **Dimensional challenge**
  The dimensional challenge in FIB machining can be classified into two categories. On the one hand, FIB technology is able to fabricate very small
structures down to the nanometre scale; however, an accurate fabrication in this dimensional scale is still a challenge because of the redeposition effect. On the other hand, the fabrication of structures over extremely large areas using FIB is also a challenge due to the low throughput and the limited range of motion within vacuum chamber.

- **Geometric challenge**
  The geometric challenge becomes severe especially in FIB three-dimensional fabrication. Although FIB is capable of machining three-dimensional micro structures using the methods reviewed in section 2.5.2, the data preparation process is very time-consuming. Challenges in improving both the machining form accuracy and the data preparation method still remain.

- **Material challenge**
  FIB machining is able to remove material from almost any solid substrate. However, when machining dielectric materials the FIB machining accuracy is degraded. The positive charges induced by the incident ions accumulate on the substrate surface where an extra electric field is formed. This extra electric field deflects and defocuses the incident ion beam in an unpredictable manner. This phenomenon results in an image drift during the fabrication process. Both the accuracies of the machining position and the corresponding profile are degraded.

- **Machining efficiency challenge**
  Low throughput is the major disadvantage of FIB machining. Due to the limitation of the beam spot size and the ion current, the material removal rate in FIB machining is limited. Although the addition of chemical enhanced etching boosts machining efficiency, the throughput is still very slow compared with other traditional fabrication techniques such as diamond turning. Therefore, FIB machining is not suitable for fabrication over large area. However, it is possible to combine the FIB technique with other machining technologies to develop a hybrid method that could be used for large area machining with high accuracy. Therefore, studies in developing this form of hybrid machining process are worth pursuing.
2.7 Summary

This chapter has presented a critical review of FIB hardware, applications and processing methods in FIB machining. Challenges in current FIB machining techniques were identified. These were the dimensional challenge, the geometric challenge, the material challenge and the machining efficiency challenge.

FIB has been widely used in many areas in terms of imaging, machining and deposition. In FIB micro machining, the beam overlap effect, the angular dependence of sputter yield and the redeposition effect make the machined surface topography evolve in an “unpredictable” way. On the one hand, they complicate the prediction of the surface topography under certain ion radiation conditions. On the other hand, they degrade the FIB machining accuracy of customer designed structures. Different simulation models and processing methods have been developed to overcome the above issues. The sputter yield can be calculated analytically based on different ion-solid interaction models or numerically by computer simulation codes that are based on the Monte Carlo method or Molecule Dynamitic method. The influence on surface topography caused by redeposition effect can be embedded into different topography simulation models. For the fabrication of three-dimensional structures, the redeposition effect can be suppressed and therefore ignored.

Processing methods such as the depth control method, slice-by-slice method and continuous slicing method give access to a controllable way of producing three-dimensional micro-structures. This survey reveals that each method has its own advantages and disadvantages in terms of machining accuracy and usability. A key finding is the need to develop a highly robust three-dimensional simulation model for the prediction of surface topography evolution. Another finding is that novel data processing and data preparation techniques which can be implemented in FIB machining also need to be developed for the purpose of manufacturing both low aspect ratio and high aspect ratio structures with high accuracy and high usability. In order to bridging the gaps in the existing research work, a three-dimensional surface modelling approach based on level set method, and a divergence compensation method for three-dimensional structures’ fabrication, together with the corresponding applications, will be presented in the following chapters of this thesis.
Chapter 3 The Development of a Deterministic Three-Dimensional FIB Machining Approach

FIB micro- and nano-machining is an enabling technique that has been growing rapidly over recent years. The development of an FIB deterministic fabrication approach to obtain micro- and nano-structures is a highly demanding task for manufacturing engineers. Such an approach can lead to breakthroughs in the manufacturability of new components and devices. However, it is very difficult to obtain structures in the ion beam sputtering process under pre-determined fabrication parameters. On the other hand, the determination of fabrication parameters to obtain accurate structures is also a challenging task. The “unpredictable” behaviour in FIB machining is mainly due to the:

- ion beam overlap effect;
- angular dependence of the sputter yield;
- redeposition effect.

All of these three factors contribute to influence the fabrication divergence achieved in FIB machining and, subsequently, degrade the form accuracy of the machined surface. A “trial and error” approach is normally adopted in FIB fabrication in order to find suitable machining parameters to obtain the intended structures. This method is expensive and time-consuming. Therefore, there is a need to carry out fundamental research on the FIB machining process and the associated effects to address the underlying necessities for predictability and productivity in FIB micro- and nano-manufacturing. Such studies will lay down the foundation for a deterministic FIB fabrication approach.

3.1 The prediction of surface topography evolution in FIB machining

3.1.1 The surface generation mechanism in FIB milling process

The formation of the surface topography during FIB fabrication is a complex process in which material removal and redeposition occur simultaneously. Figure 3-1 illustrates the surface generation mechanism of the FIB milling process. When a beam of ions bombards a work piece, some atoms will be sputtered out of the work piece surface. Some of the sputtered atoms will adhere to the machined surface due to the redeposition effect which strongly depends on the work piece material and ion beam properties.
Moreover, the local surface topography also has an important role in the redeposition effect; this effect can only take place when the motional trajectories of the sputtered atoms interfere with an existing boundary of the machined structure (see Figure 3-1, point dA'). Since the redeposition effect is random, the actual tracking of the fabrication process cannot be achieved analytically.

**Figure 3-1** Schematic illustration of the generation mechanism for a machined surface fabricated by FIB.

The prediction of this dynamic process can be achieved by using a numerical simulation method. The surface topography can be represented by a number of nodes whose trajectories evolve as a function of time during the FIB fabrication process. The normal velocity $v_\perp$ of each node is proportional to the total flux at the corresponding node. It can be described as:

$$v_\perp = \frac{F_{\text{total}}}{N}$$  \hspace{1cm} (3.1)

where $F_{\text{total}}$ is the total flux (atoms/µm$^2$/s) and $N$ is the atom density of target material (atoms/µm$^3$). The total flux at each node on the target surface consists of two parts, i.e. the flux of sputtered atoms ($F_{\text{direct}}$) caused by the incident ion beam directly and the flux
of redeposition atoms \( F_{\text{indirect}} \) which is contributed to by \( F_{\text{direct}} \). They can be described as:

\[
F_{\text{total}} = F_{\text{direct}} + F_{\text{indirect}}
\]  

(3.2)

\[
F_{\text{direct}} = \frac{F_{\text{incident}} Y(\theta) A_s}{A_t} = F_{\text{incident}} Y(\theta) \cos(\theta)
\]  

(3.3)

\[
F_{\text{indirect}} = -S_y \int \frac{F_{\text{direct}} f(\alpha) d\Omega}{dA} dA = -S_y \int f(\alpha) d\alpha d\varphi dA = -S_y \int f(\alpha) d\alpha d\varphi dA
\]  

(3.4)

As shown in Figure 3-1, \( A_s \) and \( A_t \) are the cross sectional area and the actual area on the target surface, respectively. \( Y(\theta) \) is the sputter yield at an incident angle \( \theta \). \( S_y \) and \( f(\alpha) \) are the sticking coefficient and the angular distribution of the sputtered atoms. \( d\Omega \) is the infinitesimal emission solid angle of the sputtered atoms; \( dA \) and \( dA' \) are both the infinitesimal areas on the target surface. For symmetrical structures, a rotational angle \( \varphi \) is needed to reconstruct three-dimensional surfaces.

### 3.1.2 Surface topography simulation by level set method

#### 3.1.2.1 Introduction of level set method

The level set method [137] is a numerical technique designed to track the evolution of interfaces and shapes between two different regions. This method makes it very easy to follow irregular and dynamic shapes that change along with the variation of time. A “level set” of a function \( \phi \) with \( n \) variables is a set of the form:

\[
L_c(\phi) = \{(x_1, \cdots, x_n) \mid \phi(x_1, \cdots, x_n) = c\}
\]  

(3.5)

where \( x_1 \sim x_n \) are the \( n \) variables of function \( \phi \); \( c \) is a given constant value; and \( L_c(\phi) \) represents a set of such variables that meet equation (3.5).
The idea of the level set method is to take the propagating interfaces ($L_c(\phi)$) as a certain level set ($c$) of a higher dimensional function which is called a level set function ($\phi$). The advantage is that merging and breaking, sharp gradients and cusps on the interfaces can be handled easily. Therefore it is suitable for tracking freeform interfaces. As with the evolution of the interfaces, the level set function also evolves under certain restraint conditions. The moving state of the interfaces can always be represented by the level set $c$ of the level set function. Tracing the evolution of interfaces by the level set method is actually a combination of solving level set equations and constructing the restraint conditions.

Considering a vector $x$ evolving in the time $t$ with velocity $v_\perp^2$, the level set function can be expressed as $\phi(x,t)$. At any time $t$, the moving front of vector $x$ can be embedded in the level set function in the form of:

$$\phi(x,t) = c \quad (3.6)$$

where $c$ is a given constant. By applying the chain rule on both sides of equation (3.6):

$$\frac{\partial \phi(x,t)}{\partial x} dx + \frac{\partial \phi(x,t)}{\partial t} dt = 0 \quad (3.7)$$

$$\frac{\partial \phi(x,t)}{\partial x} \frac{dx}{dt} + \frac{\partial \phi(x,t)}{\partial t} = 0 \quad (3.8)$$

the moving front of the vector $x$ at any time $t$ is then determined by solving time-dependent Hamilton–Jacobi equation in the form of equation (3.9) with an initial condition expressed in equation (3.10):

$$v_\perp(x,t) |\nabla \phi(x,t)| + \phi_t(x,t) = 0 \quad (3.9)$$

$$\phi(x,t = 0) = \Gamma \quad (3.10)$$

2 The subscript “$\perp$” indicates the direction of the velocity is perpendicular to the moving front of vector $x$. That is to say, the velocity $v_\perp$ is along the gradient direction.
where

\[ \nabla \phi(x,t) = \tilde{n} \cdot \left| \nabla \phi(x,t) \right| = n \cdot \frac{\partial \phi(x,t)}{\partial x} \]  \hspace{1cm} (3.11) 

and

\[ v_\perp(x,t) = n \cdot \frac{dx}{dt} \]  \hspace{1cm} (3.12) 

\( \tilde{n} \) is the normal direction of the moving front. \( \Gamma \) is the initial level set function. \( \Gamma - c \) is a signed distance function, which represents the distance from the point \( x \) to the moving front where a positive sign means the point is outside the moving front and vice versa. Therefore, the moving front is the data set of the points corresponding to \( \Gamma - c = 0 \). Particularly, when \( c \) is set to be zero, the signed distance function becomes \( \Gamma \); the moving front is the zero level set of the solutions derived from equation (3.9) and equation (3.10).

Here, a hidden point is that the velocity \( v_\perp \) is not only defined for the level set corresponding to the moving front of the vector \( x \) but also on the other level sets which do not have physical meanings. In order to solve equation (3.9), \( v_\perp \) is assumed to be defined for all the level sets. Therefore, both the interface and the velocity are embedded in a higher dimensional function. The newly constructed velocity is called extension velocity (\( v_{ext} \)). The extension velocity should, in the limit as one approaches the level set \( c \), yield the velocity of the level set \( c \), i.e.,

\[ \lim_{x \to c} v_{ext}(x) = v_\perp(a) \]  \hspace{1cm} (3.13) 

where \( a \) is a point on the moving front.

All in all, it can be concluded that in order to track the surface topography evolution of the propagating interfaces by the level set method, one could follow the procedures:
1. Determination of the velocity of the propagating interfaces \((v_{\perp})\) during the evolution process.

2. Construction of the initial level set function \((\Gamma)\) where the initial state of the propagating interfaces is embedded into certain level set \(c\) (see foot note 2).

3. Construction of extension velocity \((v_{\text{ext}})\).

4. Solving level set functions \((3.9)\).

5. Inferring the level set \(c\) from the solution of the level set function at time \(t\). The level set \(c\) is actually the state of the propagating interfaces at time \(t\).

The level set method can be interpreted through a basic example. Imagine two identical circles with radii of 50nm and the distance between the two centres is 120nm. The closest distance between the circumferences is therefore 20nm. If the circles expend from the centres at a speed of 1nm/s, they will be tangential to each other at 10s, after which they merge together. This process can actually be simulated by the level set method. Here \(v_{\perp}\) is 1nm/s and the two circles can be embedded into two connected cones. The extended velocity on the cones is set to be 1nm/s, which satisfies the restraint condition in equation \((3.13)\). The whole propagating process is graphically illustrated in Table 3-1. The simulation covers the propagating state from 0s to 20s. At \(t = 10s\) the two circles are at a tangent to each other as proposed. After this point, the two circles merge together with a smooth edge and connections. This example concludes that, under this theoretical framework, the surface topography of complex propagating interfaces with known velocities can be predicted accurately. When it is used to solve different cases, the most important parameter to determine is the velocity of the propagating interfaces.

\(^3\) For the sake of convenience, \(c\) is generally set to be 0.
Table 3-1 Tracing the surface topography evolution of two expending circles by the level set method.

<table>
<thead>
<tr>
<th>$t$</th>
<th>$\phi$</th>
<th>$I_0(\phi)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$t = 0 \text{ s}$</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
<tr>
<td>$t = 5 \text{ s}$</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
<tr>
<td>$t = 10 \text{ s}$</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
<tr>
<td>$t = 15 \text{ s}$</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
<tr>
<td>$t = 20 \text{ s}$</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
</tbody>
</table>
3.1.2.2 Implementation of the level set method in the simulation of FIB machining processes

The surface topography generation in FIB fabrication is a highly dynamic process. This is because the ion redeposition will change the local surface slope, which further results in a change in the local sputter yield of the machining process. This phenomenon has, therefore, posed a difficult challenge in the convergence of the simulation algorithm and computational efficiency. In this work the level set method was linked to the FIB machining model through the following aspects:

- **Velocity of the machined surface**
  According to the FIB milling model introduced in section 3.1.1, the velocity of the machining interface is always along the surface normal direction, it is equivalent to the \( v_\perp \) in equation (3.12).

- **Visibility test**
  The redeposition effect is surface topography dependent. In order to take this effect into consideration in the simulation, a visibility test between any two points on the machined surface should be carried out; the redeposition effect only occurs when the two points can “see” each other. This visibility information has been included in the level set function. Since the machined surface is always on the level set \( c \), the level set function is divided into two parts by the machined surface, one part with level sets greater than \( c \) and the other part with level sets less than \( c \). If two points can see each other, i.e. the line segment joining the two points does not intersect with the machined surface, then the level set value of the points on the connecting lines must be greater than or less than \( c \). In this case, the redeposition atoms from one point can reach the other point.

- **Extension velocity**
  Extension velocities for those points which are not on the machined surface are needed to maintain the propagation of the level set function. Under the constraint condition of equation (3.13), the most straightforward way to construct the extension velocity at such a point is by assigning the velocity at the nearest point on the machined surface as the local velocity.
**Solving level set equation**

When solving the level set function, the key concept for approximating the equations of motion is to select an approximation to the gradient operator $\nabla \phi$. Forward Euler time discretization and upwind spatial differencing are used as a robust way to solve the hyperbolic level set equation. One of the simplest upwind entropy satisfying approximations to a gradient $\nabla \phi$ is called the first order space convex [125]. The term $v_\perp(x,t)|\nabla \phi(x,t)|$ in equation (3.9) can be simplified to the following expression:

$$v_\perp(x,t)|\nabla \phi(x,t)| = \max(v_\perp,0)\nabla^+ + \min(v_\perp,0)\nabla^-$$  \hspace{1cm} (3.14)

where

$$\nabla^+ = [\max(D^{-x},0)^2 + \min(D^{+x},0)^2 + \max(D^{-y},0)^2 + \min(D^{+y},0)^2]^{1/2}$$  \hspace{1cm} (3.15)

$$\nabla^- = [\max(D^{-x},0)^2 + \min(D^{+x},0)^2 + \max(D^{-y},0)^2 + \min(D^{+y},0)^2]^{1/2}$$  \hspace{1cm} (3.16)

Here a shorthand notation is used. Right-hand derivative $D^{+x} \phi^n$ is written as $D^{+x}$, while left-hand derivative $D^{-x} \phi^n$ is written as $D^{-x}$, etc. $\phi^n$ is the approximation to the solution $\phi(x,n\Delta t)$ where $\Delta t$ is the time step in the simulation. According to Equations (3.14) ~ (3.16), the evolution of the level set function $\phi$ at any time $t$ can be solved.

**Angular dependence of sputter yield**

The sputter yield varies primarily with incident angle. The sputter yield at the time $n\Delta t$ is determined by the surface topography at time $(n-1)\Delta t$. Therefore, as long as the initial conditions of the level set function are specified, the sputter yield at any time can be derived. The velocity $v_\perp$ at the time $n\Delta t$ is then updated.
In conclusion, the level set method is introduced to model the FIB machining process. The angular dependence of sputter yield and redeposition effect have been taken into consideration. Machined surface topography at any time can be predicted by this approach. The experimental validation of the effectiveness of this method will be discussed in the following chapters.

3.2 The determination of machining parameters for obtaining high precision micro- and nano-structures

In this section, the development of a divergence compensation method is presented in order to facilitate the production of ultra precision three-dimensional structures. Key fabrication parameters, such as dwell time distribution, scanning passes, scanning pitch and scanning strategy can be determined by this method.

3.2.1 The divergence compensation method

When FIB is applied for the fabrication of three-dimensional structures, the machining accuracy can be degraded due to the finite spot size of the incident ion beam and sophisticated ion-solid interactions. There are three main aspects which impact on this:

- Firstly, the beam intensity profile is usually considered as Gaussian distribution with circular cross section [113]. The tail of the Gaussian distribution broadens the beam profile and contributes extra ion flux to the adjacent area. For each milling point, the accumulation of all the extra fluxes contributed by the adjacent points result in an unintentional increase of the local ion dose unintentionally.

- Secondly, the variation of the sputter yield under different ion incident angles also degrades the machining accuracy of FIB milling. The prediction of the milling depth based on the incident ion dose is hampered by the variation of the sputter yield.

- Thirdly, the redeposition effect, as well as the scanning strategy, can further affect the machining accuracy.

Proper divergence compensation techniques are therefore needed to overcome the above issues in order to fabricate complex structures with high accuracy using FIB.
3.2.1.1 Surface topography model

To optimize the FIB milling process, a predictive divergence compensation approach is implemented through the development of a three-dimensional surface topography generation model. In this model, the machined surface is described as a grid in the $x$–$y$ plane composed of arrays of equally spaced intervals, where the gridlines intersect at the nodes (Figure 3-2). The numerical value of each node represents the elevation of this surface point above a fiducial plane. Each node is surrounded by four other adjacent nodes, which can be combined into patches to investigate the surface normal at the target node. The surface normal direction at the target node is determined by the relative positions of the four adjacent nodes. As illustrated in Figure 3-2, the surface normal at the target node $O$ is defined as the average value of the normal directions of pitches $OAB$, $OBC$, $OCD$ and $ODA$. Once the direction of a node is defined, the separation angle between the incident ion beam and the machined surface is specified. Therefore, the sputter yield at each node can be obtained.

![Figure 3-2 Schematic illustration of surface topography model](image)

3.2.1.2 Dwell time distribution

The FIB milling depth is derived from the ion incident flux, the atom density of the target material and the sputter yield. The total incident flux at a certain node is contributed not just from the local pixel area but also from all of the adjacent areas due to the overlap effect. Therefore, the dwell time for each node can be obtained from a matrix relationship as showed in equation (3.17), which includes the ion beam distribution, ion beam overlap and angular-dependent sputter yield [129]:

$$\sum_{i,j=1}^{n} C_{ijkl} S_{ijkl} P_{xy} = \Delta Z_{ij}$$  \hspace{1cm} (3.17)
where $C_{ijkl}$ is the coefficient matrix which describes the flux contribution from the adjacent node $(k, l)$ to the local node $(i, j)$. $sp_{kl}$ and $t_{kl}$ are the scanning passes and the dwell time at node $(k, l)$, respectively. $p_x$ and $p_y$ are pitches along the pixel address scheme and $\Delta Z_{ij}$ is the milling depth increment at node $(i, j)$.

The incident ion beam current density needs to be corrected when the ion beam is presented to inclined surfaces. In this case, the number of ions per area decreases as $\cos(\theta)$, where $\theta$ is the separation angle between the incident ion beam and the surface normal. Therefore, the corrected milling depth at node $(i, j)$ can be expressed as:

$$\Delta Z_{i,j} = \sum_{k,l=1}^{n} \frac{\Phi(k,l)}{\eta} J_{kl}(i,j)Y(\theta_{ij})\cos(\theta_{ij})sp_{kl}t_{kl}p_{x}p_{y}$$  \hspace{1cm} (3.18)$$

where $\Phi(k,l)$ is the ion flux density at node $(k, l)$. $\eta$ and $J_{kl}$ are the atom density of the solid and the ion beam intensity distribution function at node $(k, l)$, respectively. Generally $J_{kl}$ follows Gaussian distribution. A three-dimensional ion beam intensity distribution is shown in equation (2.13). $Y(\theta)$ is the angular-dependent sputter yield. The summation over the indices $k$ and $l$ accounts for total dose received at node $(i, j)$ from all the pixels in the address scheme.

Equation (3.18) can be applied to each node on the machined surface to obtain a corrected FIB milling depth. In an area comprising of $N \times N$ nodes, the total milling time $p_{kl}t_{kl}$ at each node is derived from an $N \times N$ equation set composed of equation (3.18). Therefore, the dwell time distribution is calculated by equation (3.19):

$$t = \frac{Z}{C}$$  \hspace{1cm} (3.19)$$

where $t$ and $Z$ are dwell time matrix and milling depth matrix, respectively. $C$ is the coefficient matrix of the equation set (3.18).
3.2.1.3 Determination of optimized scanning passes

A multi-pass scanning method is used in FIB machining in order to reduce the redeposition effect. It is also helpful in forming a uniform bottom surface in the groove as shown in Figure 2-22(b), but the required number of passes still needs to be investigated.

For removing a quantity of material, the total ion dose should be constant and independent with the number of the scanning passes as long as the ions incident along a fixed incident angle. The relationship between the total ion dose $F_{\text{total}}$ and the number of the scanning passes $sp$ is expressed as (3.20):

$$F_{\text{total}} = I \cdot \frac{L}{v_s e} \cdot sp$$  

(3.20)

where $I$ is the ion current; $L$ is the total length of beam scanning route in each scanning pass, $v_s$ is the beam scanning speed and $e$ is elementary electronic charge. This indicates that the number of scanning passes is inversely proportional to the scanning speed. Determination of the number of scanning passes is equivalent to determining the scanning speed. Santamore et al. found that in the FIB machining process, the scanning speed has a significant effect on the sputter yield [138]. For very rapid scanning at normal incidence, each pass of the beam removes a thickness of material that is much smaller than the beam diameter. In this case, the sputter yield corresponds to the yield at normal incidence. If the scan speed is slowed down, the thickness removed in each scanning pass is at the same size as the beam diameter. The actual incident angle at the sputtering point is no longer zero even though the beam is still perpendicular to the surface. A schematic diagram of this phenomenon is illustrated in Figure 3-3. This illustrates that, under normal incident condition, the effective ion beam incident angle $\theta$ at the local milling point is widely divergent from the normal incident angle when the material removed in each scanning pass is at the same level as the beam diameter. The increase in the local incident angle leads to a variation in the sputter yield and thus causes the fabrication process unpredictable. Therefore, the material removed in each scanning pass should be much less than the beam diameter at the selected scanning speed in order to confine the divergence between the practical sputter
yield and the theoretical sputter yield applied in the machining model. The lower limit of number of the scanning passes should be controlled under this constraint.

Figure 3-3 Schematic diagram of a one-dimensional sputter model at steady state: (a) material removed in each scanning pass is much smaller than the beam diameter; (b) material removed in each scanning pass is at the same level as the beam diameter.

3.2.2 The implementation of the divergence compensation method in FIB machining

In a modern FIB systems, pattern generation in conjunction with graphic bitmap files as inputs enables the achievement of complex two-dimensional structures. Each pixel in the bitmap represents an incident beam spot. The local colour value of each pixel in the bitmap delineates the dwell time and beam blanking status. In a 24 bit RGB bitmap each pixel consists of:

- A red component – not used.
- A green component – determines if the beam is blanked, any other value other than 0 activates the beam.
- A blue component – determines the dwell time per pixel, the blue ranges from 0 to 255, which corresponds to an 8 bit colour depth.

Figure 3-4 shows an example of a FIB fabricated two-dimensional structure based on the corresponding bitmap image. It reveals the transformation from the colour scale level in the bitmap to the milling depth by varying the dwell time for each pixel. The brightest part in the bitmap corresponds to the longest dwell time, leading to a concave
structure, while the darkest part corresponds to the shortest dwell time. The flexibility in controlling the dwell time at each pixel enables a precise method for manufacturing complex three-dimensional structures by FIB milling.

![Heriot Watt University](image)

(a) (b)

**Figure 3-4** FIB milling of a two-dimensional structure. (a) The bitmap image applied; (b) the corresponding structures obtained from the bitmap.

However, the structures obtained using such a bitmap always diverge from the original design due to the intrinsic problems associated with the FIB machining mechanism, including atom redeposition, the overlap effect of the ion beam and the variation of the sputter yield under different incident angles. This divergence can be compensated for by the application of the divergence compensation method proposed in section 3.2.1; based on this, the dwell time distribution is corrected layer by layer and a series of bitmaps can be generated. In each scanning pass, the colour spread of each bitmap is identical to the dwell time distribution calculated by equation (3.19). The flowchart of the data preparation and data processing for the generation of bitmaps used in FIB machining is illustrated in Figure 3-5. As a demonstration of this process, Figure 3-6 shows a series of bitmaps for the fabrication of a hemisphere structure, where 10 bitmaps are applied.
Figure 3-5 Flowchart of the data preparation and data processing for FIB machining.
3.3 Summary

In this chapter, an approach for deterministic fabrication of three-dimensional structures is presented. This presentation is based on a modelling approach to predict the surface
generation in FIB machining process under given fabrication parameters. It was implemented based on an ion-solid sputtering model followed by a topography simulation realized by the level set method to obtain high precision required structures. The redeposition effect and the angular dependence of sputter yield are both taken into consideration.

In order to determine optimized FIB fabrication parameters, a divergence compensation method is also developed. This method was implemented in FIB machining by transferring the dwell time information onto a series of bitmaps which can be easily recognized by the system.
Chapter 4 Deterministic Fabrication of Nanodot Arrays

— A case study to overcome the dimensional challenge in FIB machining

4.1 Introduction

Recently, the fabrication of nanodot arrays on various materials has attracted intense scientific interests due to their great potentials across a wide range of technological applications. Metal nanodot arrays which exhibit unique properties based on their nano-size effects are expected to be the building blocks of new functional nano-devices, in such fields of use as catalysis, environmental remediation, DNA detection, high density data storage, and optical devices. Periodic nanodot arrays can also excite surface plasmon resonance. This phenomenon is the basis of many standard tools for measuring the adsorption of material onto planar metal surfaces (typically gold and silver) or onto the surface of metal nano-particles. When nanodot arrays are applied in a plasmonic solar cells, theoretically the light wave is supposed to be absorbed more directly without the relatively thick additional layer required in other types of thin-film solar cells [139]. Surface plasmon resonance also underpins the function of a plasmonic lens, which is a key component in the development of sub-wavelength resolution optical system for bio-imaging and nanolithography applications [10-11]. Nanodot arrays and nano-gratings are typical nanostructures used in a plasmonic waveguide to couple free space light into surface plasmon in a far field plasmonic lens. The precise and deterministic fabrication of these nanostructures is essential for their performance. For example, in a plasmonic waveguide, the diameter of a nanodot should be small enough to ensure that no propagating modes can be supported at the wavelengths of interest, while the depth of the nanodot significantly affects the intensity of transmitted light. The precise and efficient machining of periodic nanodot arrays is required in many of the applications that demand low-cost nano-patterning technologies to gain a competitive edge. Lithography tools, such as EBL (electron beam lithography) and FIB technology, allow well defined and well positioned nanodots, but they are limited by low throughput and small areas, and are not suitable for mass production when a large nanodot array area is required. In the last two decades, researchers have investigated a number of alternative and potentially low-cost nanofabrication methods, such as NIL [140-141] and μCP [142]. Soft NIL with PDMS (Polydimethylsiloxane) stamps has been demonstrated previously [143] and very high resolutions down to 10nm have been
achieved [144]. In the printing process, the surface quality of replicated structures depends on the quality the machined master structures; it cannot be improved during stamping. Therefore, the accurate machining of hard structured glass masters is essential for the preservation of structure profiles in the replication process.

In this chapter, FIB technology is applied for the deterministic fabrication of a nanodot array on a hard silicon mould. The fabrication parameters of the nanodot array were derived from the simulation model proposed in section 3.1. The hard silicon mould produced can subsequently be applied as a master to replicate the nano structures on PDMS masters by NIL. This work demonstrates a breakthrough in the dimensional challenges in FIB machining.

4.2 Deterministic fabrication of silicon mould for nanodot arrays

When FIB machining is used to fabricate nanodot arrays, the form accuracy achieved can be greatly degraded due to the redeposition in the sputtering process. This effect will cause the machined topography to diverge from the intended shape. A trial and error approach is normally used to correct the fabrication error caused by the ion redeposition since the contour of the generated nanoscale structure is very difficult to predict. On the other hand, the experimental realization of these nanostructures is complex and time consuming. Therefore, it is very important to have access to predictable and optimized geometries of nanodots during the design stage. In this work, the FIB sputtering model introduced in section 3.1 is applied to predict the surface topography of a nanodot. The simulation results can be used to guide to the FIB experiment. In the FIB sputtering model, the sputter yield $Y(\theta)$ and the angular distribution of sputtered atoms $f(\alpha)$ used in the simulation program need to be determined.

4.2.1 Determination of sputter yield

Sputter yield primarily depends on the ion incident angles and varies in the range 0° to 90°. Due to the variation of surface gradient in the ion sputtering process, the material removal rate will be significantly affected by the conspicuous variation of the sputter yield. A precise investigation on the relationship between sputter yield and incident angles is essential to this work.
The sputter yield $Y(\theta)$ was calculated using Monte Carlo simulation programs, TRIDYN [110-111]. TRIDYN simulates the ion irradiation of amorphous targets in the binary collision approximation. It allows for a dynamic rearrangement of the local composition of the target material. Therefore, the effects of high-fluence implantation, ion mixing and preferential sputtering caused by atomic collision processes can be derived. During the simulation process, heats of sublimation were used as the surface binding energies, namely 4.7 eV for the silicon substrate and 2.82 eV for the implanted gallium ions. In Monte Carlo method, the uncertainty of the simulation result is proportional to $H^{-1/2}$, where $H$ is the number of simulation histories. In this case $H$ represents the number of pseudo projectiles. A fluence increment of $10^{12}$ ions/cm$^2$ per pseudo projectile is used to guarantee the statistical quality and the precision of the results. In this work, ion fluence is estimated to be $1.27 \times 10^{14}$ ions/cm$^2$, corresponding to a history number of 127. $H=300$ was therefore used to guarantee the statistical quality. Figure 4-1 shows the comparison of the simulation result calculated by TRIDYN, Yamamura’s semi-empirical formula [93], and experimental results [94, 121, 129, 145] of the sputter yield when 20 keV and 30 keV gallium ions bombard a silicon substrate. It can be seen that the sputter yield keeps increasing when the incident ion angle varies from 0° to 80°. The sputter yield reaches its maximum value at 6 times higher than that at incident angle of 0°. It then drops to zero dramatically from 80° to 90° where only glancing incidence occurs. The comparison of the results also reveals that the simulation results obtained by TRIDYN are much closer to the published experimental data. On the contrary TRIM shows a higher sputter yield at higher incident angles with limited accuracy of around 50% due to the pure static approximation. Therefore, the calculation results from TRIDYN were chosen as the sputter yield called in the proposed surface topography simulation.
Figure 4-1 Comparison of angular dependence of sputter yield when a beam of ions bombards a silicon substrate: (a) sputter yield at 20 keV; (b) sputter yield at 30 keV.

4.2.2 Determination of angular distribution of sputtered atoms

The angular distribution of sputtered atoms generally follows a cosine distribution for normal incidence ion bombardment [87-88]. For varying ion incident angles, the angular distribution of the sputtered atoms $f(\alpha)$ is slightly different and the deviations from the cosine rule can be corrected by a power of $n$ [146]:

- TRIDYN
- Yamamura theory
- Adams D P, 2006 [94]
- Kaito H, 1999 [94]
- Frey L, 2003 [145]
- Platzgummer E, 2006 [121]
\[ f(\alpha) = \cos^\alpha(\alpha) \]  

(4.1)

In order to determine this correction factor in equation (4.1), the angular distribution function was simulated by using the Monte Carlo method. This work simulated a beam of 30keV gallium ions bombarding a silicon substrate in the x-y plane with an incident angle of 0º along the z axis as shown in Figure 4-2. The total history number was set to be 10000 to guarantee the simulation accuracy. The heat of sublimation, 4.7 eV, was taken as the surface binding energy of silicon. The lattice binding energy \(^4\) and displacement energy \(^5\) were assumed to be 2 eV and 15 eV, respectively.

![Figure 4-2 Scheme of the simulation plan.](image)

The simulated distribution of sputtered atoms in the x-y plane is shown in Figure 4-3. The coordinate of each point in Figure 4-3 is represented by the cosine of the final trajectory of sputtered atom. The angular distribution in the whole scattering space was analyzed from two orthogonal cross sections, the x-z plane and the y-z plane. As shown in Figure 4-4, in each plane the total scattering angle (\(\pi\)) was discrete with an angle step of \(\Delta\alpha\). In this work \(\Delta\alpha\) was set to be 4º to meet the statistical requirement that there were at least 10 particles falling in each angular region. All of the sputtered atoms in the whole scattering space were projected onto this plane. The number of the atoms that fall into the region \([\alpha - \Delta\alpha/2, \alpha + \Delta\alpha/2]\) was counted as \(\Delta n\). The angular distribution was the sputtered atoms per unit area:

\(^4\) The minimum energy needed to remove an atom from a lattice site.
\(^5\) The minimum energy required to knock a target atom far enough away from its lattice site so that it will not immediately return.
Chapter 4  Deterministic Fabrication of Nanodot Arrays

\[ f(\alpha) = \frac{\Delta n}{2\pi (\cos(\alpha - \Delta \alpha/2) - \cos(\alpha + \Delta \alpha/2))} \]  \hspace{1cm} (4.2)

Equation (4.2) can be further simplified and normalized as:

\[ f(\alpha) = \frac{\Delta n}{2 \sin \alpha \sin(\Delta \alpha / 2)} \]  \hspace{1cm} (4.3)

Based on equation (4.3) and the simulation results, the angular distributions of the sputtered atoms in the x-z plane and y-z plane were obtained. The two distributions are

**Figure 4-3** Simulated distribution sputtered atoms in the x-y plane.

**Figure 4-4** Schematic of the x-z (or y-z) plane in the scattering space.
shown in Figure 4-5(a) and Figure 4-5(b), respectively. Both of the distributions are fitted tightly to the cosine rule in the form of equation (4.1). In the x-z plane and the y-z plan the correction factor $n$ was found to be 1.6.

Figure 4-5 Monte Carlo simulation of the angular distribution of the sputtered atoms when 30keV gallium ions strike a silicon substrate. (a) Angular distribution of sputtered atoms in x-z plane; (b) angular distribution of sputtered atoms in y-z plane.
In this work, the simulation of FIB fabrication is assumed to start from a flat surface. However, from the start of the simulation, the machined surface will move and then becomes rough at the atomic level which results in a variation of the ion incident angles from -90° to 90°. This is expected to spread the calculated angular distribution and finally give a result not very different from a cosine function. For simplicity a cosine function with correction factor 1.6 is applied as the angular distribution function in this model and is used for further calculations.

4.2.3 Determination of sticking coefficient

The sticking coefficient $S_c$ is defined as the ratio of the flux caused by redeposition to the flux of sputtered atoms generated by the incident ion beam. It stands for the absorption degree between the redeposited atoms and the machined surface. In this model $S_c = 1$ was adopted since no chemical gas assistance is involved in the machining process.

4.2.4 Simulation of surface topography evolution

The simulation of surface topography was realized by using the level set method. According to the ion-material sputtering model, the surface velocity is proportional to the total flux which is composed of two parts: (i) the flux contributed by ion beam direct sputtering ($F_{direct}$) and (ii) the flux contributed by redeposition ($F_{indirect}$). In this work, both of these fluxes were taken into consideration.

The simulation started from a beam of gallium ions with Gaussian distribution bombarding a flat silicon substrate perpendicularly. The flat plan was embedded into a level set function as the zero level set. In the sputtering process, the dwell time was set at discrete a time steps of 1 µs. At each consecutive step, the simulation sets the zero level set as the current surface topography of the nanodot. Based on the current surface topography, the sputter yield at each pixel for the next time step was then obtained. Therefore, the direct sputtering flux $F_{direct}$ was achieved.

The calculation of $F_{indirect}$ is more complex. On the one hand, the value of $F_{indirect}$ is built on $F_{direct}$; it can only be obtained when $F_{direct}$ is determined. On the other hand, not every point on the surface can receive $F_{indirect}$ from other points; $F_{indirect}$ only worked when
the two points can “see” each other. Therefore a visibility test was needed in every time step. In level set method, the visibility test has been contained in the level set function. This can be clearly observed in Figure 4-6. If the level set function $\phi$ is always positive between two points on the surface (point O and point A), these two points are visible to each other which means the redeposition atoms from one point can reach the other point. Between the points O and B, $\phi$ is only positive in the OC segment. After passing through the moving front (point C where $\phi = 0$), $\phi$ changes to negative in CB. This means point O and B cannot see each other. Sputtered atoms from O to B are blocked at point C. Thus the redeposition will not occur between point O and point B.

All the calculations and analysis above led to the possibility of determining the surface velocity evolution with redeposition effect. However, in order to solve the level set function, the velocity was defined not only on the machined surface but over the whole domain of the level set function. Therefore, extension velocities in the whole domain had to be constructed. Extension velocities are needed to maintain the propagation of the level set function. The construction of the extension velocities should meet the

Figure 4-6 Visibility test on level set function
requirement of equation (3.13). In this work, a simple copy of the velocity from the nearest point on the machined surface was applied to construct the extension velocities.

Forward Euler time discretization and upwind spatial differencing were used as a robust way to solve the hyperbolic level set equation. The approximate solutions of the level set function at different times are shown in Figure 4-7. It indicates that the level set function evolves from a plane \((t=0\text{ms})\) to a curved surface \((t=5.5\text{ms})\). Meanwhile, the cross section of the machined structure is naturally formed by tracking the zero level set of the level set function. Due to the spatial symmetry of the nanodot, three-dimensional re-construction relative to the symmetry axis was applied as shown in Figure 4-8. Finally a quasi three-dimensional simulation program based on the level set method was developed for FIB nanofabrication.
Figure 4-7 Evolution of nanodot profile in the simulation process.
4.3 Experimental validation

Experimental work on the fabrication of nanodot arrays on a silicon substrate was carried out by using a dual beam FIB system (FEI Quanta3D FEG) with gallium ion source. As a comparison test to evaluate the simulation results, a 30keV gallium beam with current of 10pA was used to drill two series of nanodots on (100) surface of a silicon substrate. The dwell times were fixed at 5.5ms and 8.0ms, respectively. According to the simulation results, nanodot arrays with depth of 51nm and 71nm can be obtained in these situations. The FIB fabricated nanodot arrays were measured by an atomic force microscope (AFM, HYSITRON TI900 TriboIndenter). The maximum measured depths were 53nm and 76nm, respectively. A comparison of the simulation and experimental results is shown in Figure 4-9. It can be seen that the simulation results agreed well with the experimental results in terms of maximum fabrication depth with errors less than 7%. The maximum divergence takes place at the bottom part of the nanodots, where a relatively flat bottom is predicted by the simulation but a sharp bottom is observed in the measurement results. The reason for these discrepancies is the limitation of the geometry of the AFM tip which is not sharp enough to reach the sidewall of the nanodot.

This validation indicates that a good agreement between the simulation and experimental results had been obtained with simulation errors less than 10%. The modelling and
simulation program, therefore, could be used to determine machining parameters to achieve deterministic FIB nanofabrication.

Figure 4-9 Comparison of simulation and experimental results of two nanodots fabricated by FIB.

4.4 Deterministic fabrication of a silicon mould

For this evaluation, a silicon master with a pattern of periodic nanodot arrays was formed on a P type silicon substrate (100). The mouth width, depth and pitch of the nanodot array were 90nm, 120nm and 100nm respectively. The fabricated nanodot arrays are shown in Figure 4-10. In the fabrication process a 30 keV gallium beam with 50pA ion beam current was used. A dwell time of 4.2 ms determined by the simulation software was applied to this experiment. After the generation of the nanodot array, platinum deposition and ion beam cross sectioning processes were applied in order to measure the profile of the nanodots. The platinum deposition was helpful in protecting the nanodot during the cross sectioning process. Meanwhile, the contrast of the SEM micro image was also enhanced due to the existence of the platinum layer, which made it much easier to determine the profile of the nanodots. Measurement results of the depth of the nanodot confirmed that the maximum machining error was 7nm.
Figure 4-10 Cross sectional profile of the nanodot array: (a) SEM image of the cross sectional profile of the nanodot array; (b) comparison between the experimental and simulated cross sectional profiles of a single nanodot; (c) SEM image of the nanodot arrays.

4.5 Investigation of the redeposition effect
The difficulty in predicting the depth of nanodot is due to the redeposition effect. If no redeposition occurs, the milling depth of the nanodot would be proportional to the ion fluence and can be easily determined. Therefore, it is necessary to study the contribution
of the redeposition effect during the sputtering process. An accurate evaluation of the redeposition effect enables the prediction of milling depth without the simulation.

In order to study this effect, two sets of simulations were carried out using a 30keV gallium beam with 10pA ion beam current to fabricate nanodot arrays on a silicon substrate under a dwell times ranging from 0 to 10ms. In the first set of simulation the sticking coefficient $S_c$ was set to 0 in order to emulate an ideal machining condition without redeposition. In the other simulation set the sticking coefficient $S_c$ was set to 1 so as to take fully into account the influence of atoms redeposition. Figure 4-11 illustrates the variation of the depth of nanodot against dwell time from both the simulation test and experiment results under a dwell time of 5.5ms and 8ms. It was found that the milling depth increased linearly with the increase in dwell time when ion redeposition was ignored. On the other hand, there was a nonlinear increase of the FIB milling depth against dwell time, especially when the dwell time was less than 4ms. After 4ms the increase in milling depth could be regarded as linear again but with a slope lower than that without atoms redeposition. This confirms that the redeposition of atoms has significant effects in the FIB nanofabrication process. The simulation is an ideal tool for the quantitative evaluation of the effects of redeposition on the nanofabrication process. As illustrated in Figure 4-11, more than $\frac{1}{3}$ of the milling depth was reduced due to the redeposition effect.

![Figure 4-11](image_url)  
**Figure 4-11** Effect of redeposition on the nanodot fabrication process.
4.6 Summary

In this chapter, the deterministic FIB fabrication approach was successfully implemented to overcome the dimensional challenge in FIB machining.

Both the sputter yield and angular distribution of sputtered atoms were calculated by Monte Carlo simulation. The simulation results confirmed that the sputter yield primarily depends on the ion incident angles. When the incident angle was in the region of 80°, the sputter yield reached its maximum value which was 6 times higher than that at an incident angle of 0°. The angular distribution of sputtered atoms can be fitted well with a correction factor of 1.6 using the cosine rule.

The surface topography simulation based on the level set method is robustly stable and follows the surface generation in the actual FIB machining process. Ion beam direct milling and the redeposition effect were both taken into consideration.

By adjusting the sticking coefficient to zero, the redeposition effect can be nullified in the simulation program. The investigation on a net milling process proved that in the fabrication of a nanodot, more than $\frac{1}{3}$ of the milling depth was reduced due to the redeposition effect.

Based on the simulation results, a periodical nanodot array was successfully fabricated in a deterministic way by FIB machining. The experimental evaluation results have showed that the modelling and simulation program can precisely describe the generation of three-dimensional structures in the FIB machining process with less than 10% simulation error. The modelling and simulation program lay down a solid foundation to achieve deterministic nanofabrication using FIB.
Chapter 5  The Deterministic Fabrication of Three-Dimensional Structures

— Case studies to overcome the geometric challenge in FIB machining

5.1 Introduction
FIB machining has been widely used to form functional micro- and nano-structures through material removal on micro-photonics devices, novel scanning probes and micro/nano-print masters, etc. [32, 114, 147]. During the FIB machining process the sample stage is stationary whilst the ion beam scans over the defined area. With a state-of-the-art commercial FIB system it is very straightforward to obtain high precision two-dimensional structures. However, accurately and rapidly machining three dimensional structures at this scale is still challenging. By using the divergence compensation method developed in section 3.2, the optimized machining parameters can be determined in advance.

5.2 Deterministic fabrication of three-dimensional structures
In three-dimensional FIB machining, the milling depth at each pixel is determined by the sputter yield and dwell time. The dependence between sputter yield and ion incident angles was determined as the first step in this work. The divergence compensation method was then applied to calculate the corrected dwell time matrix based on the sputter yield. In the following step, the dwell time matrix was input into the FIB system in the form of bitmaps.

One assumption in the divergence compensation method is that the incident ions bombard the substrate simultaneously in each scanning pass – the scanning strategy is not taken into consideration. However, experimental results show that the scanning strategy does impact on the machined surface topography because of the redeposition effect [132]. A comparison of the structures fabricated by different scanning strategies will be carried out as a supplement of this research.
5.2.1 Determination of fabrication parameters

The divergence compensation method was integrated with bitmap milling. The corrected bitmaps with a specified number of pixels \( MN \) for the intended structures were generated using Matlab. These bitmaps were in 24 bits RGB scale, in which the local colour of each pixel represents the dwell time. The pixel number in the bitmap was determined by the following equation (5.1):

\[
\begin{align*}
M &= \frac{W}{p_x} \\
N &= \frac{H}{p_y}
\end{align*}
\]

where \( p_x \) and \( p_y \) are the pitches between adjacent pixels along the X and Y directions; \( W \) and \( H \) were the width and height of the designed pattern.

In this FIB milling experiment the multi-pass scanning method was used to reduce the divergence caused by redeposition. Twenty bitmaps were prepared for each proposed structure. For each bitmap, a wide range of dwell times can be applied depending on the size of the structure and incident ion current. For an ion current of 1nA with a beam diameter of 50nm, the maximum dwell time was set to be 300\( \mu \)s. The milling depth in each scanning pass at each pixel can be evaluated using equation (5.2):

\[
\Delta Z_{\text{max}} = \frac{t_{\text{max}} \times I \times Y}{\text{FWHM}^2 \times n \times e} \approx 35 \text{ nm}
\]  

The evaluation results show that \( \Delta Z_{\text{max}} \) is smaller than the beam diameter. The local incident angle at the milling pixel is around \( \tan^{-1}(\Delta Z_{\text{max}} / \text{FWHM}) \approx 35^\circ \). According to the relationship between sputter yield and ion incident angle illustrated in Figure 4-1(b), the sputter yield stays near constant from 0\( ^\circ \) to 35\( ^\circ \). Therefore, the practical sputter yield in the scanning process is in accordance with that used in the machining model. The constraint in section 0 is well maintained. The number of scanning passes \( sp \) for each bitmap was calculated from equation(5.3):
where $Y(0)$ is the sputter yield at 0 degree and $c$ is a coefficient which compensates for the ion beam overlap effect.

### 5.2.2 Investigation of scanning strategy

The scanning strategy in the FIB milling process also affects the machined surface topography. In the FIB milling process, the predefined pattern (Figure 5-1(d)) should first be digitized into arrays of pixels, arranged in rows and columns. The ion beam scans pixel by pixel over the pattern area along a scheduled route. As shown in Figure 5-1(a) and (b), two types of scanning strategies, raster and serpentine scans, are normally used to drive the ion beam movement. They are performed in a sequential manner where one scan line is followed by another. In the raster scan, the ion beam moves towards the same direction throughout the procedure, while in serpentine scan the scanning direction is reversed after each line. Compared with the serpentine scan, a drawback of the raster scan is that the beam always moves in a predefined direction while milling, which leads to asymmetrically shaped structures due to the redeposition effect. A straightforward solution to eliminate this kind of asymmetry is to scan the predefined pattern along the contours as it is illustrated in Figure 5-1(c). Such a vector scan can be achieved by a stream file, in which the coordinates of ion beam spot position are stored in a temporal sequence. The information in the stream file is then read into the computer memory and output to a digital-to-analog converter (DAC) to drive the FIB.

In order to investigate the influence of scanning strategies on form accuracy, a sinusoidal micro-lens array was fabricated by raster scan, serpentine scan and contour scan, respectively. The form accuracy of the micro-lens array was determined by measuring the cross-sectional profile and comparing it with the designed geometrical parameters. A layer of platinum to a thickness of 100nm was deposited onto the structure before the cross-sectioning process in order to protect the machined structure and increase the image contrast as shown in Figure 5-2(d). Figure 5-2(a) shows a sinusoidal micro-lens array with 3 µm amplitude and 10 µm period fabricated in a raster scan.
Due to the redeposition effect, asymmetric ripples appeared on the structures with a maximum height of 100nm. Figure 5-2(b) presents the same structure fabricated in a contour scan. Although the ripple profile was symmetric with the machined structure, the form accuracy was unexpectedly degraded. This divergence was believed to be caused during the beam directing process; since the ion beam was not blanked between each scanning pixel, there was always some ion dose leaked on the way from one scanning pixel to the next. This resulted in a cumulative effect which was conspicuous, particularly when the beam travelled back and forth in a finite area for a long time. The leaked ion dose caused an unpredictable divergence on the machined surface. Reducing the total travelling distance of the ion beam is believed to be beneficial to the form accuracy. Among the three scanning strategies, the ion beam travelling distance was minimized during the serpentine scan. In this case, the minimal ion dose leak was delivered. The same structure fabricated by a serpentine scan is shown in Figure 5-2(c). The surface ripple was greatly suppressed, and highly form accuracy was maintained. The measurement result showed that the amplitude of the cross-sectional profile was 2.89µm, which was only 110nm divergence from the designed structure. Among the three scanning strategies, the serpentine scan is the most reliable way for FIB micro-milling.

**Figure 5-1** (a) Raster scanning strategy; (b) Serpentine scanning strategy; (c) Contoured scanning strategy; (d) Designed surface topography of the freeform structured micro optics.

---

6 This is because all of the proposed structures in this work reach maximum depth at the points with zero surface slopes where the incident ion beam bombards vertically with a sputter yield $Y(0)$. 
82
Chapter 5     The Deterministic Fabrication of Three-Dimensional Structures

Figure 5-2 SEM images of two dimensional sinusoidal wave structures fabricated by FIB milling under different scanning strategies: (a) raster scan; (b) contour scan; (c) serpentine scan; and (d) cross sectional view of the structure fabricated by serpentine scan.

5.2.3 Experimental results and discussions
The FIB machining experiments were carried out on a dual-beam FIB system (FEI Quanta 3D FEG). The system was operated at an acceleration voltage of 30kV. The beam diameter (FWHM) and the ion current are 50nm and 1nA, respectively. Three-dimensional structures, including a parabolic structure (amplitude of 2.5µm, width of 10µm), two hemispherical structures (radius of 5µm and 500nm) and a sinusoidal structure (period 5 µm, amplitude of 0.9µm), were fabricated on a P-type silicon substrate (100) surface; the surface roughness of the silicon substrate is less than 5Å.
The sinusoidal microstructures fabricated by the conventional method and the predictive divergence compensation method are shown in Figure 5-3. With the predictive divergence compensation method the peak-to-valley form error of the machined structure has been reduced using the conventional bitmap milling approach from 200nm to 30nm. The machining accuracy was much improved; especially at the bottom part of the structure which was ameliorated to a curved profile instead of the flat one obtained using the conventional bitmap milling approach (see Figure 5-3(b)). Further FIB machining experiments indicated that a flat top/bottom could always be found on the top of the convex or the bottom of the concave shaped structures using conventional bitmap milling. The reason for this phenomenon is mainly related to the aspect ratio of the machined structure. Due to the low aspect ratio near the base, the differences in the ion dose used to fabricate the neighbouring points in the apex area of these structures are very tiny. An overlap of the Gaussian beam skirt in this region eliminates these tiny ion dose differences and results in the formation of a flat top/bottom. Since the overlap effect had been taken into consideration in the predictive divergence correction method, extra ion doses caused by beam overlap were compensated; therefore, more accurate structures were formed.

In addition, the existence of an oxide layer on the silicon substrate also contributes to the formation of a flat top/bottom on the low aspect ratio structures because the oxide layer is less sensitive to gallium ions than silicon. Figure 5-4(a) shows a low aspect ratio sinusoidal structure fabricated on a silicon substrate covered by a thin oxide layer, a flat part on the top of the structure was observed. Figure 5-4(b) shows that a precise sinusoidal feature was obtained using a silicon substrate after removing the oxide layer. Figure 5-5 and Figure 5-6 show fabricated parabolic and hemispherical microstructures on silicon substrate on which the oxide layer was removed before FIB machining. Form accuracies (p-v) of 20nm and 120nm respectively were achieved for these structures.
Figure 5-3 Comparison of sinusoidal structure fabricated by the conventional and predictive divergence compensation method: (a) cross section of the corrected sinusoidal structure; (b) cross section of the sinusoidal structure fabricated by conventional bitmap milling; (c) intended sinusoidal shape; and (d) comparison of the sinusoidal structures fabricated by both methods.

Figure 5-4 Comparison of sinusoidal structures fabricated on: (a) silicon substrate with a thin oxide layer; and (b) silicon substrate after removing the oxide layer.
Chapter 5  The Deterministic Fabrication of Three-Dimensional Structures

Figure 5-5 Comparison of parabolic microstructures fabricated by the conventional and predictive divergence compensation method: (a) cross section of the corrected parabolic structure; (b) cross section of the parabolic structure fabricated by conventional bitmap milling; (c) intended parabolic shape; and (d) comparison of the parabolic structures fabricated by both methods.
Figure 5-6 SEM images of hemisphere structures with 5µm radius fabricated by the predictive divergence compensation method and conventional method: (a) 52° tilt view; (b) cross section cutting through the diameter; (c) cross section of the hemisphere fabricated by the predictive divergence compensation method; (d) cross section of the hemisphere fabricated by the conventional method; and (e) sketch of the designed hemisphere; and (f) comparison of hemispherical structures.
The effectiveness of this divergence compensation method at the micron scale has been demonstrated through comparisons between the experimental results and the intended structures. This methodology is also effective for FIB milling at the submicron scale. A hemispherical structure with 480nm radius (500nm intended) was fabricated by using the divergence compensation approach as shown in Figure 5-7. Along the circumference a reduction of 20nm was found. Such relative divergence is primarily due to the redeposition effect which is more pronounced in the nanometre range.

![Cross section of nano hemisphere with 500nm radius fabricated on silicon; a reduction of 20nm along the radius was found due to redeposition.](image)

It is worth mentioning that although the fabricated structures were very close to the designed geometries, there were still divergences along the curved surface. Such divergences were mainly caused by atom redeposition and the limited resolution of the dwell time. The redeposition process is inevitable within the FIB fabrication process due to its randomness. Although redeposition can be reduced by using the multi-pass scanning method, there is still certain amount of sputtered atoms deposited on the machined structure, especially for high aspect ratios and nano-structures. Besides, the limited resolution of dwell time contributes to the fabrication divergence of structures with a low aspect ratio. A user specified maximum dwell time is assigned to each bitmap. In terms of the local colour, the dwell time for each pixel in the bitmap is linearly interpolated between zero and the maximum dwell and rounded to the value...
from a fixed dwell time table. For low aspect ratio structures, the dwell times are very close between adjacent pixels. Differences between dwell times may be omitted due to rounding error. This phenomenon also caused divergence of the fabricated structures from the designed shape.

5.2.4 Optimization of the divergence compensation method

The limitation of the divergence compensation method is that due to the computing of dwell time at each pixel can be very time consuming. For a milling area comprising $M \times N$ pixels, the equation set with a coefficient matrix of dimensions of $M^2 \times N^2$ needs to be solved. The huge amount of calculation is due to the consideration of the overlap effect where the ion dose at each pixel is influenced by the ion dose from all the other pixels. In order to integrate this correction method with a commercial FIB system and enable it to be applied online, an optimization of the divergence compensation approach is carried out through the theoretical and experimental evaluation of the overlap effect in order to reduce the processing time required.

The overlap effect is characterised by normalized pixel spacing $p_s / d_f$, where $p_s$ is the pixel spacing along the X and Y directions and $d_f$ is the beam diameter. It was found that $p_s / d_f$ should be equal to or smaller than 0.673 in order to have a uniform scanning ion flux in channel milling [114]. In this case, the extra dose from the adjacent pixel contributes almost 33% of the peak dose shown in Figure 5-8(a). The overlap here can be reduced by increasing the pixel spacing; indeed, when $p_s / d_f$ equals to 1.0 only an extra dose of 7% is contributed. To evaluate the influence of the overlap effect under such condition the ion current was kept at 1nA as that used in the above experiments. The variation rate of the ion dose is around $1.4 \times 10^{13}$ ions/ms as shown in Figure 5-8(b). The variation of milling depth can be estimated based on the ion dose variation. Taking the fabrication of the hemispherical structure as an example, there are around 50,000 pixels in the bitmap and the total fabrication time is 10 minutes. The average machining time on each pixel is 12ms. According to equation (5.3), $1.4 \times 10^{13}$ ions/ms result in a depth variation of 160nm; however, because of the redeposition effect, the actual depth variation is actually less than this. Such depth variation is in the same order as the divergence between the fabricated structure and the intended structure. Validation tests on hemispherical structures are shown in Figure 5-8(c) and these indicate that, by applying $p_s / d_f = 1$ without the overlap effect correction, the
machined profile coincides with the intended structure very well with a maximum divergence of only 150nm. Further increases in the normalized pixel spacing will result in a deeper milling cavity and a flat base due to a bigger depth variation. This can be clearly observed in Figure 5-8(c) and (d) with $p_x/d_f = 1.2$ and $p_x/d_f = 1.5$ present. Therefore, it is recommended that the overlap effect can be suppressed by choosing the pixel spacing equal to the beam diameter. The same result has also been verified on the sinusoidal and parabolic structures.

![Graphs](image_url)

**Figure 5-8** Influence from normalized pixel spacing in the machining process: (a) overlap of ion current under different pixel spacing; (b) overlap effect on depth distribution for Gaussian FIB milling (uniformed pixel spacing $p_x/d_f$ is indicated); (c) comparison of hemisphere structures fabricated under different normalized pixel spacing without consideration of the overlap effect (“OC” indicates the overlap correction); and (d) depth variation and the contribution of overlap effect under different normalized pixel spacing.
5.3 Deterministic fabrication of optical sensors

5.3.1 Fibre-top cantilever and fibre-side cantilever

FIB machining is a promising technique for the fabrication of MEMS devices and micro-optical components with high quality surface finishes. In this work, accelerometers based on micro-optical fibres were successfully fabricated by the three-dimensional deterministic FIB machining technique. The measurement of acceleration was realised by monitoring the displacement of a cantilever which was fabricated by FIB sputtering. Two types of designs, a fibre-top cantilever and a fibre-side cantilever were fabricated by FIB machining for this work.

Cantilevers have been widely used in the measurement of accelerations. They are superior in relative simple design and high feasibility. By varying the cantilever dimensions it is possible to tune the range, sensitivity and frequency response of the system. It is feasible to integrate a cantilever into an optical fibre by carving tiny mechanical beams directly on its cleaved edge. Former researchers have successfully fabricated miniature cantilevers onto the end of optical fibres by using FIB machining for advanced microscopy [53]. Subsequent research indicates that laser machined cantilevers are also possible [148]. The principles behind these fibre-cantilever based sensors are almost the same – by forming a Fabry–Pérot optical cavity between the cantilever and a reference plane, interference occurs between the multiple reflections of light in the cavity. Such an optical readout technique is inherently safe and immune to electromagnetic interference and crosstalk. It can offer a few nanometres displacement measurement at a MHz readout rate.

The two types of fibre based accelerometers are illustrated in Figure 5-9. In the fibre-top accelerometer, a Fabry–Pérot optical cavity is formed between the top surface of the fibre and the under surface of the cantilever. The incident light reflects three times at the fibre-to-air interface, the air-to-cantilever interface and the cantilever-to-metal interface. Due to the interference between the three reflected light beams, interference fringes can be found in the output signal. The length of the Fabry–Pérot optical cavity can be obtained from the interference signal and, therefore, the displacement, velocity and acceleration of the cantilever can be measured by monitoring the displacement of the cantilever. Similarly with the fibre-top accelerometer, the cantilever can be carved on the sidewall of an optical fibre to form a fibre-side accelerometer. The key
component in the fibre-side accelerometer is a mirror at 45° relative to the fibre core as it is illustrated in Figure 5-9(b). With the aid of the micro-mirror, the incident light can be reflected horizontally to the side-cantilever. Interference occurring at the interfaces enables the detection of the motion state of the cantilever. With the addition of the top cantilevers this raises the possibility of 3-axis acceleration measurement using 3 fibres or potentially a single multi-core fibre, offering an all-optical three axes accelerometer with a sub-millimetre width.

![Three-view drawings of a fibre-top cantilever (a), and a fibre-side cantilever (b).](image)

**Figure 5-9** Three-view drawings of a fibre-top cantilever (a), and a fibre-side cantilever (b).

### 5.3.2 Fabrication of fibre cantilevers by FIB

Due to the low material removal rate in FIB machining, the fabrication was initially carried out by using a picosecond laser to bring it close to the designed shape. A ridge with thickness of 50 µm was formed at the top of the fibre. FIB machining was used to carve the Fabry–Pérot optical cavity from the side of the ridge. A high current intensity (50nA at 30kV, dwell time 1µs) was used for removing large cross sections with a gallium ion beam. The beam followed a serpentine scan along the length direction of the cantilever. For fibre-top cantilever, the FIB sputtering depth did not affect the performance of the accelerometer as long as the ions shot through the ridge. Therefore, conventional ion beam machining without divergence compensation was applied. After
a roughing cut by FIB, an ion beam cleaning process was then carried out to minimize the inclined angle of the sidewall. Additionally, the surface finish on the sidewall could also be improved by the ion beam cleaning process. A SEM image of the fire-top cantilever is shown in Figure 5-10.

![Figure 5-10](image)

**Figure 5-10** Fibre-top cantilever fabricated by a combination of laser and FIB machining.

For the fibre-side cantilever, the divergence compensation method was applied to fabricate the micro-mirror at the centre of the fibre-top surface. The ion beam was incident perpendicularly to the fibre core in an area of 10µm × 10µm. Since the mirror surface was a plane with a constant gradient, the sputter yield was the same at each pixel on the mirror. Therefore the dwell time distribution was unchanged in each scanning pass. Only one bitmap was needed for the whole fabrication process. According to the divergence compensation method, the pixel spacing was set to be the same as the beam diameter. A fabricated fibre-side cantilever is shown in Figure 5-11. The inclined angle of the mirror was measured by a SEM viewed from 38° to the fibre top surface. It was found that the error in the fabrication was ±0.1°. A performance test was carried out by coupling the fibre to the optical fibre interferometer readout system. In this system an amplified-spontaneous-emission (ASE) source with a FWHM of 30nm and a centre wavelength of 1550nm was used. An optical spectrum analyser is employed to read the interference signal. The incident light that impinged on the fibre-side cantilever was reflected and the demodulation of intensity signal was observed as shown in Figure 5-12, which provided further proof of the high form accuracy of the mirror and the effectiveness of the divergence compensation method.
Figure 5-11 Fibre-side accelerometer fabricated using FIB machining. (a) ~ (b) SEM image of the fibre-side accelerometer under different magnification; (c) a close-up view of the 45º micro mirror at the centre of the fibre; (d) a close-up view of the surface finish on the fibre-side cantilever.
5.4 Summary

In this chapter, the deterministic fabrication approach with the embedded divergence compensation method was applied to overcome the geometric challenges in FIB machining. It has been shown that applying this method can help achieve high precision three-dimensional structures. The major results can be summarised as follows:

- In FIB machining process, the serpentine scanning strategy performs better than raster scan and contour scan due to the shortest beam travelling distance.

- Compared with the conventional FIB machining method, the divergence compensation method can reduce the divergence caused by atom redeposition and that the machined surface form accuracy can be dramatically improved, e.g. a 6-fold improvement for the parabolic microstructure. A less than 3% relative divergence was consistently achieved for the basic microstructures manufactured in this work and, due to a more pronounced redeposition effect, for nano-hemispherical structures the relative divergences were in the order of...
4%. These results show that the fabrication divergence caused by the overlap effect and the angular dependent sputter yield are greatly reduced using this approach.

- A flat top/bottom was found in low aspect ratio convex/concave structures whilst using conventional bitmap FIB milling; this phenomenon was mainly related with the aspect ratio of the structures. Applying the divergence compensation approach and removal of an oxide layer of silicon substrate before FIB milling, will remove these features.

- The overlap effect can be suppressed by carefully choosing the pixel spacing.

- A case study of fabricating fibre-top and fibre-side accelerometers shows that FIB is an ideal tool for developing of prototyping micro-optical devices in small area. In the fibre-side accelerometer, the key part – a 45° micro mirror was successfully obtained by FIB deterministic fabrication. The performance of these sensors also proved the effectiveness of the divergence compensation method developed.
Chapter 6 Deterministic Fabrication of Nanoscale Diamond tools

— A case study to overcome the material and machining efficiency challenge in FIB machining

FIB machining is an ideal technology for obtaining micro and nano structures. However, the low material removal rate as well as the gallium ion implantation effect limits the applications of FIB technology. Over the years there have been demonstrations of other methods which are capable of forming sub-micron structures, such as e-beam lithography, dip-pen lithography and laser writing. These structures could then be replicated using NIL or moulding. However, NIL only works on a small scale. Meanwhile, both the master and the work piece must be flat. The replication of nano structures on curved surfaces is still a challenging task. For nanostructures to become ubiquitous they will have to be mass produced at low cost.

In this chapter, the deterministic fabrication approach is applied to obtain a nanoscale multi-tip single crystal diamond (SCD) tool. The tool can be used to replicate nanostructures on a large area and mass production by SCD turning. SCD turning has been exploited successfully to generate a wide range of optical structures from discrete optical components to large area micro-optic based films such as brightness enhancement films for LCD displays [149]. It is suitable at the industrial scale production with a high throughput. However, the size of the cutting tools limits the application of SCD turning in nanotechnologies. Because of the extreme hardness of diamond, it is very difficult to machine SCD tools by routine micromachining techniques. The choice of shaping techniques is very limited. The literature reports that laser [150] and FIB methods [151] can be used to shape diamond. By applying advanced ion-milling techniques to trim diamond tools, it is possible to increase the complexity and ultimate dimensional resolution of diamond machined products. As shown in Figure 6-1, complex structures down to nanometres can be transformed and replicated over an extremely large scale.
6.1 Introduction

In the last few years, the feasibility of fabricating micro tools by FIB sputtering has been successfully demonstrated by many researchers. Micro-cutting and micro-milling tools made of diamond [152], cobalt M42 high-speed steel, and C2 tungsten carbide [153-154] were successfully developed. These tools’ dimensions range from 15µm to 100µm. Varieties of tool geometries have been produced with the help of FIB sputtering as shown in Figure 6-2. However, the development of cutting tools with nano level features is still a difficult task.
Figure 6-2 Varieties of micro cutting tools fabricated by FIB machining: (a) Single tip SCD tool shaped by FIB sputtering [152]; (b) Two tipped SCD tool shaped by FIB sputtering [151]; (c) Diffractive-optical-elements shaped micro-tool made by FIB [155]; (d) hemispherical micro-tool fabricated by FIB [155].

The need for industrial applications is the main drive and need to decrease the tool size down to nanometre level. There are many examples in nature of why nanostructures could be useful. They can be found in many insects, plants and living organs. For example, moth’s eyes have nano-bumps on their surface to absorb more visible light, the surface of a butterfly wing has multilayer nanoscale patterns which create optical interferences and the nanostructures on the filaments of the edelweiss flower absorb ultraviolet light. Non-optical examples include the nanostructures on Mayfly wings to prevent them from folding, lotus leaf nanostructures showing super-hydrophobic properties that could be useful for self cleaning surfaces and gecko feet structures for adhering the surface. Other applications include wire grid polarizers and diffraction gratings. Additionally, the production of photonic products such as plasmonic solar cell panels, wire grid polarizers and diffraction gratings potentially benefits from SCD turning if the tool size is scaled down to the sub-micron scale. Therefore, it is absolutely necessary to develop nanoscale SCD tools that can meet these extensive requirements.
6.2 Challenges in fabrication of nanoscale SCD tools by FIB

In this work, the FIB machining accuracy is essential to tool performance and tool life. However, the achieved form accuracy of the tool will be degraded particularly where the size of the structures is at the same level as the beam spot size. Some nano-effects that are generally negligible in micro-fabrication must be taken into consideration in nanofabrication. In nano SCD tool fabrication, there are five main issues which need to be overcome, including:

- **Formation of a sharp cutting edge**
  According to the machining mechanics of the cutting process as it is shown in Figure 6-3, the cutting edge need to be extremely sharp to improve the finish of the machined surface. Additionally, a blunt cutting edge can also cause more tension applied on the tool tip and lead to a reduction of the tool life. However, edges of the facet formed by the ion beam are always rounded due to the Gaussian distribution of the beam. To make a nano SCD tool that has super cutting performance, fabrication of facets with sharp edges becomes the prime task.

![Figure 6-3 Illustration of the SCD tool cutting process.](image)

- **Ion beam drifting during the fabrication process**
  Since diamond is a type of dielectric material, an additional electric field will be generated when it is bombarded by charged particles such as gallium ions. Consequently, the incident ion beam will be deflected before it reaches the diamond surface, which greatly deteriorates the machining accuracy. Therefore,
the accumulated charges on diamond must be conducted out of the system efficiently.

- **Ripples generated on the diamond surface**
  Ion-bombardment of solid surfaces is known to cause the formation of periodically modulated structures often referred to as ripples [156]. This phenomenon has been observed for various materials such as single crystal materials (Diamond [152], Si [157], Ge [158], Cu [159] and Ag [160]) and other materials (SiO₂ [161] and graphite [162]). The ripples on the diamond tool surface will increase its surface roughness and, therefore, must be eliminated.

- **Redeposition effect in the FIB sputtering process**
  The principles of the redeposition effect have already been introduced in the previous chapters. It is a universal effect which occurs during the FIB sputtering process. Both surface roughness and machining form accuracy are negatively affected. The redeposition ratio⁷ mainly depends upon the aspect ratio and the profile of the fabricated structures. In a large proportion of FIB milling applications, the redeposition effect should be suppressed as much as possible.

- **Contribution from beam tail**
  The beam tail effect is generated from the Gaussian distribution of the ion beam. The contribution depends on the beam spot size and the pitch between adjacent scanning pixels. Extra material removed by the dose contributed by the beam tail will degrade the form accuracy of nano-SCD tools.

### 6.3 Solutions

#### 6.3.1 Measures to form a sharp cutting edge

In order to obtain a sharp cutting edge using FIB, the cutting tool was fabricated from the clearance facet. The schematic diagram illustrated in Figure 6-4 shows the method to produce a sharp cutting edge by FIB machining. It implies that the cutting edge near to incident ion beam is rounded off due to the truncation of Gaussian beam profile,

---

⁷ Redeposited ratio is defined as the ratio between the number of redeposited atoms and the number of total sputtered atoms.
while other edge away from the beam becomes much sharper. This effect can be clearly observed under a SEM when a beam of ions sputtering on the edge of a diamond cutting tool. As shown in Figure 6-5, the ion beam with a current of 15nA was incident along the z direction. The upper edge of the facet which was closer to the ion source was rounded, while the lower edge was protected by the material above and thus retained the sharpness. Measurement result showed that the edge radius of the lower edge was less than 50nm.

Therefore, paying careful attention is required in order to produce a sharp cutting edge when using FIB sputtering, especially for SCD tools with complex structures or multiple cutting edges, the orientation of the tool facet has to be adjusted several times. It was reported that for SCD tools with single tip and double tips, the orientation of the tools were adjusted four [152] and six [151] times respectively. In general, the tool rotation/sputter sequence and the location of facets are critical for the forming of tool characteristics.

![Figure 6-4](image-url)  
*Figure 6-4 Schematic diagram of cutting edge formation [163].*
6.3.2 Solution to reduce Ion beam drifting

The ion beam drifting effect occurs on all dielectric materials. Generally a metal-coated layer will be applied onto the tool to provide protection for the tool edge and also prevent charge-up during FIB sputtering. This metallic layer should be thick enough to effectively conduct the charges induced by the incident ions out of the tool’s body effectively. Meanwhile, the coated layer should also have a smooth surface finish and with a negligible thickness compared to the tool size. Currently there are many coating techniques, such as chemical vapour deposition and electron beam evaporation, which meet the above requirements.

A specific requirement in SCD tool fabrication is that the rake facet should be kept clean to guarantee that samples cut by the SCD tool would not be contaminated with any impurities attached to the rake facet. This can be achieved by ion beam assisted chemical vapour deposition. By scanning an area with the beam, the precursor gas will be decomposed into volatile and non-volatile components; the non-volatile platinum remains on the surface as a deposition. Not only the thickness, but also the location of the deposited platinum can be controlled precisely with nanometre accuracy. The coated platinum is confined to the diamond tip on the clearance facet. Additionally, the deposited platinum can also serve as a sacrificial layer to protect the underlying diamond from the destructive sputtering of the beam.

Figure 6-5 Upper (a) and lower (b) edges of a diamond facet sputtered by FIB.
6.3.3 Solution to reduce ripples generated on the diamond surface

The underlying mechanism of this ripple formation can be traced to a surface instability caused by the competition between curvature-dependent sputtering and surface diffusion processes. The ripples are generally produced by off-normal incident ion beam. The orientation of the ripple depends on the beam incident angles [156]. For incident angles less than a critical angle from the normal, the wave vector of the modulations is parallel to the component of the ion beam in the surface plane. The wave vector is perpendicular to this component for the incident angles close to grazing, see Figure 6-6. The wavelength of these ripples typically depends on the ion incident angle. According to Bradley and Harper’s theory [156], the wavelength approaches infinite when the ion incident angle approaches $\pi/2$. That is to say that ideally a ripple-free surface can be formed when the ion incident angle is $\pi/2$.

![Figure 6-6](image)

**Figure 6-6** Dependence of ripple orientation on the angle of incidence $\theta$ as shown in the inset. (a) Orientation for small incident angle; (b) orientation for incident angle close to $\pi/2$.

Experimental results showed that a lower beam current was helpful in eliminating the ripple effect. In this experiment, two rectangular cavities with identical dimensions were fabricated by FIB on a diamond block. Two different ion currents 15nA and 7nA were applied for the two cavities, respectively. SEM images of the surface morphologies of the sidewalls are shown in Figure 6-7. Periodical ripples with amplitude of 200nm were found on the sidewall sputtered by 15nA ions, while a smooth surface was formed on the sidewall sputtered by 7nA ions. Further investigation proved that a better surface finish could be achieved when the ion current was less than 7nA.
This was because the inclined angle of the sidewalls fabricated under different ion currents was different. Since the ion beam profile follows a Gaussian distribution, the sidewalls fabricated by FIB are not perfectly vertical. For certain materials, the inclined angle of the sidewall is determined by the beam diameter which depends on the beam current; a bigger diameter results in a more inclined sidewall (rough milling) while a smaller spot size results in a more vertical sidewall (fine milling). When the ion current was kept at 15nA, the beam diameter was 182nm, which was almost twice as much as the beam diameter under 7nA ion current. Therefore, the inclined angle of the sidewall was bigger under a 15nA ion bombardment. In other words, the incident angle was less close to \( \pi/2 \). According to Bradley and Harper’s theory, the ripples formed on the sidewall were more pronounced in this case.

![Figure 6-7 Cavities sputtered by FIB: (a) with an ion current of 15 nA and (b) with an ion current of 7 nA.](image)

It can be concluded that the ripples generated in ion beam sputtering process can be suppressed by using a low beam current of less than 7nA. However, merely decreasing the beam current will lead to a very time-consuming process. With regard to the machining efficiency, it is recommended that in order to fabricate a ripple-free SCD tool each facet needs to be polished by a low current ion beam.

### 6.3.4 Solution to overcome the redeposition effect

For concave structures with high aspect ratio, the sputtered atoms are less likely to escape from the cavity and are therefore deposited onto the sidewalls. The redeposition
effect can be ignored if there are only non-concave structures on the tool tip. However, for tools with notch or jagged shapes in the cutting edge, small amounts of material from one side of the edge could redeposit at the opposite side. As shown in Figure 6-8, a rectangular cavity with a length of 750nm was fabricated on the edge of a diamond block. In order to minimize the ripples on the sidewalls and improve the machining accuracy, a polishing process with an ion current of 30pA was carried out. The polishing process followed three independent steps as shown in Figure 6-8(a) and was called an “edge to edge” process, i.e. one edge was finely polished followed by another. An SEM image of the three polished edges is shown in Figure 6-8(b) where the bulk of amorphous material was found on the edge that was first polished due to the redeposition effect. The machining accuracy was therefore degraded. Special scanning strategies need to be developed to minimize the redeposition effect when polishing nanoscale SCD tools. Similar to the multi-pass scanning method in micro fabrication, a “multi-edge” polishing method can be applied in an FIB cleaning process. Multi-edge polishing means the edges that need to be polished are processed simultaneously. As illustrated in Figure 6-8(c), the beam spot was controlled scanning along the overall profile of the rectangular structure. The scanning path started from commenced 100nm from the edge. Focused ions were deflected along the scanning path until a specific milling depth was achieved. Then the ion beams moved onto the next scanning path. The pitch between adjacent scanning paths was 10nm. The scanning path approached the edges and stopped until the scanning path overlapped with the edges. The advantage of this method is that the deposited atoms on each edge are immediately removed by the following scan. Therefore, the redeposition effect is suppressed. The structure polished by this method is shown in Figure 6-8(d); comparing this with Figure 6-8(b), the polished structure possesses much better form accuracy.
Figure 6-8 FIB polished nano structures by applying different scanning strategies. (a) ~ (b) An “edge to edge” polishing process and polishing result achieved; (c) ~ (d) A “multi-edge” polishing process and polishing result achieved.

Experimental results show that the “multi-edge” polishing process is helpful in minimizing the redeposition effect on the fabrication of a nano-SCD tool with concave rectangular structures. From this evidence, it is assumed that other structures with more complex profiles can also be polished in the same way.
6.3.5 Evaluation of the beam tail effect

Due to the scattering of the ions emitted from the LMIS and the limitation in the focusing ability of the ion column, a beam of focused ions has a finite size which is measured by beam diameter (FWHM). As shown in Figure 2-4, in the FIB system used in this work the beam diameter varies from 7nm to 1700nm under different ion beam currents. Therefore, the beam tail effect is more pronounced in nanofabrication and cannot be ignored. However, the often-quoted beam diameter of a beam profile does not provide a sufficient description of the beam characteristic. Serious mistakes could be made if one relies only on beam diameter to develop a FIB operation. For example, in a circuit repair application where two parallel metal lines need to be deposited, the minimum separation between the lines without causing a short circuit is not determined by the beam diameter but decided primarily by the tail of the FIB profile. Therefore, it is necessary and useful to develop simple and sensitive methods for evaluating the effect caused by beam tail in the FIB machining processes.

An accurate evaluation of the effect caused by the beam tail for a finely focused ion beam is difficult using conventional methods. Generally, the beam is scanned across an abrupt edge. The sputtering depth caused by the beam tail can be measured either by SEM or AFM. For SEM, the implantation of ions can change the secondary electron contrast during the measurement, while for AFM the profile of the structure is also difficult to measure due to its high aspect ratio. The AFM tip cannot reach the sidewall of the structure. In this work, the contribution from beam tail was evaluated by topography simulation based on level set method, which can avoid the above difficulties.

Before the topography simulation, the sputter yield of diamond under various incident angles needs to be determined. In this work, the sputter yield was calculated using the Monte Carlo simulation code TRIDYN. The calculations modelled impingement of 20keV and 30keV gallium ions onto a flat diamond surface. The heat of sublimation, namely 7.41eV, was taken as the surface binding energy of diamond. The bulk binding energy and displacement energy were assumed to be 3eV and 28eV [164]. The calculation results were shown in Figure 6-9 and were compared with the experimental results reported by other researchers [152, 165]. For 30keV incident ions, the calculated sputter yield agreed well with experimental results with a relative divergence of 20% in
the range between 0° and 60°. For 20 keV ions, the maximum divergence was found between 60° and 85° where the calculated sputter yield was almost twice as much as the experimental results. This divergence is primarily attributed to the ripples generated during the sputtering process. Due to the existence of the ripples with wavelengths comparable with the beam diameter, the ion incident angle varied with the surface profile. For certain incident angles the measurement results were actually an integration of the sputter yield under all the possible angles all over the bottom surface and sidewalls. However, this dynamic change on the surface profile was not taken into consideration in TRIDYN.

![Graph](image)

**Figure 6-9** Plot of the sputter yield of diamond (atoms/ion) vs. ion beam incidence angle. Six data sets are shown including calculation results by Monte Carlo method and experimental results reported by other researchers [152, 165].

In the following topography simulation program, the surface diffusion effect is not taken into account and the sputtered surface is treated as a smooth surface. Therefore, the sputter yield calculated by TRIDYN is applied in this model. The simulation imitated a beam of ions with a kinetic energy of 30 keV bombarded onto an abrupt edge made of diamond. In this simulation program the ion current was set at 50 pA which corresponds to a beam diameter of 19 nm. To guarantee the stability, the Courant-Friedrichs-Lewy (CFL) condition must be satisfied, which limits the advancement of the surface to a maximum of one grid spacing in each time step. Therefore, in the
surface topography evolution process the time step and the grid size were set to 25\(\mu\)s and 1nm \(\times\) 1nm, respectively.

The level set functions evolved with sputtering time are presented in Figure 6-10. The surface topography profiles, which are represented as the zero level set, are embedded naturally in the level set functions. A comparison of the cross sections sputtered by the beam tail is shown in Figure 6-11. The erosion on the abrupt edge caused by beam tail is clearly observed. It indicates that the erosion effect was much more pronounced at higher ion dose. As the incident ion dose increases, the side wall of the edge became more abrupt. Meanwhile, the erosion area is also broadened. It can be estimated that the erosion area caused by the beam tail is about three-times as the beam radius. For a beam of focused ions with 19nm diameter, only half of the beam (9.5nm) can impact on the edge. However, the erosion can expand to 25nm away from the edge.

Figure 6-10 Level set functions evolved with sputtering time: (a) \(t = 2.5\)ms; (b) \(t = 7.5\)ms; (c) \(t = 12.5\)ms; and (d) \(t = 17.5\)ms.
Figure 6-11 Erosion on the edge when fabricated by a beam of ions with a 19nm beam diameter.

The contribution from the beam tail cannot be ignored in nanofabrication, especially in the fabrication of nanoscale SCD tools. Generally the clearance angle of SCD cutting tools is 10°, which means the angle contained by the clearance facet and rake facet is 80°. An illustration of this relationship is shown in Figure 6-12. Due to the approximately vertical clearance facet, the thickness of diamond on the tip is very different (thickness at point p₁ is much less than the thickness at point p₂). Therefore, when a uniform ion dose was applied on the clearance facet, the thinner part (p₁) on the tip would be over sputtered. In this case, the contribution of the beam tail at point p₁ is much more pronounced; the ion damage and the impurity levels in the surface layer of diamond exposed by the FIB were typically higher. The over sputtered part on the diamond tip could be totally removed. As shown in Figure 6-12(c), a reduction in the size of the tip would occur due to the extra ion dose contributed by beam tail. In order to avoid this effect, the ion dose at each point on the clearance facet should not be uniform but proportion to the local depth. Although the clearance facet would still be affected by the beam tail, the ion dose contributed by the beam tail is not sufficient to break through the diamond block. The geometric profile on the rake facet remains the same as the designed profile.
Figure 6-12 Contribution from beam tail effect: (a) side view of the diamond tool tip; (b) schematic sketch showing the different sputtering depths at points $p_1$ and $p_2$; (c) sketch plan view of the diamond tip indicates a shrinkage of the tool geometry caused by the beam tail effect.

### 6.4 Fabrication of nanoscale diamond tool

Nano SCD tools with multiple chisel-tips can be used to fabricate metallic masters which enable the printing of sub-micron structures on various materials at an industrial scale. One such designed nanoscale tool is shown in Figure 6-13. Periodical chisel structures with a height of 0.6µm and a tip width of 0.15µm are designed on the tool tip.

In order to guarantee the expected performance and the capability required in the desired products, such as brightness enhancement films and optical waveguide panels, the deviation tolerances of these nano structures need to be confined to ±5%. In this work, a tool blank was mounted on a 5-axis motorized stage having a 300nm motion resolution and 360° rotational motions with a minimum of 0.1° increments. The secondary electrons emitted during the FIB fabrication process were collected to provide images of the fabricated tool for the purpose of process monitoring. The experimental process consisted of four steps:

- Before the FIB fabrication process, a special sample preparation process was carried out to minimize the image drift during ion bombardment.
• During the fabrication process, a beam of 30keV ions scanned over the diamond tip to remove the redundant material. This was a rough cut to form the cutting point structures.

• Low ion current was used to finely trim the profile of the structures as an ion beam cleaning process.

• Vertexes of the triangular diamond tips were cut off by applying a low ion current and the cutting point tips were obtained.

![Figure 6-13 Geometrical profile of a nano SCD tool.](image)

6.4.1 Sample preparation
Before the FIB machining process, the diamond tool needs to be carefully prepared to suppress the image drift. The bottom part of the diamond was painted with a layer of silver (Quick drying silver paint @ Agar Scientific). The painted part directly contacted with an aluminium stage directly over a broad contact area in order to increase the charge conducting efficiency. Next, a layer of platinum with a thickness of 3nm was deposited onto the tip of the diamond tool using FIB induced deposition. In the deposition process, a pixel dwell time and refresh time (the time between two subsequent complete pattern scans) must be defined to allow sufficient build up of the platinum at each pixel before the ion beam returns to that pixel. The pixel dwell time must be sufficiently long enough to decompose the platinum as completely as possible. Concurrently, the dwell time should also be short enough to remove the minimum
amount of diamond material via ion sputtering. It has been reported that the sputtering effect on the substrate can be suppressed almost completely by selecting appropriate scanning parameters [57], in which case the pixel spacing is typically five times wider for deposition than for milling; the dwell time is typically 10 times shorter and the refresh time is much longer in order to enable the precursor gases to adsorb on the sites where the decomposition reaction has occurred.

In this experiment, the horizontal field width (HFW) of the ion beam scanning area was set at 300μm. The resolution of the scanning area was 512 pixels×442 pixels. The view field was scanned by the ion beam with a 7nA current under which the beam diameter (FWHM) was 102nm. The scanning dwell time $t_d$ at each pixel was set to be 50ns. Therefore, the pixel spacing $p_x$ was:

$$p_x = \frac{HFW}{512} = 586 \text{ nm} \approx 5 \times \text{FWHM} \quad (6.1)$$

Generally, in FIB induced deposition the refresh time is in the range 2~5ms [166] to allow the platinum precursor to fully decompose. In this experiment, the refresh time $t_r$ was:

$$t_r = t_d \times 512 \times 442 = 11.3 \text{ ms} \quad (6.2)$$

which is sufficient to decompose the precursor.

During the deposition process, the motorized stage was moved slowly to expose more area under beam radiation. Once the region covered with silver paint was connected with the FIB sputtering point by the deposited platinum layer, the prepared SCD tool was ready for FIB fabrication. This tool was shown in Figure 6-14, where the platinum layer can be clearly observed due to a change in the contrast under the SEM. The advantage of this method is that the charged particles can be conducted out of the SCD tool efficiently. Meanwhile, since the platinum was only coated on the flank facet of the SCD tool, the rake facet was kept clear and, therefore, less impurity would be transferred to the work piece cut by this SCD tool.
Three-dimensional deterministic fabrication of a diamond tool

To avoid the over sputtering effect and to minimize the erosion caused by beam tail on the cutting edge, the ion dose at each point on the clearance facet needs to be precisely controlled proportionally to the local depth of the diamond block. Therefore, a three-dimensional FIB fabrication process was carried out.

In this work, the divergence compensation method proposed in section 3.2.1 was applied to optimize the FIB machining parameters. Since the minimal dimension on the diamond tip is only 150nm, the beam diameter must be kept much less than this in order to guarantee machining accuracy. An ion beam current of 50pA was applied in this process. In this case, the beam diameter was 19nm which was approximately $\frac{1}{10}$ of the minimal dimension on the tip. The pixel spacing was matched to the beam diameter so that the beam overlap effect could be suppressed. In order to minimize the contribution from the beam tail, a positive offset along the normal direction of the tool profile is necessary. The offset distance was set at 30nm based on the simulation results. In the scanning process, the maximum dwell time where the pure white pixels were located was set to 25.5µs. Dwell times at other pixels were proportional to the local tones of the bitmap. An example of the bitmap created for four tooth tip is shown in Figure 6-15.
The incident ion beam followed a serpentine scan starting from the upper left corner of the bitmap and was blanked while the beam passed through the tips. The total machining time was approximately 3 hours.

![Figure 6-15 Bitmap created for a four-tooth SCD tool.](image)

The rough cutting process was followed by ion beam cleaning with a current of 10pA under which the beam diameter was 13nm. The beam scanning path followed the edge of the designed profile to minimize the redeposition effect. For each cutting line, the beam repeatedly scanned over with dwell times proportional to the local thickness of the diamond tip. The dwell time distribution and scanning path in the cleaning process are shown in Figure 6-16.

![Figure 6-16 (a) Distribution of dwell time and (b) the scanning path over the machining area in the ion beam cleaning process.](image)

For the last step, a 10pA ion current was used to cut off the vertexes of the triangular diamond tips. Tool tips with a height of 600nm were obtained. Schematic procedures to form a nano SCD tool from an original diamond tip using the FIB sputtering is shown in Figure 6-17.
Figure 6-17 The FIB machining procedure for nano SCD tools with periodical chisel structures: (a) fabrication of trapezoidal structures on the diamond tip; (b) the ion beam cleaning process; and (c) cutting off the vertexes of the triangular tips.

6.5 Results and discussion

For the sake of contrast, the original SCD tool is shown in Figure 6-18(a). Figure 6-18(b) ~ (c) show SEM images of a FIB fabricated nano SCD tool. An ion-induced secondary electron image (taken from the clearance facet) of the diamond tip with a magnification ×20000 is shown in Figure 6-18(d). Periodical chisel structures with
finely finished edges are successfully obtained. The ripple-free side walls on the diamond tip are clearly observed in Figure 6-18(c). The surface texture of the diamond tip was measured by white light interferometer (Zygo New View 5000) as shown in Figure 6-19. A surface roughness $R_a$ of 1nm was obtained on both of the clearance facet and the rake facet.

**Figure 6-18** A nanoscale SCD tool fabricated by FIB machining. (a) Original SCD tool; (b) a lower magnification view of the same tool trimmed by FIB; (c) ~ (d) lateral and top view of the nanoscale SCD tool.
Chapter 6  Deterministic Fabrication of Nanoscale Diamond tools

Figure 6-19 Surface roughness on the diamond tip: (a) Surface roughness on the clearance facet; (b) surface roughness on the rake facet.

Both the structure profile and the cutting edge radius of the diamond tip were measured by the SEM equipped with the FIB system. After the FIB sputtering process, the tool was flipped 180º for the measurement. The SEM image was taken from the rake facet where the sharp cutting edge is situated. The measurement results of the form accuracy are shown in Figure 6-20(a) and are listed in Table 6-1. The relative error is controlled within -2%~5%. The maximum divergence in the profile is -10.2nm. This value is very close to the ion beam resolution which is 7nm at 30kV at beam coincident point. Figure 6-20(b) shows an SEM image of the cutting edge which was cleaned using an ion current of 10pA from the clearance facet. The edge was formed by the clearance facet and the rake facet at a relative angle of 81º. The measurement result shows that the tool cutting edge radius is achieved at a minimum value of less than 40nm.

---

8 The coincident point is the optimal working point of dual beam system that both the ion beam and the electron beam focus on at the same point. At the coincident point, the beam remains focused and almost does not shift when the stage is tilted or rotated in any direction.
Figure 6-20 Measurement of dimensions of nano-SCD tool: (a) SEM images of four nano-structures on the tool tip; (b) SEM image (taken from side clearance facet) of the cutting edge.
Table 6-1 Form accuracy of the fabricated nano structure on diamond tip

<table>
<thead>
<tr>
<th>Tip dimension (nm)</th>
<th>Designed</th>
<th>Experiment</th>
<th>Divergence</th>
<th>Relative Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tip 1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tip width</td>
<td>150.0</td>
<td>152.8</td>
<td>2.8</td>
<td>1.9%</td>
</tr>
<tr>
<td>Base width</td>
<td>450.0</td>
<td>458.5</td>
<td>8.5</td>
<td>1.9%</td>
</tr>
<tr>
<td>Tip length</td>
<td>600.0</td>
<td>594.0</td>
<td>-6.0</td>
<td>-1.0%</td>
</tr>
<tr>
<td>Tip 2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tip width</td>
<td>150.0</td>
<td>156.4</td>
<td>6.4</td>
<td>4.3%</td>
</tr>
<tr>
<td>Base width</td>
<td>450.0</td>
<td>466.2</td>
<td>16.2</td>
<td>3.6%</td>
</tr>
<tr>
<td>Tip length</td>
<td>600.0</td>
<td>594.0</td>
<td>-6.0</td>
<td>-1.0%</td>
</tr>
<tr>
<td>Tip 3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tip width</td>
<td>150.0</td>
<td>155.2</td>
<td>5.2</td>
<td>3.5%</td>
</tr>
<tr>
<td>Base width</td>
<td>450.0</td>
<td>466.2</td>
<td>16.2</td>
<td>3.6%</td>
</tr>
<tr>
<td>Tip length</td>
<td>600.0</td>
<td>594.0</td>
<td>-6.0</td>
<td>-1.0%</td>
</tr>
<tr>
<td>Tip 4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tip width</td>
<td>150.0</td>
<td>147.8</td>
<td>-2.2</td>
<td>-1.4%</td>
</tr>
<tr>
<td>Base width</td>
<td>450.0</td>
<td>451.7</td>
<td>1.7</td>
<td>0.4%</td>
</tr>
<tr>
<td>Tip length</td>
<td>600.0</td>
<td>589.8</td>
<td>-10.2</td>
<td>-1.7%</td>
</tr>
</tbody>
</table>

Although the fabrication process was carefully controlled and various optimizations carried out, there are inevitable errors both in the fabrication and the measurement processes, which have escaped the numerous checks that the author has conducted. The source of the errors can be traced from:

- **Measurement error**
  All the measurements were taken under the SEM. The boundary of the structure was judged by naked eye and any cognitive bias was credited to human error.

- **Defocus of the ion beam**
  The ion beam may not be focused perfectly on the diamond tip, thus reducing the machining accuracy.

- **Redeposition effect**
  The redeposition effect has been suppressed as much as possible in this work but cannot be totally eliminated. The residual redeposited atoms may contribute towards the errors.

- **Resolution of the bitmap and stream files**
  The resolution of bitmap was determined by the pixel spacing during the ion beam scanning process, while the resolution of the stream file is determined by
the view field of ion beam. The location accuracy of the ion beam was limited by the finite resolution in the bitmap and stream files and a rounding error introduced into the result.

- **Alignment of SCD tool**
The SCD tool was fixed on a customized designed platform as shown in Figure 6-21. The orientation of the tool can be manually adjusted by the four screws mounted at each corner of the platform. However, the orientation of tool was not perfectly aligned with the ion beam due to the limitation of the stage accuracy. Therefore, the machining area was not the defined outlined area but the projected area on the tool facet. This effect introduced an alignment error into the result.

![Figure 6-21](image_url)

**Figure 6-21** Design of a customised platform for machining nano-SCD tools. One or two tool blanks can be mounted on the stage with adjustable orientations by the four screws at each corner of the stage.

- **System stability**
The stability of the LMIS, ion columns and chamber pressure _etc._ could also introduce errors into the system. Additionally, the changes in external environment such as vibration and electromagnetic interference may also have contributed to system errors.
The cutting performance of the nano-SCD tool was tested on a Precitech Freeform 700 ultra precision diamond turning machine. This machine features an X-axis that provides 350mm of travel, a Z-axis that provides 300mm of travel, and a Y-axis that provides 150mm. The spindle C-axis has a load capacity of 50kg. The position accuracy of the linear axes is less than 0.2µm. The FIB machined nano-SCD tool was mounted on this machine to fabricate nano-grating array on an electroless nickel substrate which is mounted on the C axis as shown in Figure 6-22(a). In the cutting trial, the C axis of the machine is locked. The depth of cut and cutting speed were chosen as 0.1µm and 1000mm/min respectively. The fabricated micro-grating array is shown in Figure 6-22(b), where colourful interference fringe can be clearly observed. A SEM image of the nano-grating array with a magnification of 15,000 is shown in Figure 6-22(c). Figure 6-22(d) shows the cross-sectional view of the nano-grating array. The image was taken by the SEM with a relative angle of 38º to the substrate surface. Nano-grating array with good form coherence was successfully obtained. Using such a nano scale diamond tool in diamond turning process to fabricate nano-structures, the material removal rate ($MRR$) is:

$$MRR = 4S_{cs}v_c = 1.8 \times 10^3 \mu m^3/s$$  \hspace{1cm} (6.3)

where $S_{cs}$ is the cross section area of each nano-structure and $v_c$ is the cutting speed.

The material removal rate is 30,000 times higher than purely FIB machining with ion current of 1nA. The machining efficiency can be further improved by fabricating more nano-structures on the diamond tool tip.
6.6 Summary

In this chapter, deterministic fabrication of nanoscale SCD cutting tools with extremely precise dimensions was demonstrated using FIB machining. Complex tool facet shapes with cutting edge radii of less than 40nm were obtained successfully. By controlling the ion dose proportional to the local thickness of the diamond tip, the over-sputtering effect was effectively suppressed. Meanwhile, the total machining time was also reduced by 50% compared with a two-dimensional FIB machining. From experiments using a FIB to sputter SCD tools, it was found that:

- A sharp cutting edge can be obtained by leaving the edge on the far side of incident ion beam;
• Image drift during the fabrication process can be effectively suppressed by a hybrid coating technique;

• Ripples on the sidewalls can be eliminated by applying a small ion current polishing process;

• The redeposition effect was greatly reduced by customising the ion beam scanning strategies;

• An extra ion dose contributed by the beam tail (19nm diameter) introduced an erosion of 25nm on the edge. The erosion can be compensated by an offset in the bitmap.

The experimental results demonstrate that it is feasible to fabricate nanoscale SCD tools by FIB machining. The diamond tools developed in this work can be used to machine nano-gratings over extreme large area with a high efficiency. Compared with purely FIB machining, the material removal rate was improved 30,000 times and, therefore, the machining efficiency challenge in FIB machining is overcome.
Chapter 7  Conclusions and Recommendations for Future Work

7.1  Research Assessment
The initial objective of this research is to develop modelling and simulation approaches for the deterministic fabrication of three-dimensional micro- and nano-structures by FIB. With this aim the thesis starts from a thorough investigation on the influence of the primary fabrication parameters in FIB machining, going on to provide a modelling and simulation approach which can accurately predict the surface generation of a FIB machining process. This approach is achieved by embedding an ion-solid sputtering model into the level set method. A divergence compensation method is also developed which is used to predict and obtain fabrication parameters to compensate the machining error for given structures in FIB machining. Case studies have been carried out as validations of the developed approach. By applying the deterministic fabrication approach, several existing challenges in FIB machining, including the dimensional, geometric, material and machining efficiency challenges have been overcome.

The novelty and contribution to knowledge from this research lie in:

- The in-depth investigation of the FIB machining process and the influence of fabrication parameters, which serve as the fundamentals to improve the FIB machining processes.

- The development and application of a modelling and simulation method to accurately predict the generated three-dimensional surface topography in FIB machining process.

- The development of a divergence compensation method which can predict and obtain fabrication parameters to compensate for the machining error in given structures during the FIB machining process.

- The development of the deterministic fabrication method based on the surface topography model and the divergence compensation approach.
• The demonstration of how to apply the proposed deterministic fabrication approach to obtain accurate micro- and nano-structures to overcome the existing challenges in FIB machining.

• The development of the fabrication approach to accurately obtain nanoscale SCD tools by FIB machining, laying down a solid foundation for the development of a novel processing method suitable for large-area nanofabrication with high throughput in the future.

7.2 Conclusions
In this research, the deterministic fabrication of micro- and nano-structures by FIB has been successfully achieved. The sputter yield and the distribution of sputtered atoms are calculated using the Monte Carlo method. The FIB machining process is modelled and simulated based on an ion-solid sputtering model combined with the level set method. The machined surface topography generation is actually predicted. The divergence compensation method was also implemented in FIB machining to obtain fabrication parameters for accurately machining micro- and nano-structures. Existing challenges in FIB machining in terms of dimension, geometry, material and machining efficiency were overcome. Case studies including the fabrication of a silicon mould used for printing a nanodot array, the fabrication of optical sensors, and the fabrication of nanoscale diamond tools were carried out to validate the proposed approach. The major results come from this research can be summarised as follows:

• The angular dependence of sputter yield, beam overlap effect and redeposition effect are the three primary factors which degrade the form accuracy in the FIB machining process.

• The modelling and simulation program developed can precisely predict the generation of three-dimensional structures in FIB machining process with an error of less than 10%. The modelling and simulation program provide a solid foundation to achieving deterministic nanofabrication by FIB. The dimensional challenge in FIB machining can be also overcome by using this method.

• In the fabrication of the nanodot array by FIB, more than 1/3 of the milling depth was reduced due to the redeposition effect.
The divergence compensation method can reduce the divergence caused by the variation of sputter yield, overlap effect and redeposition effect. The machined surface form accuracy can be dramatically improved by using this method. A less than 3% relative divergence was consistently achieved for the basic microstructures. The geometric challenge in FIB machining has been shown to overcome by using this method.

In the FIB three-dimensional fabrication processes, the overlap effect can be suppressed by carefully choosing the pixel spacing.

In the fabrication of dielectric materials, the image drift can be effectively suppressed by a hybrid coating technique which overcomes the material challenge in FIB machining.

By using the proposed deterministic fabrication method, it is feasible to fabricate nanoscale diamond tools with high accuracy by FIB. Sharp cutting edge can be obtained by leaving the edge on the far side of incident ion beam. The ripples on the sidewalls can be eliminated by applying a small ion current polishing process. The beam tail effect can cause extra damage outside the FIB sputtering area. The erosion area caused by the beam tail effect on an open edge is three-times that of the beam radius. The redeposition effect can be suppressed by customising the ion beam scanning strategies. The machining efficiency challenge in FIB machining can be overcome by combining the FIB machining technique with the SCD turning technique.

### 7.3 Recommendations for future work

As regarding future work, the following is recommended:

- Further development of the topography simulation method based on level set method. Although the current method is a highly robust method with high accuracy, the main limitation of this method being applied on a larger scale is the computing time. For larger scale simulations, parallel computing techniques may be applied, but the fundamental solution is to improve the algorithm to
decrease the programming complexity. There have already been several schemes that can be used to improve the efficiency in the level set method, such as the narrow band level set method and fast marching method [167]. The simulation efficiency may further be improved by introducing these schemes into the current surface topography simulation model.

- Based on the divergence compensation method, it is feasible to further develop CAD/CAM (computer aided design/manufacture) tools to create a seamless data flow to the FIB system. Such CAD/CAM tools should satisfy the stringent requirements towards the geometric and dimensional accuracy of three-dimensional structures imposed by various applications.

- Developing nanoscale diamond tools with a large tip width (~100µm). SCD tools with large tip width and multiple nanoscale teeth will result in a high machining efficiency. Meanwhile, it requires more from the FIB machining process. Chemical active gases such as water can be used to enhance the sputter yield in the machining process. The FIB machining performance and machining accuracy under such conditions needs further evaluation.

- The nature of the ion beam machining process is energy transformation. It can be interpreted as a beam of energy absorbed by the machined surface. From this perspective, there are other technologies which are based on the same principle, such as plasma machining and laser machining. It may be possible to transplant the deterministic fabrication method in this research onto these technologies.
References


References


References


References


References


