

Advanced Micro and Nano Fabrications for Engineering Applications

**A Submission for the Degree of Doctor of Science
(DSc) at the University of Birmingham**

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Synopsis

This document is a compilation of my selected research publications in micro and nano fabrications. The papers are largely arranged in chronological order to show the development of research interests. The research works are grouped into three sections.

Section one consists of 34 research papers on micro fabrication in various materials. The research was motivated by the development of a finger nail sized micro engine as explained in Papers 1 and 2. Such a microengine was proposed based on the fact that hydrocarbon fuels have about 100 times more energy per unit weight than lithium batteries. This makes it possible for a microengine to outlast batteries and replace them in portable devices, including notebook computers and handheld electronics. Among many challenges in this exploration, development of a suitable microfabrication process is a formidable one. This process needs to be able to produce microcomponents with sufficient accuracy in volume production to maintain the manufacturing costs reasonable. The engine materials should be high temperature resistant. It is apparent that the components of these processes can find many other applications. Such a process did not exist at the time. Therefore, our research effort was devoted in this area to change the situation.

The approach we adopted was evolved from MicroElectroMechanical Systems (MEMS), which was largely about silicon wafer processes at the time. However, silicon wafers and their processes are not suitable for making microengines mainly for two reasons: (a) silicon is not a high temperature resistant material; (b) the fabrication technology was only suitable for fabricating components 500 μm in thickness, while the engines require 1000 μm components. We set out to develop a couple of new processes. These

processes involve making ultrathick micromoulds, and producing high temperature resistant components from the moulds.

In developing the micromoulding process, SU-8 resist from MicroChem was used. Papers 3 to 6 report the research in this area in detail. At the time, thick SU-8 components could only be fabricated using x-ray from a synchrotron, which was not available to majority users. The research team set out to develop a UV lithography based ultrathick SU-8 process to suit general users. The design of the engine requires a strict vertical geometry and ultra-thickness on the piston and the cylinder for the prevention of the leakage. Conventional SU-8 processes often produce a trench with a wide top and narrow bottom profile, which is common for negative photoresist. The T-shape becomes more pronounced when the thickness of SU-8 layer is approaching 500 μm or more. Many factors contribute to this dimensional change, such as the volume change of the SU-8 resist during polymerization, chemical diffusion of crosslinking agents and several illuminations parameter. Therefore, the study of SU-8 optical properties in the near UV range is essential for optimizing the process of ultra-thick SU-8 layers. In the research, the relationship between the prebake time and UV light absorption property, as presented in paper 3-5, and use and without use of a filter, paper 15, were studied and optimised. Microstructures up to 1000 μm in thickness were fabricated and an aspect ratio of 40:1 was achieved. As SU-8 resist moulds are not easy to remove, another then new and removable negative tone resist KMPR was studied and up to 180 μm thick quality structures were developed, paper 6. These processes were leading in the field at the time and contributed to the development of thick microstructures significantly. The excellent micromoulds from these processes are the foundation of producing components of various high temperature resistant materials.

Three micromould based processes for producing high temperature resistant components were studied. The first process is deep structure electroforming as presented in papers 6 and 31. In this process, thick resist was used to form micromoulds on a gold plated silicon wafer, and nickel structures were electro-deposited from the exposed gold layer upwards. A micro nickel Wankel engine was manufactured through the process. This process provided an alternative way to produce metallic microcomponents from x-ray based LIGA process.

The second micromould based process is soft lithography for producing high precision microceramic components. The ceramic materials used were alumina and zirconia. The process starts with high quality master moulds, being SU-8, Si, or metal. Next negative PDMS soft moulds are produced from the master moulds. Then well prepared ceramic slurry is filled in the PDMS moulds to form green bodies before they are sintered in a furnace to produce the final strong ceramic components. The main technical challenges in the process were producing defect free and high density components. To meet the challenge, my team studied rheology of ceramic slurries, adding a vacuum process after filling slurry into moulds, and optimizing sintering cycles. High quality microengine components were produced. Extensive characterisation work was carried out to analyse the shrinkage, hardness, strength, surface roughness and grain size of the microceramic structures. The research in dealing with the technical challenges and attempts made towards solving problems are best reflected in papers 7 to 21.

The third micromould based process is also soft lithography, but for producing metallic components. Stainless steel 316L powder was the main material used in the research. Papers from 22 to 34 report the research from fabrication to characterisation of the results.

Section two of the document includes some research activities and achievements on nanocomposite materials embedded in metallic and ceramic matrices. This area is a natural extension of the research included in the first section. In the first of the two approaches covered by paper from 32 to 35, micro components of Ni based nanocomposite were obtained by electrochemical co-deposition of Ni and materials, such as carbon nanotubes and ceramic nanoparticles, into microfabricated photoresist moulds in a nickel sulfamate bath. Experiments indicate that the coexistence of CNTs and Al₂O₃ nanoparticles in the nickel bath not only help their dispersion, but also improve the content of Al₂O₃ nanoparticles embedded in the nickel matrix. The microhardness testing on the microcomponents shows that the hardness of nickel with incorporation of Al₂O₃ nanoparticles and MWCNTs is improved. A more comprehensive study is to determine the effects of the operating parameters on the mechanical and thermal properties of Ni-Al₂O₃/CNTs nanocomposite. Characterisation shows that both the hardness and Young's modulus of the composite materials have improved.

Papers 36 to 40 present research into ceramic-graphene platelet composite fabrication process in order to achieve improvement in toughness of ceramics. In the case of GPL-ZrO₂-Al₂O₃ composite, powder mixtures were prepared by ball milling using zirconia balls as the milling media. GPL/ZTA ceramic samples were fabricated using spark plasma sintering. Analysis of SEM images shows that GPLs are well dispersed in the ceramic matrix microstructure and undamaged after high temperature sintering. Nearly fully densified samples are obtained at a sintering temperature of 1550 °C. The addition of only a 0.81 vol% GPLs into ZTA composites resulted in a 40% increase in fracture toughness. Pull-out of GPLs, crack bridging and crack deflection have been observed

and believed as the causes of increased toughness. The presented work shows graphene nanofillers have potential to improve the fracture toughness of ceramic composites considerably and lead to a variety of light and strong ceramics to suit engineering applications.

Section 3 includes the papers to reflect the research in developing nanostructure fabrication processes. One type of nanostructures is nanoarrays. Ordered nanostructure arrays have many functional applications, such as surface plasmonics, high density data storage, photonic devices, nano-filtration, and chemical and biological sensors. Standard lithography techniques such as X-ray, E-beam and focused ion beam lithography have low productivity and high cost. We developed light interference lithography, nanosphere lithography, and soft lithography to produce various nanopatterns and pillars. The nanopatterns were made in gold, silver, resists, PDMS, and nickel to suit both sensing and imprinting applications. The study is most represented by papers from 41 to 47.

Another type of nanostructures studied is graphene oxide (GO) films and patterns as reported in papers 48 and 49. A highly efficient process developed in the team involves forming GO films using Langmuir-Blodgett (LB) -based method, followed by thermal reduction of GO sheets with argon protection. Different thermal reduction temperatures result in different degrees of reduced GO and optical and electrical properties of the reduced GO films, making them potentially beneficial for transparent conductor in optoelectronic devices.

Hybrid structures of reduced GO sheets/ZnO nanorods were fabricated which can significantly suppress defect emission and enhance UV emission. The intensity ratio between the UV and defect emission is improved by a factor of up to 14 times. In

the process, reduced graphene oxide sheets were obtained by heat treatment of the graphene oxide (GO) under argon protection. The hydrothermal method was used to produce ZnO nanorods since it was a process of low cost, low temperature, large area uniformity and environmentally friendly method.

In summary, the research contained in this DSc submission shows a continuous exploration and development of novel micro/nano fabrication processes. Although the submission covers research activities spanning 15 years, from 2000 to 2015, many of the research results represent the top technology of the time. They have contributed to the ever progressing manufacturing capability of the world. The research has encompassed both theoretical and experimental studies, contributing to the understanding of the processes and materials involved.

This document is dedicated

To my parents

JIANG Guo Qi (1919-2010)

WANG Rong Qing (1924 -)

who gave me the first education and encouraged me to progress throughout my career.

To my sister

Kaijun Jiang

who cared and guided me in my early education stage from which I benefit in my
entire life.

To my wife

Yuxia Cui

who has been a wonderful companion with support, encouragement, and
care.

To my sons

Vincent Yuan Jiang

& Andrew Yuan Jiang

who have given me enormous joy, inspiration and encouragement to contribute
towards building a better society for their generation.

Acknowledgment

I feel very fortunate that I have had excellent research fellows and PhD students working as a team in developing challenging but interesting micro/nano fabrication techniques. Their intelligence, diligence and patience in research made our numerous achievements possible. I especially grateful to the following researchers who have made significant contributions in our technological exploration:

Peng Jin; Jung-Sik Kim; Chen-Han Lee; Zhenggang Zhu; Hossein Ostadi; Majid Malboubi; Xueyong Wei; Hany Hassanin; Mohamed Imbaby; Xianzhong Chen; Mohammadkhani; Jian Liu; and Feng Han.

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Contents

| | |
|--|---------|
| Synopsis | i |
| Contents | vii |
| List of submitted works | viii |
| Section One: Micro fabrication | 1 |
| Section Two: Nanocomposite materials | 179 |
| Section Three: Nanostructure fabrication | 272 |
| Appendix: Full publications of the candidate | Appx. 1 |

List of Submitted Works

The submission includes 49 selected papers published in reputable international academic journals. All of the works were completed in my research group. I played a leading role in the majority of the publications (95%) by proposing research directions and ideas, initiating research work, design of experiments, writing or modifying publications. The extent of my contributions to the listed publications is indicated by the following abbreviations:

| | | |
|--|----------------------------|----------------|
| Contribution to initiation and design of experiments | Major Moderate Minor | 1A 1B 1C |
| Contribution to direction and execution of research | Major Moderate Minor | 2A 2B 2C |
| Contribution to writing of publications | Major Moderate Minor | 3A 3B 3C |

| No. | Title of Publications | Extent of contribution |
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[The full-text published papers have been redacted from the e-thesis in order to avoid copyright infringement.]

Appendix: Full publications of the candidate

BOOK:

1) M. Malboubi and K Jiang, "Gigaseal Formation in Patch Clamping: With Applications of Nanotechnology", Springer, 2014 edition (31 July 2013), ISBN-10: 3642391273, ISBN-13: 978-3642391279

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