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3D complex shaped- dissolvable multi level micro/nano mould fabrication

Abdulbast Kriama

Thesis submitted to the School of Engineering, University of Glasgow, for the degree of Doctor of Philosophy

University of Glasgow

November 2010

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Acknowledgements

First and foremost I would like to thank the almighty God (ALLAH) for giving me the knowledge, strength and patience to complete this work. May His blessings continue to shower on Prophet Mohammad (peace be upon him). I pray that He continues the same the rest of my life.

I would like to show my sincere appreciation to my advisor, Dr. Graham Green, for his support, encouragement and guidance throughout this work. His knowledgeable insights, outstanding perception and friendly personality showed me not only how to be an engineer, but how to be a man. The experience of working under his supervision in his research group will have profound impact on my future career development. This dissertation work would not have been possible without his continuous support and enthusiasm for applied research. My special thanks go to Dr Phil Dobson for his helping to complete this work.

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Finally, I want to express my sincere gratitude to my wife, Hanan for her longtime support and unconditional love! She always makes me smile even facing the greatest difficulty.
Abstract

There is growing interest in the development of fabrication techniques to cost effectively mass-produce high-resolution (micro/nano) 3D structures in a range of materials. Biomedical applications are particularly significant.

This work demonstrates a novel technique to simultaneously fabricate a sacrificial mould having the inverse shape of the desired device structure and also create the desired device structure using electroplating deposition techniques. The mould is constructed of many thin layers using a photoresist material that is dissolvable and sensitive to UV light. At the same time the device is created in the emerging mould layers using Gold electroplating deposition technique. Choosing to fabricate the mould and the 3D structures in multiple thin layers allows the use of UV light and permits the potential cost-effective realization of 3D curved surfaces, the accuracy and geometric details of which are related to the number of layers used.

In this work I present a novel idea to improve the LIGA process when using many masks to deposit multi thin layer over each other. Moreover, this technique can be utilized to produce a curved surface in the vertical direction with any diameter. Practically, a 2 µm thickness of layer is applied in the proposed technique. However, a layer of 0.5 µm or less can be deposited. An example is provided to explain the novel fabrication process and to outline the resulting design and fabrication constraints. With this technique, any structure could be made and any material used.

The work employs conventional techniques to produce a 3D complex shape. By using conventional techniques with multi layers to produce a 3D structure, many problems are expected to occur during the process. Those problems were mentioned
by many researchers in general but have not been addressed correctly. Most researchers have covered those problems by leaving the conventional and using a new technique they invented to produce the required product. However, in my work I have addressed those problems for the first time and I offered a new and effective technique to improve the MEMS technology and make this technology cheaper. This was achieved by using a research methodology requiring a rigorous review of existing processes, as outlined above, then by proposing a concept design for an improved process. This novel proposed process was then tested and validated by a series of experiments involving the manufacture of demo-devices. The conclusion is that this new process has the potential to be developed into a commercially implementable process.
3D complex shaped- dissolvable multi level micro/nano mould fabrication

Declaration

This thesis contains no material which has been accepted for the award of any other degree or diploma, except where due reference is made in the text of the thesis. To the best of my knowledge, this thesis contains no material previously published or written by another person, except where due reference is made in the text of the thesis.

_________________________________________

Abdulbast Kriama                      Glasgow, 28/02/2011
Contents
Acknowledgements.................................................................................................................. II
Abstract................................................................................................................................... IV
Declaration............................................................................................................................... VI
List of Figures ........................................................................................................................ XI
List of table.............................................................................................................................. XIV
List of Acronyms..................................................................................................................... XV
Chapter 1 ................................................................................................................................ 1
Introduction ............................................................................................................................ 1
  1.1. Definition of MEMS.................................................................................................... 2
  1.2. MEMS Application ................................................................................................. 3
  1.3. Motivation ................................................................................................................... 5
  1.4 Objectives .................................................................................................................... 7
  1.2. Research Program .................................................................................................... 9
  1.3 Structure of the Thesis .............................................................................................. 10
Chapter 2 ............................................................................................................................. 12
Literature review ................................................................................................................... 12
  2.1. History of the MEMS .............................................................................................. 13
    2.1.1. General Historical Overview ........................................................................... 14
  2.2. Review of MEMS fabrication process...................................................................... 23
    2.2.1. General description .......................................................................................... 23
    2.2.2. Depositing thin film ....................................................................................... 27
    2.2.3. Electrodeposition ............................................................................................ 30
    2.2.4. Photolithography ............................................................................................. 33
    2.2.5. Etching processes .......................................................................................... 41
    2.2.6. Bulk Micromachining ...................................................................................... 46
    2.2.7. Surface Micromachining ................................................................................ 47
    2.2.8. Mould Micromachining ................................................................................. 50
  2.3. Comparative study of previous research .................................................................. 59
    2.3.1. Using UV light instead of X Ray ................................................................... 59
    2.3.2. Utilizing three layers ....................................................................................... 61
    2.3.3. Using dry photo resist .................................................................................... 64
5.5. Problems in the third level ................................................................. 136
5.6. Results of investigation ................................................................. 142
5.7. Conclusion .................................................................................. 147
Chapter 6 .......................................................................................... 148
Conclusions and future work ............................................................ 148
Conclusions ...................................................................................... 149
Future work ...................................................................................... 153
References ........................................................................................ 154
Appendix ............................................................................................ 162
A.1 ...................................................................................................... 164
Drawing of the demo-device1 ............................................................. 164
A1.1 ..................................................................................................... 170
Drawing of the demo-device2 ............................................................. 170
A.2 ...................................................................................................... 186
Some pictures of the demo-device2, which were built with 5 layers, have been taken by using Scanning Electron Microscopy (SEM). ................................................................. 186
A.3 ...................................................................................................... 194
Some pictures of the demo-devices1, which were built with 9 layers, have been taken by using Scanning Electron Microscopy (SEM). ................................................................. 194
A.4 ...................................................................................................... 200
Pictures of removing the SU8 and not solving ..................................... 200
A.5 ...................................................................................................... 203
The surface of the photoresist after the development and before electroplating process 203
A.6 ...................................................................................................... 208
The surface of the photoresist after the electroplating process and appearing of lips ..... 208
A.7 ...................................................................................................... 210
Scheme of the treated photoresist surface ......................................... 210
A.8 ...................................................................................................... 212
Appearing of bubbles in the second level .......................................... 212
A.9 ...................................................................................................... 217
Appearing of bubbles under the second level ..................................... 217
A.10 .................................................................................................... 220
Some pictures of wrinkled surface in the photoresist ......................... 220
Some pictures of wrinkled surfaces in the demo-devices, which were built with 3 layers, have been taken by using Scanning Electron Microscopy (SEM) ......................................................... 224

Paper which we participated in a conference in Barcelona in Spain in 2008................................. 232

List of Figures

Figure 1 the Intel 4004 Microprocessor, Busicom calculator[16] ................................................................. 16
Figure 2 the process of multi layer using seed layers[28] .................................................................................. 18
Figure 3 hot Embossing[32] .......................................................................................................................... 19
Figure 4 glancing Angle Deposition[35] .......................................................................................................... 21
Figure 5 spiral microcoil using a 3D-LIGA process[38] .................................................................................... 22
Figure 6 the light passes through the mask ....................................................................................................... 24
Figure 7 transfer the 2D configuration ............................................................................................................. 25
Figure 8 Deposited material ............................................................................................................................ 25
Figure 9 process of the electroplating .............................................................................................................. 30
Figure 10 the electroplating process to depositing gold .................................................................................... 32
Figure 11 the Photolithography processes ..................................................................................................... 34
Figure 12 spin coating process ........................................................................................................................ 35
Figure 13 Photomask ........................................................................................................................................ 38
Figure 14 the aligned process .......................................................................................................................... 38
Figure 15 Positive and Negative resist ........................................................................................................... 39
Figure 16 isotropic etching ............................................................................................................................... 44
Figure 17 anisotropic etching .......................................................................................................................... 44
Figure 18 sensitive material after developing, bulk micromachining .............................................................. 46
Figure 19 etching in the thin film ...................................................................................................................... 46
Figure 20 the thin film after removing the sensitive material, bulk micromachining ....................................... 46
Figure 21 sensitive material after developing, surface micromachining ......................................................... 48
Figure 22 the deposited thin film, surface micromachining .............................................................................. 48
Figure 23 the thin film after removing the sensitive material, surface micromachining ................................... 48
Figure 24 Moulding: material flow into mould master ...................................................................................... 51
Figure 25 Stamping: the stamp modifies material already on the wafer .......................................................... 51
Figure 26 the LIGA process .............................................................................................................................. 57
Figure 27 The LIGA process utilizes X-ray masks and a synchrotron source[87] .............................................. 60
Figure 28 The process utilizes standard optical masks and a standard UV source[87] ................................. 61
Figure 29 the Riston technique by lamination[92] ............................................................................................ 64
Figure 30 utilizing dry photo resist by using multilayer process[92] ................................................................. 65
Figure 31 3D of the micro coil[96] .................................................................................................................... 67
Figure 32 3D multilevel mould of micro coil[96] ............................................................................................. 67
Figure 33 3D multilevel mould of micro coil[96] ............................................................................................. 68
Figure 34 a process for fabricating multi level metal structures[98] ............................................................. 70
Figure 35 a turbine produced by using six layers[98] ..................................................................................... 71
Figure 36 a turbine which was produced with six levels[98] ......................................................................... 72
Figure 37 principal process steps of hot embossing[101] ................................................................................ 75
Figure 38 3D Direct-Write[37] ........................................................................................................................ 76
Figure 39 Schematic diagram of projection micro-stereo lithography[44] ......................................................... 77
Figure 40 the primary steps in gray-scale technology[104] .......................................................................... 78
Figure 41 Fabricated structures with inclined exposure[84] ........................................................................... 78
Figure 42 process of 3D X-LIAG lithography with unscrewing[86] ........................................ 79
Figure 43. Transfer of the 2D structure [52] ........................................................................... 84
Figure 44 the XYZ planes ........................................................................................................ 84
Figure 45 the multi layer planes .............................................................................................. 85
Figure 46 the demo-device1 .................................................................................................... 87
Figure 47 the mould of the demo-device1 ............................................................................. 87
Figure 48 depositing a seed layer ......................................................................................... 88
Figure 49 depositing a photo resist ....................................................................................... 88
Figure 50 the first mask ........................................................................................................... 89
Figure 51 exposing the photo resist with UV light ................................................................. 89
Figure 52 the developed photo resist (the mould) .................................................................. 89
Figure 53 deposition of gold .................................................................................................. 90
Figure 54 deposition of the second seed layer ....................................................................... 90
Figure 55 the demo-device1 .................................................................................................. 91
Figure 56 the cleaning process .............................................................................................. 91
Figure 57 removing the photo resist .................................................................................... 92
Figure 58 removing the seed layer ....................................................................................... 92
Figure 59 the final product ..................................................................................................... 92
Figure 60 demo-device2 ......................................................................................................... 95
Figure 61 the first layer of the demo-device2 after developing .............................................. 100
Figure 62 the first layer of the demo-device1 after developing ............................................ 100
Figure 63 electroplating equipment in JWNC ....................................................................... 101
Figure 64 experiment of electroplating ................................................................................ 102
Figure 65 the cathode ............................................................................................................. 103
Figure 66 the second layer of the demo-device2 after the deposition of the seed layer 105
Figure 67 the second layer of the demo-device2 after developing ........................................ 106
Figure 68 demo-device2 after fabrication 1 ......................................................................... 109
Figure 69 demo-device2 after fabrication 2 .......................................................................... 110
Figure 70 demonstration of making a device with curve surface ........................................ 111
Figure 71 demo-device1 after fabrication (1) ......................................................................... 112
Figure 72 demo-device1 after fabrication (2) ....................................................................... 112
Figure 73 removing of the SU 8 .......................................................................................... 115
Figure 74 the photoresist after developing .......................................................................... 117
Figure 75 the photoresist after electroplating ...................................................................... 118
Figure 76. The lips after one hour in the oven ..................................................................... 122
Figure 77 the lips after one hour and fifteen minutes in the oven ......................................... 122
Figure 78 the lips after one hour and thirty minutes in the oven .......................................... 123
Figure 79 the lips after one hour and forty-five minutes in the oven .................................... 123
Figure 80 the deposited ratio ............................................................................................... 126
Figure 81 the designed holder ............................................................................................... 127
Figure 82 bubbles appeared in the second level under the photoresist ......................... 129
Figure 83 bubbles appeared under the second level ......................................................... 130
Figure 84 bubbles over the second seed layer ................................................................. 130
Figure 85 bubbles under the seed layer ....................................................................... 131
Figure 86 the height for some bubbles ........................................................................ 131
Figure 87 wrinkled surfaces ........................................................................................ 133
Figure 88 wrinkled surface .......................................................................................... 136
Figure 89 the thickness of the photoresist “AZ4562” after the development .......... 137
Figure 90 The thickness was 5.230 μm after 10 minutes on a hotplate at 100°C ....... 137
Figure 91 the shrinking in the photoresist ..................................................................... 138
Figure 92 3D microscope pictures of the demo-device1 .................................................. 139
Figure 93 demo-device2 with 5 layers ......................................................................... 145
Figure 94 demo-device1 with 6 layers .......................................................................... 146
Figure 95 the multi layer planes ................................................................................... 149
Figure 96 the division of the require mould ................................................................... 150
List of table

Table 1 the Advantages and Disadvantages the evaporation and sputtering deposition [46] ................................................................. 28
Table 2 the Advantages and Disadvantages the CVD deposition mouthed [4, 28, 41, 48] ........... 29
Table 3 utilizing polyimide by using multilayer process [55] .................................................. 63
Table 4 the technique of the sacrificial metallic mould [97] ..................................................... 69
Table 5 the photoresist thicknesses for demo-device ............................................................ 96
Table 6 the photoresist thicknesses for demo-device ............................................................ 96
Table 7 the thickness of az4562 and S1818 layers .................................................................. 97
# List of Acronyms

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>MEMS</td>
<td>Micro Electro Mechanical Systems</td>
</tr>
<tr>
<td>NEMS</td>
<td>Nano Electro Mechanical Systems</td>
</tr>
<tr>
<td>MST</td>
<td>Micro Systems Technology</td>
</tr>
<tr>
<td>HF</td>
<td>hydrofluoric acid</td>
</tr>
<tr>
<td>3D</td>
<td>three dimensional</td>
</tr>
<tr>
<td>RGT</td>
<td>resonant gate transistor</td>
</tr>
<tr>
<td>MOS</td>
<td>Metal-Oxide-Semiconductor</td>
</tr>
<tr>
<td>IC</td>
<td>integrated circuit</td>
</tr>
<tr>
<td>TIJ</td>
<td>Thermal Inkjet Technology</td>
</tr>
<tr>
<td>LIGA</td>
<td>lithography, electroforming, and moulding</td>
</tr>
<tr>
<td>AFM</td>
<td>atomic force microscope</td>
</tr>
<tr>
<td>SCREAM</td>
<td>Single Crystal Reactive Etching and Metallization</td>
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<tr>
<td>EFAB</td>
<td>electrochemical fabrications</td>
</tr>
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<td>DRIE</td>
<td>Deep Reactive-Ion Etching process</td>
</tr>
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<td>SUMMiT</td>
<td>Sandia Ultra-planar, Multi-level MEMS Technology</td>
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<td>DNA</td>
<td>Deoxyribonucleic acid</td>
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<td>Glancing Angle Deposition</td>
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<td>physical Vapor Deposition</td>
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<td>CVD</td>
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<td>HDCVD</td>
<td>High Density Plasma CVD</td>
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<td>UV</td>
<td>Ultraviolet</td>
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<td>RIBE</td>
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<td>ion beam LIGA</td>
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<td>PMMA</td>
<td>polymethylmethacrylate</td>
</tr>
<tr>
<td>SU 8</td>
<td>Organic Resin Solution</td>
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<tr>
<td>AZ4562</td>
<td>methoxy-propyl acetate (PGMEA)</td>
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</table>
Chapter 1

Introduction
1.1. Definition of MEMS

To make small machines which are almost invisible to human eyes has been one of the dreams of mankind. This dream was achieved by a technique called ‘MEMS’. It is an acronym for four words ‘Micro Electro Mechanical System’ and generally refers to the devices that integrate electrical devices and mechanical structures at the micrometer \((10^{-6} \text{ m} = 0.000001 \text{ m})\) scale. Similarly, NEMS is an acronym for ‘Nano Electro Mechanical System’, having devices on nano meter \((10^{-9} \text{ m} = 0.000000001 \text{ m})\) scale.

MEMS combine two systems together, Micro-Electronics System and Micro-Mechanical System. Micro-Electronics Systems consist of electronic circuits on silicon chips and Micro-Mechanical Systems are the structures and moving parts of the MEMS device. MEMS are also referred to as micromachines (in Japan), or Micro Systems Technology - MST (in Europe) or microelectromechanical systems (in US)\(^1\).

Micro Electro Mechanical Systems is a term coined around 1989 by Prof. R. Howe to describe an emerging research field, where mechanical elements, like cantilevers or membranes, had been manufactured at a scale more akin to microelectronics circuits. Prof. Howe demonstrated techniques to fabricate micro beams from polycrystalline silicon films. Following this demonstration, Prof. Howe built the prototype polysilicon MEMS, the first fully integrated micromechanical and microelectronic system which he demonstrated in use as a chemical vapour sensor\(^2, 3\). In addition, Micro-Electro-Mechanical Systems (MEMS) consist of extremely tiny mechanical elements, often integrated mechanical elements, sensors, actuators, and electronics on a common silicon substrate through microfabrication technology\(^4\). Previously, sensors and
actuators were the most costly and unreliable part of a macro scale sensory-actuator-electronics system. However, with the advent of this technology “MEMS” these complex electromechanical systems are being manufactured using batch fabrication techniques with drastically reduced cost and improved reliability of the sensors and actuators.

1.2. MEMS Application

As mentioned, MEMS is a class of systems that are physically small. This system technology is miniaturized systems which comprise sensors, actuators and electronic functions thereby opening up a whole range of new applications which would not be possible with purely microelectronic systems. These systems have both electrical and mechanical components.

Sensors and actuators are the two main categories of MEMS. A sensor is a device that measures information from a surrounding environment and provides an electrical output signal in response to the parameter it measures. An actuator is a device that converts an electrical signal into an action. It can create a force to manipulate itself, other mechanical devices, or the surrounding environment to perform some useful function.

More recently a wide range of MEMS applications prototypes have been developed. MEMS are highly miniaturized devices that integrate a number of functions including fluidics, optics, mechanics and electronics on a single silicon chip using traditional integrated circuit process technology. MEMS merge sensing, actuating and computing into miniature systems that enable enhanced levels of perception, control and performance.
The field of Micro Electro Mechanical System has evolved during the last thirteen years from the techniques developed for the microelectronics industry. Nowadays, there are numerous possible applications for MEMS and Nanotechnology, here are a few applications of current interest:

1. Accelerometers

An example application includes advanced sensor technology used for airbag deployment for the automotive industry.

2. Biotechnology

Another example of a successful MEMS application is the optical device which is the miniature disposable pressure sensor used to monitor blood pressure in hospitals.

3. Inkjet printer head

One of the most successful MEMS applications is the inkjet printer head. Inkjet printers use a series of nozzles to spray drops of ink directly onto a printing medium.

More applications relate MEMS technology will be mentioned in the next chapters.
1.3. Motivation

Because of the high performance that Micro electro mechanical systems have shown at low cost, the demand for this technique has increased in various fields. Therefore, Micro electro mechanical systems have attracted researchers in many application fields. Hence the dreams of manufacturing mini devices become a reality, as a result of conducting a great variety of basic research into this technology and recently the fabrication technique as a general machining tool in micro scale is also a significant subject.

Since the use of conventional micro machining processes, such as anisotropic etching and surface micro machining, to make the first transistor from silicon in 1947, these techniques were suitable to build devices with two dimensional structures. Until this day, researchers have innovated and developed techniques such as ink jet nozzle arrays in 1978 and in 1982 the silicon was used to fabricate some mechanical components. Recently, in 2007, hybrid techniques used two or more techniques to improve the performance of MEMS. In the next chapter “History of MEMS” I will discuss the developments of MEMS.

In the 90’s a multi layer process was able to produce a three-dimensional structure with dimensions of the order of micrometers. The researchers succeeded in producing a special device and they used three layers and offered new vistas for researchers interested in this technology. From this date until today the researchers have tried to use this technique for an unlimited number of layers. Researchers in some areas succeeded in producing a device with 5 levels, but they did not use conventional techniques. They manipulated the different known conventional techniques to provide a hybrid technique. However, these hybrid techniques usually need high cost of manufacturing time and processing.
On the other hand, all researchers are having great difficulties creating three dimensional (3D) shapes with a high aspect ratio. Moreover, the selection of materials is limited to silicon or glass. Even the LIGA process, which can be used to create a micro structure with a high aspect ratio, cannot create general 3D structures with a curved surface. Also, all researches using the conventional technique in the multi layer process, mentioned that many unexpected problems appeared during the process, such as Lips, wrinkled surfaces and bubbles. After considering previous research that used the multi layer process in their technique, I did not find any research that treated these problems carefully. Most researchers have mentioned these problems in general although some researchers have reported that the problem is in the formation of gas bubbles. That is why all researchers have stopped using the conventional multi layer technique to produce 3D structure.

These issues have made serious limitations on the design and function of MEMS applications. On this account, a new micro fabrication technique satisfying real 3D by using multi layer moulds and a wide material selection is investigated for the mould by the author. In this thesis, a new method is proposed and verified experimentally.
1.4 Objectives

The objective of this work is to find and design a technique which can manufacture a complex 3D shape in micro/ nano scale: this technique should:

- be easy to use and low-cost,
- be suitable for different materials,
- save time, “if compared with any other techniques”.

To build a 3D structure device with a curved surface in the vertical direction was an aim for many researchers. None of them have been able to reach this aim in an easy way or by using conventional processes, but they have achieved it in complex and expansive ways. With the proposed technique, I will be able for the first time in the MEMS technology to fabricate a curved surface in the vertical direction of the device.

The proposed technique is based on improving conventional techniques and develops a hybrid technique using multiple techniques in one. I use a hybrid technique which consists of:

- LIGA, LIGA can produce 3D structure, but not a curved surface,
- thin film technique, this technique can produce a 3D structure, but not a curved surface,
- multi layer technique, which had many problems addressed in this work.
To make a 3D structure, there has to be a thin film deposited and then applied to the LIGA process, one layer on top of the other. The whole structure can then be stacked together using multi layers to meet the device requirement. This technique has improved the conventional technique. This improvement has made the MEMS technology cheap and efficient compared with any previous technique. With this technique I improved the LIGA process and any structure could be made using any material.

In this work, the author has researched a suitable material for the dissolvable mould and the technique that should be followed. All of the information available from previous research was somewhat conflicting and it was not clear which information should be followed. It was decided to do experiments to collect information about these materials and discover if they were good for these techniques or not. The experiments had two goals; to make demo-devices and discover the best material for the mould in this technique.

This work has solved many problems appearing in MEMS technology by using the conventional technique for the first time. The author has conducted many experiments to address many problems such as lips, bubbles and wrinkled surfaces and has found a good and effective technique to solve them.
### 1.2. Research Program

<table>
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<td>Identification of difficulties on building 3D structure in MEMS</td>
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<td>Participate conference paper</td>
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<td>Second year report</td>
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<td>11</td>
<td>Prepare to journal paper</td>
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</table>
1.3 Structure of the Thesis

To achieve the objectives of this research, the very first step was to understand the development of the field of micro electro mechanical system. In chapter 2 presents a Literature review in MEMS and I divided it in three sections. First section gives us a general introduction to the history of the MEMS and its development during these fifty years and to give the reader a good knowledge about this technique that I improved its performance by producing directly a device with 3D structure. The MEMS fabrication is a set of technologies used to manufacture structures with micrometric features. This task is a new technology and it is different from the traditional fabrication techniques such as milling, drilling, etc. In section 2 gives us an overview of the MEMS fabrication that helps the reader to understand the technique that I have used and improved. In the section 3, I am going to discuss some of the previous research and compare it with the proposed technique. The thesis concludes with a summary of this work and recommendations for future research. A number of important outcomes of this research are highlighted.

I will present in chapter 3 a guide line and demonstrate my proposed technique and how a device with a curve could be produced. This technique is a hybrid technique which consists of LIGA and thin film technique. The detailed explanation of this technique is in chapter 4. In this chapter I demonstrate how a device can be built by repeating two masks and when a successful product has been produced through this procedure, I have applied this technique using multi masks to fabricate a relatively complex shaped-device. Chapter 5 shows the problems and difficulties that I met during this work and a discussion about how these problems can be treated.
Chapter 1

Introduction

The author has contributed to the MEMS technology a new technique which can build a 3D complex shape device using conventional techniques. Therefore, he has offered a technique which saved time and cost if compared with any other techniques. The author published the idea in a conference in Barcelona in Spain in 2008 and submitted the work to Proceedings of the Institution of Mechanical Engineers, Part B, and Journal of Engineering Manufacture in June 2010. Both the paper conference and the journal paper are attached in Appendix A.12, A.13.
Chapter 2

Literature review
2.1. History of the MEMS

The field of Micro Electro Mechanical Systems (MEMS) has evolved during the last fifty years from techniques developed for the microelectronics industry. The technology for fabrication of microstructures in silicon has clearly played a key role in revolutionizing the impact of microelectronics on society. The fact that computers, that were once the size of entire rooms, have been replaced by small laptop computers of much higher performance illustrates this clearly.

For a comprehensive understanding of this technique of the MEMS technology I should know the history and the development that it has had during the last years. Consideration of the development that this technique has had, gives us a vision of how the researchers attention and their attempts have and still are ongoing to improve the performance of this technology. They have researched a way to make a device with a 3D structure in micro/nano scale. During these years this technology had amazing development which has started in the fifties of the last century with the discovery of the transistor and then the “integrated circuits (ICs)” which was a limited function and until today which the MEMs device interned in all of my devices that have an unlimited number of functions.

Until today researchers still try to support this technique to build directly a device with a 3D structure. Like any research, all of these researches have advantages and disadvantages. In this chapter I have tried to put in the hand of the reader a general introduction to the history of the MEMS and its development during these fifty years and to give him a good knowledge about this technique that I improved to produce directly a device with a 3D structure which I think will help mankind in our life.
2.1.1. General Historical Overview

In 1947 at Bell Telephone Laboratories, the first transistor was invented by William Shockley, John Bardeen, and Walter Brattain\(^5\). The first transistor was made from germanium, a semi conductive chemical material.

In 1954, Charles S. Smith discovered the Piezoresistive Effect in Silicon and Germanium in Bell Telephone Laboratories\(^6\). He found that silicon and germanium could sense air or water pressure better than metal. These significant discoveries led to the development of the concept of “integrated circuits (ICs)”. In 1958, Jack Kilby in Texas Instruments made the first “integrated circuits (ICs)”. This IC was built as a “Solid Circuit” on one germanium chip: 1 transistor, 3 resistors, and 1 capacitor, which was made all together on one substrate and the whole device was made to a very small scale \(^4, 7\).

In 1959, Richard Feynman gave a talk “There's Plenty of Room at the Bottom” at a meeting of the American Physical Society. In his talk he introduced the possibility of manipulating matter on an atomic scale and he challenged his audience to design and build a tiny motor or to write the information from a page of a book on a surface by using a scale of 1/25,000\(^8\).

In the 1950s, the use of electropolishing HF (hydrofluoric acid) showed the potential for deep etching in silicon substrates\(^9\). However the first silicon (pressure) sensor was fabricated using isotropic etching by Honeywell in 1962. This technology was a combination of wet and dry etching and oxidation\(^10\).

In the 1960’s, H. A. Waggener developed a technique called “Electrochemically Controlled Thinning of Silicon”. This technique is known
now as the bulk micromachining process. Bulk micromachining is a way to etch the bulk of the silicon substrate leaving behind the desired geometries. Since its foundation, bulk micromachining has remained a very powerful method for fabricating micromechanical elements such as micro-fluidic channels, nozzles, diaphragms, suspension beams and other moving or structural elements. The first surface micromachined structures were fabricated in the 1960s using metal mechanical layers\textsuperscript{[11]}. Surface micromachining involves the formation of mechanical structures in thin films formed on the surface of the wafer. The early 1980s saw the growth of silicon based surface micromachining using polysilicon as the mechanical layer\textsuperscript{[12]}.

In Westinghouse 1964, Harvey Nathanson produced the first fabricated MEMS device. His device joined a mechanical component with electronic elements and was called a resonant gate transistor (RGT)\textsuperscript{[13, 14]}. The RGT was a gold resonating MOS (Metal-Oxide-Semiconductor) gate structure, in which a layer of material is deposited between structural layers for mechanical separation and isolation. This layer is removed during the release etch to free the structural layers and to allow mechanical devices to move relative to the substrate. A layer is releasable when a sacrificial layer separates it from the substrate. The device was around one millimetre long and it responded to a very narrow range of electrical input signals. It was utilized as a frequency filter for ICs\textsuperscript{[14]}. The application of the sacrificial layer technique to micromachining in 1985 gave rise to surface micromachining, in which the Si substrate is primarily used as a mechanical support upon which the micromechanical elements are fabricated\textsuperscript{[5]}.

The world's first single complete chip microprocessor was generated and made in 1971 and was introduced by Intel as the ‘’The Intel 4004’’. The first application of the processor has to power the Busicom calculator\textsuperscript{[15]}, figure 1.
Since 1970, the complexity of chips has doubled every two to three years and engineers have tried to minimize the chip and at the same time contain several functions.

In 1979 Hewlett Packard developed the Thermal Inkjet Technology (TIJ). In this technique the ink squirts through an array of nozzles onto paper. The inkjet nozzle array was the first MEMS devices to have three-dimensional structures with dimensions. The minimum dimension of manufactured devices and ICs has decreased from 20 microns to the sub micron levels of today\(^{17, 18}\).

The field of MEMS evolved during the 1980s giving the world the first product of micromachining parts (such as pressure-sensor diaphragms or accelerometer suspension beams) for Si microsensors\(^5\).

In 1982, Kurt Petersen published a paper, which was called "Silicon as a Mechanical Material". The paper provided information on material properties and etching data for silicon and was instrumental in enticing the scientific community into exploration of these areas\(^ {19}\). In the same year a team at the Karlsruhe
Nuclear Research Center in Germany, developed a new process called LIGA\textsuperscript{[20, 21]}. The abbreviation LIGA is an acronym for the German words, “Lithographie”, “Galvanik”, and “Abformung” (lithography, electroforming, and moulding). This process was developed under the leadership of Prof. Dr. E.W. Becker and Dr. W. Ehrfeld, using X ray lithography for mask exposure, electroforming to form the metallic parts\textsuperscript{[20]} and moulding to produce micro parts with plastic, metal, ceramics, or their combinations\textsuperscript{[22]}. This process is important in microsystems manufacturing because it allows for the manufacturing of high aspect ratio microstructures. High aspect ratio structures are very thin, or narrow, and tall, such as a channel. LIGA can achieve ratios as high as 100:1 and LIGA structures have precise dimensions and low surface roughness\textsuperscript{[21]}.

In 1986, Binnig and Gerber from IBM Research Laboratory in Al-maden, California developed a micro device called the atomic force microscope (AFM). The AFM is a device that maps the surface of an atomic structure by measuring the force acting on the tip of a micro scale cantilever with a sharp tip or probe at its end. The cantilever is usually silicon or silicon nitride. The ultimate resolution of the AFM is down to about 10 Å\textsuperscript{[23]}.

In 1980s the development of using this technique “MEMS” was very big and had many successfully researched and there were many new applications such as the Sensonor Crash sensor (Airbag) in 1985\textsuperscript{[24]}, the first rotary electrostatic side drive motors in 1988 by Muller and the first polysilicon surface micromachined MEMS device integrated with circuits and lateral comb drive emerged where structures move laterally to the surface in 1989\textsuperscript{[25]}.

In the 1990s, a great increase in the number of devices and applications greatly expanded the sphere of influence of MEMS. For example, but not
exclusively, in 1992 at Cornell University, a bulk micromachining process was
developed called Single Crystal Reactive Etching and Metallization (SCREAM).
It was developed to fabricate released microstructures from single crystal silicon
and single crystal Gallium Arsenide (GaAs)\textsuperscript{[26]}. 

In 1993, Ikuta and Hirowatari developed a new process called IH process
(Integrated Harden Polymer Stereo Lithography)\textsuperscript{"} to conceive 3D objects with a
high aspect ratio and a curved surface. This process is applicable for micro
structures made of both polymer and metals and mainly used the micro stereo
lithography and electro plating and it had been proposed in MEMS’93\textsuperscript{[27]}.

In the same year a new technique appeared which was called multi layer
process. Figure 2 shows the conventional plating-through-mask technology and
its multi layer formation. Most fabrication processes for thick metallic
microstructures have their basis on this technology. The thick photo resist mould
blocks the current flow in the electroplating bath, so that the electroplating
happens only on the open-seed area. This technique has advanced in the last years
such as the technique called EFAB in California\textsuperscript{[15]}.

![Figure 2 the process of multi layer using seed layers\textsuperscript{[28]}](image-url)
In 1994, Bosch, a company from Germany, developed a special Deep Reactive-Ion Etching (DRIE) process. The DRIE process stands for Deep Reactive Ion Etching and is a dry etch micromachining method\textsuperscript{[29]}.

In 1998, another surface micromachining foundry began. This one was started at Sandia National Laboratories and the process was called SUMMiT IV. This process uses a five-layer polycrystalline silicon and later evolved into surface micromachining process. SUMMiT is an acronym for “Sandia Ultra-planar, Multi-level MEMS Technology\textsuperscript{[30]}.

In the same year, Kim and Mark demonstrated how a copper coil could be manufactured using microsurface machines and electroplating\textsuperscript{[31]}.

In addition, the 1990s witnessed the development of many techniques in this technology: such as embossing technology which appeared in the Karlsruhe Nuclear Research Center in Germany as part of LIGA process\textsuperscript{[32]}.
The increasing interest shown in sensors, actuators and other micro flow devices based on microfabrication techniques stems from the fact that they offer advantages such as small size, fast response times and small sample and dead volumes. The microfabrication techniques make multi-function systems possible, integrating sensors (e.g. for flow and pressure), actuators (e.g. valves and pumps), electronics and passive components (e.g. channels, valves and filters)\[33]\.

MEMS devices became commercially available in the early 2000s and it entered into our lives, in mobile phones, in automobiles and in home devices. Although time and cost have been reduced by using this technology, researchers continue trying to improve this technique and they are still discovering new ways to combine MEMS sensors and actuators with emerging bioMEMS technology. Applications include drug delivery systems, insulin pumps, DNA arrays, lab-on-a-chip (LOC), glucometers, neural probe arrays, and microfluidics, just to name a few. The area of bioMEMS has only just begun to be explored. Research and development at this time is occurring at a very rapid pace.

Since 2000 there has been a huge growth in the number of devices and many applications have appeared, (too many to mention individually). Nevertheless, I am going to mention a few particularly relevant examples:

In 2001, Metz, Holzer and Renaud developed a polyimide-based microfluidic device. They present a paper which describes the fabrication of the flexible microfluidic channels in various shapes and with a wide range of dimensions\[34].
To improve the Three-dimensional structures of the MEMS/NEMS device, in 2003 Kennedy, Jensen and Brett found a new way, which used the unique Glancing Angle Deposition (GLAD) technique to deposit a thin film\cite{35}, Figure 4.

![Figure 4 glancing Angle Deposition\cite{35}](image)

In year, 2005, an application from Moon, Lee and Kwon appeared which improved the LIGA process. They demonstrated a novel fabrication technology of the microneedle array. The fabrication technology consists of a vertical deep X-ray exposure and a successive inclined deep X-ray exposure with a deep X-ray mask whose pattern has a hollow triangular array\cite{36}.

In 2004, Karam and Casler introduced a novel technique “A new 3D, direct-write, sub-micron microfabrication process that achieves true optical, mechatronic and packaging integration on glass-ceramic substrates”. In this technique a laser beam was used to make a pattern\cite{37}.

The researchers knew the difficulty in designing a 3D structure in this scale and they researched and are still researching to find a technique to make it perfectly. Figure 5 is an example device which was produced in 2006 by Harutaka
Mekaru et al. The researchers introduced a paper “Fabrication of a spiral microcoil using a 3D-LIGA process”. In this paper they improved the LIGA process and they developed the 3D X-ray lithography and the warm injection moulding technology with the unscrewing release mechanism\textsuperscript{[38]}. 

![Spiral microcoil using a 3D-LIGA process](image)

**Figure 5**: Spiral microcoil using a 3D-LIGA process\textsuperscript{[38]}

In this section I have offered some development in the MEMS technology and I have demonstrated its history. Microsystems continue getting smaller. The applications and growth for MEMS and NEMS are endless and will continue to find their way into many aspects of our everyday lives. In this short history I have seen the development of this technology, in the beginning a small integrate circle with limited functions, then a computer the size of a house then in these days to a hand size computer with high performance. All of these developments have happened only in the last 50 years. From this the reader can see the amount of research that brought this technology to this efficiency and I can see the amount of demand to improve this technology more.

After this introduction to the MEMS history and its development, I have attempted to clarify the most important developments that have taken place in this new technology, but I know fully that I did not give the subject the correct attention, because it will take us a lot of time, and I have chosen to focus on the crucial points only. Now it is time to understand the procedure needed to produce a device with this technique.
2.2. Review of MEMS fabrication process

As seen in the first section this technique is a new technique if it is compared with any other processes such as casting or milling. This Micro fabrication is a set of technologies used to manufacture structures with micrometric features. This task can unfortunately not rely on the traditional fabrication techniques such as milling, drilling, turning, forging and casting because of the scale. The fabrication techniques have to come from another source\textsuperscript{[1]}. This technique needs a special environment and must take place in an area which is called clean room. In this section I will give an overview of the MEMS fabrication “only the conventional techniques” that helps the reader to understand the technique that I have used and improved.

2.2.1. General description

MEMS technology is based on a number of tools and methodologies that are used to form small structures with dimensions in the micrometer scale. The important parts of this technology have been adopted from integrated circuit technology. For example, most devices are installed and built on wafers of silicon, such as ICs. The structures are realized in thin films of materials. They are patterned using lithography methods\textsuperscript{[39, 40]}.

Generally, the lithography method starts by a surface preparation step like almost all microfabrication processes. In order to remove moisture, the wafers are baked\textsuperscript{[41]}. The second step is the depositing of a thin film of metal on a substrate wafer usually silicon, in a process called deposition process or coating the substrate wafer directly with a special organic polymer (sensitive material), which is sensitive to light or X-ray, using a spinning process and called spin-coating. In
this process the polymer is poured on the substrate which is then set to high-speed rotation, spreading the photo resist in a thin-film with very uniform thickness. The polymer solvent is then evaporated by baking on a hotplate or in an oven. This is the baking process. Then a patterned mask is used via lithography methods, this mask has the negative shape of the desirable device in 2D, then is aligned on the organic polymer. The organic polymer is then exposed to ultraviolet (UV) light of a suitable wavelength, by photolithographic imaging in a process called pattern transfer. In this process the light passes through the shape in the mask onto the sensitive layer, Figure 6, the light changes the property of considered zone. The exposure time depends on the thickness of the organic polymer.

Figure 6 the light passes through the mask

Next the sample is immersed in a special chemical solution, which removes the material that is exposed to UV light and leaves holes on the organic polymer, Figure 7, which represents the inverse of the desirable device structure. This process is called development\textsuperscript{[42, 43]}.
In some processes these holes are filled with a desirable material, Figure 8, this is the process that will be used in this research. In other processes the thin metal films deposited on the substrate wafer or the substance wafer is etched with features corresponding exactly to those holes, using the etching process. All of these processes will be discussed later.
In effect, this organic polymer is used as a sacrificial layer. This sacrificial layer is required to keep the other layers separated as the structure is being built up but is dissolved in the very last step allowing selective parts of the structure freedom to move \(^{[28]}\). The 2D configuration of the desirable device is drawn on a mask and the light passes through the mask to transfer the 2D onto the sensitive layer. The conversion into 3D depends on the thickness of the deposited layer or on the depth of the etching process. Thus this process is called 2.5D \(^{[27, 44, 45]}\). In other words, with this technique, I can only build a device that I can draw in 2 dimensions. This is the main disadvantage of MEMS technology. It cannot make the structure of the desirable device directly in 3D. In recent years researchers have developed many methods to improve the performance of the MEMS technology.
2.2.2. Depositing thin film

The ability to deposit thin films of substrate material is one of the major steps in MEMS processing. To make a device I should create a layer over this substrate wafer and I call this layer a thin film. How can I make this thin film over the substrate material? The process to make it is called deposition of thin film. Common deposition films include polysilicon, silicon nitride, silicon dioxide, tungsten, and aluminium. There are a number of different techniques that facilitate the deposition or formation of very thin films. MEMS deposition technology can be classified in two groups: chemical reaction and physical reaction\[4, 41\].

2.2.2.1. Depositions resulting from physical reaction

In this process the deposited material is physically moved on to the substrate material. In other words, there is no chemical which forms the deposited material on the substrate material. Like chemical deposition, physical deposition comprises many different techniques\[4\].

Within the physical deposition process there are many different types of technique to do this process but the common one is physical Vapor Deposition (PVD). PVD covers a number of deposition technologies in which a material is released from a source and transferred to the substrate. The two most important technologies are evaporation and sputtering. Table 1 shows us the Advantages and Disadvantages of each.
Evaporation | Sputtering
---|---
**Advantages** |  high film deposition rates  
  excellent purity of the film because of the high vacuum condition used by evaporation |  synthesis of ultrathin films with minimal impurity  
  easily controllable process parameters |
**Disadvantages** |  step coverage is more difficult to improve by evaporation than by sputtering |  High capital expenses are required  
  The rates of deposition of some materials (such as SiO2) are relatively low |

Table 1 the Advantages and Disadvantages the evaporation and sputtering deposition\textsuperscript{[46]}

2.2.2.2. Depositions resulting from chemical reactions

Chemical deposition is a chemical process for depositing thin films of various materials. In this process the substrate layer is exposed to one or more volatile materials, which react and/or decompose on the substrate surface to produce the desired deposit\textsuperscript{[4, 28, 41, 47]}. Under the chemical deposition process there are many different types of technique to do this process but the common one is Chemical Vapor Deposition (CVD).

Chemical vapor deposition (CVD) can be defined as a deposition technique, in which chemical components react in the vapor phase on a surface, to form a solid film. In this process the substrate material is placed inside a reactor to which a number gas is supplied. The fundamental principle of the process is that a chemical reaction takes place between the source gases. The product of reaction is a solid material which condenses on all surfaces inside the reactor\textsuperscript{[28, 48]}. The four most important CVD technologies in MEMS are Atmospheric pressure CVD (APCVD), Low Pressure CVD (LPCVD), Plasma Enhanced CVD
(PECVD), and High Density Plasma CVD (HDCVD). Table 2 shows us the Advantages and Disadvantages of each.

<table>
<thead>
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<th>Atmospheric pressure CVD (APCVD)</th>
<th>Low Pressure CVD (LPCVD)</th>
<th>Plasma Enhanced CVD (PECVD)</th>
<th>High Density Plasma CVD (HDCVD)</th>
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</thead>
<tbody>
<tr>
<td><strong>Advantages</strong></td>
<td>Simple reactor design</td>
<td>Excellent purity</td>
<td>Low temperature</td>
<td>Low temperature</td>
</tr>
<tr>
<td></td>
<td>High deposition rates</td>
<td>Excellent uniformity</td>
<td>High deposition rates</td>
<td>High deposition rates</td>
</tr>
<tr>
<td></td>
<td>Low temperature</td>
<td>Good step coverage</td>
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<td></td>
<td></td>
<td>Lange wafer capacity</td>
<td>Better film composition</td>
<td>Better film composition</td>
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<td></td>
<td></td>
<td>Greater control of</td>
<td>adjustment</td>
<td>adjustment</td>
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<tr>
<td></td>
<td></td>
<td>thickness and resistivity</td>
<td>Wider control of</td>
<td>Wider control of film density</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>film stress</td>
<td>film stress</td>
</tr>
<tr>
<td><strong>Disadvantages</strong></td>
<td>Particle contamination</td>
<td>High temperature</td>
<td>Chemical and particle</td>
<td>Plasma induced film damage</td>
</tr>
<tr>
<td></td>
<td>Gas phase reactions</td>
<td>Slow deposition rate</td>
<td>contamination</td>
<td>Process damage</td>
</tr>
<tr>
<td></td>
<td>Poor step coverage</td>
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<td>Low film density</td>
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</table>

Table 2 the Advantages and Disadvantages the CVD deposition mouthed$^{[4, 28, 41, 48]}$

Within the chemical deposition process there are many other different types of technique. For example, oxidation, dry oxidation, wet oxidation, selective oxidation, electro deposition, epitaxy, and thermal oxidation$^{[48]}$
2.2.3. Electrodeposition

Electroplating or electrodeposition is a plating process that uses electrical current to deposit a desired material from a solution and coat a conductive surface with a thin layer of the material. In this process a substrate is placed in a liquid solution (plating solution) Figure 9. The part to be plated (the substrate wafer) is the cathode of the circuit. The anode is made of the metal to be plated on the part. Both components are immersed in the plating solution, which contains one or more dissolved metal salts.

![Diagram of electroplating process](image)

**Figure 9** process of the electroplating

The anode and cathode are both connected to an external supply of direct electrical current. The anode is connected to the positive terminal of the supply, and the cathode (the substrate wafer) is connected to the negative terminal. When the external power supply is switched on, the metal at the anode is oxidized from the zero valence state to form cations with a positive charge. These cations associate with the anions in the solution. The cations are reduced at the cathode to...
deposit in the metallic, zero valence state. For example, in an acid solution, 
copper is oxidized at the anode to \( \text{Cu}^{2+} \) by losing two electrons. The \( \text{Cu}^{2+} \) 
associates with the anion \( \text{SO}_4^{2-} \) in the solution to form copper sulphate. At the 
cathode, the \( \text{Cu}^{2+} \) is reduced to metallic copper by gaining two electrons. The 
result is the effective transfer of copper from the anode source to a plate covering 
the cathode (the substrate wafer)\textsuperscript{[49]}. In other words, when an electrical potential 
is applied between a conducting area on the substrate and a counter electrode in 
the liquid, a chemical redox\textsuperscript{[50]} process takes place resulting in a deposition of a 
layer of material, which happens spontaneously on the substrate, and usually 
some gas generation at the counter electrode\textsuperscript{[51, 52]}. Standard electroplating 
solutions exist for a range of metals such as copper, gold and nickel, including all 
noble metals.

In many 3-D structures fabrication the use of gold electroplating is 
preferred because of its excellent corrosion resistance, solderability and 
bondability and its high electrical and thermal conductivity\textsuperscript{[51]}. In this work, gold 
electroplating will be used to form the desired device. It is also used to fabricate 
elevated structure of the MEMS switches\textsuperscript{[53]}. The electroplating system in the 
University of Glasgow uses an electrical current to reduce cations of a desired 
metal from a plating solution and coat a conductive object with a thin layer of the 
metal. The anode and cathode in the electroplating cell are both connected to an 
external supply of direct current. The plating current was set by this equation.

\[
\text{Plating Current (mA)} = (\text{sample holder area} - \text{sample area}) \text{ mm}^2 \times 0.013\textsuperscript{[54]}
\]

Controlling the thickness of the electroplated object generally depends on 
altering the time the object spends in the salt solution. The longer it remains 
inside the bath, the thicker the electroplated shell becomes. It depends on the
temperature of the solution and on the current\textsuperscript{[55, 56]} as well. Of course there must also be an adequate amount of metallic ions in the bath to continue coating the object. The shape of the object will also have an effect on the thickness. Sharp corners will be plated thicker than recessed areas. This is due to the electric current in the bath and how it flows more densely around corners\textsuperscript{[51, 52]}.

The electroplating process for gold or any other material is shown schematically in Figure 10. In this process, the substrate is typically covered with a conductive, which acts as the plating base and an underlying adhesion; also it prevents contamination of the plating solution (barrier layer). Typically these metal seed layers are deposited using sputtering or evaporation. The seed layer for gold electroplating usually is two level metallization such as Ti/Au or NiCr/Au for good adhesion. The substrate is then patterned with a photoresist, and gold is selectively electroplated into the resist-free areas. Following plating of the structures to the desired height, the photoresist mask is stripped and the metal plating base is removed by wet etching.
2.2.4. Photolithography

The second step in the MEMS manufacturing is Lithography or Photolithography. The lithography is a word which comes from the Greek words “λίθος - lithos, 'stone' and γράφω - grapho, 'to write'. Literally translated as writing on stone”, the lithographic printing technique has been known since the 18th century\cite{57}. Through this technique a stone slab was used onto which an oil or fat was applied to divide the slab into hydrophobic and hydrophilic regions. Ink applied to the slab would adhere to only the hydrophilic regions and when paper was brought into contact with the slab the ink would transfer to the paper producing a copy of the hydrophilic regions on the slab.

Here the lithography in MEMS fabrication the 'stone' used is called a photomask and contains the pattern, and the 'paper', onto which the pattern will be 'printed', is known as the substrate or the thin film on which deposited.

To write on these substrates or the thin film there are numerous lithographic techniques. But the three techniques most commonly used are optical lithography, electron beam lithography and imprint lithography. In optical lithography a source of illumination is directed through a mask that contains the desired pattern, on to the top of the substrate. This is similar to how a slide projector produces an image of the slide on a projector screen. In this work I will use optical lithography. In electron beam lithography a beam of electrons is applied on the substrate surface which is mounted on a motorised stage. Carefully moving the sample and detecting the electron beam across the whole of the substrate's surface allows the desired pattern to be traced out\cite{58, 59}. With imprint lithography a deformable layer on top of the substrate is directly structured using a moulding process\cite{59, 60}. 
Lithography is the one major process in MEMS fabrication by which the geometric patterns that define the devices are transferred from a reticle (also called a photomask, or mask) to a light-sensitive chemical material (photoresist, or simply "resist") which has been deposited on the substrate wafer. It uses UV light or X-ray radiation to transfer the geometric pattern from the mask onto the resist material. This method is used because it allows exact control over the shape and size of the objects it makes, and because it can make many patterns over an entire surface simultaneously, Figure 11. The main disadvantage to this method is that it requires a flat substrate to start with, and it can require extremely clean operating conditions\cite{4, 61}.

Figure 11 the Photolithography processes

Photolithography work flow consists of the following major steps when viewed from the point of view of the wafer:
1. Preparation of the photosensitive film (photoresist)

2. The Photomask and Alignment of the substrate wafer

3. Exposure of the photoresist


2.2.4.1. Preparation of the photosensitive film (photoresist)

The silicon wafer or the deposited material is coated with a light-sensitive material (Photoresist layer) or resist material layer or sacrificial layers as some like to call it. These resists materials are often produced in liquid (as a solvent) form and are typically cast onto the substrate by spin coating. The spin coating is the standard resist application method, Figure 12. A few millilitres of resist is applied on the substrate wafer at the centre of it and slowly rotated. Then acceleration increased to ca. 10000 rpm spreads the resist over the wafer towards the edges, leaving a very uniform layer with thicknesses range from 0.1 μm up to 500μm depending on the rotated speed.

Figure 12 spin coating process
The most common method of coating planar surfaces is spin coating in MEMS fabrication. However, there are other techniques for special coating such as spray coating for a uniform resist layer over a nonplanar surface\textsuperscript{[62, 63]}.

The main parameters for film thickness control are not only the spin speed but also the viscosity and solvent evaporation rate. Spin coated films fill cavities and recesses because they are liquids during spin coating. This is advantageous for gap filling and smoothing\textsuperscript{[4, 40]}. The remaining solvent evaporates during the bake process and hardens the photoresist.

The purpose of this layer is to produce movable parts or to help to produce 3D micro machining. For example, a suspended beam cantilever can be built by depositing and structuring a polymer (sacrificial layer), which is then selectively removed at the locations where the future beams must be attached to the substrate (i.e. the anchor points). The structural layer is then deposited on top of the polymer and structured to define the beams. Finally, the polymer is removed to release the beams, using a selective etch process that will not damage the structural layer. There are many possible combinations of the structural/sacrificial layer. The combination chosen depends on the process. For example, it is important for the structural layer not to be damaged by the process used to remove the polymer\textsuperscript{[64]}.

Once the resist is spun on, the substrate is then baked to remove the remainder of the solvent. The product is a uniform solid resist coating on the substrate.
2.2.4.2. Baking process

The baking process\[^{65, 66}\] is the step during which almost all of the solvents are removed from the photoresist coating. The baking process plays a critical role in photo-imaging. The photoresist coatings become photosensitive, or imageable, only after baking. Over-baking will degrade the photosensitivity of resists by either reducing the developer solubility or actually destroying a portion of the sensitizer. Under-baking will prevent light from reaching the sensitizer. Positive resists are incompletely exposed if considerable solvent remains in the coating. This under-baked positive resists are then readily attacked by the developer in both exposed and unexposed areas, causing less etching resistance. The bake of thick resist coatings is a decisive factor determining the patterning result. A much higher solvent amount has to be evaporated from the coating. This requires long bake times or/and higher temperatures. Degradation of the photoactive compound may occur as a consequence of this and the function of the resist deteriorates\[^{66}\].

There are two techniques for the bake process, one technique uses only a heating step before the pattern transfer process and the development process, and not after that, and this technique is called soft bake\[^{40, 67}\]. The other technique uses two heating steps, one before the pattern transfer process and the development process and one after these processes, and these are called prebaking and postbaking\[^{4, 28, 68}\].
2.2.4.3. The Photomask

A photomask, Figure 13, is an opaque plate made from glass or quartz with holes or transparencies that allow light to shine through in a defined pattern. Where the mask is blocking the light (Ultraviolet or X-ray), the photoresist on the wafer is not exposed. Where it is transparent, light falls onto the wafer and the photoresist is exposed\cite{1, 4, 40}.

![Figure 13 Photomask](image)

**Aligned process:**

After the bake process the photoresist is then exposed to UV or X-Ray radiation. Firstly the substrate wafer is aligned under the desired mask, Figure 14. In this crucial step the mask must be aligned correctly with previous layer on the substrate wafer.

![Figure 14 the aligned process](image)
2.2.4.4. Exposure process and Development of patterns

The most common resist for positive resists is phenolic Novolak, which is soluble in alkaline developers. Diazonaphthoquinine (DNQ), a photoactive compound, acts as an inhibitor; and the unexposed resist is therefore non-soluble in developer. Upon exposure, DNQ decomposes and releases carboxylic acid, which makes the exposed resist soluble. More information in reference [40].

Figure 15 Positive and Negative resist
The organic polymer becomes soluble when exposed to ultraviolet light, and which is used in photolithography, and temporarily holds the pattern of the required made device. When the resist material is exposed to radiation (Ultraviolet or X-ray), this leads to a chemical change in the properties. These changed areas are dissolved from the layer during development, leaving a relief-like structure in the photoresist coating. The photoresist is classified into two types, positive and negative\cite{1}, Figure 15.

When the photoresist is exposed to a light, the light leads to a change in the chemical structure of the resist so that it becomes more soluble in the developer. The exposed resist is then removed by the developer solution, leaving holes in the layer of the underlying material. In other words the mask will be an exact copy of the pattern which is to remain on the wafer. This process is called a positive resist.

When the photoresist is exposed to a light, the light polymerizes the exposed surface in the photoresist, and it will be more difficult to dissolve. Therefore, the negative resist remains on the surface wherever it is exposed, and the developer solution removes only the unexposed portions. Masks used for negative photoresists, therefore, contain the inverse of the pattern to be transferred. This process is called a negative resist\cite{1, 4, 40}.
2.2.5. Etching processes

In order to form a functional MEMS structure on a substrate, it is necessary to etch the substrate wafer or the deposited thin films related to the required device. The desired pattern is transferred from the photo resist to the underlying film or the substrate wafer by a process known as etching. Etching is a process which can be classified as two processes, wet etching and dry etching\[^{69}\], to remove unwanted portions of conductive or resistive material. In other words, etching is the process by which entire films, particularly sections of films or the substrate wafer, are dipped in acid which eats into the exposed surface revealing the 3D structure. This process plays an important role in the fabrication sequence\[^{4}\].

2.2.5.1. Wet etching

Wet etching involves the use of liquid chemical reactants to etch the desired material. Wet etching is performed by immersing the wafers in an appropriate solution or by spraying the wafer with the etchant solution.

Common Chemicals used in Wet Etches are:

- HF used for glass/silicon etch
- Nitric Acid used in silicon etch
- Phosphoric for Aluminum etch
- Ammonium Hydroxide in RCA Clean
- Acetic Acid as buffer agent\[^{4, 28, 41, 47}\]
Wet etching has some advantages: low cost, low damage to the wafer, high selectivity, and high throughput. However, it has some disadvantages: limited resolution; higher safety risks due to the direct chemical exposure of the personnel; high cost of etchants in some cases, poor anisotropy, and poor process control (temperature sensitivity)\[70]\.

2.2.5.2. Dry etching

Dry Etching is a type of etching process that does not use any liquid chemicals or etchants to remove materials from the wafer. In contrast to the wet etching process, dry etching can have a high degree of directionality, resulting in highly anisotropic etch profiles. Dry etching may be accomplished by any of the following\[69]\:

- Through chemical reactions that consume the material, using chemically reactive gases or plasma
- Physical removal of the material, usually by momentum transfer
- A combination of both physical removal and chemical reactions

There are various types of dry-etch processes, ranging from physical sputtering and ion-beam milling to chemical-plasma etching. Reactive ion etching(RIE), the most common dry-etch technique, uses a plasma of reactant gases to etch the wafer, and thus is performed at low pressure in a vacuum chamber\[47]\. As mentioned, in 1994 a technique called the Bosch process appeared\[71]\. This technique used a special Deep Reactive-Ion Etching (DRIE) process. The DRIE process is a dry etch micromachining method. In this a gas is first used to etch the pattern, then deposit and develop the polymer. Then the gas
is used to etch to make the pattern. The same steps are repeated to obtain the desirable high\textsuperscript{[28]}. There are other types of etching used in dry etching techniques; Ion Mill, Plasma Etching, Reactive-Ion-Beam Etching (RIBE), Electron Cyclotron Resonance (ECR) and Inductively Coupled Plasma (ICP)\textsuperscript{[69]}

Dry etching has some advantages and disadvantages. The advantage is that high anisotropic etch can be achieved using sidewall polymerization techniques, independent of material composition. The disadvantages are:

- High Ion damage
- Expensive
- Difficulty in obtaining smooth etched sidewalls
- Some methods requires high temperature\textsuperscript{[28, 69]}

The major advantage of dry over wet etching is anisotropy. It is important to understand the range of directionality that may be obtained using dry-etching techniques. There are four basic mechanisms; sputtering; purely chemical, Ion-Enhanced Energy-Driven Mechanism, and Ion-Enhanced Inhibitor\textsuperscript{[69]}. 
2.2.5.3. Isotropic and Anisotropic Etching:

The researchers classify the wet etching in two types: isotropic and anisotropic etching\cite{72}. The technique of fabrication of Harvey Nathanson\cite{11} is known as isotropic etching Figure 16. Isotropic etching removes material from a substrate or the thin film using a chemical process. The material is equally removed in all directions due to the etch rate being uniform in all directions.

![Figure 16 isotropic etching](image)

Anisotropic etching differs from isotropic etching in that the removed material is dependent on the crystallographic orientation of the silicon crystal. The etch rate (the amount of material removed per unit of time) varies greatly for the different crystal planes. The silicon can then be etched away selectively creating a variety of structures including v-shaped grooves, pyramid-shaped mesas and micro-chambers, Figure 17, in other words, the etching rate is different in horizontal and vertical directions\cite{4, 47}.

![Figure 17 anisotropic etching](image)
Both isotropic and anisotropic techniques can be employed in wet and dry etching.

There are a number of MEMS microfabrication technologies. In general, researchers classify lithography technology into three different manufacturing processes as traditional fabrication of MEMS\cite{1, 37, 39, 42}. Surface micromachining makes completely assembled mechanical systems. Bulk micromachining uses either etches that stop on the crystallographic planes of a silicon wafer or etches that act isotropically to generate mechanical parts. These techniques combine with wafer bonding and boron diffusion allowing complex micromechanical devices to be fabricated. The third technique is Mould micromachining.
2.2.6. Bulk Micromachining

Bulk micromachining is an old technique. Researchers have known about this technique for nearly 40 years and they have used it to enable the MEMS micromachining. As mentioned, Harvey Nathanson in Westinghouse 1964\textsuperscript{[11]} and his fabrication of silicon transistors brought about the process of etching in silicon and started the technique of “bulk micromachining”. This technique is a fabrication technique which builds mechanical elements by starting with a silicon wafer, and then etching away unwanted parts, and being left with useful mechanical devices to form three dimensional mechanical structures such as cantilevers, diaphragms, and channels\textsuperscript{[72]}.

![Figure 18 sensitive material after developing, bulk micromachining](image1)

![Figure 19 etching in the thin film](image2)

![Figure 20 the thin film after removing the sensitive material, bulk micromachining](image3)
In the process a substrate wafer is selectively etched, using lithography to transfer a pattern from a mask to the surface. This is sometimes done to “undercut” structures that are required to physically move; to form membranes on one side of a wafer, or to make a variety of trenches, holes, or other structures\cite{73,74}, Figure 18, 19 and 20.

The underlying thin film or the substrate can be subsequently etched. The etching process can be done using wet etching or through reactive ions known as dry etching\cite{74}.

2.2.7. Surface Micromachining

Until the early 1980s, bulk micromachining was the most common form of machining micrometer scales. While bulk micromachining creates devices by etching into a substrate wafer, surface micromachining techniques build up the structure in layers of thin films on the surface of the substrate wafer (or any other suitable substrate)\cite{64}. Surface micromachining starts with a substrate wafer or thin film and grows layers on top. These layers are selectively etched by photolithography and either a wet etch or a dry etch. Surface micromachining can involve as many layers as is needed with a different mask (producing a different pattern) on each layer. Modern fabrication uses this technique and can use many layers. Micromachining is a younger technology and the number of layers is still limited\cite{64}.

The surface micromachining showed the viability of the micromechanics technology. Moving motors, gears, accelerometers and uncounted structures have been fabricated and tested. Significant freedom in device design is allowed using this technology\cite{26,75}. In most surface micromachining processes the underside of
the MEMS structural layer, resulting from chemical vapour deposition or physical deposition on the sacrificial layer, is planar. The addition of surface topography on the underside of the structural layer requires patterning of the sacrificial layer before deposition of a conformal structural layer\cite{76, 77}, Figure 21, 22 and 23.

![The sensitive material after developing, surface micromachining](image1)

Figure 21 sensitive material after developing, surface micromachining

![The deposited thin film](image2)

Figure 22 the deposited thin film, surface micromachining

![The thin film after removing the sensitive material, surface micromachining](image3)

Figure 23 the thin film after removing the sensitive material, surface micromachining

Later, a technique appeared which used both techniques as a hybrid technique known as SCREAM (single crystal silicon reactive etching and metallization) process\cite{4, 26}. The SCREAM process is a dry bulk micromachining process\cite{78} and uses Reactive Ion Etching (RIE)\cite{71} to both define and release the structures. SCREAM requires two masks to define the structural elements and the metal contacts. The process is explained in [26].
The great advantages bulk micromachining has over surface micromachining is that bulk techniques can be used quickly and uniformly over a large wafer surface area. This quality makes bulk micromachining a relatively cheap process, in terms of both money and time. The disadvantage of bulk micromachining is that it is not easily integrated with microelectronics, as surface micromachining is. This flaw is due to the isotropic nature of wet etching, which limits line width resolution. Surface micromachining is useful for forming free-moving microstructures, including basic rotating structures that cannot be realized using bulk micromachining techniques. Surface micromachining is highly anisotropic, but since it relies on high speed, high energy ion collisions, there is a high potential for radiation damage to the surface of the wafer, and this sometimes results in poor selectivity\textsuperscript{70}. This radiation often reduces the yield for surface micromachining processes, making the process more costly than bulk micromachining. Also, surface micromachining techniques are often much more time-consuming than bulk micromachining techniques.
2.2.8. Mould Micromachining

Since the establishment of MEMS technology researchers have tried to improve a method to make a 3D structure using this technology. In Bulk and surface Micromachining technology the 3D structure depends on the thickness of the deposited layer or on the depth of the etching process and the most common material in use is silicon. Both methods have the same problem, ‘high surface damage’. Another technique to make MEMS devices is the Micro moulding process. This refers to the fabrication of microstructures using moulds to define the deposition of the structural layer. In the micro moulding process a mould in the inverse shape of the required structure is developed. This mould is then filled with a structural material and later the mould is etched or removed leaving the desired structure behind in the structural material. Micromoulding is an additive process, in that the structural material is deposited only in those areas constituting the microdevice structure. In contrast, bulk and surface micromachining are examples of subtractive micromachining processes, which feature blanket deposition of the structural material followed by etching to realize the final device geometry\textsuperscript{[4, 47]}.

2.2.8.1. Stamping technique

One microfabrication technique to make MEMS devices which is included under the Micro-moulding process is stamping. In mould micromachining, a mould in the inverse shape of the desired structure is developed. A molten or liquid material is used and later the mould is removed leaving the desired structure behind in the structural material, Figure 24.
In stamping, there is no transport of material: the polymeric material, which is on the wafer to begin with, is modified locally by the stamp, Figure 25.

The mould micromachining can be further divided into methods that use reusable or disposable moulds as resist material. In stamping, the researchers can distinguish two cases: 2D-surface processes and 3D-volume processes, which have rather different requirements for stamp masters. For more information about these techniques I refer to these references [40]. One of the most prominent micro moulding processes is the LIGA process.
2.2.8.2. LIGA

A widely known micromolding process is the LIGA. LIGA is an abbreviation for the German words, “Lithographie”, “Galvanoformung”, and “Abformung” (lithography, electroforming, and moulding). As mentioned, the LIGA process was developed by the Institute for Microstructure Technology of Forschungszentrum Karlsruhe in Germany (Institute for Nuclear Engineering) in 1986 under the leadership of Prof. Dr. E.W. Becker and Dr. W. Ehrfeld, using X-ray lithography for mask exposure, electroforming to form the metallic parts\cite{20} and moulding to produce micro parts with plastic, metal, ceramics, or their combinations\cite{22}. These three steps make it possible to mass-produce micro components at a low-cost. The main advantage is that the LIGA process delivers consistently smoother, straighter sidewalls, resulting in better of parts fit and smoother operation. Other advantages are:

- structures with extreme high aspect ratios (ratio between the height of a structure and the minimum lateral dimension) can be fabricated;
- there are no limits concerning the two-dimensional shape of a device;
- a broad range of materials (polymers, metals, ceramics) can be used;
- with advanced scanner systems “real” three dimensional structures can be fabricated;
- partly movable, more complex structures can be fabricated using sacrificial layer techniques\cite{79}

Virtually any shape that can be drawn in 2 dimensions and which has vertical sides (thickness) between 100 microns to several mm can be produced currently.
LIGA has only recently been accepted as a proven, commercially available production process. Today, millions of high precision parts are fabricated using LIGA production processes. However, the high production cost of LIGA process, due to the fact that it is not easy to access an X ray source, limits the application of LIGA\cite{80}. Another disadvantage of the LIGA process is that it relies on the fact that structures fabricated using LIGA are not truly three dimensional, because the third dimension is always a straight feature. The quality of the fabricated structures often depends on secondary effects during exposure and effects like resist adhesion. A similar technique, UV LIGA, relying on thick UV resists is a useful fabrication with less precision. Modulating the spectral properties of synchrotron radiation, 3D components with different size regimes can be fabricated using X ray lithography. On the other hand, only 2D structures can be made in shallow photoresist layer and 3D built from the thickness of deposited layer, from that engineers have described this process ‘LIGA’ 2.5D and is not a complete 3D structure \cite{38}.

There are many lithographic techniques in existence but only a limited number of techniques are able to produce the high aspect ratio. The LIGA process is one of the processes currently used to produce microstructures. LIGA has achieved the vertical sidewalls of tall structures with good dimensional control over the entire height and with the LIGA process; microstructures’ height can be up to hundreds of microns to millimetre scale, while the lateral resolution is kept at the submicron scale because of the advanced X ray lithography. Various materials can be incorporated into the LIGA process, allowing electric, magnetic, piezoelectric, optic and insulating properties in sensors and actuators with a high-aspect ratio, which are not possible to make with the silicon-based processes. Besides, by combining the sacrificial layer technique and LIGA process, advanced MEMS with moveable microstructures can be built\cite{80}. The use of X ray
lithography to pattern PMMA (polymethylmethacrylate) layers is an ideal technique to achieve high-aspect-ratio structures with submicron resolution in very thick resists. However, the cost of the X ray source (synchrotron radiation) is very expensive and the availability of high aspect ratios in very thick resists is not only interesting, it is often a requirement for fabricating micromechanical structures for MEMS or packaging applications, hence it would be very attractive if near UV resists with lower resolution (microns to tens of microns) and lower aspect ratios but otherwise having characteristics similar to PMMA/X ray were available. Such an optical-lithography-based LIGA process technique would have great potential for low-cost MEMS fabrication. IBM\textsuperscript{[81, 82]} in 1989 reported a new resist (SU-8) that has the potential to fulfil the above profile. The SU-8 is a negative photoresist and was developed by Gelorme et al\textsuperscript{[82]}, near-UV photoresist based on EPON SU-8 resin (from Shell Chemical). The key properties that make the SU-8 so attractive for ultra thick resist applications are its very low optical absorption in the near-UV range. This leads to uniform exposure conditions as a function of thickness, which gives rise to vertical sidewall profiles and hence good dimensional control over the entire structure height\textsuperscript{[83, 84]}. SU-8 polymer used as a resist is negative tone which apparently crosslinks sufficiently to repel swelling in an organic developer. This high crosslink density produces the undesired side-effect of making it difficult to remove.\textsuperscript{[81, 82, 85]}

The type of LIGA depends on the irradiation that is used in the exposure step in this process and it can be varied for a comparison of the behavior in matter of some of these types. In X LIGA, synchrotron X ray radiation is used to irradiate high aspect ratio masks. X LIGA technology has today a limited potential for industrial applications because of the high cost related to the exposure procedure and the manufacturing of masks. Furthermore, the thickness of the structures cannot be controlled. UV LIGA is much cheaper but has a
minimum feature size larger than 1 mm\cite{22}. However, only 2D structures can be made in shallow photoresist layers. Electron beams can be highly focused and can produce very fine structures, well below 100 nm. However electrons are very light and therefore scatter easily in material which results in a loss of resolution at depth. IB LIGA (ion beam LIGA) has been developed as an alternative LIGA technique. It uses high energy (MeV) light ions, mostly protons. IB LIGA can also be collimated or focused for use as a (maskless) direct-write tool. For more detailed comparison of various lithographic techniques see reference \cite{22}.

### 2.2.8.3. Visit: LIGA manufacturing centre

One of the tasks done in this work was visiting a manufacturing centre using LIGA process. The aim of this visit was to stand close to this technology and to realize its steps well. In July 2008 and after one year researching and studying previous research, I decided to visit that centre located in Germany. This centre is now called “Karlsruher Institut für Technologie (KIT)”. In this centre LIGA first appeared in the world and it is the main centre using this technique. I went there and I spent two days in this centre. I have learned there how to use the LIGA to produce some gears and others structures which are utilized in constructing watches. In this centre the researchers used another technique which is more advanced than the LIGA this process is called hot embossing, in which a dry photoresist is used rather than conventional photoresist. I have discussed with the researchers about the difficulties associated with the fabrication processes, especially by using multi levels to produce 3D structures. They agreed that many problems occur when they used multi levels and they avoided to use this technique for producing any structure. Then I asked them how a device which has a curvature in the vertical direction could be made. The answer was not clear; they avoid manufacturing a device which has these shapes.
After my visit to this centre, I knew that producing a device with complex shape with any techniques is still a big challenge. That supported my decision to enter the challenge of finding a new strategy or improving a technique which can make this device
2.2.8.4. LIGA process

The LIGA process involves the following steps:\(^4\):

1. A very thick (up to hundreds of microns) resist layer.
2. The resist layer is exposed to collimated UV light or X-ary radiation
3. Then the resist is developed.
4. Metal is electrodeposited onto the primary substrate.
5. The resist is removed or stripped, resulting in a freestanding metal structure.

A schematic diagram of the LIGA process flow is shown in Figure 26 below:

![Fig 26 schematic diagram of the LIGA process](image)

Figure 26 the LIGA process
In the last years the improvement in each single step in this technique has seen big advances. There are many researchers who have succeeded in finding or improving ways to build a 3D structure in micro scale. However, there is still difficulty in producing directly the right 3D structure, which I produced and I will demonstrate that in the next chapter.

Since the LIGA has been in existence, it has opened a big field for researchers to think of a technique to build a 3D structure. Especially in the last few years there has been successful research such as, in 2007 Harutaka et al[86], has fabricated a spiral microcoil with a technique in which the LIGA was improved. There was another technique which improved the LIGA and the stamping technique in 2007[32]. And there are many researches which I cannot enumerate. In the next section I will discuss the comparative study of previous researches.
2.3. Comparative study of previous research

As previously mentioned, multi layer fabrication appeared in micro technology at the beginning of the nineties. Researchers have identified many techniques to perform the LIGA process and each one was able to produce the desired device. At the end of each research in the summary the researcher said about his technique “this technique can be used to produce a device with many layers”. During my research I found a lot of research which used multi layers. So some used two layers, some three, some four and some six and all of them, as the researchers said in their conclusion can be applied for many layers. They have all succeeded in producing the required device. However, I have followed all of these techniques and I cannot apply these techniques for more than three or four layers.

In this section I am going to discuss some of this research and compare it with the proposed technique. I will start with one of the first developments in the LIGA process and then I will follow the history of evolution.

2.3.1. Using UV light instead of X Ray

As mentioned, one of the first pieces of research that used UV light instated of X-ray appeared in 1993. Frazier and Allen presented a paper “Metallic Microstructures Fabricated Using Photosensitive Polyimide Electroplating Moulds”. In this paper an UV light state X-ray was used. Research that was all conducted before in the LIGA processes for the creation mould, a photoresist material (typically PMMA-based) is deposited, patterned (typically using synchrotron radiation X-ray) and electroplated. The researchers used in this paper a process for the fabrication of ‘electroplated microstructures using
photosensitive polyimide. The researchers said: (Using this technology, structures made of a variety of electroplated metals can be fabricated. Vertically integrated structures which exploit the multilayer ability of the polyimides used can also be realized. Finally, processes for surface micromachining using this process for the fabrication of movable electroplated micro actuators can be achieved[87]).

The researchers in this paper used a material (Ciba-Geigy Probimide) as a photosensitive material. The baking process was classified into two stages; the first stage was at 80º for 15 min and the second was at 110º for 20 min. The researchers offered a new technique for researchers to improve the LIGA technology.

In the following pictures I want to demonstrate the steps for both techniques, using the x-ray radiation and the UV light.

Figure 27 The LIGA process utilizes X-ray masks and a synchrotron source[87]
2.3.2. Utilizing three layers

A technique has been developed that uses a multi layer process to build three-dimensional microstructures, such as micro-inductors have conventionally been fabricated by multilayer UV photolithographic patterning of thick resist layers and electroplating. This usually involves a process of seed layer deposition, resist spin coating, UV exposure and electroplating to mould the resist patterns. The process is then repeated, using intermediate seed layers if necessary, to realize the three-dimensional metal microstructure. The final steps involve chemical removal of the resist and plating seed layers. An example of structures made using this approach include a three-dimensional coil with ten windings\textsuperscript{[88]}. 

Figure 28. The process utilizes standard optical masks and a standard UV source\textsuperscript{[87]}
In this paper the researchers built a 3D coil using only three layers of organic resists (AZ4000 series, HOECHST) with a high-aspect-ratio, the height was 60 µm. In addition, one seed layer was deposited in this process. The photo resist was not directly confined between two seed layers. The researchers followed the same technique conducted in the reference [89] for the baking of the photo resist, where the baking temperature must not exceed 130°C. The temperature used in the second layer should be less than that in the first layer, about 15°C[89], they said that this is enough to stop shrinking in the photoresist. However, they did not mention the effect of baking time on the photoresist behaviour through the process. The temperature of the solution in the electroplating process was at 40°C.

The same group of researchers presented other papers[90, 91], they used the same process and they did not use more than three layers to produce a device such us micro channels. In these papers they used the same technique except increasing the temperature of the electroplating to 50°C.

Because of the emergence of some unexpected problems by using multi layer with conventional photo resist, such as those which appeared in my work, researchers tried and are still trying to find a good technique to use the multilayer technique. So I found many of the researches that treated these problems by using different techniques. In the 1996, Konaka et al. proposed a technique “Single- and Multi-layer Electroplated Microaccelerometers”[55]. In this paper the authors demonstrated how a device could be fabricated using multi layer, but they utilized polyimide (Ciba-Geigy Probimid 349) and they fabricated only 2 levels. Their technique is summarized Table 3.
1. The process starts with oxidized (300 nm) wafers as a substrate.

2. A polyimide is spun onto the wafer and soft baked for 10 min at 120º

3. After that the polyimide is cured at 350º for 1 hour in nitrogen

4. A seed layer is deposited

5. A photo-sensitive polyimide “Ciba-Geigy Probimid 349” is then spin on the substrate and soft bake for 30 minutes at 110 degrees C.

6. Transferee imaging using mask aligner to build electroplating molds

7. The photo-sensitive polyimide are then developed

8. Nickel is used as a structural material and is deposited using standard electroplating techniques.

9. To build the upper electrode, a second Ti/Cu/Cr layer is deposited

10. repeated the same steps from point 5

11. When the electroplating in the second level is complete, the photo-sensitive polyimide molds and The copper sacrificial layer are etched

---

**Table 3 utilizing polyimide by using multilayer process**

This paper reports succeeds in producing a device with two levels and proposes this technique be used on other material (Ciba-Geigy Probimid 349) rather than that used in the conventional process. However, the researchers **did**
not mention about the shrinking in the photoresist or the expanding in the seed during the baking process of the photo resist in second level. In this research the researchers used various temperatures (25º, 40º, 50º) during the electroplating process and they said that satisfactory accelerometers could be fabricated using a bath temperature of 25º.

2.3.3. Using dry photo resist

In the same year (1996) another new technique to release a 3D structure was presented by a group in the Swiss Federal Institute of Technology in Switzerland\textsuperscript{92}. In this technique a dry photo resist was utilized as a mould rather than conventional photoresist. In this paper, the authors developed the proposed technique according to Application of the Riston by lamination. The 'Riston' negative working film was introduced in 1970 by DuPont\textsuperscript{93}. The photosensitive film is sandwiched between polyolefine and mylar protective foils. As shown in Figure 29, the polyolefine foil is removed prior to lamination of the resist against the substrate at defined temperature and pressure.

![Figure 29 the Riston technique by lamination\textsuperscript{92}](image)
The researchers used multilevel metal electroplating in a negative dry film resist mould (Riston). The process was implemented in 4 levels. The technique is following, in Figure 30:

1. The substrate is coated with a positive photoresist (2 µm) to form a sacrificial layer and a gold seed layer is evaporated.

2. After removal of its protective polyolefine sheet, the Riston (50µm) is laminated at 110°

3. Then the mylar sheet\textsuperscript{[55]} is removed and the Riston is exposed to UV light.

Figure 30 utilizing dry photo resist by using multilayer process\textsuperscript{[92]}
3. The Riston is then developed. Electroplating of nickel (50µm) is then performed bath at 50°.

4. The mould is dissolved in KOH 1.5 wt.% at 50°.

5. For the creation of multilevel 3D structures, the steps (2) and (3) are repeated.

In this research only two seed layers were used, one is direct on the substrate material and the second is between the level one and two. Using dry photo resist rather than conventional photo resist has many advantages:

1. No liquid handling and spinning and absence of solvents,

2. High, stable over-wafer thickness uniformity,

3. Simple development\textsuperscript{[92]},

However, the thickness is quite high (20-50µm) in this paper\textsuperscript{[92]}, it is difficult to get the thickness smaller than 10 µm and the minimum thickness of dry photo resist is 10µm\textsuperscript{[94]}. In my technique the thickness of the layer may be 0.5 µm.
2.3.4. Effect of soft bake process

Due to the problems encountered in the conventional process, many researchers have tried to change the time and the temperature of baking. In 1998 a paper was presented in which a micro coil was fabricated with the multilayer process\textsuperscript{[95]}. The authors divided the soft bake process into two steps. The first step, soft bake 1, was performed at an intermediate temperature of 85°C for 2 hours in an oven. In the second step, soft bake 2, the photo resist was heat-treated at an elevated temperature of 115°C for 3 minutes on a hotplate\textsuperscript{[95, 96]}.

The researchers fabricated the device only with two levels and the used two seed layers; one is it directly on the substrate layer. The photo resist is not confined between two seed layers. The technique is shown in Figure 31, Figure 32 and Figure 33.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure31.png}
\caption{3D of the micro coil\textsuperscript{[96]}}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure32.png}
\caption{3D multilevel mould of micro coil\textsuperscript{[96]}}
\end{figure}
In this paper the researchers did not mention the shrinking in the first layer of the photoresist during the baking process in the second level.

2.3.5. Using sacrificial metallic mould

Another technique has been developed that used metal moulds rather than conventional photoresist as the sacrificial material to void the unexpected problems by using the conventional photo resist. In 1999, Jun-Bo Yoon et al published a new technique to fabricate a 3D structure. The researchers presented a mould called “Sacrificial Metallic Mould” (SMM)\textsuperscript{[97]}. The researchers mentioned that thermal stability of the lower photo resist layer made the fabrication yield poor. This problem resulted from the thermal reflow of the lower photoresist layer during the second level photo resist baking. These problems cannot be completely avoided by using polyimide. Therefore, the conventional plating through mask technology has inevitable limitations on the number of levels\textsuperscript{[97]}. In this technique one seed layer was deposited in the first level and the metal mould also serves as a seed layer for electroplating the next layer of the structure. The technique is shown in Table 4:
1. First a wafer is passivated with some dielectric material, then electrical contacts are formed, and the first-and-last seed metal is deposited on the entire surface.

2. The bottom electrode (first level metal) is formed by conventional resist patterning and electroplating (Fig. a).

3. The photo resist is moulded with conventional way and it is patterned for the SMM and UV exposed, followed by the electroplating of the SMM (Fig. b).

4. The SMM is formed by electroplating (Fig. c).

5. Another photo resist is moulded for the second level electrode is patterned (Fig. d).

6. During the patterning process, it is developed together with the previously exposed photo resist mould underneath (Fig. e).

7. The post and the second-level electrode are simultaneously formed by single-step electroplating (Fig. f).

Finally, etching the SMMs (Fig. i).

Table 4 the technique of the sacrificial metallic mould\(^\text{[97]}\)

In this paper the researchers have succeeded in solving the problem but they used a long procedure and time. They used many chemical solutions to etch the mould and etch the photo resist. These may cause some effect on the body and the structure of the desired device.
2.3.6. Utilization of a hybrid process

Multi layer three-dimensional structures have developed well in this decade. A technique was developed in 2002 that used a hybrid approach consisting of UV photolithographic patterning of spin-coated resist layers, laser patterning of laminated resist layers and electroplating with intermediate seed layers\(^{[98]}\). The laser is particularly suited for ablation of thick resists built up from multiple laminated layers. The technique is shown in Figure 34:

![Figure 34 a process for fabricating multi level metal structures\(^{[98]}\)](image_url)

Figure 34 shows an outline of the process, which involves repeated application of a basic process cycle comprising:

1. Seed layer deposition;
2. Resist deposition and patterning;
3. Electroplating

Repeated these steps, deposited a seed layer in needs.
In this paper, the researchers report success in fabricating a device produced by using six layers, they did not use all levels with the conventional photoresist, but they fabricated the first three levels and then laminated 50 µm of dry photoresist and then they deposited two levels of conventional resist. This layer of dry resist was treated as an isolator between the first three levels and the following levels. Here only two seed layers were used and the conventional photoresist is not confined directly between two seed layers. The dry photoresist was deposited in the middle of the process and separated the conventional photoresist from each other. They utilized a conventional photoresist for layer thickness less than 20 µm and for the thickness more than 20 µm it was utilized the dry photoresist. In this paper a turbine was produced as shown in Figure 35.

![Figure 35 a turbine produced by using six layers](image)

Here the structure of the turbine helped them to produce it. All the levels were directly over the first level. In each subsequent pattern, the deposited
material had an interface, or in other words, Figure 36, the deposited material in the second pattern was positioned completely over that in the first pattern.

Figure 36 a turbine which was produced with six levels\textsuperscript{98}

This structure helped to spin the photoresist on the deposited material rather than a seed layer. In this paper the researchers did not mention about the time and the temperature that they used in the baking process. Also, the shrinking in the photo resist or the extension in the seed layer or in the structure of the device was not reported.
2.3.7. Study of conventional resist and compared with polyimide resist

Previous researchers studied the behaviour of the photo resist AZ4562 and compared with the polyimide PI2611 by using multi layer to produce a coil\cite{99} in a thesis in 2003. The author said “it was hoped to present various different sample results for the final comparison. However, due to the problems encountered in the AZ4620 process, completed coils were not available to present the samples fabricated in that method”.

The steps of the process are:

1. First level
   - Seed layer deposition
     Shipley 1818, **Soft bake @ 90º for 30 min**
     Patterning, **Hard bake @ 12 º for 30 min**
     Copper etching in copper etchant
     Wafer soaked in heated acetone

2. Second level
   - Deposition of polyimide
   Electroplating

3. Third level
   - Spinning of AZ4620
     **Soft Curing (4min @ 20º + 4min @ 60º + 4min @ 100º + 4min @ 60º + 4min @ 20º)**
     Exposure in specified time and developer
     **Hard bake @ 60º for 15 min**
     Electroplating

4. Forth level
   - Deposition of a seed layer
   Spinning of 1818
     **Soft bake @ 60º for 15 min**
   Patterning
     **Soft bake @ 60º for 15 min**
   Electroplating
In this work, the researcher tried to use the conventional photo resist with multi layer and he tried to change the bake process. As shown in the technique the researcher used soft bake and hard bake. For the soft bake the temperature was increased from 20º to 100º for four min in each temperature. As a result, the researcher said that “due to the problems encountered in the process, completed coils were not available”. Unexpected problems are those which have appeared in my research, such as lips, bubbles or wrinkles. These problems are discussed in chapter 5 of this thesis.

2.3.8. Other techniques to produce 3D structure

MEMS technology has entered to many application fields, such as telecommunication, medicine, and iotechnology. However, in recent years, the technology has been faced with the enormous challenge of promoting industrialization. In particular, the current process for fabricating MEMS parts is considered to be one factor in this issue for the following reasons:

1. Equipment expense is comparatively high.

2. Applicable materials are limited.

3. Production of 3D structural components is difficult\textsuperscript{[100]}. 

For a solution to these problems, the researchers have improved and found new techniques to produce sub-micrometer-scaled parts. In the next section I attempt to give an overview of some of this improvement.
2.3.8.1 Hot embossing

Because of the problems that I mentioned above, many researchers avoided the technique (using the conventional photoresist in multi layers process) to produce a 3D structure and tried to develop the LIGA process without this technique. The techniques proposed in [101] involved applying hot embossing on micro moulding to manufacture directly a three-dimensional form. Figure 37 shows one such technique.

![Figure 37 principal process steps of hot embossing](image)

The researchers report successfully producing a device with 3D structure. However, they used **only one layer and didn’t follow the conventional way to build the structure**. The researchers produced this technique to avoid the problems in the conventional technique.
2.3.8.2. 3D Direct-Writing

Another technique tried to develop the process of the micro moulding which appeared in 2004. The authors called the technique 3D Direct-Write\textsuperscript{[37]}. The procedure of this technique is described in [37]. In this paper the laser patterning process was used, Figure 38.

![Figure 38 3D Direct-Write\textsuperscript{[37]}](image)

The researchers did not use the conventional process and they proposed a new technique because of many problems and they reported in the paper that “Extending the number of layers, in a surface micro-machining process from four to five layers (extending the height of the machine from 10 μ to 12 μ) could take months or years due to the impact of thermal cycles during deposition of the fifth layer on the residual stresses in Layers 1-4, and the corresponding effect of these residual stresses on Layer 5.” \textsuperscript{[37]} These problems that the researcher mentioned in his paper were met in my research as I will discuss and treat in chapter 5.
2.3.8.3. Stereo lithography

As well another researcher pursued another way to improve the LIGA process or the micro mould process. As mentioned in the previous chapter that Stereo Lithography technique appeared in micro scale in 1993\cite{27} and the authors mentioned that the LIGA cannot create a curved surface. In 2005 a technique was proposed, which improved the performance the stereo lithography using digital micro-mirror dynamic mask\cite{44}. The procedure of this technique is described in [44], Figure 39.

![Schematic diagram of projection micro-stereo lithography](image)

Figure 39 Schematic diagram of projection micro-stereo lithography\cite{44}

In 2009 another research appeared which improved the stereo lithography using a dynamic mask\cite{102} Both of these researches have used the technique in the paper in 1993, and the researchers have only improved the steps of this technique. The technique has succeeded in producing a true 3D structure. However, this technique needs special equipment and it is difficult to produce high precision structures\cite{103}. All of the papers in micro stereo lithography mentioned that the conventional process used to make micro moulds, has many problems to build a curved surface in the axial direction of the required mould. In my work I
have proposed a technique in which a curved surface in the axial direction can be fabricated and I have explained the procedure in chapter 3.

### 2.3.8.4. UV light variables

1. A technique is proposed to directly produce 3D structures. This technique depends on controlling the intensity and beam directions of exposing light. Some researchers used gray-scale lithography\textsuperscript{[104]} as in Figure 40 and the procedure of this technique is described in the paper [104], same used oblique beam light such as in [84], Figure 41 and same used a moveable substrate.

Figure 40: The primary steps in gray-scale technology\textsuperscript{[104]}

Figure 41: Fabricated structures with inclined exposure\textsuperscript{[84]}

78
2. Another technique appeared in 2007 as research appeared and proposed a technique in which the 3D-LIGA was achieved by the technical development of 3D X-ray lithography and warm injection moulding replication technology with a unscrewing release mechanism[86]. The technique is shown in Figure 42.

![Figure 42 process of 3D X-LIAG lithography with unscrewing][86]

The researchers succeeded in producing a device such as spiral micro coil. And they did not use the conventional process in this paper, but with this technique I can produce only symmetric devices
2.4. Conclusion

The short introduction in the first section gives the reader a good knowledge about this technique that I will improve its performance to directly produce a device with a 3D structure which I think will help mankind in our lives.

In the section 2, I have explained the most common techniques “the conventional techniques” used in the MEMS technology and I have focused on the technique that I used and improved such as the LIGA process and the electroplating process.

In the section 3, I have presented some previous research, in which researchers have reported attempts to use conventional material in a multi layer process to produce a 3D structure. These researchers have succeeded in promoting the LIGA process and I have seen that many researchers have succeeded in producing a 3D structure like micro turbine in [98] or a micro coil in [88, 89, 96]. They have all succeeded in producing the required device. And all of the researchers confirmed in his conclusion that I can apply the technique for many layers. I will however be able to demonstrate in the following chapters, that it is not possible to realise 3 or 4 layers using the techniques described in this literature review. In addition, many researchers showed the conventional process has many problems to fabricate a 3D structure, especially by using multi layers as stated in [37, 97, 99].

Some researchers applied with the conventional resist by using multi layer an assistant substance to avoid these problems, such as in [98] the researchers used a dry photoresist in the fourth level. This dry photoresist deposited is treated
as a separator between the conventional photoresists. In [97] the researchers used a sacrificial metallic mould to overcome the problems in the conventional resist.

In [95, 96] the researchers tried to solve these problems by using variable steps in the baking process. They succeeded in producing a 3D device by using 3 layers, but in the technique one seed layer was used. In [99] the baking temperature fluctuated between 20º, 60º, 100º, 60º and 20º for eliminating the problems associated for the conventional process. In spite of that, these problems appeared during the fabrication of the demanded device.

In [27, 37, 44, 86, 101, 102, 105] all researchers confirmed that the conventional process has many unaccepted problems during the operation. Because of these problems, all of the researchers proposed many new techniques to fabricate a 3D structure.

All the previous problems have appeared in my work as well, such as lips, bubbles and wrinkles. However, after many experiments, I found a good solution to treat these problems which I will discuss in chapter 5.
Chapter 3

Concept design of process
3.1. Introduction

Because of the growing demand for MEMs devices, especially after the big success of projects in different fields such as in medicine or in the manufacture of aviation, this has encouraged the researchers to innovate or improve a technique which is effective and cheap and not complex in fabrication, to be utilized in MEMs technology. As I mentioned in the previous chapter, the manufacturing process in this technology unfortunately is not the same process that I know from traditional fabrication techniques such as milling, drilling, turning, forging and casting. The LIGA technology is introduced here because it is very useful for the construction of a 3D structure. It has succeeded in producing a high aspect ratio structure device, but in this work I attempt to obtain a high width to thickness ratio of the layer. In other words, I use a hybrid technique which consists of LIGA and thin film technique. To make a 3D structure, there has to be a thin film deposited and then applied to the LIGA process one layer on top of the other. The whole structure can then be stacked together by using multi layers to meet the requirement and in this chapter I will show a guide line and demonstrate how a device with a curve could be produced by using this proposed technique.
3.2. Objective

The LIGA process uses a mask with the shape of the desired structure device to allow 2D information to be transferred onto a resist material, Figure 43.

![Figure 43. Transfer of the 2D structure](image)

It becomes possible to fabricate a mould with the inverse shape of the desired structure. The 3D structure depends on the thickness of the resist material, as illustrated in Figure 44.

![Figure 44. The XYZ planes](image)
Chapter 3

Concept design of process

The LIGA process has had good success in providing a way to produce devices with a high aspect ratio structure [106], but it is not possible to make any structure in the ZX plane or in the ZY plane, as shown in Figure 44. For example, it would not be possible to create features like a curved surface in the Z direction. LIGA uses the shadow of a mask and can only transfer a 2D structure. In other words, a device made with LIGA can only be drawn on the XY plane, as illustrated in Figure 43. The LIGA process has been developed to create 2.5D structures [107]. For this reason researchers have tried, and continue to try, to find new ways to improve the capability of the LIGA technology.

Micro mould or LIGA has undergone significant progress during the last decade using many different techniques to fabricate 3D structures; a notable example of this is the fabrication of a Spiral Micro coil [88, 89, 96]. However, difficulties remain in terms of fabrication of a curved surface or the creation of a structure on a curved surface with LIGA process [37]. In other words, it is not yet possible to build a structure on the XZ plane or on the YZ plane, Figure 44. In reference to the previous researches, to produce a curved surface in Z, Figure 45 direction using multi layers from conventional photo resist, many expected problems and difficulties may appear during the operation.

Figure 45 the multi layer planes
The main objective of this work is determining how the fabrication of 3D complex structures and their high-volume reproduction can be improved. Most previous projects have tried to obtain a layer with high-aspect-ratio using the conventional process, but in this work I try to obtain a high width to thickness ratio of the layer. This work presents a novel idea to improve the LIGA process when using many masks to deposit multi thin layer over each other Figure 45. Moreover, this technique can be utilized to produce a curved surface in the Z direction with any diameter as shown in Figure 45. Practically, a 2 µm thickness of layer is applied in the proposed technique. However, a layer of 0.5 µm or less can be deposited.

With my proposed technique, I have saved time and cost if I compared my technique with any other. For example, if I need to make a device involving a curvature surface with a 50µm radius and the proposed technique divided the mould to 10 layers. Each layer needs about two and half hours and plus two hours for dehydration to be completed. To complete the device with 10 layers I need a total of 45 hours, whereas one layer in another technique, such as stereo lithographic, will need one week.
3.3. Steps of the method

The proposed process for producing a multilayer device employs for example Auto CAD software to design the completed design. The design can then be divided into stacked 2D layers, the number of layers being determined by the complexity of the device. Each of these 2D patterns can then used to produce a photolithography mask. The following steps could then be employed to fabricate the 3D device:

To demonstrate this method a sample device was made in Figure 46, with a relatively complex 3D structure.

The mould was drawn as “the inverse of the demo-device1” using Auto CAD software. The drawn mould was divided into nine layers, determined by the complexity of the device in Figure 47. The drawings of those layers are in Appendix A.1.
After thorough cleaning, the substrate is coated in a thin layer (e.g. 50 nm) of a conductive material such as gold on the upper surface, Figure 48. This layer will act as a seed layer for the electroplating process. To ensure good adhesion between the sample and the seed layer, a layer of 50 nm of titanium was deposited.

3.3.1. Preparing the photo resist

- A layer of resist material (e.g. AZ4562 or S1800) is spin coated on top of the seed layer. The resist dilution and spin speed are controlled to ensure the resulting film is the same thickness as the corresponding 2D layer of the demo-device1.

- To solidify the photoresist, both the sample and the resist are placed in an oven or on a hotplate, Figure 49.

3.3.2. Exposing the photo resist using UV light

In this step the first mask, which has the same shape as the first layer of the demo-device1 mould Figure 50, is held and installed in a machine. This machine
can expose the photo resist with UV light, Figure 51. The exposure time has been shown to depend on the thickness of the layer.

Figure 50 the first mask

Figure 51 exposing the photo resist with UV light

3.3.3. Developing the resist

Due to the UV light, the properties of the photo resist are changed. The sample is then immersed in a chemical solution. That solution removes the material that was exposed in the UV light and leaves holes in the sample, Figure 52, that represent the mould of the demo-device1.

Figure 52 the developed photo resist (the mould)
3.3.4. Depositing the desired material in the mould

Electroplating is used to deposit the desirable material in the mould (e.g. gold, nickel), ensuring the deposited material is the same thickness as the resist mould of the demo-device1, Figure 53.

At this step the first level is completed, and the same process is used for the second level as in the first level. A 50 nm gold and 50 nm titanium seed layer is deposited over the first level as shown in Figure 54. This layer covers all of the first level. The gold layer has two functions: one is to protect the first layer of the photo resist from the UV light and the second is to help the electrical connection. The titanium layer is used to ensure strong adhesion between the gold layer and the photo resist.

At this point the same steps previously done in the first level are repeated using the next mask but with a different thickness of resist. These steps will be repeated for all nine masks. When all the steps are finished the demo-device1 is
complete, ‘the electroplated 3D structure’ the demo-device1 requires releasing from the encapsulating photoresist and seed layers mould, Figure 55. The demo-device1 must be cleaned from the photo resist and the seed layers.

3.3.5. Cleaning process

To clean the demo-device1 from the resist and the seeds layers I should follow these steps. I start by exposing the ninth level to UV light “Flood-Expose” (i.e. without a mask), Figure 56. This layer can then be removed using resist developer. This reveals the top most seed layer.

Once the resist layer has been removed the demo-device1 starts to appear, Figure 57. The upper layer now is the last seed layer; to clean it the sample should first be immersed in a chemical solution (e.g. potassium iodide/Iodine for gold) and then be immersed in another chemical solution HF (hydrofluoric acid) to etch this seed layer. In this step the seed layer is removed; the next level is shown in Figure 58.
The steps “exposing UV light and chemical etching” should be repeated for all levels. To clean the inside surface the sample should be immersed in a warm bath of acetone.
3.4. Conclusion

The multi layer process has existed since 1993 and has made good progress during recent years. Most of the research has reported that the use of the conventional technique has many problems and they have used different techniques and they have succeeded in building a 3D structure. However, the technique that they followed cannot be used for producing any other devices. In other words, all researchers have presented a new technique or improved other techniques just for special devices that they want to build. But if I followed their technique, many problems will be met in the fabrication stage. In Chapter 2, in section 3 “comparative study of previous research” I have discussed these matters more widely.

By using my technique I overcame the problems that all researchers have met or mentioned in their research, which I will discuss in chapter 5 “. In my technique I have improved the conventional technique and this improvement has made the technique cheap and efficient compared with any provirus techniques. With my technique any structure could be made using any material.

In this chapter I put in the hands of the reader the steps of the proposed technique and what I have attempted to achieve. In the next chapter I will explain the proposed technique by using it to produce some devices that have quite a complex 3D structure.
Chapter 4

Fabrications and Experiments
4.1. Introduction

This technique, as was explained, depends on the division of the mould into many layers that represent the masks made during fabrication. This means that many masks will be used, and the fabrication of a mask is very expensive. My intention at this point is simply to demonstrate the technique. To that end, I started by designing some devices that can be made with just two masks and with repeated use of the two masks during the fabrication steps, the desired device will be built. In this chapter I am going to explain in detail how a device can be built by repeating two masks. When a successful product has been produced through this procedure, I have applied this technique using multi masks to fabricate a relatively complex shaped-device1.

![Figure 60 demo-device2](image)

I started with the simplified devices in Figure 60 (demo-device2), which generally have dimensions of 110x360x28 µm. I first drew the moulds of the devices and divided each one of them into five layers. Layers 1, 3 and 5 have the same shape and I can use the same mask for them. Layers 2 and 4 have the same shape and I can use the same mask for them. For the demo-device1 in Figure 46, the general dimensions are 750 µm length and 625 µm width and has nine levels. The drawings of these layers are in Appendix A.1, A1.1.
Firstly the masks for the demo-devices were designed using lithographic CAD software “L-Edit”. I drew special marks in all masks “align marks” and then produced using E-beam lithography on commercially prepared mask plates. When I was able to produce the demo-device2, I designed the nine masks for the demo-device1.

As mentioned, the thickness of the photoresist layers differs from level to level. In this method the key material to making the 3D structure is the photoresist and since it originally uses liquid material it has to be dehydrated as much as solid material. The photoresist thickness for each layer is shown in Table 5 for the demo-device 2:

<table>
<thead>
<tr>
<th>Level</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness in µm</td>
<td>2</td>
<td>11</td>
<td>2</td>
<td>11</td>
<td>2</td>
</tr>
</tbody>
</table>

Table 5 the photoresist thicknesses for demo-device2

For the demo-device1, the photoresist thickness for each layer is:

<table>
<thead>
<tr>
<th>Level</th>
<th>1&amp;9</th>
<th>2&amp;8</th>
<th>3&amp;7</th>
<th>4&amp;6</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness in µm</td>
<td>5</td>
<td>1</td>
<td>2</td>
<td>1</td>
<td>4</td>
</tr>
</tbody>
</table>

Table 6 the photoresist thicknesses for demo-device1
I used the more common materials here in JWNC (James Watt Nanofabrication Centre), which are AZ4567\(^{[108]}\) and S1800\(^{[109]}\), a wide range of data exists on these resists\(^{[67, 95, 96, 108, 109]}\), however it is often contradictory. To test the thicknesses obtainable with these resists I conducted some basic experiments. The resist film thickness was measured as a function of spin speed, as shown in Table 7.

<table>
<thead>
<tr>
<th>spin speed [rpm]</th>
<th>1000</th>
<th>2000</th>
<th>3000</th>
<th>4000</th>
<th>6000</th>
</tr>
</thead>
<tbody>
<tr>
<td>AZ 4562 [µm]</td>
<td>11.5</td>
<td>9.3</td>
<td>7.5</td>
<td>6.5</td>
<td>5.3</td>
</tr>
<tr>
<td>S1818 [µm]</td>
<td>3.9</td>
<td>2.7</td>
<td>2.1</td>
<td>1.7</td>
<td>1.5</td>
</tr>
</tbody>
</table>

Table 7 the thickness of az4562 and S1818 layers

These demo-devices were built on a silicon wafer. I ordered 4 inch wafer of silicon and then I cut it in many samples. Each sample for demo-device2 has square area of “2.5x2.5 cm\(^2\)”. A hundred of moulds of the demo-device2 were built on it. Each mould has 110 µm length and 360 µm width.

For the demo-device1, I cut the silicon wafer into many samples. Each sample has square area of “2x2 cm\(^2\)”. A hundred moulds of demo-device1 were built on it. Each mould has 750 µm length and 625 µm width and the space between the moulds is 50 µm. The template has length 7950 µm and width 6700 µm and with the align marks has 1.5x1.4 cm\(^2\).

Before starting the fabrication of these demo-devices, the sample, which the demo-devices will be built on, should be cleaned. In this technology there is a
special way to clean the samples. The sample should be immersed in an ultrasound bath for 5 min:

In Acetone \([\text{(CH}_3\text{)}_2\text{CO}]\)

In Isopropanol \([\text{(CH}_3\text{)}_2\text{CH OH}]\),

In Methanol \([\text{CH}_3\text{ OH}]\).

The sample was then dehydrated for 10 min at 200°C on a hotplate or at 90°C in an oven for more than 30 min.
4.2. The first level

A seed layer of 50 nm titanium and 50 nm of gold were evaporated over the entire sample surface. The gold layer helps the electroplating process to connect electricity and to make good adhesion between the sample and the gold layer where a layer of titanium is deposited. For the demo-device2 I used S1818 photo resist in the first level. Related to Table 5, this photo resist was spun at a speed of 3000 rpm to obtain a thickness of 2.1 µm (Table 7), it was then left at room temperature for 15 minutes\textsuperscript{[108]}. It was then baked on a hotplate at 90ºC for 30 minutes (or an oven at 90ºC for 75 minutes), to allow the majority of the solvent to evaporate without disrupting the photoresist layer. At this level the time and the temperature for baking are not important; it only needs sufficient time to solidify the photo resist. However if a bake temperature greater than 120ºC was used for longer than 30 minutes difficulties were encountered during development. For the demo-device1, the photoresist for this level was AZ4562 and the thickness was 5.3 µm (Table 7) and I followed the same steps.

Following this, the sample for demo-device2 was left at room temperature for 30 minutes before UV exposure. In the exposure process the first mask was held and installed in the machine over the sample in a machine called “Ma6 (SUSS Mask Aligner MA6\textsuperscript{[110]}).” In this machine the photo resist can be exposed with UV light. The exposure was carried out using 365 nm wavelength light, at an intensity of 7.2 mW for 15s and before being left at room temperature for a further 30 minutes. It has been shown that this ‘rest’ is required to allow evolution of N\textsubscript{2} (a product of resist exposure) from the resist layer without disrupting the film. The sample was developed for 40 seconds using developer (Microposit concentrated, from the CHESTECH company) diluted 1:1 with water. For the demo-device1, it was the same process just the exposure time was 20 s
and the AZ developer (400K developer, from the GB Microchemicals company) diluted 1:4 with water for 2 minutes. Figure 61 and Figure 62 shows a typical optical micrograph of the developed resist at this level.

Figure 61 the first layer of the demo-device2 after developing

Figure 62 the first layer of the demo-device1 after developing
4.2.1. Electroplating

As mentioned above, to deposit the gold I used an electroplating process to build the devices. The facility in the JWNC was limited and there were many difficulties which appeared in my work during this experiment which will be discussed in the next chapter. For example, estimating the deposition rate and controlling the temperature of the electroplating solution. However, here I will explain the proposed technique that I found to control the deposition rate of the gold. The technique used here did not result in any damage to the photoresist. The technique was executed by using the instruments shown in Figure 63.

Figure 63 electroplating equipment in JWNC
4.2.1.1. Electroplating experiment

As shown in Figure 64, the experiment consists of a Thermostatically Controlled Water Bath a power supply, a thermometer, a jug of electroplating solution, an anode which was covered in gold, a cathode which consists of two parts; a holder and a sample and a PVC Stirrer.

The cathode consists of a holder made from a non-conductor of electricity and installed four access points for the electrical conductivity. These four points are connected to the power supply in the negative output. The sample was installed over this holder in these four points. To stake the sample over the holder I used wax. The holder was heated and I put then the wax over it between the four points. When the wax melts, I installed the sample over it and left the holder at room temperature, Figure 65.
The areas in the sample, that are beside the four points, should be cleaned from the photoresist and opened a field in the photoresist on the seed layer, but this process must be carried out very carefully to avoid any damage in the mould. Then these four points were connected with an area in the sample by using an Electrodag 1415 (conductive paste).

The electroplating solution consisted of; Gold Sulphite, Sodium Sulphite, Tri-Potassium Citrate and Arsenic compound. This solution was provided by the company Enthone (UK) Ltd[111].

I took 80 ml of this solution and poured it into a jug. This jug was then put in the water bath. After that the thermostatical control was switched on and set at 28°C. When the temperature of the electroplating solution reached the required temperature, I then put the anode and the cathode into the jug. Then I set up the PVC Stirrer at 100 rpm. The sample was connected to the power supply in the negative output and the anode was connected to the positive output.
The value of plating current that was needed in the electrical circuit was dependant on the area that I wanted to be deposited and can be counted using this form.

\[
\text{Plating Current (mA)} = \text{area mm}^2 \times 0.013^{[54]}
\]

The area in the sample which will be deposited is not the area of the demo-device, but includes the aligned market area and the area which I have cleaned from the photoresist and the four points in the holder and the area of the (conductive paste) area which connected the seed layer with four points.

The area in the sample was 3.54 mm\(^2\). This is the area of the mould of the demo-device2 in the first layer. And the area in the demo-device1 was 178.92 mm\(^2\). The other areas were difficult to count, especially the area of the conductive paste. All of the other areas were estimated approximately. For the demo-device2 I have used 4 mA as value for the plating current and for the demo-device1 6 mA.

When the current was selected, I set the power supply with the selected value and switched it on. After many experiments it was found that the time to deposit 2.1 \(\mu\text{m}\) with these conditions was 8 minutes and 20 seconds. I met many problems when I was doing these experiments and I will discuss the difficulty in the next chapter.
4.3. The second level

Prior to starting the second level, the sample was cleaned with water and blow-dried in air before being left at room temperature for two hours. The sample was then placed on a hotplate for 30 minutes at 50°C to remove any remaining water before being left at room temperature. To start building the second level a seed layer of titanium of 50 nm and 50 nm of gold was deposited over the entire sample surface Figure 66.

Figure 66 the second layer of the demo-device2 after the deposition of the seed layer
I then spun AZ4562 at 1500 rpm to obtain nearly 11 µm. The sample was then baked on a hotplate at 50ºC for 45 minutes. After that I brought the second mask, which has the same shape of the second layer that was divided from the demo-device2 mould and aligned it to the first level and exposed the sample for 20s with the same illumination source as used in the first level. The exposed AZ4562 was developed in AZ developer (400K developer, from the GB Microchemicals company) diluted 1:4 with water for 2.5 minutes (Figure 67). The thickness was nearly 11 µm.

Figure 67 the second layer of the demo-device2 after developing
Gold electroplating was used with the same conditions as described previously with the alteration that 45 minutes were required to deposit 11 µm.

4.4. Third and subsequent levels

For the third and fifth levels I repeated the same steps as used for the first level and I used the first mask and the same photo resist and thickness. However, I found that the time and temperature for baking are key parameters, which caused many defects, such as bubbles and wrinkles. This issue will be discussed in the next chapter. These defects required us to do some experiments to find the appropriate time and temperature for these levels. I found that the appropriate temperature and time were at 50°C for 45 minutes. In the fourth level I followed the same steps as the second level, using the same mask, the same thickness and the same photo resist.

Prior to starting any level, the sample should be cleaned with water and blown dried in air and left at room temperature for two hours. After that I place the sample on a hotplate for 30 minutes at 50 °C.

4.5. Cleaning process

After completion of the fifth level, the 3D structure requires releasing from the encapsulating photoresist and seed layer mould. Firstly: -

- I exposed the top level of photoresist to UV light using a “Flood-Exposure”. The layer can then be removed using photoresist developer. This reveals the topmost seed layer.

- The seed layer was removed by using two steps
  1. The sample immersed in a gold etching solution to just in 5 seconds to etch the 50 nm of the gold
2. The second step is that, the sample then was immersed in another chemical solution, “HF”, to etch the 50 nm of titanium.

These steps are repeated for each layer in this order, with a final clean being achieved by immersing the sample in warm acetone.

After removal of the resist and seed layers the completed devices were inspected using Scanning Electron Microscopy (SEM) Figure 68 and Figure 69. In these figure the demo-devices appeared with a good surface and the structure of it is completely made with the feature that I wanted.
Figure 68 demo-device2 after fabrication 1
Figure 69 demo-device2 after fabrication 2
4.6. Conclusion

In this chapter I have shown that I was able to produce a device with five layers successfully and I overcame all of the problems that appeared during this manufacturing process and that many researchers experienced when using the multi layering technique and I will discuss that in the next chapter. This technique was then applied to produce the device in Figure 46. In this device, I want to show how a device with a curved surface in Z direction can be produced Figure 70.

![Diagram of a device with curved surface](image)

Figure 70 demonstration of making a device with curve surface

The next pictures in Figure 71, Figure 72 are for this device and was inspected using Scanning Electron Microscopy (SEM). More pictures are attached in A2

This device was built with six layers and did not complete the remaining three layers because the device is symmetric. As shown in the pictures the devices have good surfaces and are without any defects. More pictures are attached in A3
Figure 71 demo-device1 after fabrication (1)

Figure 72 demo-device1 after fabrication (2)
Chapter 5

Results and Discussion
5.1. Introduction

As mentioned in the previous chapters, in this technique the key material is the resist material, and it should be kept in the sample until all steps are completed and should be of acceptable properties;

1. While this technique uses multi levels the photoresist should have good transparency to align the levels.

2. To make the 3D structure, the photoresist in the first level will support the second level. In other words, the advance level is built over the previous level and since the photoresist used is originally a liquid material, to obtain the required thickness, it should be able to solidify.

3. When all steps are completed the produced device is ready, but it is still covered and surrounded by the photoresist mould and should pick the device out of the photoresist mould. The technique can be done only when the photoresist has the dissolvable property.

I searched in JWNC (James Watt Nanofabrication Centre) for a photoresist which has good properties and will be stable for this technique. The more common materials identified in JWNC to use are:

1. The negative photo resist SU 8\textsuperscript{[112]} (Organic Resin Solution)\textsuperscript{[113]}
2. The positive photoresist AZ4562 (methoxy-propyl acetate (PGMEA))\textsuperscript{[108]}
3. The positive photoresist S1800\textsuperscript{[109, 114]}
4. The positive photoresist polyimide\textsuperscript{[115]}
I researched the properties of these materials. The information was collected from a diverse range of sources \cite{67, 95, 96, 108, 109, 112, 115} and from the experienced people here in JWNC. Every researcher based his technique on the shape produced, and what he wants to make. However, it is often contradictory. During the study there was conflicting information and at times I did not know which to follow. I decided to run experiments to collect information about these materials and discover whether they were suitable for these techniques or not. The experiments had two goals: to construct these demo-devices and to discover the best material for this technique. As a result of these experiments I found that:

1. All of these photoresist are transparent. It can be aligned to a level with previous level by using any of these photoresists.

2. The polyimide is one commended form of photoresist but its preparation to use takes a long time compared with other type of photoresist. The baking time is too long and the bake temperature is very high and up to 300°C. To transfer the image from the mask to the Polyimide it needs another type of photoresist\cite{116, 117}. The processes with Polyimide take a long time.

Figure 73 removing of the SU 8
3. The SU-8 is a negative, epoxy-type, near-UV photoresist based on EPON SU-8 epoxy resin (from Shell Chemical)\textsuperscript{[83]} that has been originally developed, and patented (US Patent No. 4882245 (1989) by IBM. The photoresist SU 8 has a problem with dissolving after the baking process. After some experiments and investigation I found that the photoresist SU 8 can be removed, Figure 73, by using a complex method\textsuperscript{[112]} but not dissolved, as in Figure 73 or in A4. The proposed technique in this work depends on the used resist material to be soluble and not only removable. In other words, the resist material must be dissolved at the end of the procedure. More than that, the temperature for baking SU 8 is very high, at up to 200ºC\textsuperscript{[83]}.

4. The most common material here in JWNC or in the manufacturing to use is the photoresist AZ4567 and S1800. I researched the properties of these materials. Both of them have good properties dependant on the bake temperature, they can be easily dissolved and the bake temperature is variable, it can be from 50ºC to 113ºC. From the previous review, as mentioned, the information was collected and there was a big difference between them and I could not know which information I should follow. Then I decided to do some experiments to do the demo- devices only with these both photoresists.

When fabricating the demo-device I found that precise process control was required in order to ensure the device was produced without defects. Some parameters were found to be extremely sensitive to variation and could result in failed fabrication. Specifically, the time and temperature of the soft bake\textsuperscript{[105]} . The dehydration time and the temperature of the electroplating solution were all critical. In this chapter, I will discuss the individual difficulties and problems that appeared.
5.2. Problems in the first level

When I started doing the fabrication, the steps that I followed were taken from the supplier. For the photoresist AZ4562 the bake temperature was 90°C for 10 minutes on a hotplate\textsuperscript{[108]} and for the photoresist S1818 the bake temperature was 115°C for 60 seconds on a hotplate\textsuperscript{[109]} . Then the photoresist was developed. The surface of the photoresist was flat as in

Figure 74 and in Appendix A.5. This measurement was done with a machine called “Tektak”.

![Figure 74](image)

In the picture the horizontal axis in micro meter (\(\mu m\)) and the vertical axis in angstroms (\(Å\)) 1 micrometer = 10 000 angstroms (1 \(\mu m = 10 000 \ Å\)).
After the electroplating process I found that the edges formed a lip, Figure 75 and in Appendix A.6. These lips appeared when I hard-baked the photo resist and cleaned the surface with dry etching as well.

![Rise corner “lips”](image)

**Figure 75** the photoresist after electroplating

After considering previous research I didn’t find any research that treated this problem although all the researchers mentioned this problem in general, like in the thesis from Jing Sun[99] or in a paper which was published by Jun-Bo in IEEE[97]. And because of it most researchers have stopped using the conventional
technique and used other techniques to solve these problems. They have succeeded in producing the required device with multi layers. However, they used different techniques and they did not mention that they have attempted to treat these problems. In chapter 2 in section 3 “Comparative study of previous researches” I have discussed some researches and compared them with my research.

Firstly, in this level I carried out for the photoresist two steps only; the bake process and the electroplating process. I hypothesized that the electroplating solution may influence the photo resist or that the temperature of the solution was too high (it was between 50°C and 60°C), or perhaps the baking time or temperature was not sufficient.

The Thermal treatment time and temperature are difficult parameters to optimize. In fact, if the treatment is too short or too mild, the resist will not be hard enough and it will flow on the substrate surface. On the other hand, if the thermal treatment is too long or the temperature is too high, the photoresist will not be sensitive to exposure\textsuperscript{[105]}.

When I started fabricating demo-devices, I identified many techniques for baking. Some use soft baking and some use pre- and hard-baking. Soft baking consists of just one bake after spinning the photo resist and before the UV exposure. Pre- and hard-baking involves a bake after spinning and another bake after development. There are many methods of soft baking. The manufacturer of AZ 4500 series says that the bake temperatures should not exceed 90°C\textsuperscript{[108]} and for SI 8100 series 115°C\textsuperscript{[109]}. The temperature is not the only parameter here; the time is as well. Some leave the sample in an oven for 20 minutes\textsuperscript{[118]} at 100°C, some for 30 minutes at 120°C\textsuperscript{[67]}, some for 60 minutes and some divided the soft
bake process in two steps. The first step, soft bake 1 was performed at an intermediate temperature of 85°C for 2 hours in an oven. In the second step, soft bake 2, the photo resist was heat-treated at an elevated temperature of 115°C for 3 minutes on a hotplate\cite{95, 96}. Similar to the soft bake, the pre- and hard-bake uses many possible methods.

I first tried to change the temperature of the electroplating solution but the manufacturer recommended I follow the technical data sheet \cite{54, 111} in which it states that the temperature should be around 50°C. I then thought that the baking time was insufficient and that could cause the rising up of the edges; I could change this easily. When I started making the demo-devices, I followed the manufacturer’s technique, which says not to go over 90°C and to soft bake the photo resist for 20 minutes. I then ran some experiments in which I used different times and temperatures for soft baking, to determine appropriate values.

In these experiments I used different temperatures and different times. I tried 90°C, 120°C, 140°C and 160°C for 30 minutes. I found that with a temperature greater than 120°C for 30 minutes, I had difficulties in developing the photo resist. I stayed then with 90°C and tried different lengths of times.

I did another experiment. In this experiment I fixed the bake temperature at 90°C and I changed the time. The bake time was in four steps at one hour, one hour and fifteen minutes, one hour and thirty minutes and one hour and forty-five minutes. The baking was in an oven. After that I ran the electroplating deposition to deposit the cold for all four samples and I left the samples in the electrosolution for the same time, 2 hours. I discovered that the longer I left the sample in the oven, the more the lips decreased.
The sample that had been baked at 90ºC in the oven for one hour had lips with a height of more than 400 nm, Figure 76. When the sample was left in the oven for a long time its height was reduced or disappeared, Figure 79. I found that the sufficient soft bake time for the photoresist “AZ4562 and S1818” is one hour to an hour and thirty minutes, and the sample should be in an oven at 90ºC. In the pictures below I can see the reduction of the lips; height, depending on how long they spent in the oven. The original pictures are attached in Appendix A.7. In the picture the horizontal axis in micro meter (μm) and the vertical axis in angstroms (Å).
Figure 76. The lips after one hour in the oven

Figure 77. The lips after one hour and fifteen minutes in the oven
Figure 78 the lips after one hour and thirty minutes in the oven

Figure 79 the lips after one hour and forty-five minutes in the oven
As a result, I observed that leaving the sample in the oven for one hour and forty-five minutes at 90°C had the best results for soft baking at this level. However, this temperature and time period caused problems in the second level, which will be discussed in the next section.
5.3 Electroplating process

As mentioned, the facility in the JWNC was limited to the electroplating process. I used the equipment in it, but I met a problem in how I could control the temperature in a water bath, and after a delay of more than four months, from December 2008 to April 2009, I had new equipment in which the temperature was controlled.

When I started the experiment the condition of the experiment was the same that all researchers followed in this laboratory and the same way that supplier of the electroplating solution recommended following. The recommend temperature was between $50^\circ$C and $60^\circ$C\textsuperscript{[111]}. After the first experiment I found the surface of the photoresist had some deformation that I called “lips”. This problem was solved by increasing the time of the baking. But when I baked the photoresist in the second level or subsequent levels with this technique many problems appeared, such as bubbles or wrinkling surfaces. Thus I decided to conduct some experiments to find out if the electroplating solution affects on the photoresist or the temperature is the cause of the deformation. The first experiment consisted of placing the sample in the electroplating solution at room temperature for two hours without doing the electroplating process. The result was that the surface of photoresist id not changes. That means the electroplating solution did not affect the photoresist at the room temperature. Then the sample was placed in the electroplating solution at $50^\circ$C for two hours without doing the electroplating process. I found that the surface of the photoresist was deformed. After that I tried to reduce the temperature of the solution. After a lot of research and many experiments I found many researchers use low\textsuperscript{[55, 56]} temperatures and some of them recommend not going above $35^\circ$C and said the high temperature served only to increase the deposition rate on the cathode. I did the experiment at
28°C and the surface of the photoresist was not deformed and did not have any wrinkling.

Another problem appeared at this step in the work. I found a problem in controlling the deposition ratio. When I conducted the experiments I found that the solution lost some of its properties. I found the colour of the solution after using became dark and the quantity became small. The deposited ratio in the solution was decreased every time when I used the same solution Figure 80. In this figure I can see the ratio of the deposition by using a new solution is high and when I used the same solution again the ratio decreased. In other words, the ratio of the deposition in the electroplating solution is dependent on the age of the solution.

![Figure 80 the deposited ratio](image-url)
The area that I wanted to deposit was very small and it was in the demo-device1 178.9275 mm$^2$ and in the demo-device2 was 3.5466 mm$^2$. However, the solution quickly lost its properties and the deposited rate was very low. The solution is very expensive. And using a new solution for each single process was not available. I investigated why the solution is quickly becoming old and losing a lot of its properties, although I used a very small area. When I started doing the experiments the cathode was composed of two parts a holder and the sample. The holder was made from a conductive material. Its area was 3969 mm$^2$. During the electroplating process, the gold was deposited in this area as well. The gold was deposited in the area that was not needed in my work. Because of that I designed a new holder from non conductive material, as shown in Figure 81, to resolve this problem.

![Figure 81 the designed holder](image)

By reducing the area that should be deposited and the fact that the deposition will be only in the area in the mould of the photoresist, the life of the
electroplating solution stayed for a longer time and the rate of decline in the depositing ratio was slim. With this holder I could control the deposited ratio.

Taking into account the experience of the relevant work, another expected problem appeared in the next level. The source of this problem was in the first level. As I mentioned, in this level the sample was immersed in the electroplating solution for a long time, which may be more than two hours. The photoresist seemed to absorb some of the solution. When I started doing this step, the sample was immersed for 50 minutes in the solution to deposit 5 $\mu$m. After that the sample was cleaned with water and blown dried in air, as I had been recommended and as was used in the JWNC, and not left at room temperature or any thermal dehydrate, but it had taken directly to deposit the second seed layer. By baking the photoresist in the second level the photoresist in the first level still saturated with the solution and by heating the solution started to evaporate. Because there was no way for the gas to escape it evaporated from the photoresist then formed bubbles, which I will discuss in the next section.

In an attempt, to solve this problem I took the sample after the electroplating process and cleaned it with water and blow dried it in air. But the sample was not then left in the room temperature for some time, the sample was placed directly on a hotplate to thermal dehydrate. This technique caused a wrinkling in the surface of the photoresist which I will deduce in the next section.
5.4. Problems in the second level

In the second level, I started with depositing a 50 nm seed layer of gold only without titanium on the top of the first level. This second layer covered the entire first level. When I baked the second photo resist at 90ºC for 30 minutes I found a lot of black spots, as shown in the view under the microscope in Figure 82 and Appendix A.8.

Figure 82 bubbles appeared in the second level under the photoresist

I repeated the process by using another sample but other spots appeared, as in Figure 83 and in Appendix A.9, I determined these black spots to be gas bubbles.
Chapter 5

Aftet investigation I found that these bubbles may be confined between the first level and the second seed layer Figure 84 or between the second seed layer and the second layer of the photo resist Figure 85.
I measured these bubbles and found some of them were as high as 20µm Figure 86.

Figure 85 bubbles under the seed layer

Figure 86 the height for some bubbles
I researched the cause for these bubbles and why they had not appeared in the first level. After investigation it was found that many parameters could cause this phenomenon:

1. Dehydration

As mentioned, I immersed the sample in a liquid ‘electroplating solution’ during the electroplating process. The sample stayed in the solution for about one hour and was then taken out. Because the liquid temperature was more than 50°C, the photo resist may have absorbed some of it and become saturated. When I baked the photo resist at the second level in an oven at 90°C the liquid tried to evaporate but could not escape because the seed layer restricted it. This may have caused bubbles to form under the second seed layer.

To avoid this phenomenon I have to allow sufficient time for dehydration and I should follow these steps:

- Try to perform the deposition process of gold by electroplating in one immersed step, deposition of gold thickness should be performed in one immersion in the electroplate solution and do not take the sample from the solution and clean/dry it and place it again in the solution.

- After electroplating the sample should be cleaned with water and dried with air. The sample should then be left at room temperature for a minimum of two hours. The sample can then be thermally dried.
Chapter 5

Results and Discussion

- After many experiments it was found that sufficient time for thermal drying in this technique is 45 minutes on a hotplate and the temperature should be at 50°C, and not more than 60°C.

2. Adhesion and wrinkled surfaces

After investigation it was found that the seed layer was just 50 nm of gold when I started making the demo-devices. The surface in the first level, on which I deposited this gold seed layer, was made from silicon and in the second level was the surface of the photo resist. I observed that the surface of the photo resist had some wrinkles. After the electroplating, the surface of the deposited material and the surface of photo resist were not identical and there were some differences between their heights.

Figure 87 wrinkled surfaces

These wrinkles and differences between the heights may cause the restriction of gas through the deposition of gold seed layer between the second
seed layer and either the first or the second level contacted photo resist layers. When I baked the photo resist at the second level in an oven at 90°C, the restricted gas, which was under the second seed layer, could not escape because the seed layer was sealed. So this situation caused bubbles under the second seed layer, Figure 87 and in Appendix A.10. But if the restricted gas was over the second seed layer, it could not escape because the second photo resist was sealed and thus the bubbles might appear over the second seed layer Figure 87.

The second thought was that these wrinkles may cause weakening or dislocation of the adhesive surfaces. I immersed the sample in a liquid ‘electroplating solution’ and leaked some of this liquid in the area, which showed a weakening or breaking of the adhesion or restriction of some other gases. During the baking of the photo resist in the second level at 90°C the liquid or other restricted gas in these areas tried to escape but could not do so. This caused the bubbles.

To address this problem and avoid the appearance of the bubbles I had to follow the dehydration steps mentioned above. Secondly, I used adhesive material between the photo resists and seed layers. Here I used a seed layer consisting of two materials; first I deposited 50 nm of titanium and then 50 nm of gold. Thirdly, I dealt with soft baking and determined a sufficient temperature and time. This will be discussed in the next point.
3. Soft baking

From the experiments it was observed that the thickness of the photo resist decreases when it is left in the oven for a long time. On the other hand the photo resist shrinks during the baking process, and some gas from it evaporates. When I baked the photo resist in the second level, the photo resist in the first level would be baked as well and some gas (N$_2$ from the photoresist solvent or from H$_2$O from the electroplating solution) might be released from it that had been restricted under the second seed layer and over the photo resist in the first level and caused some bubbles.
5.5. Problems in the third level

Having followed the technique steps, the first step in the third level was the deposition of the gold seed layer. I then spun the photo resist on this seed layer then baked it at 90°C for one hour and thirty minutes. Following this, I observed under the microscope, that the photo resist and the seed layer were wrinkled as in Figure 88 and in Appendix A.10.

![Figure 88 wrinkled surface](image)

During the experiment I found that the photoresist layer lost a little bit of its hight by leaving it more time in the bake process or in the thermal drying. In Figure 89 the thickness was 5.451 μm and after bost bake the thickness was 5.239 μm, Figure 90 (Appendix A.10). This led us to do an other experiment and I found that as the photoresist was left in the oven, it lost gas and became small.
Figure 89 The thickness of the photoresist “AZ4562” after the development was 5.230 μm after 10 minutes on a hotplate at 100°C.

Figure 90 The thickness was 5.451 μm.
As shown in Figure 91, the photo resist shrank when left in the oven at 90°C. In this experiment I took 4 samples and I prepared them in the same way. At baking process the sample was placed in oven at 90°C for different times, 60 minutes, 75 minutes, 95 minutes and 110 minutes. As result I found the photoresist thickness in the sample, which was left 60 minutes, was 9029 nm, while the photoresist thickness in the sample, which was left 110 minutes was 8319 nm. The shrinking was nearly 1 μm in a time of less than an hour. However, the material of the demo-devices were made from gold and one of the properties of gold is the extention under high temperatures. At first the photo resist and the seed layer adhered to each other. While I baked the photoresist, it shrank and the seed layer and the structure of the demo-devices in the first and the second level expanded. These movements caused the photo resist to become dislocated from the surface of the seed layer. After more baking the photo resist became small and
the seed layer became long. There was no way for it to extend, and the seed layer then pushed on the top photo resist and these deformations appeared.

I completed this level and then dissolved the photo resist on all levels and etched the gold seed layer. I cleaned the sample and took some pictures using the 3D microscope. This picture Figure 92 (Appendix A.11) shows us how the device became wrinkled and deformed.

![Image of wrinkled device](image-url)

Figure 92 3D microscope pictures of the demo-device
A temperature of 90ºC for the time of one hour and forty-five minutes in an oven was suitable in the first level and gave the photo resist good properties, such as hardness and a decrease of dark erosion. However, using this temperature at the subsequent levels caused many problems. This was researched and I observed that the soft baking was too hot and too long for the subsequent levels and needed to change. These defects required us to do some experiments to find more appropriate baking times and temperatures and it was found that the appropriate temperature and time for soft baking are 50ºC for 45 minutes, for all subsequent levels.

However, if the temperature for the soft bake is reduced, it may lead to the formation of lips in the subsequent levels. For this reason, many separate experiments were done for the photo resist. I took some samples and spun some of the photoresist over it. Then it was baked at 50ºC for 30 minutes. Then I exposed it to UV light and developed it. The photoresist was only patterned. The sample was placed on a hotplate at a different temperature. When the temperature was more than 60ºC, I observed that, the area of photo resist that was exposed by UV light was extended if it was exposed to heat before development. Furthermore, I observed that lips appeared beside the exposed area after the electroplating process. On other hand, the temperature of the electroplating solution was high and led to an increase in the corners. Further work is required to identify the cause of these lips.

As a result, I decided to first reduce the temperature of the electroplating solution to avoid this problem. I ran many experiments with different temperatures. It was observed the temperature acts only to accelerate the deposition rate. The electroplating process was done at 28ºC and I obtained good results. In support of my result temperature I found many papers agreed [55, 56] that
the temperature should not exceed 35°C in the solution when depositing gold with electroplating and the process can be done at 25°C. Secondly, I controlled the exposure time spectral output of light sources.
5.6. Results of investigation

After investigation and many experiments it was found that soft bake and dehydration are the most effective parameters. In the literature review it was found that everyone uses a different time and temperature for soft baking related to his technique. In the proposed technique it was observed, as a result of the experiments, that the appropriate time and temperature used for soft baking that conducted the operation successfully was 50°C for 45 minutes on a hotplate. After each electroplating the sample should be cleaned with water and dried with air and then left at room temperature for a minimum of two hours. The sample can then be thermally dehydrated.

The demo-device1 was fabricated by following these steps:

1. Clean the sample as usual

2. Dehydrate the sample at 90°C for one hour in an oven

3. Deposit a seed layer of titanium and gold (Ti 50 nm, Au 50 nm)

4. Spin the primer 30s@400rpm and delay 2 minutes to help the adhesive

5. Spin the photo resist S1818 30s@3000 rpm and delay 10 minutes

6. Soft bake on a hotplate at 50°C for 45 minutes and delay 30 minutes at room temperature

7. Align the first mask and expose for 15s; then delay for 30 minutes
8. Develop with Micro-developer and water at a ratio of 1:1 for 50s

9. The thickness should be 2.1µm

10. Use electroplating solution for 8 minutes and 20s

11. Clean with water, dry with air and leave at room temperature for a minimum of two hours

Second level:

1. Dehydrate the sample at 50ºC for 45 minutes on a hotplate

2. Deposit a seed layer of titanium and gold (Ti 50 nm, Au 50 nm)

3. Spin the photo resist az4562 for 30s@6000 rpm and delay 10 minutes

4. Soft bake on a hotplate at 50ºC for 30 minutes and delay 5 minutes

5. Spin the photo resist az4562 for 30s@6000 rpm and delay 10 minutes

6. Soft bake on a hotplate at 50ºC for 45 minutes and delay 30 minutes at room temperature

7. Align the first mask and expose 20s; then delay 30 minutes

8. Develop using AZ400k developer with water at a ratio of 1:4 for 2 minutes
9. The thickness should be 10.5µm

10. Use electroplating solution for 45 minutes

11. Clean with water, dry with air and leave at room temperature for a minimum of two hours

Third level:

1. Dehydrate the sample at 50ºC for 45 minutes on a hotplate

2. Deposit a seed layer of titanium and gold (Ti 50 nm, Au 50 nm)

3. Spin the photo resist S1818 for 30s@3000 rpm and delay 10 minutes

4. Soft bake on a hotplate at 50ºC for 45 minutes and delay 30 minutes at room temperature

5. Align the first mask and expose for 15s; then delay 30 minutes

6. Develop with Micro-developer with water at a ratio of 1:1 for 50s

7. The thickness should be 2.1µm

8. Use electroplating solution for 8 minutes and 20s

9. Clean with water, dry with air and leave at room temperature for a minimum of two hours
For the fourth level the same steps as in the second level were repeated; for the fifth level the same steps as in the third level were repeated.

A demo-device2 with a good surface (Figure 93 and Appendix A.2,) was then obtained. This technique was used for producing other devices containing six layers (Figure 94 and Appendix A.3).

![Figure 93 demo-device2 with 5 layers](image)

Figure 93 demo-device2 with 5 layers
Figure 94 demo-device1 with 6 layers
5.7. Conclusion

This work proposes a novel technique in MEMS technology. The basis of the new technique is that the required mould should be fabricated layer by layer. Consequently, the multi-layer executed shape will represent the final product shape. The number of layers depends on the desired shape of the mould. In particular, if there are curved surfaces in the axial direction of the required mould, numerous thin layers should be used to achieve the desired surface quality. These layers will be made from a dissolvable material that is sensitive to light. The layers will be dissolved just after all processes are complete. As in the other MEMS techniques, masks are used to build the desired mould features. However, in this technique a mask with the same shape of the particular layer in the required mould is used for each layer individually. Therefore, more experiments are needed to construct a relationship between the accurate exposure time and the thickness of the treated layer. Also it is beneficial to investigate the effect of the developer time on the accuracy of the features desired in each level.
Chapter 6

Conclusions and future work
Conclusions

The main objective of the work was to determine how the fabrication of 3D complex structures and their high-volume reproduction can be improved. In this work, a novel technique has been proposed in MEMS technology. This technique improves on the existing conventional techniques and the LIGA process. The LIGA process has succeeded in producing a high aspect ratio structure device, but in this work I attempt to obtain a high width to thickness ratio of the layer. In other words, a hybrid technique has been used which consists of LIGA and thin film techniques. To make a 3D structure, there has to be a thin film deposited and then applied to the LIGA process one layer on top of the other. The whole structure can then be stacked together by using multi layers to meet the requirement and chapter 3 has shown how a device with a curve could be produced by using this proposed technique.

This work presents a novel idea to improve the LIGA process when using many masks to deposit multi thin layers over each other (Figure 95). Moreover, this technique can be utilized to produce a curved surface in Z direction with any diameter as shown in Figure 95. Practically, a 2 µm thickness of layer is applied in the proposed technique. However, a layer of 0.5 µm can be deposited.

Figure 95 the multi layer planes
The basis of the new technique depended on division of drawing mould of the required device to many layers, Figure 96. The shape of each layer in this technique works as a mask for each level. The number of layers depends on the desired shape of the mould. In particular, if there are curved surfaces in the axial direction of the required mould, numerous thin layers should be used to achieve the desired surface quality. These layers will be made from a dissolvable material that is sensitive to light. The layers will be dissolved after all the processes are complete. The thickness of each layer in the fabricated mould is the same of layer that has been divided in the drawing mould of the required device.

As in the other MEMS techniques, masks are used to build the desired mould features. However, in this technique a mask having the same shape of the particular layer in the required mould is used for each layer individually. Therefore, it needs more experiments to construct a relationship between the accurate exposure time and the thickness of the treated layer. Also it is beneficial to investigate the effect of the developer time on the accuracy of the features desired in each level.

Since 1993, researchers have known about the multi layer process technique and have made good progress during the last years. Most of the
research has reported that the use of the conventional technique has many
problems and they have used different techniques and succeeded in building a 3D
structure. Moreover, all previous problems have appeared in my work as well,
such as lips, bobbles and wrinkles. However, by using my technique I overcame
the problems that all researchers have met or mentioned in their research which I
have discussed in chapter 5 “Results and Discussion”.

As a result, I observed that one hour and forty-five minutes at 90°C had the
best results for soft baking at the first level and the “lips” disappeared. However,
this temperature and time period caused problems in the subsequent level, which
was in the form of bubbles of gas. These bubbles may appear under or over the
second seed layer. In the third level another problem appeared, “wrinkled
surface”. To avoid this phenomenon I have to allow sufficient time for
dehydration and I should follow these steps:

- The appropriate temperature and time for soft baking is 50°C for 45
  minutes, for all subsequent advanced levels. In the first level, the
  photoresist can be baked at 90°C for 30 minutes.

- Try to perform the deposition process of gold by electroplating in one
  immersed step, deposition of gold thickness should be performed in one
  immersion in the electroplate solution. Do not take the sample from the
  solution and clean/dry it and place it again in the solution.

- The temperature of the electroplating solution in the experiment should
  not go above 28°C in all levels.
• After electroplating the sample should be cleaned with water and dried with air. The sample should then be left at room temperature for a minimum of two hours. The sample can then be thermally dried.

• After many experiments it was found that sufficient time for thermal drying in this technique is 45 minutes on a hotplate and the temperature should be at 50ºC, and not more than 60ºC.

• An adhesive material should be deposited between the photo resists and seed layers

In my technique I have improved the conventional technique and this improvement has made the technique potentially cheap and efficient compared with any previous techniques. With my technique any structure could be made. The technique used the most common photoresist which is easy to align and dissolve.

In addition, the work has also identified the critical nature of the electroplating solution temperature in order to achieve a reliable and high-quality fabrication process. In particular it has been demonstrated that a temperature of 25ºC is required.
Future work

Although it has been possible to demonstrate a workable fabrication process future work is necessary to further investigate and explain the interaction of temperature, time and UV light exposure on the behaviour and properties of the resist material. It will be useful to conduct a study the lips in the photoresist and why they appeared, when exposed to the high temperature and what is the relationship between the light and the lips.

In the appendix A.2, some pictures are attached for demo-devices which were built with 5 layers and inspected using Scanning Electron Microscopy (SEM). The demo-device in Figure 46 I built six layers of nine and did not complete the remaining three layers because the device is symmetric. The demo-device had good surfaces and was without any defects, some pictures of this demo-device are attached in appendix A.3.
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Appendix

List of Appendix

A.1, drawing of the demo-device1

A1.1, drawing of the demo-device2

A.2, some pictures of the demo-device2, which were built with 5 layers, have been taken by using Scanning Electron Microscopy (SEM).

A.3, some pictures of the demo-device1, which were built with 9 layers, have been taken by using Scanning Electron Microscopy (SEM).

A.4, pictures of removing the SU8 and not solving

A.5, the surface of the photoresist after the development and before electroplating

A.6, the surface of the photoresist after the electroplating process and appearing of lips

A.7, scheme of the treated photoresist surface

A.8, appearing of bubbles in the second level

A.9, appearing of bubbles under the second level

A.10, some pictures of wrinkled surface in the photoresist

A.11, some pictures of wrinkled surfaces in the demo-devices, which were built with 3 layers, have been taken by using Scanning Electron Microscopy (SEM).

A.12, Paper which we participated in a conference in Barcelona in Spain in 2008

A.1

Drawing of the demo-device1
A1.1

Drawing of the demo-device2
<table>
<thead>
<tr>
<th>Dimension (mm)</th>
<th>Material, etc.</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>30</td>
<td></td>
</tr>
</tbody>
</table>

Diagrams of the fabricated parts.
A.2

Some pictures of the demo-device2, which were built with 5 layers, have been taken by using Scanning Electron Microscopy (SEM).
Appendix
A.3

Some pictures of the demo-devices, which were built with 9 layers, have been taken by using Scanning Electron Microscopy (SEM).
Appendix
A.4

Pictures of removing the SU8 and not solving
A.5

The surface of the photoresist after the development and before electroplating process
Appendix

The surface of the photoresist after the electroplating process and appearing of lips.
A.7

Scheme of the treated photoresist surface
A.8

Appearing of bubbles in the second level
A.9

Appearing of bubbles under the second level
Appendix
Appendix

![Image of microchip components](image-url)
Appendix

A.10

Some pictures of wrinkled surface in the photoresist
A.11

Some pictures of wrinkled surfaces in the demo-devices, which were built with 3 layers, have been taken by using Scanning Electron Microscopy (SEM)
Paper which we participated in a conference in Barcelona in Spain in 2008
3D Structures: multi-layer micro/nano fabrication

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Abstract

There is keen interest in the development of fabrication techniques to cost effectively mass-produce large areas of high-resolution (micro/nano) features in a range of materials. Applications include high-density storage devices and textured curved surfaces for medical implants such as arterial stents. A key technical challenge is the fabrication of curved surfaces, 3D features and the efficient transfer of these high-resolution features onto curved surfaces. This paper describes recent work aimed at meeting the challenges outlined above via a development of the well-known LIGA (“Lithographie”, “Galvanik”, and “Abformung”) process. The emerging new process is provisionally termed ‘3D structure: multi-layer micro/nano fabrication’ and comprises the production of a multi-layered mould having the inverse form of the desired feature/device and fabricated from a dissolvable material that is also sensitive to UV light.

Introduction

In recent years MicroElectroMechanical Systems (MEMS) have developed well and have found application in almost all technology. This technique has also had a major influence on the cost of products [1]. Since the establishment of MEMS technology engineers have tried to improve the method to make 3D structures. In bulk and surface micromachining technology the 3D structure is defined by the thickness of the deposited layer or by the depth of the etching process. Both methods have the same problem ‘high surface damage’. One fabrication techniques that successfully addresses the above problems is LIGA [2], it is an acronym for the German words, “Lithographie”, “Galvanik”, and “Abformung” (lithography, electroforming, and moulding), and was introduced in 1986 [3]. Structures are created in 2D and third axis depends on the thickness of the resist material. With the development, that LIGA has enjoyed MEMS technology has moved forward considerably. Nowadays it is possible to fabricate devices with a 3D structure. However, difficulties remain with the fabrication of curved surfaces and the addition of information...
Appendix

onto a curved surface. The work described in this paper seeks to improve the performance of the fabrication of 3D complex structures. With this ability it may be possible to fabricate a complex structure and perhaps even a curved structure. For example it may become possible to use this technique to apply information onto the surface of an Arterial Stent \[^4\]

which is used to improve blood flow in the body's arteries.

The LIGA process uses a mask which has the shape of the desired structure device to allow 2D information to be transferred onto a resist material.

![Fig 1 transfer the 2D structure \[^5\]](image)

It becomes possible to fabricate a mould that has the inverse shape of the desired structure. The 3D structure depends on the thickness of the resist material, as illustrated in Figure.

![Fig 2 the XYZ planes](image)

The LIGA process has had good success in providing a way to produce devices with a high aspect ratio structure \[^6\]

but it is not possible to make any structure in the ZX plane.
Appendix

or in the ZY plane, Figure 2. For example, it would not be possible to make tubes go through an object or projection or bulge. LIGA uses the shadow of a mask and can only transfer a 2D structure. In other words, a device that we make with LIGA can only be drawn in the XY plane, illustrated in Figure 2. The LIGA process has been developed to create 2.5D structures\(^5\). For this reason engineers have tried and continue to try to find ways to improve the process. They have found many ways to make 3D structures and a notable example of this is the fabrication of a Spiral Microcoil using LIGA \(^7\). However difficulties remain with the fabrication of a curved surface or the creation of a structure on a curved surface with LIGA process. In other words, at it is not yet possible to build a structure in the XZ plane or in the YZ plane (Figure 2).

**Development of 3D structure in the LIGA process**

In this section we will explain how it may be possible to fabricate a mould which has the inverse shape of a desired structure. The mould is built from a material which is dissolvable and is sensitive to UV-light as well. The desired structure has a curved shape. The mould will be divided into many layers. These layers have a very small thickness. In others words, we divide the mould in layers and each layer has just a 2D structure. This means that we can then use UV-light instead of X-ray because the thickness of the resist material using UV-light is limited and UV-light is much cheaper than X-ray \(^8\).

**The steps of the method**

The first step of this novel method is to draw the inverse of the desired device with any software such as Auto CAD as a complete ‘3D Solid Model’. Next we divide the mould into many layers as thin as possible, it is envisaged that there could be more than 100 layers required. Each layer has a different shape and some of the layers are similar but they have small differences from each other. Each layer will be used in this method as a mask for one process. Each layer has a number related to its position in the mould. A reference mark must be added to each layer. It must be noted that because of the deposition technique used we must refresh the reference mark on each subsequent layer to ensure that the multi-layers will all align accurately.

The process progresses according to the following steps. To help understand we will explain with an example object, illustrated in Figure 3, how to draw the mould:
Appendix

1. Prepare a substrate layer ‘silicon wafer’;

2. Coat the substrate layer with a resist material such as ‘PMMA’, the thickness of this layer is the same as the thickness of the layer that we have divided in the mould. Here we could use spin coating or sputter deposition\(^9\);

3. Take the first mask, which has the same shape of the first layer of the mould, Figure 4, and hold it over the resist material;

4. Expose the mask to a UV-light, the light passes through the mask onto the resist material and transfers the mask shape onto the resist material and causes to change the properties of the resist material as well;

5. Develop the resist material by drowning it in chemical solution, the chemical solution removes the material that is exposed in UV-light. Now we have a cavity which has the same shape of the mask, fig 5;
6- Deposit a desired material such as Aluminium onto the resist material. The deposited material will fill the cavity on the resist material. In this step the CVD ‘Chemical Vapor Deposition’ or electroplating method could be used to deposit material.

7- In order to prepare the surface for the next step, clean the previous surface of the resist material which has been covered by the deposited material in the previous step (6). This leaves just the material that filled the cavity.

When this step is finished, we have the substrate layer and over it is the resist material with a cavity which has been filled with material such as Aluminium fig 6. We need the resist material to support the next steps. When we finish all steps then we can dissolve the resist material.
Fig 7 the object in mould

After this step 6 we bring the second mask and repeat the steps from step 2 to step 7, fig 21 with all masks that we have made from the mould. When the process is completed we can dissolve the resist material and leave the desired device with 3D structure, fig 8.

Fig 8 dissolve of resist material and lift the device

Conclusion

In this work, a novel technique in MEMS technology has been proposed. The basis of the new method is that a mould should be divided into many layers. These layers are made from a dissolvable material that is also sensitive to light. The layers will dissolve only when all processes are completed. In this method masks are used, which contain a large number of copies of each mould’s layer, which can be used repeatedly. This saves a lot of time and money in total.

The LIGA process, modified with this method, has a good push forward to improve its performance in MEMS technology. However, fabrication using this technique is a very challenging task due to the high number of the layers, the alignment between the layers and the exposition the UV light on the resist material by using the second mask and the
others. The substantial technical challenges associated with this new technique forms the basis of on-going research being undertaken at the University of Glasgow.

References

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Appendix

A.13

Nano/Micro fabrication of 3D device structures using a multi-layered mould approach

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Abstract

There is growing interest in the development of fabrication techniques to cost effectively mass-produce high-resolution (micro/nano) 3D structures in a range of materials. Biomedical applications are particularly significant.

This work demonstrates how to simultaneously fabricate a sacrificial mould having the inverse shape of the desired device structure and also create the desired device structure using electro-deposition techniques. The mould is constructed of many thin layers using a photo-resist material that is dissolvable and sensitive to UV light. At the same time the device is created in the emerging mould layers using Gold electro-deposition technique. Choosing to fabricate the mould and the 3D structures in multiple thin layers allows for the use of UV light and permits the potential cost-effective realization of 3D curved surfaces, the accuracy and geometric details of which are related to the number of layers used. An example is provided to explain the novel fabrication process and to outline the resulting design and fabrication constraints.

1. Introduction

In recent years MicroElectroMechanical Systems (MEMS) have developed significantly and have enjoyed wide ranging and cost-effective application [1-3]. In the early 1960’s MEMS development focused on diffusion and etching of bulk wafers, an approach called bulk Micromachining. Surface Micromachining, a technique developed in the 1980s, has more recently given the field a real boost. Structures are generally built from thin layers of materials. The process consists of depositing thin films of metal or crystalline material on a substrate layer (usually silicon), that are subsequently patterned. Patterning is normally achieved, in a technique called photolithography, by depositing a layer of material that is
sensitive to light (photoresist), and exposing it to light through a mask (this mask has transparent and opaque regions in the shape of the desired device). The exposed regions of photoresist can be selectively etched away, whilst leaving the unexposed regions untouched. When no longer required, the unexposed regions of photoresist can also be etched away using a suitable solvent. In effect, a sacrificial layer is introduced. This is a material that keeps other layers separated as the structure is being built up but is dissolved in the very last step, allowing release of the desired structural [4-6].

As mentioned above, the 2D shape of the desired device is drawn on a mask, with the light passing through the mask to transfer the 2D image into the photoresist layer. The third dimension of the pattern depends on the thickness of the thin films used or on the depth of any etching process. This is a disadvantage of surface micromachining, as it employs the use of thin films it cannot create highly 3D structures.

Since the establishment of MEMS technology researchers have tried to find improved methods to make 3D structures. One fabrication technique that successfully addresses the above problems is LIGA [7]; this is an acronym from the German words “Lithographie”, “Galvanik”, and “Abformung” (lithography, electroforming, and moulding), and was introduced in 1986 [8]. Structures are still created in 2D, with the third axis dimension controlled by the thickness of the resist material. However by employing X-ray illumination and thick resists very high aspect ratios can be achieved, for example gears for watch movements [9]. LIGA has found many applications in MEMS fabrication. A particular advantage of the process is that allows a mould having the inverse shape of the desired structure to be created.

The LIGA process has had good success in providing a way to produce devices with a high aspect ratio structure [6], but it is not possible to make any features in the ZX or ZY plane. For example, it would not be possible to create features with a curved surface in the Z direction. As traditional LIGA uses the same photo-mask principle as described previously it can still only transfer a 2D image and is therefore designed to create 2.5D structures [10]. LIGA has made significant progress during the last decade, using many different techniques to fabricate 3D structures. A notable example of this is the fabrication of a Spiral Micro coil [7]. However, difficulties remain with regard to the fabrication of a curved surface or the creation of a structure on a curved surface. In other words, it is not yet possible to build a structure with detail in the XZ plane or in the YZ plane (where Z is the height of the
structure). For this reason researchers have tried and continue to try to find new ways to improve the capability of the LIGA technology.

Another technique has been developed that uses multi layer process to build three-dimensional microstructures using multi-layer UV photolithographic patterning of thin resist layers followed by electroplating. This usually involves a process of seed layer deposition, resist spin coating, UV exposure and electroplating to mould the resist patterns. The process is then repeated, using intermediate seed layers if necessary, to realize the three-dimensional metal microstructure. The final steps involve chemical removal of the resist and plating seed layers. Examples of structures made using this approach include a three-dimensional coil with ten windings [11]. Precise process control is essential in this technique if a high quality mould (and hence device) is to be produced. A 3D coil has been fabricated using only three layers of organic resists (az4000 series, HOECHST) with a high-aspect-ratio, the height was 60 µm. In addition, only one seed layer was deposited in this process, whereas the photo resist was not directly confined between two seed layers. The authors follow the same technique described in [12] for the baking of the photo resist, where the baking temperature must not exceed 130°C. The temperature used in the second layer was 15°C less than that in the first layer, to prevent shrinking of the first layer of photo resist. However, the authors do not detail the effect of baking time on the photo resist behavior throughout the process.

Further, another technique has been developed that uses metal moulds rather than photo resist as the sacrificial material [13]. The metal mould also serves as a seed layer for electroplating the next layer of the structure. The authors claim that thermal reflow of the lower photo resist layer resulted in a poor fabrication yield. Therefore, the conventional plating through mask technology exhibits a limitation on the number of levels that can be produced.

Multi layer three-dimensional structures have also been fabricated using a hybrid approach consisting of UV photolithographic patterning of spin-coated resist layers, laser patterning of laminated resist layers and electroplating with intermediate seed layers [14]. The laser is particularly suited for ablation of thick resists built up from multiple laminated layers. In this case only two seed layers are used and the wet photo resist is not confined directly between them. The dry photo resist is deposited in the middle of the process and separated by the layers of wet photo resist. The minimum thickness of dry photo resist that can be deposited is 20 µm, it is also a good material to produce layers with a high-aspect-ratio.
Previous researchers have studied the behavior of the photo resist az4562 and compared it with polyimide PI2611 by using multi layers to produce a coil [15]. Jing SUN states “it was hoped to present various different sample results for the final comparison. However, due to the problems encountered in the az4620 process, completed coils were not available to present the samples fabricated in that method”.

Considering the difficulties encountered by previous researchers, it is anticipated that producing a curved surface in the Z plane (Figure 1) will be difficult due to the many layers required to achieve the necessary detail and accuracy.

![Figure 1: An example curved structure produced using multiple resist layers](image)

The main objective of the work described here is therefore to determine how to achieve the quality fabrication of complex 3D micro-structures, enabling their future high-volume reproduction. As we have seen, most previous researchers have tried to obtain a single layer with a high aspect ratio however in this work we employ a more conventional high width to thickness ratio. This paper therefore presents a novel technique to improve the LIGA process by using many masks to deposit multiple thin layers (Figure 1). Moreover, this technique can be uniquely utilized to produce a curved surface in the Z direction with any diameter. A 2 µm thickness of resist layer will be used to best demonstrate the application of the proposed technique but a layer of 0.5 µm can be deposited.

The following section of this paper will provide an outline summary of the proposed novel fabrication technique and then, in subsequent sections, proceed to describe the technical challenges encountered and overcome when attempting to fabricate each layer of a prototype test device.

2. Research methodology
The research methodology guiding the research activity was informed by the following research hypothesis. ‘That a reliable process can be conceived and developed to allow the fabrication of complex 3D micro-structures by using a multilayer electro-deposition approach. CAD software can be used to prepare a 3D model of the desired design that is then divided into stacked 2D layers, the number of layers being determined by the complexity of the structure. The 2D pattern associated with each can then be used to produce a photolithography mask.’

Therefore it can be envisaged that the following specific steps can then be employed to fabricate a 3D micro-structure:

• After a thorough cleaning, the substrate is coated in a thin layer (e.g. 50 nm) of a conductive material such as gold on the upper surface. This layer will act as a seed layer for the electroplating process.

• A layer of resist material (e.g. az4562 or S1800) is spin coated on top of the seed layer. The resist dilution and spin speed are controlled to ensure the resulting film is the same thickness as the corresponding 2D layer of the device. The sample is then baked to remove solvent from the resist film.

• The resist is exposed to UV light through the appropriate mask plate, after exposure the resist is processed and developed to expose a 2D mould with a seed layer at the bottom.

• The mould is filled with metal (e.g. gold, nickel) using electroplating, ensuring the deposited material is the same thickness as the resist mould.

• This process is then repeated for each subsequent level. A seed layer is deposited over the previous level, protecting the underlying layer of photo resist from the UV light and acting as a seed layer for the subsequent electroplating step.

After completion, the electroplated 3D structure requires releasing from the encapsulating photoresist and seed layer mould. To achieve this we:

• Expose the top level of photoresist to UV light using a “Flood-Exposure” (i.e. without a mask). This layer can then be removed using photoresist developer. This reveals the top most seed layer.

• The seed layer can be removed using an appropriate metal etch solution (e.g. potassium iodide/Iodine for gold).
These steps are repeated for each layer with a final clean being achieved by immersing the device in warm acetone.

3. Demonstrated Fabrication

The proposed fabrication process, outlined in the previous section, depends on the division of the mould into many layers, meaning that many masks may be required. To simplify and minimize the cost of the development process we designed test devices that could be made with just two repeating masks (Figure 2). However the developed process would still be compatible with designs requiring many different masks.

![Figure 2. Demonstration devices consisting of five layers with two alternating masks](image)

The devices shown in Figure 2 have dimensions of 110x360x28 µm. Layers 1, 3 and 5 are 2 µm thick and have the same shape, permitting use the same mask. Layers 2 and 4 are 11 µm thick, have the same shape and can thus be used a second mask. The masks were designed using lithographic CAD software “L-Edit” and then produced using E-beam lithography on commercially prepared mask plates.

As mentioned above the thickness of the photo resist can be controlled from level to level to produce the appropriate 3D structure. The photoresist materials selected were az4567[16] and S1800[17], since a wide range of data is available on these resists [16-20]. To test the thicknesses obtainable with these resists a number of basic experiments were conducted. These experiments allowed the resist film thickness to be measured as a function of spin speed, as shown in Table 1.
3.1. The first level

A seed layer of 50 nm titanium and 50 nm of gold were evaporated over the entire sample surface before S1818 photo resist was spun on for the first level. The photo resist was then spun at a speed of 3000 rpm to obtain a thickness of 2.1 µm (Table 1) and then left at room temperature for 10 minutes before being baked on a hotplate at 90°C for 30 minutes (or an oven at 90°C for 75 minutes), to allow the majority of the solvent to evaporate without disrupting the photoresist layer. At this point the exact time and temperature for baking were found to not be important; only requiring sufficient time to solidify the photo resist. However if a bake temperature greater than 120°C was used for longer than 30 minutes difficulties were encountered during development. Following this the sample was left at room temperature 30 minutes before UV exposure through the mask plate. Exposure was carried out using 365nm wavelength light, at an intensity of 7.2 mW for 15s before being left at room temperature for a further 30 minutes. It has been shown that this ‘rest’ is required to allow evolution of N2 (a product of resist exposure) from the resist layer without disrupting the film. The sample was developed for 40 seconds using Micro developer (Microposit concentrated from the Chestech company) diluted 1:1 with water. Figure 3 shows a typical optical micrograph of the developed resist at this level.

### Table 1: The thickness of AZ4562 and S1818 layers spun at various speeds.

<table>
<thead>
<tr>
<th>Spin Speed [rpm]</th>
<th>1000</th>
<th>2000</th>
<th>3000</th>
<th>4000</th>
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<tbody>
<tr>
<td>AZ4562 [µm]</td>
<td>11.5</td>
<td>9.3</td>
<td>7.5</td>
<td>6.5</td>
<td>5.3</td>
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<tr>
<td>S1818</td>
<td>3.9</td>
<td>2.7</td>
<td>2.1</td>
<td>1.7</td>
<td>1.5</td>
</tr>
</tbody>
</table>

The structure which represents the mould of demo-device1

110 µm
This structure was filled with gold in next step. As mentioned above, for this step electroplating is used to deposit gold into the holes. To deposit 2 µm requires 10 minutes. We have followed to deposit the gold the same process in the date sheet[21, 22]. However we found that the temperature of the solution in the electroplating process, which is made from components: Brightener No6, BDT-X Complex and BDT 510 B form the company Enthone[21], should not exceed 28ºC and the current should be 4 mA.

3.2. The second level

Prior to starting the second level, the sample was cleaned with water and blown dried in air before being left at room temperature for two hours. The sample was then placed in an oven for 30 minutes at 50º to remove any remaining water before being left at room temperature.

A seed layer of 50 nm titanium, and 50 nm of gold, was evaporated over the entire sample surface. A layer of az4562 was spun onto the sample at 1500 rpm to obtain an 11 µm thick film (Table 1). The sample was then baked on a hotplate at 50ºC for 45 minutes. The second mask was then used by aligning it to the first level, already on the substrate, and exposing the sample for 20s with the same illumination source as used in the first level. The exposed az4562 was developed in AZ developer diluted 1:4 with water for 2.5 minutes (Figure 4).

Figure 4 the second layer of the demo-device1 after developing

Gold electroplating was used with the same conditions as described previously with the alteration that 45 minutes were required to deposit 11 µm.

3.3. Third and subsequent levels

The third and fifth levels were produced by repeating the same steps as the first level. However it was found that the baking time and temperature were much more sensitive, with
the wrong conditions resulting in many defects, such as bubbles and wrinkles (detailed below). It was also found that a temperature and time of 50ºC for 45 minutes eliminated these defects. The fourth level followed the same procedure as the second level.

After completion of the fifth level, the 3D micro-structure requires releasing from the encapsulating photoresist and seed layer mould. Firstly: -

• The top level of photoresist was exposed to UV light using a “Flood-Exposure”. The layer was then removed using photoresist developer. This revealed the top most seed layer.

• The seed layer was removed by using a gold etching, exposing the layer of photoresist beneath.

These steps are repeated for each layer with a final clean being achieved by immersing the device in warm acetone. After removal of the resist and seed layers the completed devices were inspected using Scanning Electron Microscopy (SEM) (Figure 5).
4. Discussion

When fabricating this test device we found that precise process control was required in order to ensure the device was produced without defects. Some parameters were found to be extremely sensitive to variation and could result in failed fabrication. Specifically, the time and temperature of the soft bake, the dehydration time and the temperature of the electroplating solution were all critical.

4.1. Problems in the first level

Figure 6a shows a surface profile scan of the as-developed photoresist, showing it to be flat except for the developed features. Figure 6b shows the same sample after exposure to the electroplating solution. It clearly indicates the formation of a lip at the edge of each feature. The length of the lips is approximately 400 nm. This feature was still seen to form when the sample was hard baked.

Figure 6 a) Photo resist after developing    b) Photo resist after electroplating

It was judged that there were two possible variables influencing the formation of the lips:

• the quality and temperature of the electroplating solution (normally between 50-60°C)
• the generally recommended baking time and temperature was not sufficient
It is interesting to note at this point that the literature details many different techniques for baking photoresist materials. Some researchers suggest using soft baking and some pre- and hard-baking. Soft baking consists of just one bake after spinning the photo resist and before the UV exposure. Pre- and hard-baking involves a bake after spinning and second bake after development. The manufacturer of the AZ 4500 series recommends that the bake temperatures should not exceed 90°C [16] and for the SI 8100 series a maximum of 115°C is advised [17]. Recommended bake times also vary between manufacturers, with 20 minutes at 100°C [16], 30 minutes at 120°C [18] or on a hotplate for 3 minutes at 95°C [23] all being suggested. Some researchers even propose dividing the soft bake process into two steps [19, 20].

Therefore in an attempt to prevent the lip formation we addressed each of the above possible variables in turn:

- It was observed that the quality and temperature of the electroplating solution did not appear to influence the formation of the lips. Variation of the temperature was seen to act only to accelerate the deposition rate. Thereafter the electroplating process was undertaken at 28°C and we obtained good results. This conclusion and approach found support in the literature [24, 25].

- Baking temperature was systematically varied over 90°C, 120°C, 140°C and 160°C for 30 minutes. It was observed that a temperature greater than 120°C for 30 minutes would result in difficulties with developing the photo resist. However, it was also observed that at 90°C the longer the sample was left in the oven the less prominent the lip formation. After a considerable period of testing it was concluded that a soft bake of one hour forty five minutes at 90°C gave the optimum result. It should be noted that the two little blips on the central surface are considered normal variations.

4.2. Problems in the second level

When the second level of photo resist was baked at 90°C for 30 minutes microscopic black spots were seen to form (Figure 7a). This phenomenon was repeatable (Figure 7b); upon investigation it was determined that these black spots were gas bubbles.
Further observation suggested that these bubbles may be confined above (figure 7a) or below (figure 7b) the seed layer, resulting in a slight variation in appearance. It was felt that two main factors could cause this phenomenon to occur, each was systematically investigated in turn:

1. Dehydration

The sample was immersed in a warm (50°C) liquid for around one hour during the electroplating process. During this time the photoresist may have absorbed some of this liquid which would rapidly evaporate during the 90°C bake of the subsequent level. However this evaporating liquid would be unable to escape due to the overlaying seed layer, resulting in bubbles under the second seed layer. To avoid this phenomenon the following steps were employed:

- The electroplating of the gold was carried out in one single step, with the sample never taken out for inspection and then replaced.

- After electroplating the sample was cleaned with water and blown dry with nitrogen. The sample was then left at room temperature for a minimum of two hours before any baking procedure. This permitted the absorbed liquid to evaporate slowly, avoiding bubble formation.

- Thermal drying was employed (after two hours at room temperature) for 45 minutes on a hotplate with a temperature of 50°C, and never more than 60°C. Again this dried the sample slowly, avoiding the formation of bubbles.

2. Adhesion and wrinkled surfaces
After the first level, all subsequent gold seed layers were deposited onto the surface of underlying photo resist, which was observed to have some wrinkles (Figure 8). This resulted in a rough surface finish after electroplating.

![Figure 8: Wrinkled surfaces](image)

One possible explanation for the resist roughness was of the occurrence of trapped bubbles, described in the previous section. Another possibility was that these wrinkles resulted from poor adhesion between the seed layer and the photoresist. The first possibility was solved by following the dehydration steps mentioned above. The second was eliminated by employing an adhesion layer (50 nm of titanium) between the photo resist and the gold seed layer.

### 4.3. Problems in the third level

After deposition and baking (90°C for one hour and thirty minutes) of the third layer of photo resist it was observed that both it and the underlying seed layer had again become wrinkled (Figure 9a).

![Figure 9a: Wrinkled surface](image)
This wrinkling can be explained as a result of the differential expansion of the gold and adhered photo resist during the bake process. This becomes particularly clear upon inspection of a released device in a Scanning Electron Microscope (Hitachi S4700) (Figure 9b).

As we have seen an oven bake of 90ºC for one hour and forty-five minutes was employed without problem in the first level, giving benefits such as resist hardness. However, using this temperature at the subsequent levels was clearly causing additional problems. Systematic experimental investigations revealed that a soft bake of 50ºC for 45 minutes for the second and all subsequent levels eliminated the problem of wrinkles. However, as described previously, such a low temperature soft bake carries the risk of causing the formation of resist lips which has treated by using lowe temprature in electroplating.

5. Results

As a result of the systematic experimental investigations described above a reliable procedure for multi-level photo lithography/electroplating has been developed and can now be summarized as follows:

1. Clean the sample
2. Dehydrate the sample at 90ºC for one hour in an oven
3. Deposit a seed layer of titanium and gold (Ti 50 nm, Au 50 nm)
4. Spin the primer 30s@400rpm and delay 2 minutes to help the adhesive
5. Spin the photo resist S1818 30s@3000 rpm and delay 10 minutes
6. Soft bake on a hotplate at 50ºC for 45 minutes and delay 30 minutes at room temperature
7. Align the first mask and expose for 15s; then delay for 30 minutes
8. Develop with Micro-developer and water at a ratio of 1:1 for 50s
9. The thickness should be 2.1µm
10. The operating conditions for electroplating:

the temperature of the solution should be at 25ºC
the current should be 4 mA

for 8 minutes and 20s

11. Clean with water, dry with air and leave at room temperature for a minimum of two hours

Second level:

1. Dehydrate the sample at 50°C for 45 minutes on a hotplate
2. Deposit a seed layer of titanium and gold (Ti 50 nm, Au 50 nm)
3. Spin the photo resist az4562 for 30s@6000 rpm and delay 10 minutes
4. Soft bake on a hotplate at 50°C for 30 minutes and delay 5 minutes
5. Spin the photo resist az4562 for 30s@6000 rpm and delay 10 minutes
6. Soft bake on a hotplate at 50°C for 45 minutes and delay 30 minutes at room temperature
7. Align the first mask and expose 20s; then delay 30 minutes
8. Develop using AZ400k developer with water at a ratio of 1:4 for 2 minutes
9. The thickness should be 10.5 µm
10. The operating conditions for electroplating:

the temperature of the solution should be at 25°C

the current should be 4 mA

for 45 minutes

11. Clean with water, dry with air and leave at room temperature for a minimum of two hours

Third level:

1. Dehydrate the sample at 50°C for 45 minutes on a hotplate
2. Deposit a seed layer of titanium and gold (Ti 50 nm, Au 50 nm)
Appendix

3. Spin the photo resist S1818 for 30s@3000 rpm and delay 10 minutes

4. Soft bake on a hotplate at 50°C for 45 minutes and delay 30 minutes at room temperature

5. Align the first mask and expose for 15s; then delay 30 minutes

6. Develop with Micro-developer with water at a ratio of 1:1 for 50s

7. The thickness should be 2.1µm

8. The operating conditions for electroplating:
   - the temperature of the solution should be at 25°C
   - the current should be 4 mA
   - for 8 minutes and 20s

9. Clean with water, dry with air and leave at room temperature for a minimum of two hours

For the fourth level the same steps as in the second level are repeated; for the fifth level the same steps as in the third level are repeated.
Appendix

The effectiveness of this new process and be demonstrated with reference to the demo-device1 shown in Figures 10. The technique was then extended to produce a device consisting of six layers.

6. Conclusion

This paper has, for the first time, described the development of a novel technique using multi-layered electroplating technique allowing the fabrication of complex 3D micro - structures.

The work has also identified the critical nature of the electroplating solution temperature in order to achieve a reliable and high-quality fabrication process. In particular it has been demonstrated that a temperature of 25oC is required.
Appendix

In addition it has been possible to merge current thinking about the LIGA process and with developments in surface micro-machining to permit the fabrication of complex 3D micro-structures using a reliable process.

Although it has been able to demonstrate a workable fabrication process future work is necessary to further investigate and explain the interaction of temperature, time and UV light exposure on the behavior and properties of the resist material.

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