Replication of metal-based microscale structures by compression molding: a combined experimental and finite element analysis study

Jing Jiang
Louisiana State University and Agricultural and Mechanical College, jjiang3@lsu.edu

Follow this and additional works at: https://digitalcommons.lsu.edu/gradschool_dissertations

Part of the Mechanical Engineering Commons

Recommended Citation
https://digitalcommons.lsu.edu/gradschool_dissertations/2339
REPLICATION OF METAL-BASED MICROSCALE STRUCTURES BY COMPRESSION MOLDING: A COMBINED EXPERIMENTAL AND FINITE ELEMENT ANALYSIS STUDY

A Dissertation

Submitted to the Graduate Faculty of the
Louisiana State University and
Agricultural and Mechanical College
in partial fulfillment of the
requirements for the degree of
Doctor of Philosophy

in

The Department of Mechanical Engineering

by

Jing Jiang
B.S., University of Science and Technology of China, Hefei, China, 1999
M.S., University of Science and Technology of China, Hefei, China, 2002
May 2008
To my parents,

and my family
Acknowledgements

In the journey of my dissertation research, I am deeply indebted to my faculty co-advisors, Dr. Wen Jin Meng and Dr. Glenn Sinclair for their dedication and support throughout my research. I am thankful to Dr. Meng for his direction on the experimental component of this thesis. I am grateful to Dr. Sinclair for sharing his insights on finite element analysis.

I would like to thank Dr. Dorel Moldovan, Dr. Yitshak Ram, Dr. Suresh Moorthy, and Dr. Luis Lehner to be my dissertation committee members. I thank you all for your time and effort in the process of completing my dissertation.

I also thank Dr. Edgar Lara-Curzio and Chris Stevens, who helped me in the execution of high temperature uniaxial tensile testing at the High Temperature Materials Laboratory of Oak Ridge National Laboratory.

The other members of Dr. Meng’s research group, Dongmei Cao, Bo Shi, Fanghua Mei, Bin Lu, Yang Mu, have been my valuable partners.

# Table of Contents

Dedication ................................................................................................................................. ii  

Acknowledgments .................................................................................................................. iii  

List of Tables ............................................................................................................................. vi  

List of Figures ........................................................................................................................... vii  

Abstract ........................................................................................................................................ x  

Chapter 1. Introduction. ............................................................................................................. 1  
1.1 Overall Objective .................................................................................................................. 1  
1.2 Microfabrication Techniques ............................................................................................... 2  
1.2.1 Photolithography ........................................................................................................... 2  
1.2.2 LIGA Technique ............................................................................................................ 3  
1.2.3 Micro-Injection-Molding ............................................................................................... 4  
1.2.4 Microcasting ................................................................................................................. 5  
1.2.5 Micromilling .................................................................................................................. 6  
1.2.6 µEDM ............................................................................................................................ 7  
1.2.7 Selection of Microfabrication Techniques for Metal-Based Microstructures ............... 7  
1.3 Insert Fabrication Techniques ............................................................................................. 8  
1.3.1 Insert Fabrication (w/o Surface Engineering) ............................................................... 9  
1.3.1.1 Insert Fabricated by LIGA ......................................................................................... 9  
1.3.1.2 Insert Fabricated by µEDM ....................................................................................... 10  
1.3.1.3 Insert Fabricated by Combining of LIGA with µEDM ............................................ 11  
1.3.2 Surface Engineering of Mold Insert ............................................................................. 11  
1.4 Potential Applications ......................................................................................................... 13  
1.5 References ......................................................................................................................... 14  

Chapter 2. Fabrication of High-Aspect-Ratio Microscale Ta Mold Inserts with µEDM .......... 18  
2.1 Introduction ......................................................................................................................... 18  
2.2 Experimental Procedures ................................................................................................... 19  
2.3 Results and Discussion ....................................................................................................... 23  
2.4 Summary ............................................................................................................................ 34  
2.5 References ......................................................................................................................... 34  

Chapter 3. Fabrication of High-Aspect-Ratio Microscale Mold Inserts by Parallel µEDM .... 36  
3.1 Introduction ......................................................................................................................... 36  
3.2 Experimental Procedures ................................................................................................... 37  
3.3 Results and Discussion ....................................................................................................... 41  
3.4 Summary ............................................................................................................................ 51  
3.5 References ......................................................................................................................... 52
List of Tables

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Table 5.1</td>
<td>Element numbers in meshes</td>
<td>85</td>
</tr>
<tr>
<td>Table 5.2</td>
<td>Normalized peak contact stresses, $\sigma_{\text{max}}$, for the two load levels</td>
<td>88</td>
</tr>
<tr>
<td>Table 5.3</td>
<td>Load step convergence check of pressure, $p$ (MPa)</td>
<td>92</td>
</tr>
</tbody>
</table>
# List of Figures

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure 1.1</td>
<td>Schematic of the photolithography process</td>
<td>2</td>
</tr>
<tr>
<td>Figure 1.2</td>
<td>LIGA process</td>
<td>4</td>
</tr>
<tr>
<td>Figure 1.3</td>
<td>Sketch of microcasting process</td>
<td>5</td>
</tr>
<tr>
<td>Figure 1.4</td>
<td>Ni microposts array made by LIGA</td>
<td>9</td>
</tr>
<tr>
<td>Figure 1.5</td>
<td>Molded Pb by Ni microposts</td>
<td>10</td>
</tr>
<tr>
<td>Figure 1.6</td>
<td>Ta insert made by µEDM</td>
<td>10</td>
</tr>
<tr>
<td>Figure 1.7</td>
<td>As-electrodeposited Ni pattern</td>
<td>12</td>
</tr>
<tr>
<td>Figure 1.8</td>
<td>Ta pattern transferred from Figure 1.7</td>
<td>12</td>
</tr>
<tr>
<td>Figure 2.1</td>
<td>Ta blank insert</td>
<td>19</td>
</tr>
<tr>
<td>Figure 2.2</td>
<td>High precision µEDM with digital control panel</td>
<td>19</td>
</tr>
<tr>
<td>Figure 2.3</td>
<td>Electro-chemical-polishing setup</td>
<td>20</td>
</tr>
<tr>
<td>Figure 2.4</td>
<td>Deposition setup, including load lock, ICP generators, and balanced magnetron sources</td>
<td>21</td>
</tr>
<tr>
<td>Figure 2.5</td>
<td>The custom built, high vacuum, high temperature, instrumented micromolding machine</td>
<td>22</td>
</tr>
<tr>
<td>Figure 2.6</td>
<td>An SEM image of one as-µEDMed Ta blank</td>
<td>23</td>
</tr>
<tr>
<td>Figure 2.7</td>
<td>Typical surface morphology of the as-µEDMed Ta blank surfaces</td>
<td>24</td>
</tr>
<tr>
<td>Figure 2.8</td>
<td>TEM characterization of the modified surface layer of the as-µEDMed Ta piece</td>
<td>24</td>
</tr>
<tr>
<td>Figure 2.9</td>
<td>Chemical characterization of the as-µEDMed Ta surface with XPS</td>
<td>26</td>
</tr>
<tr>
<td>Figure 2.10</td>
<td>Typical surface morphology of the as-ECPed Ta blank surfaces</td>
<td>29</td>
</tr>
<tr>
<td>Figure 2.11</td>
<td>Typical surface morphology of the Ti-C:H coated Ta insert surfaces</td>
<td>30</td>
</tr>
<tr>
<td>Figure 2.12</td>
<td>Molded features on the Al disk molded at 361 °C</td>
<td>31</td>
</tr>
<tr>
<td>Figure 2.13</td>
<td>Molded features on the Cu disk molded at 410 °C</td>
<td>32</td>
</tr>
</tbody>
</table>
Figure 5.2 Aluminum stress strain curves at 360°C: (a) $\dot{\varepsilon} = 0.0003/s$, (b) $\dot{\varepsilon} = 0.001/s$ and $\dot{\varepsilon} = 0.005/s$ ................................................................. 81

Figure 5.3 Approximation of deformation in aluminum after indentation........................................... 82

Figure 5.4 Coarse meshes: (a) indentation application, (b) single punch test problem.. 85

Figure 5.5 Comparison of FEA and exact contact stresses for the single punch test problem................................................................. 89

Figure 5.6 Comparison of FEA and analytical interior stresses for the periodic test problem on the $y$-axis.............................................................................. 90

Figure 5.7 Molding response: normalized molding pressure $p/\sigma_Y$ vs normalized molding depth $d/b$ with different meshes..................................................................................... 93

Figure 5.8 Comparison of molding response: FEA and experiment................................. 94

Figure 5.9 Molding response regimes......................................................................................................................... 98

Figure 5.10 Contact stress distribution at different indentation depth: (a) coarse grid, (b) fine grid................................................................. 99

Figure 5.11 Contact stress distributions in the fully plastic regime................................. 100

Figure 5.12 FEA interior stresses on the $y$-axis: $\sigma_y/p$ and $\sigma_x/p$............................................. 101

Figure 5.13 Yield region propagation (a) Yield region depths for $p/\sigma_Y = 3.2$ (b) Propagation of yield region depths................................................................. 101

Figure 6.1 Room temperature replication of microscale Al structures by compression molding: (a) an intact a-Si:N coated Si insert after one Al molding; (b) the corresponding replicated Al structure................................................................. 108

Figure 6.2 Geometry for the FEA model of indentation of an elasto-plastic slab by a periodic array of rectangular punches................................................................. 109

Figure 6.3 Al1100O stress strain curve (room temperature).................................................. 111

Figure 6.4 Progression of applied pressure as indentation progresses into the elasto-plastic regime................................................................. 111

Figure 6.5 Progression of contact stress concentration as indentation progresses into the elasto-plastic regime. The two insets show $\sigma_c$ distributions at $d/b$ values of $4 \times 10^{-5}$ and 0.85, respectively. The arrows link the insets to their corresponding $K_c$ values................................................................. 112
Abstract

Fabrication of microscale Ta mold inserts by micro-electrical-discharge-machining (µEDM) is reported. Morphology, chemistry, and structure of the near-surface region of as-machined Ta blanks have been characterized by scanning electron microscopy, X-ray photoelectron spectroscopy, and transmission electron microscopy. A TaC surface layer forms on as-machined Ta surfaces. This altered surface layer was removed by electro-chemical-polishing. Further modification of Ta insert surfaces was accomplished by deposition of a conformal Ti-containing hydrogenated carbon coating. We demonstrate successful replication of high-aspect-ratio microscale structures (HARMS) in Al and Cu by compression molding with such surface-engineered Ta mold inserts. In addition, a hybrid microfabrication technique, combining micropattern definition with LIGA (Lithographie, Galvanof ormung, Abformung) fabricated Ni microstructures with parallel micropattern generation with µEDM, was used to fabricate micropattern with some geometrical complexity on elemental Ta and 304 stainless steel.

Also, the results of instrumented micromolding of Al are studied. Measured molding response was rationalized with companion high-temperature tensile testing of Al using a simple mechanics model of the micromolding process. The present results suggest that stresses on the mold insert during micromolding are determined primarily by the flow stress of the molded metal at the molding temperature and the frictional traction on the sides of the insert. The influence of strain rate was also considered.

In addition, the elasto-plastic response of an Al block indented by a periodic array of long smooth strip punches made of a relatively rigid material is studied through finite element analysis (FEA). First, elastic test problems, for which analytical solution exist, are carried out to
calibrate the FEA mesh. Results demonstrate that satisfactory accuracy is achieved for key, peak, contact stresses near the edge-of-contact region and interior stresses. Second, indentation response is tracked with FEA into the elasto-plastic regime. Results show that the yield region within the indented material approaches a self-similar state as indentation progresses.

Finally, Al molded by Si inserts at room temperature is studied through experiment and FEA.
Chapter 1. Introduction

1.1 Overall Objective

Metal-based high-aspect-ratio microscale structures (HARMS) have potential applications as building blocks in micro-heat-exchangers, micro-electromagnetic-relays, and micro-chemical-reactors, etc. Compared with HARMS based on Si and plastics, metal-based HARMS have high strengths and high thermal conductivities. One key to commercial realization of any of those microdevice prototypes is a low-cost, high-throughput fabrication technology. Replication of metal-based HARMS by compression molding is proposed in this study. We will fabricate Al-based HARMS by compression molding, study the mechanics of molding process, and simulate the molding process through a commercial finite element analysis (FEA) code- ANSYS. In the following, we briefly review current microfabrication techniques and motivate the technique of replication by compression molding.

1.2 Microfabrication Techniques

Microfabrication is a collection of technologies which are utilized in making microdevices. It has wide-ranging applications in various fields of engineering and science. Historically, the bulk of research activities in microfabrication have focused on techniques originated from fabrication of microelectronic devices,\(^1\) notably the technique of photolithography.\(^2\) Its main application is producing three dimensional microscale structures out of Si by bulk and surface micromachining. In the past two decades, applications of microelectromechanical systems (MEMS) have gained increasing attention in both technological and commercial areas. Development of MEMS requires sound fabrication and manufacture technologies for making three dimensional structures and devices with overall dimensions on the order of a few to a few hundred micrometers. As a result of the intense research over the past two decades, a number of new
technologies have been developed and put into practice. These technologies include LIGA technique,\textsuperscript{2, 3, 4} micro-injection-molding,\textsuperscript{5, 6, 7, 8, 9, 10} microcasting,\textsuperscript{10, 11, 12, 13, 14} precision micromilling,\textsuperscript{15, 16, 17, 18} micro-casting,\textsuperscript{10, 11, 12, 13, 14} micro-electrical-discharge-machining (µEDM),\textsuperscript{19, 20, 21} etc. Other technologies, such as compression molding or hot embossing\textsuperscript{22} and laser ablation,\textsuperscript{23} show promising results as well. This section reviews the present status of relevant technologies as reported in the literature.

1.2.1 Photolithography

Photolithography is a basic technique which can be used to transfer a pattern from a photomask to the surface of a substrate. It has a similarity to the conventional lithography used in printing and shares some of the fundamental principles of photographic process. Photolithography involves a combination of mask fabrication, substrate preparation, spin coating of photoresist, pre-baking, exposure, post-baking, and developing. The main procedure is illustrated in Figure 1.1.

![Figure 1.1 Schematic of the photolithography process\textsuperscript{2}](image)
Mask fabrication is the first step of photolithography. For the application of ultraviolet (UV) lithography, a blank mask consists of a substrate glass deposited with a chromium (Cr) film and a photoresist, for example AZ®. Pattern can be defined through a Pattern Generator followed by etching. Clean Si wafer is the most commonly used substrate. Hydrofluoric acid (HF) and nitric acid (HNO₃) are used to remove the oxide layer on the surface if necessary. With a spinner, photoresist can be coated on top of the Si wafer. The desired thickness depends on the viscosity of photoresist and the spin speed. After baking, the photoresist is exposed under an illumination source for some time, which is based on the photochemical properties and thickness of the photoresist. After the exposed photoresist is baked, the next step is to dissolve the exposed (positive photoresist) / unexposed (negative photoresist) photoresist. The developing time is the key to obtaining a high quality structure. In general, the quality of the product depends on the exposing light source, type of photoresist, the dimension and aspect ratio of the structure. The final step is etching of the uncovered area.

1.2.2 LIGA Technique

LIGA is the German acronym for lithography (lithographie), electroplating (galvanof ormung), and molding (abformtechnik). LIGA was initiated by the Karlsruhe Nuclear Research Center (Kernforschungszentrum Karlsruhe) in Germany and X-ray lithography was employed in the original version of the technique. As has been discussed in 1.2.1, the X-ray lithography involves a thick layer of X-ray resist, high-energy X-ray radiation exposure and development. A three dimensional resist structure will be achieved after the X-ray lithography is finished. Subsequent electroplating fills the resist mold with a metal and, after resist removal, a free standing metal structure results. The metal shape may be a final product or serve as a mold insert for precision plastic injection or compression molding. Figure 1.2 (see next page) shows
the LIGA process schematically. It is capable of creating very finely defined microscale structures of over 1000µm in height.

(a) Synchrotron radiation

(b) Development

(c) Electroplating

(d) Mold insert

(e) Mold filling

(f) Mold release

Figure 1.2 LIGA process

Due to the high cost of synchrotron radiation and the slow speed of electrodeposition, metal microscale structures made from electrodeposition into deep resist structures made by X-ray lithography are very expensive. This limits the application of LIGA for mass production.

1.2.3 Micro-Injection-Molding

Micro-injection-molding is a manufacturing technique for making parts from thermoplastics, metal and ceramic powders. Micro-injection-molding technique was originally used to fabricate compact discs (CDs). In recent years, it has been further developed into an autonomous technology with novel machines and processes being designed for various microtechnical applications.

For micro-injection-molding of thermoplastic polymers, molten plastic is injected at a high pressure into a mold, which contains the negative pattern of the desired shape. After the plastic is cooling down, the desired microscale structure is achieved. Several processes are
employed for this technology: Injection molding, reaction injection molding, injection compression molding, and thermoforming.

For micro-injection-molding of metal and ceramic powders, the fine powder materials are mixed with a binder in approximately equal volumetric quantities to form a feedstock. The binder normally is a mixture of mostly organic components with different molecular weights. It allows plastic flow of the feedstock during shaping by injection molding to the green compacts. After the feedstock was injected into the mold, debinding is done by pyrolysis, by extraction with water of an organic solvent, or by catalytic depolymerization of one or more of the binder components. Sometimes these processes are used in combination. Debinding is followed by sintering the loose parts to dense compacts. The heating and cooling rates and dwell time at various temperatures play significant roles in sintering.

1.2.4 Microcasting

Microcasting is an alternative process for the production of metallic microstructure. Compared to other microfabrication techniques, microcasting extends the material range from electroformable metals and alloys to nearly all meltable alloys. Figure 1.3 shows the general steps of microcasting process.

(a) plastic master, (b) embedded in ceramic slip, (c) hollow form, (d) metal melt filled mold, (e) single cast structure
First, a plastic master is fabricated by injection molding (a). Second, the plastic master is embedded in a ceramic slip (b). Next, after drying the ceramics mold is sintered and during this process the master is lost by melting and pyrolysis (c). Then, the preheated ceramic mold is filled with a metal melt by centrifugal or pressure casting (d). Finally, the cast structure is cleaned and the microscale structure is separated after removing the mold from the casting.

The final cast structure loses some quality as compare to the master. Surface roughness is sometimes a problem. The grain size, microstructure and mechanical strength of cast structures are significantly affected by mold temperature.

1.2.5 Micromilling

In conventional machining, the milling process is very versatile and capable of creating three-dimensional features and structures. Adaptation of this process at the microscale leads to the rapid and direct fabrication of micromolds. It is used to manufacture microscale structures and tooling inserts for micro-injection-molding and hot embossing. As compared to other microfabrication techniques, the advantages of ultra-precision micromilling techniques include accurate machining of complex features, such as curvilinear shapes, and the ability to work with a variety of workpiece geometries. The minimum size of the machined structure is subject to the size of the end mill. Recent study demonstrates that end mill with 10µm in diameter was achieved through fabrication with a focus ion beam (FIB).\(^{15}\)

A number of manufacturing constraints limit the application of this technology. The primary disadvantage of micromilling is the expense and the upkeep of a delicate milling machine. Another main constraint is that micromilling is a serial substrative manufacturing technique, in which cuts are made sequentially. This leads to long machining time and frequent tool replacements.
1.2.6 μEDM

μEDM is a machining method in which a voltage is applied through a dielectric fluid medium between the tool electrode and the workpiece. An electrical discharge is generated when the electrode and workpiece are positioned close to each other. A section of the workpiece is melted/vaporized and removed by the electrical discharge. The spark temperature is reported to be in the range of 8000 to 12000°C.\(^5\) Machining is carried out by repeated melting/vaporizing and removal with a pulsating electrical discharge.

μEDM enables machining to tolerances of a few microns, which makes this technique highly competitive with other technologies, such as laser ablation. This process is also well suited for machining hard materials which may not be feasible using conventional cutting equipment. The process can also produce very smooth surfaces by reducing the discharge intensity while reducing the gap between electrode and component to sub-micron levels.

The limitation of μEDM is that the workpiece has to be electrically conductive.

1.2.7 Selection of Microfabrication Techniques for Metal-Based Microstructures

In the above discussion, several microfabrication techniques are summarized. Now, we need to think about which fabrication technique we should choose to fabricate metal-based HARMS quickly and easily.

In fact, each microfabrication technique has its own limited application range. Electroforming of lost plastic mold (first two steps of LIGA), micro-injection-molding and microcasting have more complicated manufacturing processes, which cause the microfabrication to be more time-consuming and of higher cost. Micromilling and μEDM are more direct machining techniques. However, with μEDM, the process itself is time-consuming; with micromilling, efficiency, tool wear, burr occurrence, and surface roughness of the micromachined structures are, and remain to be, challenging issues. Therefore, a more direct, less time-consuming, and more cost-
effective microfabrication technique needs to be developed for low-cost, high-throughput production of metallic HARMS.

The conventional LIGA process protocol includes molding plastics-based structure as the end step. A similar idea was proposed by Cao et al.,\textsuperscript{26, 27} in which metals are molded instead of plastics. They fabricated an array of Ni microposts on a substrate with LIGA technique and used it as the mold insert to directly fabricate metal structures on Pb and Zn plates by compression micromolding. Their results show that this LIGA-like technique is feasible, valid, and efficient. The present study is based on this idea. In addition, stronger mold inserts are made by µEDM or combination of µEDM and LIGA.

1.3 Insert Fabrication Techniques

This section focuses on the techniques which are used to fabricate inserts used in our current research in high temperature metal micromolding.

In the process of microscale compression molding of metals, molding inserts play a significant role. In general, the molding process occurs at high temperatures, for example, 400°C to 500°C for Al and Cu molding. Here two issues of material selection need to be considered. The first is that the insert needs to be strong enough to sustain the high stresses during molding and demolding, i.e., no plastic deformation should occur during this process. The second is the chemical/mechanical properties of the near-surface regions of the insert must be adequate to allow repeated molding cycles without damage to the insert and the molded metal part, i.e., no chemical reaction should occur between the mold insert and the molded material and the wear rate and friction force should be small enough in the whole process. These two issues form the challenges that the present study aims to address.
1.3.1 Insert Fabrication (w/o Surface Engineering)

Based on the first consideration, strong and refractory metals are considered. Several microfabrication technologies were applied to fabricate mold inserts. In what follows, a review of these related microfabrication techniques is given.

1.3.1.1 Insert Fabricated by LIGA

Good mechanical properties and electroformable property make Ni the first consideration for mold insert fabrication. In fact, Ni has a relatively high yield stress, 370MPa at room temperature, enough for many molding applications; its melting temperature is 1455°C, which is relatively high comparing with Pb and Al and guarantee it to be strong enough during Pb and Al molding.

Cao etc used an array of Ni microposts made by LIGA as the mold inserts (Figure 1.4) to transfer the pattern onto Pb (Figure 1.5, see next page).26

![Figure 1.4 Ni microposts array made by LIGA](image)

Figure 1.4 Ni microposts array made by LIGA26
An alternative is to use refractory metals for inserts. Fabrication can be accompanied with µEDM. We chose Ta as the insert bulk. The melting temperature of Ta is 2996°C. Figure 1.6 shows an example of a Ta insert.
1.3.1.3 Insert Fabricated by Combining of LIGA with μEDM

LIGA-made Ni insert and μEDM-made Ta insert have their limits: For electrodeposited Ni insert, its structure is micro-/nano-crystalline and will undergo significant grain growth at elevated temperatures. After one molding experiment, the insert undergoes one annealing, and the yield strength decreases significantly. This decrease limits the molding temperature. For μEDM-made Ta insert, the shortcoming is obvious: Only simple geometric patterns are possible. We proposed to combine LIGA with μEDM to machine molding inserts. We first define a negative pattern using Ni by LIGA (Figure 1.7, see next page), followed by pattern transfer onto Ta substrate by μEDM (Figure 1.8, see next page).

1.3.2 Surface Engineering of the Mold Insert

Based on the second consideration, hard coatings were deposited on the mold inserts to improve their surface chemical/mechanical properties. Here the coating must satisfy two criteria. First, the coating needs to be synthesized with desired mechanical, tribological, and chemical properties and it must adhere strongly to the surface. Second, the coating needs to cover topologically complex surfaces in a conformal fashion.

Plasma assisted vapor deposition of amorphous hydrogenated carbon (a-C:H) and metal-containing hydrocarbon (Me-C:H) thin films have been studied for over a decade. By incorporating several atomic percent of metals, for example, Ti or W, the microstructure of a-C:H thin films has been shown to consist of nanocrystalline metal carbide clusters embedded in an a-C:H matrix (MeC/a-C:H). Me-C:H thin films possess moderately high hardness, low friction coefficient, and low wear rate, and have been shown to be beneficial when applied to macro-scale mechanical components. Recently, a low pressure, high-density, inductively coupled plasma (ICP) assisted hybrid chemical/physical vapor deposition (CVD/PVD) technique has been used to deposit a-C:H
and Ti-C:H thin films over a wide range of compositions. We used this Ti-C:H coating by ICP assisted hybrid CVD/PVD technique to modify the near surface properties of the mold insert.

Figure 1.7 As-electrodeposited Ni pattern

Figure 1.8 Ta pattern transferred from Figure 1.7
1.4 Potential Applications

Replication of HARMS by direct compression molding is fast and simple and offers potential advantages in production cost and throughput. Potential applications include: Micro-heat-exchangers for electronic cooling systems, micro-electromagnetic-relays, etc.

Increasing miniaturization of semiconductor devices has led to increased power densities. The resulting higher device temperatures have become a serious problem affecting the performance and reliability of electrical devices. In the electronic cooling system design, the most important goal is to reduce the overall thermal resistance. Tuckerman and Pease attached a Si-based heat sink with microchannels to the inactive side (back) of an electronic chip and arranged a close-loop for water, which is pumped through the microchannels to take away the heat generated. Due to the small size of the microchannels, the heat transfer coefficient is very high. Tuckerman and Pease demonstrated that this setup had very small thermal resistance (as low as $9 \times 10^{-6} \text{K/(W/m}^2\text{)}$) for a pumping power of 1.84W. Our replication technique matches the needs for microchannel heat exchangers very well. Cu-based and Al-based HARMS have very high thermal conductivities and better mechanical properties than Si and can be used in harsh environments, such as high vibration and high temperatures.

Another potential application is micro-electromagnetic-relay fabrication. Most MEMS relays are based on silicon fabrication and cannot be used for power applications. Ni-based micro-electromagnetic-relays made by UV-LIGA have been reported. It can be used for power applications. As has been discussed in former sections, LIGA is a time-consuming process and has low product throughput. Considering our fabrication strategy of combining of LIGA and \(\mu\)EDM, we can first fabricate a Ni-based negative pattern of desired pattern by UV-LIGA. Second, the Ni pattern will be transferred to Ta or other refractory metals. The successful
transfer of complex pattern has been shown in Figures 1.7 and 1.8. Third, we can try to mold Ni using the insert made with former method. Pb, Al, and Cu have already been molded successfully. Ni molding will be investigated by increasing the molding temperature and using high-quality inserts. Since the molding insert can be used repeatedly, the cost of replicating one micro-electromagnetic-relay should be lowered.

1.5 References

1 W. M. Moreau, Semiconductor lithography: principles and materials, Plenum, New York, 1988

2 M. Madou, Fundamentals of Microfabrication, CRC Press LCC, 1997


6 L. Merz, S. Rath, V. Piotter, R. Ruprecht, J. Haußelt, Powder injection molding of metallic and ceramic microparts, Microsystem Technologies, 10(3), 202-204, March, 2004


<table>
<thead>
<tr>
<th>Reference</th>
<th>Authors</th>
<th>Title</th>
<th>Journal</th>
<th>Volume Issue</th>
<th>Page Numbers</th>
<th>Publication Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>G. Baumeister, K. Mueller, R. Ruprecht, J. Hausselt</td>
<td>Production of metallic high aspect ratio microstructures by microcasting</td>
<td>Microsystem Technologies</td>
<td>8(2-3)</td>
<td>105-108</td>
<td>May, 2002</td>
</tr>
<tr>
<td>12</td>
<td>G. Baumeister, R. Ruprecht, J. Hausselt</td>
<td>Replication of LIGA structures using microcasting</td>
<td>Microsystem Technologies</td>
<td>10(6-7)</td>
<td>484-488</td>
<td>October, 2004</td>
</tr>
<tr>
<td>14</td>
<td>G. Baumeister, R. Ruprecht, J. Hausselt</td>
<td>Microcasting of parts made of metal alloys</td>
<td>Microsystem Technologies</td>
<td>10(3)</td>
<td>261-264</td>
<td>March, 2004</td>
</tr>
<tr>
<td>16</td>
<td>C. Friedrich, P. Coane, M. Vasile</td>
<td>Micromilling development and applications for microfabrication</td>
<td>Microelectronic engineering</td>
<td>35(1-4)</td>
<td>367-372</td>
<td>February, 1997</td>
</tr>
<tr>
<td>17</td>
<td>D. Adams, M. Vasile, G. Benavides, A. Campbell</td>
<td>Micromilling of metal alloys with focused ion beam-fabricated tools</td>
<td>Precision Engineering – Journal of the International Societies for Precision Engineering and Nanotechnology</td>
<td>25(2)</td>
<td>107-113</td>
<td>April, 2001</td>
</tr>
<tr>
<td>18</td>
<td>A. Tseng</td>
<td>Recent developments in micromilling using focused ion beam technology</td>
<td>Journal of Micromechanics and Microengineering</td>
<td>14(4)</td>
<td>R15-R34</td>
<td>April, 2004</td>
</tr>
<tr>
<td>19</td>
<td>K. Takahata, N. Shibaike, H. Guckel</td>
<td>High-aspect-ratio WC-Co microstructure produced by the combination of LIGA and micro-EDM</td>
<td>Microsystem Technologies</td>
<td>6(5)</td>
<td>175-178</td>
<td>August, 2000</td>
</tr>
</tbody>
</table>

24 MicroChemicals company, Germany

25 D. M. Cao, Replication of metal-based high-aspect-ratio microscale structures by high temperature micromolding, Louisiana State University, Electronic Thesis & Dissertation Collection, etd-07012004-160623, August, 2004

26 D. M. Cao, W. J. Meng, K. W. Kelly, High-temperature instrumented microscale compression molding of Pb, Microsystem Technologies, 10(4), 323-328, May, 2004

27 D. M. Cao, D. Guidry, W. J. Meng, K. W. Kelly, Molding of Pb and Zn with microscale mold inserts, Microsystem Technologies, 9(8), 559-566, October, 2003

28 D. M. Cao, W. J. Meng, Microscale compression molding of Al with surface engineered LiGA inserts, Microsystem Technologies, 10(8-9), 662-670, November, 2004

29 D. M. Cao, J. Jiang, R. Yang, W. J. Meng, Fabrication of high-aspect-ratio microscale mold inserts by parallel µEDM, Microsystem Technologies, 12(9), 839-845, August, 2006


32 K. Bewilogua, H. Dimigen, Preparation of W-C-H coatings by reactive magnetron sputtering, Surface Coating Technology, 61(1-3), 144-150, 1993


36 W. J. Meng, E. I. Meletis, L. E. Rehn, P. M. Baldo, Inductively-coupled plasma assisted deposition and mechanical properties of metal-free and Ti-containing hydrocarbon coatings, Journal of Applied Physics, 87(6), 2840-2848, 2000


40 J. D. Williams, W. Wang, Microfabrication of an electromagnetic power relay using SU-8 based UV-LIGA technology, Microsystem Technologies, 10(10), 699-705, December, 2004
Chapter 2. Fabrication of High-Aspect-Ratio Microscale Ta Mold Inserts with µEDM

2.1 Introduction

Metal-based micro-electro-mechanical systems (MEMS) have important advantages over Si-based MEMS. Examples of metal-based passive and active MEMS include micro-heat-exchangers\(^1\) and micro-electromagnetic-relays.\(^2\) Construction of these nontraditional metal-based MEMS often requires the fabrication of metal-based high-aspect-ratio microscale structures (HARMS), with heights on the order of several hundreds µm. Fabrication technologies which can produce metal-based HARMS economically and with high fidelity are critical to commercial realization of metal-based MEMS.

The micro-electrical-discharge-machining (µEDM) process, derived from the conventional EDM process\(^3\) and adapted for micromachining, can generate HARMS features in a wide range of engineering materials. The µEDM process has been investigated for fabricating HARMS out of metals\(^4\) and electrically conducting ceramics,\(^5\) and offers a potential alternative for fabricating microscale mold inserts.

In this chapter, we report successful fabrication of Ta HARMS by µEDM. To determine the suitability of µEDMed Ta HARMS as mold inserts, detailed morphological, chemical, and structural characterization of as-µEDMed Ta surfaces was carried out. Engineering of as-µEDMed Ta surfaces was achieved through electro-chemical-polishing (ECP) followed by deposition of a conformal Ti-containing hydrogenated carbon (Ti-C:H) coating. Successful replication of HARMS in Al and Cu by compression molding with such surface-engineered Ta mold inserts was demonstrated.
2.2 Experimental Procedures

![Ta blank insert](image1.jpg)

Figure 2.1 Ta blank insert

![High precision µEDM with digital control panel](image2.jpg)

Figure 2.2 High precision µEDM with digital control panel

Ta mold inserts were fabricated from arc-melted Ta (99.9%) rods. As-received Ta rods were machined into square insert blanks, with a square active area of 9500µm×9500µm, 2000µm in height (Figure 2.1). The top surface of the active area on the insert blank was mechanically polished with SiC abrasive papers down to 1200 grit size. Insert fabrication from the Ta blank involved three main steps: µEDM of the active area, ECP of as-µEDMed microscale Ta features, and deposition of a conformal Ti-C:H coating over the microscale Ta features.

A SARIX High Precision Micro-Erosion Machine (Model SR-HPM-B) was used for Ta µEDM (Figure 2.2). Blade electrodes were made from 500µm thick Mo sheet metal. The µEDM parameters of discharge frequency, on-time width, and maximum voltage were set respectively at 150kHz, 4µsec, and 100V. A commercial fluid, IonoPlus/3000, was used as the dielectric medium for the µEDM process. According to the fluid supplier, this fluid is predominantly hydrocarbon-based, with trace amounts of sulphur.
As-μEDMed Ta blanks were ECPed for a total of 20min in a mixed acid solution of H₂SO₄/HF(49%) with a volume ratio of 6 to 1. ECP setup is shown in Figure 2.3. ECP was carried out in the constant-current mode, with the Ta blank acting as the anode and a graphite rod, 6.15mm in diameter, forming the counter electrode. ECP was conducted for 10min at 350mA (current density 90mA/cm²), during which the bias voltage ranged between 1.7 to 2.0V, and for an additional 10min at 200mA (current density 50mA/cm²), during which the bias voltage was ~1.3V.

Following ECP, a conformal Ti-C:H coating was deposited over the microscale Ta features in an inductively coupled plasma (ICP) assisted hybrid chemical/physical vapor deposition tool (Figure 2.4, see next page). ECPed Ta blanks were cleaned in acetone and methanol, loaded into the deposition chamber, and surface-etched for 5min in a pure Ar ICP. Immediately after the surface-etch, a pure Cr interlayer was deposited followed by a 10min deposition of a Ti-C:H top layer. Further details of the deposition process and characterization of conformal Ti-C:H coatings over HARMS have been reported previously.
Figure 2.4 Deposition setup, including load lock, ICP generators, and balanced magnetron sources

Scanning electron microscopy (SEM) examinations of Ta mold inserts in various stages of processing, as well as HARMS in Al generated by compression molding, were carried out on a Hitachi S-3600N microscope operated at 15kV. Surface chemical compositions of as-received Ta bulk and as-μEDMed Ta blank were analyzed with X-ray photoelectron spectroscopy (XPS) on a Kratos AXIS165 system. The monochromatic Al Kα excitation source was operated at an anode bias of 15kV and a current of 15mA. Depth profiling was accomplished by ion etching with an argon ion gun operated at a bias voltage of 5kV and a filament discharge current of 15mA. Depth profiling of as-μEDMed Ta surfaces was accomplished by up to 34 Ar⁺ etching cycles, the duration of each cycle was 20min. XPS spectra were collected from the as-μEDMed Ta surface before the first etch cycle and after each etch cycle. One survey spectrum was collected at a spectral resolution of 0.5eV/channel and a speed of 100ms/channel. Two high-resolution scans of the Ta4f and C1s spectral regions were acquired at a spectral resolution of 0.1eV/channel and a speed of 300ms/channel. The specimen representing the as-received Ta bulk
was prepared through mechanically cutting, mechanical polishing, and ECP in an H$_2$SO$_4$/HF(49%) solution with a volume ratio of 6 to 1.

Figure 2.5 The custom built, high vacuum, high temperature, instrumented micromolding machine

Structural characterization of as-µEDMed Ta surfaces was carried out with cross-sectional transmission electron microscopy (TEM). Two cross-sectional TEM specimens were prepared and examined on a JEOL JEM2010 microscope operated at 200kV. One specimen was prepared by gluing the as-µEDMed Ta surface to a Si wafer slice, and another by gluing the as-µEDMed Ta surface to a slice of mechanically polished bulk Ta. Both specimens were prepared following standard procedures of mechanical polishing followed by ion milling.\(^8\)

Compression micromolding of pure Al (Al-1100H14, 99%) and pure Cu (99.9%) with Ti-C:H coated Ta mold inserts were carried out in a high-vacuum, high-temperature, compression molding apparatus (Figure 2.5).\(^9\) Al and Cu starting materials were in the form of circular disks,
35.5mm in diameter and 6.4mm in thickness. Top surfaces of Al and Cu disks were mechanically polished with SiC abrasive papers down to 1200 grit size prior to being molded.

2.3 Results and Discussion

The overview of a typical as-µEDMed Ta blank and surface morphology of one typical rectangular protrusion are shown in the SEM micrograph of Figure 2.6 and Figure2.7 (see next page).

The µEDM process created trenches on the top surface of the active area of the Ta blank, and resulted in an array of 12, nominally-identical, rectangular protrusions (Figure 2.6). Each rectangular protrusion has dimensions of ~400µm in height, ~9500µm in length, and ~150µm in width. The protrusion center-to-center spacing is ~780µm. Figure 2.7(a) shows a higher magnification SEM micrograph of one typical rectangular protrusion. No change in morphology of the protrusion top surface is observed. The sidewall surface of the protrusion is clearly modified by the µEDM process. Figure 2.7(b) shows the presence of nodules and cracks on the protrusion sidewall surface. Similar surface morphologies are observed at the bottom of the
Figure 2.7 Typical surface morphology of the as-µEDMed Ta blank surfaces trench. Figure 2.7 shows that a modified layer exists on the portion of the as-µEDMed Ta surface in contact with the arc discharge, regardless of surface orientation. From cross-sectional SEM examinations, the thickness of the modified surface layer is estimated to be less than 5µm.

Figure 2.8(a) shows a low magnification TEM bright-field (BF) micrograph of the near-surface region of an as-µEDMed Ta piece. The presence of a surface crack is evident, in agreement with SEM observations of µEDM induced surface cracks shown in Figure 2.8. Figures 2.8(b) and 2.8(c) (see next page) show selected area diffraction patterns (SADP) obtained from one near-surface region in two orientations. Both SADPs can be indexed to a
cubic system, with a lattice parameter of ~4.45Å. The SADP shown in Figure 2.8(c) indicates that the structure of the near-surface region exhibits face-centered-cubic symmetry. These results are thus incompatible with the as-µEDMed Ta near-surface region being elemental Ta with a body-centered-cubic symmetry. Considering that TaC possesses a B1 structure with a lattice parameter of 4.46Å, SADPs shown in Figures 2.8(b) and 2.8(c) suggest that the structure of the modified layer on the as-µEDMed Ta surface is B1-TaC. Consistent TEM results were obtained from both cross-sectional specimens.

Figure 2.9(a) (see next page) shows a series of XPS survey spectra obtained from an as-µEDMed Ta surface, and the same surface after 340min and 680min of Ar⁺ etching, respectively. For reference purposes, the spectrum obtained from an as-ECPed bulk Ta surface after a 5min Ar⁺ etch is also included. In addition to Ta4f, Ta4d, Ta4p, and Ta4s spectral signatures, Figure 2.9(a) shows that oxygen and carbon contaminants are present on the as-µEDMed Ta surface. The Ar⁺ etch completely removes the O1s signature, indicating that oxygen on the as-µEDMed Ta surface arises from specimen handling in air, and that there is little or no oxygen contamination within the near-surface modified layer. In contrast, the C1s signature remains
Figure 2.9 Chemical characterization of the as-μEDMed Ta surface with XPS (Figure con’d.)
even after 680 min of \( \text{Ar}^+ \) etching, indicating that carbon atoms are incorporated into the near-surface region of Ta after \( \mu \text{EDM} \). The absence of O1s or C1s signatures above the background level from the briefly \( \text{Ar}^+ \) etched, as-ECPed Ta bulk indicates that ECP is effective in removing contaminants from Ta surfaces, and confirms the purity of the Ta bulk.

The corresponding high-resolution C1s and Ta4f spectra are shown in Figures 2.9(b) and 2.9(c), respectively. The C1s peak obtained from the as-\( \mu \text{EDMed} \) Ta surface is located at a binding energy of 284.7 eV, consistent with the presence of graphitic carbon on the surface. The C1s peaks obtained from the same surface after multiple \( \text{Ar}^+ \) etching cycles exhibit a clear down-shift in binding energy, from 284.7 eV to 282.8 eV. The latter C1s binding energy is consistent with that of TaC.\(^{10}\) Similarly, the Ta4f\(_{7/2}\)/Ta4f\(_{5/2}\) doublet peaks obtained from the as-\( \mu \text{EDMed} \) Ta surface are located at binding energies of 23.3 eV and 25.2 eV, respectively. This doublet exhibits a down-shift in binding energies after multiple \( \text{Ar}^+ \) etch cycles to 22.8 eV and 24.6 eV. These binding energies, 22.8 eV and 24.6 eV, are again in agreement with previously observed values.
for TaC. The XPS data thus corroborate the TEM results, and indicate the formation of a surface TaC layer as a result of Ta µEDM. No C1s signature was detected from the briefly Ar⁺ etched, as-ECPed Ta bulk. The corresponding Ta4f doublet peaks are located respectively at 21.6eV and 23.4eV, in good agreement with those of elemental Ta. This again indicates that ECP is effective in removing contaminants from Ta surfaces, and confirm the purity of the Ta bulk.

The formation of the TaC surface layer helps to explain the presence of surface cracks after Ta µEDM. Although detailed formation mechanisms are presently unclear, one surmises that the TaC surface layer forms during the sudden surface temperature rise resulting from the arc discharge. The carbon atoms needed for the Ta-C reaction are supplied from the hydrocarbon-based dielectric fluid. The temperature decreases rapidly as one travels from the surface into the Ta bulk. During the cooling phase, the surface layer with the higher temperature undergoes more thermal contraction than the cooler bulk, and is therefore placed under a tensile thermal stress after cooling. The TaC surface layer is expected to be more brittle than the Ta bulk, and cracks as a result of such tensile thermal stresses. The nodular morphology of as-µEDMed Ta surfaces greatly increases its roughness, and the presence of numerous surface cracks weakens it structurally. The nodular and cracked surface is also unsuitable as a substrate for coating deposition. These factors make the as-µEDMed Ta blanks unsuitable for usage as mold inserts without further surface modification.

Therefore, as-µEDMed Ta blanks were further ECPed under conditions given above. Figure 2.10(a) (see next page) shows a SEM image of a typical µEDMed rectangular protrusion after the ECP process. Comparison with Figure 2.7(a) clearly shows that the TaC surface layer has been completely removed, and the protrusion sidewall surfaces are nodule- and crack-free.
Figure 2.10(b) shows a higher magnification view of the protrusion sidewall. The morphology of the as-ECPed Ta surface reflects the grain structure of the Ta bulk. Facets created on as-ECPed surfaces reflect differences in etch rates for different grains. Similar morphologies are observed on trench bottom surfaces. The width of the Ta protrusions decreased from ~170µm on the as-µEDMed Ta blank to ~160µm after ECP.

(a) Low-magnification view of one protrusion

(b) Close-up view of the sidewall surface

Figure 2.10 Typical surface morphology of the as-ECPed Ta blank surfaces

Figure 2.11(a) (see next page) shows a SEM image of a typical rectangular protrusion after depositing a conformal Ti-C:H/Cr coating over the as-ECPed Ta blank. Complete coating
Figure 2.11 Typical surface morphology of the Ti-C:H coated Ta insert surfaces

(a) Low-magnification view of one protrusion

(b) Close-up view of the sidewall surface

(c) High-magnification view of the Ti-C:H coating surface morphology
coverage is seen at transitions from the protrusion top surface to the sidewall surface. The higher magnification view of the sidewall surface shown in Figure 2.11(b) shows that the coating covers the large scale surface facets uniformly, leaving no exposed Ta metal. The fine scale surface morphology of the Ti-C:H coating, shown in the SEM image of Figure 2.11(c), consisting of μm/sub-μm scale undulations, is typical of plasma deposited Ti-C:H coatings.\textsuperscript{11}

The efficacy of Ti-C:H coated Ta inserts for compression molding of metals at elevated temperatures is demonstrated through examples shown in Figures 2.12 and 2.13 (see next page).
The molded features on Al, shown in Figure 2.12, were created at a disk temperature of ~361°C, with the insert heated to ~371°C. The molded features on Cu, shown in Figure 2.13, were created at a disk temperature of ~410°C, with the insert heated to ~423°C. Both molding runs followed the procedures reported in other papers. The SEM image in Figure 2.12(a) shows that the rectangular protrusions on the Ta insert were faithfully replicated into rectangular channels on the Al disk, with an average width of ~160µm. Depth of molding is measured to be ~220µm. The higher magnification view of one typical channel on the Al disk shown in Fig
2.12(b) demonstrates that molded Al features have sharp sidewall to bottom transitions. Similar morphologies are observed on the Al disk top surfaces and the bottom surfaces of channels, suggesting little or no material flow at the surface during molding. Sidewall roughness observed in Figure 2.12(b) originates partly from roughness of the protrusion sidewalls on the Ta insert. Detailed mechanisms responsible for sidewall roughness generation remain to be elucidated. Similar discussions on sidewall roughness apply to molded features on the Cu disk shown in Figure 2.13. The depth of molding on the Cu disk is ~350µm. The tilted low magnification view of the molded features on Cu disk in Figure 2.13(a) shows that the top surface between two channels is somewhat convex upward, indicating that material pile-up exists during compression molding of Cu. Similar observations regarding pile-up are made in molded Al. Figure 2.13(b) demonstrates that sidewall, bottom, and even corners of channels in Cu are clean. No foreign material is trapped in the channels, as corroborated by energy dispersive X-ray spectroscopy analysis. Although replication of Al HARMS by micromolding was reported in 2004,\textsuperscript{12} to our knowledge, this is the first time that successful replication of Cu-based HARMS by high temperature micromolding is demonstrated.

The molding stress or applied pressure is calculated by dividing the total molding force with the normal contact area between the insert and the metal disks during molding. For the case of Al molding at ~361°C, the maximum molding stress is ~46MPa. In contrast, the maximum molding stress reached as high as ~187MPa during the Cu molding at ~410°C. Even under such high stresses, no visible deformation of the Ti-C:H coated Ta inserts was observed after Al and Cu molding, demonstrating sufficient strength of the Ta inserts in molding of Al and Cu at elevated temperatures. The present results show that μEDM is an effective alternative for fabricating refractory metal based HARMS inserts for high temperature metal micromolding. To
prolong the life of surface engineered refractory metal mold inserts and improve the quality of replicated features, the insert fabrication process needs to be optimized. Future optimization would include tuning the \( \mu \)EDM parameters to decrease the thickness of the modified surface layer and the surface roughness, altering the ECP process to remove the modified surface layer more efficiently and leave a smoother surface, and investigating coating chemistry and structure to increase coating adhesion and wear resistance.

2.4 Summary

Successful fabrication of surface engineered, microscale, Ta mold inserts was demonstrated with a three step process of \( \mu \)EDM, ECP, and conformal Ti-C:H coating deposition. Characterization of the morphology, chemistry, and structure of the near-surface region of as-\( \mu \)EDMed Ta blanks showed the formation of a TaC surface layer on as-machined Ta surfaces. Further surface engineering of as-\( \mu \)EDMed Ta blanks by ECP and conformal coating deposition yields Ti-C:H coated Ta mold inserts. Successful replication of HARMS in Al and Cu was achieved by compression molding with such surface-engineered Ta mold inserts.

2.5 References

1 C. Harris, K. Kelly, T. Wang, A. McCandless, S. Motakef, Fabrication, modeling, and testing of micro-cross-flow heat exchangers, JMEMS 11, 726, 2002

2 J. D. Williams, W. Wang, Microfabrication of an electromagnetic power relay using SU-8 based UV-LIGA technology, Microsystem Technologies, 10(10), 699-705, December, 2004


4 G. L. Benavides, L. F. Bieg, M. P. Saavedra, and E. A. Bryce, High aspect ratio mesoscale parts enabled by wire micro-EDM, Microsystem Technologies, 8(6), 395-401, September, 2002

5 K. Takahata, N. Shibaike, and H. Guckel, High-aspect-ratio WC-Co microstructure produced by the combination of LIGA and micro-EDM, Microsystem Technologies, 6(5), 175-178, August, 2000


9  D. M. Cao, W. J. Meng, K. W. Kelly, High-temperature instrumented microscale compression molding of Pb, Microsystem Technologies, 10(4), 323-328, May, 2004


12 D. M. Cao, W. J. Meng, Microscale compression molding of Al with surface engineered LIGA inserts, Microsystem Technologies, 10(8-9), 662-670, November, 2004
Chapter 3. Fabrication of High-Aspect-Ratio Microscale Mold Inserts by Parallel µEDM

3.1 Introduction

Commercial realization of nontraditional microscale devices and systems, such as microchemical-reactors, micro-heat-exchangers, and micro-electromagnetic-relays, demands fabrication technologies which can achieve economical mass production of high-aspect-ratio microscale structures (HARMS) made of metals or ceramics. Al-based HARMS, by virtue of its favorable combination of thermal conductivity and density, are important for heat transfer applications. The construction of electromagnetic microdevices, on the other hand, requires fabrication of Ni- or Fe- based HARMS.

The LIGA (Lithographie, Galvanof ormung, Abformung) process, combining deep lithography, electrodeposition, and molding replication, represents one important strategy for fabricating metal-based HARMS. In the conventional LIGA approach, mold inserts are formed by electrodeposition into lithographically defined recesses in polymeric resists. Ni is most often used. Electrodeposition of Ni typically leads to formation of nano-/micro- crystals, which undergo significant grain growth if subsequently heated to high temperatures. Yield strength of electrodeposited Ni microspecimens decreases by more than 50% over the room temperature value when heated to ~400°C. Attempts to electrodeposit alloys with higher high-temperature strengths have been made. Examples include Ni-Fe, Ni-Mo, and Ni-Co-Fe. These codepositions involve complex electrochemical reactions which limit the achievable composition range. Electrodeposited alloys also may not possess the optimal micro-/nano- scale structures for high temperature use.

The micro-electrical-discharge-machining technique (µEDM) has been used to fabricate metal-based HARMS, and offers an alternative to electrodeposition for fabricating mold inserts.
out of high temperature metals/alloys. Using µEDM, we have successfully fabricated HARMS mold inserts with simple geometries out of refractory metals such as Ta.\textsuperscript{10} Traditional µEDM technique utilizes a single electrode in performing a cut. The use of cylindrical pin electrodes for hole drilling, rectangular blade electrodes for groove cutting,\textsuperscript{11} or wire electrodes for cutting of more complex shapes\textsuperscript{12} are all examples of serial subtractive machining. Micropatterns can be generated by µEDM with a single electrode by orbiting, which significantly increases the total machining time. Electrode wear during orbiting necessitates frequent electrode replacements, and leads to shape inaccuracies of the machined part. It would be difficult/time-consuming for purely serial subtractive techniques to produce multiple microscale features in close proximity over large areas.

The feasibility of parallel pattern generation with µEDM was demonstrated by Guckel et al., who showed that LiGA fabricated, patterned Ni electrodes can be used in combination with µEDM to produce HARMS with complex shapes out of WC/Co.\textsuperscript{13} Parallel µEDM, in which multiple microfeatures are generated on the work piece simultaneously with a lithographically-defined, patterned electrode, offers advantages of fabrication speed and pattern repeatability. In this chapter, the potential of parallel µEDM as a technique for fabricating HARMS mold inserts out of high temperature compatible metals/alloys are investigated.

### 3.2 Experimental Procedures

The initial pattern definition was accomplished with the LIGA approach using SU-8 resists, employing a broad-band ultraviolet (UV) exposure source with wavelength ranging from 310 to 410nm. To achieve a target thickness of 1000µm, SU-8/100 resist was used for its high viscosity. The fabrication process is shown in Figure 3.1. (see next page)
A 30nm/10nm Cr/Au electrodeposition seed layer was sequentially electron beam evaporated onto 100mm diameter Si wafers (Figure 3.1(a)). About 10g of SU-8/100 was poured onto the wafer surface, and spun at 400rpm for 30sec (Figure 3.1(b)). SU-8 coated Si wafers were pre-baked at 110°C for 10h (see Figure 3.2, next page), and exposed to UV radiation through a mask at a dose of 2000mJ/cm². After post-baking exposed SU-8 at 110°C for 20min, the wafer was developed faced down in SU-8 developer for 1hour (Figure 3.1(c)). An additional 0.5h of development occurred with ultrasonic agitation to ensure complete development. Elemental Ni was electrodeposited into developed SU-8 recesses (Figure 3.1(d)).
Figure 3.2 Baking curve: The pre-bake data is above the lines; the post-bake data is under lines. Example: 20°C/1h means dwelling time at 20°C is 1 hour; 30min means ramping time.

Figure 3.3 Ni electrodeposition setup

The Ni electrodeposition setup is shown in Figure 3.3. The total electrolyte volume of Ni sulfamate bath is of ~46L. The concentrations of Ni sulfamate, boric acid, and lauryl sulfate (wetting agent) were ~88g/L, ~39g/L and ~1g/L, respectively. The pH value of the bath was kept at ~3.7 by adding KOH. The electrolyte was agitated and cleaned by recirculating flow through two 10µm filters. The bath temperature was kept constant at 41°C. All electrodeposition occurred in a constant-current mode. Electrodeposition of Ni HARMS into lithographically defined SU-8 recesses with heights ~1000µm followed by overplating took about twelve days, with the current
density respectively set at 4 and 8mA/cm\(^2\) for the first two days, and then set at 17mA/cm\(^2\) for the following ten days. After completion of electrodeposition, the remaining SU-8 resist was removed by heating the specimen in air at \(~750^\circ\)C for 1h, followed by ultrasonic cleaning in acetone and methanol.

Parallel \(\mu\) EDM experiments were carried out on arc-melted elemental Ta (99.9%) and 304 stainless steel (SS304) with electrodeposited Ni HARMS as electrodes. Pattern transfer via parallel \(\mu\) EDM was attempted in three different Ni HARMS electrode geometries: an array of parallel microchannels with two different widths (Channel), an array of hollow gears with teeth on the external diameter (Gear-I), and an array of hollow gears with teeth on both the external and internal diameters (Gear-II). Patterns on Ni electrodes with the first two geometries were transferred onto two machined Ta insert blanks, with a square active area of 9500\(\mu\)m×9500\(\mu\)m and a height of 2000\(\mu\)m. The top surface of the active area on the Ta insert blank was mechanically polished with SiC abrasive papers down to 1200 grit size. Patterns on Ni electrodes with the third geometry were transferred onto SS304 disks 1 inch in diameter, with the top surface mechanically polished with SiC abrasive papers down to 1200 grit size.

A SARIX High Precision Micro-Erosion Machine (SR-HPM-B) was used for parallel \(\mu\) EDM (see Figure 2.2). During \(\mu\) EDM, the discharge parameters of frequency, on-time width, and maximum voltage were set respectively at 120kHz, 5\(\mu\)sec, and 90V. A commercial dielectric fluid, IonoPlus/3000, was used as the dielectric medium for the \(\mu\) EDM process. According to the fluid supplier, this fluid is predominantly hydrocarbon-based, with trace amounts of sulphur.

Surfaces of as \(\mu\) EDMed Ta blanks using the Ni Channel electrodes were further modified by electrochemical polishing (ECP) (see Figure 2.3). The ECP process lasted for a total of 20min in a mixed acid solution of \(\text{H}_2\text{SO}_4/\text{HF}\) (49%), with a volume ratio of 6 to 1. ECP was carried out
in the constant-current mode, with the Ta blank acting as the anode and a graphite rod, 6.15mm in diameter, forming the counter electrode. ECP was conducted for 10min at a total current of ~350mA, during which time the bias voltage ranged between 1.7 to 2.0V, and for an additional 10min at 200mA, during which time the bias voltage was ~1.3V.

Scanning electron microscopy (SEM) examinations of the electrodeposited Ni HARMS, the corresponding parallel µEDMed structures, and surface conditions after the ECP process, were carried out on a Hitachi S-3600N microscope operated at 15kV. Surface chemical compositions of an as-µEDMed Ta blank and the same Ta blank after the ECP process were analyzed with X-ray energy dispersive spectroscopy (EDS).

3.3 Results and Discussion

Figure 3.4 A SEM image of an as-electrodeposited Ni HARMS (Channel), with an array of parallel microchannels with two different widths

The overview of an as-electrodeposited Ni Channel HARMS is shown in Figure 3.4. An array of nominally-identical, rectangular, Ni protrusions are present in a 1cm×1cm area. The widths of each Ni protrusion are respectively 270µm on the wide side and 180µm on the narrow side. The corresponding widths of each channel between two adjacent Ni protrusions are 130µm
on the narrow side and 220µm on the wide side, respectively. The height of all Ni protrusions is 
~1000µm. All the Ni protrusions have well defined sharp edges and corners, even at the 
transitions from wide to narrow channels.

(a) An overview of the as-µEDMed Ta

(b) A higher magnification view of one typical Ta protrusion

Figure 3.5 Examination of an as-µEDMed Ta blank with the Ni Channel HARMS
(Figure con’d.)
(c) A close-up view of the typical sidewall surface morphology of as-μEDMed Ta protrusions

(d) A typical EDS spectrum collected from the sidewalls of as-μEDMed Ta protrusions

Figure 3.5(a) (see previous page) shows an overview of the Ta blank after μEDM with the Ni HARMS shown in Figure 3.4 as the electrode. An array of rectangular trenches with two widths was created, in correspondence with the array of rectangular Ni protrusions on the electrode. The widths of each trench are respectively ~330 μm on the wide side and ~230 μm at the narrow side. The widths of each Ta protrusion between two adjacent trenches are ~70 μm on
the narrow side and ~170µm on the wide side, respectively. The depth of all trenches is ~460µm. The non-uniform contrast of the image indicates that the Ta surfaces were modified by the µEDM process.

Figure 3.5(b) shows a higher magnification SEM image of one typical Ta rectangular protrusion. It is evident that the morphology of the top and sidewall surfaces was modified by the µEDM process. The modified top surface suggests that arcing occurred near the end of the µEDM process between the channel bottom of the original Ni structure and the top surface of the Ta protrusions, resulting in the modified layer on the top surface of the Ta protrusions. In a higher magnification view, Figure 3.5(c) shows the presence of nodules and cracks on the sidewall surface of the Ta protrusions. Similar surface morphologies were observed at the bottom of the Ta trenches, as well as in previous studies of µEDMed Ta surfaces using rectangular blade electrodes. Figure 3.5(d) shows a typical EDS spectrum collected from the sidewall of one Ta protrusion. In addition to the Ta peaks in the spectrum, the presence of C, S, and Ni peaks is observed. The presence of C and S on the as-µEDMed Ta surface arises from the presence of the C- and S-containing dielectric fluid during µEDM, and is in accordance with our previous observations. The presence of Ni on the as-µEDMed Ta surface results from erosion of the Ni electrode during µEDM, and is consistent with observed electrode wear.

Our previous study of Ta µEDM showed that an altered surface layer, consisting of a B1-TaC layer several µm in thickness, forms due to reaction with the hydrocarbon dielectric fluid. The formation of the brittle TaC surface layer accentuates the formation of surface cracks. The nodules on as-EDMed Ta surfaces greatly increase its roughness, and the presence of numerous surface cracks weakens it structurally. The nodular and cracked surface morphology makes it unsuitable for further surface engineering by coating deposition. The as-µEDMed Ta pieces
therefore cannot be used directly as inserts for compression molding of reactive metals. Thus, the surfaces of as-μEDMed Ta blanks were further cleaned by the ECP process.

(a) An overview of the as-ECPed Ta HARMS

(b) A higher magnification view of one typical Ta protrusion after ECP

Figure 3.6 Examination of a parallel μEDMed Ta blank after electrochemical polishing (ECP) (Figure con’d.)
(c) A close-up view of the typical sidewall surface morphology of as-ECPed Ta protrusions

(d) A typical EDS spectrum collected from the sidewalls of as-ECPed Ta protrusions

Figure 3.6(a) (see previous page) shows an overview of the same array of trenches on Ta after the ECP process. In contrast to Figure 3.5(a), a uniform contrast is observed after ECP. The presence of some dents on the top surface of some Ta protrusions indicates slight over-etching. After the ECP process, the widths of each trench are enlarged respectively to ~355µm on the wide side and ~255µm on the narrow side. The widths of each Ta protrusion between two
adjacent trenches are correspondingly decreased to ~45\(\mu\)m on the narrow side and ~145\(\mu\)m on the wide side, respectively. The ECP process led to some rounding at the transitions between the wide and narrow Ta protrusions.

Figure 3.6(b) shows a SEM image of a typical Ta protrusion after ECP. Comparison between Figures 3.6(b) and 3.5(b) clearly shows that the altered surface layer has been completely removed, and the sidewall surfaces of the Ta protrusions are free of nodules and cracks. Similar morphologies are observed on trench bottom surfaces. Figure 3.6(c) shows a higher magnification view of the Ta protrusion sidewall, the morphology of which reflects the grain structures of the Ta bulk. Facets created on as-ECPed Ta surfaces reflect differences in etch rates for different Ta grains. EDS spectra were collected from the top and sidewall surfaces of the Ta protrusions after ECP, a typical one of which is shown in Figure 3.6(d). The presence of only Ta peaks in the spectrum corroborates the complete removal of the altered surface layer.

Another example of parallel \(\mu\)EDM is shown in Figure 3.7. (next page) An overview of the electrodeposited Ni Gear-I electrode is shown in Figure 3.7(a), which consists of an array of hollow gears ~1000\(\mu\)m in height. A total of 20 teeth are present on the external diameter of each gear, with diameters at the tooth tip and root of ~1100\(\mu\)m and ~900\(\mu\)m, respectively. The width at the half height for the teeth is ~80\(\mu\)m. The internal diameter of the hollow gears is ~687\(\mu\)m. Figure 3.7(b) shows the result of pattern transfer onto a Ta blank through parallel \(\mu\)EDM with the Ni electrode. The pattern, as defined by the protruding Ni areas on the electrode, is transferred in entirety onto the Ta surface as trenches. The depth of the gear-shaped trenches on Ta is ~230\(\mu\)m.

Figure 3.8(a) (see p49) shows an overview of the electrodeposited Ni Gear-II electrode, consisting of an array of hollow gears ~1000\(\mu\)m in height. A total of 20 teeth are present on both the external and internal diameters of each gear. For the external teeth, the diameters at the tooth
(a) An overview of an as-electrodeposited Ni HARMS (Gear-I), with an array of micro gears with teeth on the external diameter

(b) Part of a Ta blank after parallel µEDM with the Ni Gear-I HARMS electrode

Figure 3.7 Parallel µEDM of Ta blanks in other geometries
tip and root are ~1570µm and ~1284µm, respectively. The width at the half height for the external teeth is ~113µm. For the internal teeth, the diameters at the tooth tip and root are ~873µm and ~1098µm, respectively. The width at the half height for the internal teeth is ~72.5µm. Figure 3.8(b) shows the array of gear-shaped trenches created on the surface of a SS304 specimen through parallel μEDM with the Ni electrode, with a trench depth of ~370µm. A higher magnification view of a typical gear-shaped trench in SS304 is shown in Figure 3.8(c). Vertical sidewalls are generated by the parallel μEDM process. The dimensions of the gear-shaped trenches are enlarged by ~20% as compared with those of the original Ni gears. The nodular morphology of the trench sidewall surfaces is similar to that observed in as-μEDMed Ta, and is typical of the μEDM process.

(a) An overview of an as-electrodeposited Ni HARMS (Gear-II), with an array of micro gears with teeth on the external and internal diameters
Figure 3.8 Parallel μEDM of SS304 blanks in other geometries
(Figure con’d.)
(b) Part of a SS304 blank after parallel μEDM with the Ni Gear-II HARMS electrode

(c) A close-up view of the typical sidewall surface morphology of as-μEDMed trenches in SS304

It should be noted that the three parallel μEDM tests described above were performed with the same set of discharge parameters and the same Ni electrode height of ~1000μm. The significant difference in depths of the features generated by parallel μEDM, ~460μm for the Channel geometry in Ta, ~230μm for the Gear-I geometry in Ta, and ~370μm for the Gear-II
geometry in SS304, indicates a large discrepancy in Ni electrode wear rate and points to the variability of the parallel μEDM process due to variations in electrode geometry and materials. Mechanisms responsible for material erosion during μEDM are complex.\textsuperscript{15, 16} Differences in the geometry of the original Ni HARMS electrode result in differences in flow of the dielectric fluid, which has important influences on the μEDM process. Our preliminary observations suggest that the dimensions of the original Ni structures influence their wear, with smaller Ni structures wearing faster than the larger ones. Data shown in Figures 3.7 and 3.8 suggests that the type of material on which parallel μEDM is carried out also influences electrode wear, and consequently the depth of transferred micropatterns. The combined influence of these factors on the parallel μEDM process remains to be fully understood.

Results shown in Figures 3.6, 3.7, and 3.8 illustrate the potential of the hybrid LiGA/parallel-μEDM approach. The use of lithography for generating Ni HARMS electrodes facilitates the creation of deep, complex two-dimensional micropatterns on microscale mold inserts made of high temperature compatible metals/alloys. The use of these mold inserts for molding replication of metal-based HARMS is thus well suited for design and fabrication of metallic HARMS based microdevices. The sidewall surface roughness of μEDMed/ECPed parts is increased to what is typical in the conventional LiGA process. Additional strategies for improving the sidewall surface roughness deserve further investigation.

3.4 Summary

The hybrid process for fabricating HARMS mold inserts out of high temperature metals/alloys, combining UV-LiGA definition of Ni patterned electrodes and parallel-μEDM, has been investigated. Microscale mold inserts were successfully fabricated in elemental Ta and 304 stainless steel. The hybrid LiGA/parallel-μEDM strategy offers a credible alternative to
conventional LiGA regarding fabrication of meso- and micro-scale mold inserts, and enables them to be fabricated out of high-temperature compatible metals/alloys not achievable with electrodeposition.

3.5 References


3. J. D. Williams, W. Wang, Microfabrication of an electromagnetic power relay using SU-8 based UV-LIGA technology, Microsystem Technologies, 10(10), 699-705, December, 2004


11. M. Murali, S. H. Yeo, A novel spark erosion technique for the fabrication of high aspect ratio micro-grooves, Microsystem Technologies, 10(8-9), 628-632, November, 2004

12. A. Schoth, R. Forster, W. Menz, Micro wire EDM for high aspect ratio 3D microstructuring of ceramics and metals, Microsystem Technologies, 11(4-5), 250-253, April, 2005
13 K. Takahata, N. Shibaike, H. Guckel, High-aspect-ratio WC-Co microstructures produced by the combination of LiGA and micro-EDM, Microsystem Technologies, 6(5), 175-178, August, 2000


Chapter 4. Experiments and Modeling for Microscale Compression Molding of Metals at Elevated Temperatures

4.1 Introduction

Metal-based microscale devices can have improved performance as compared to their Si-based counterparts, one example being the case of micro-heat-exchangers.\textsuperscript{1} In other instances, metal-based microdevices possess functions not achievable using Si-based materials, for example micro-electromagnetic-relays.\textsuperscript{2} Prototypes of metal-based micro-heat-exchangers and micro-electromagnetic-relays have been reported in the literature.\textsuperscript{3, 4} Metal-based microdevices may also function better than Si-based micro-electro-mechanical systems when subjected to high stresses, high temperatures, and other harsh conditions.

Realization of most metal-based microdevices requires the fabrication of high-aspect-ratio microscale structures (HARMS). Metal-based HARMS can be fabricated by combining X-ray/UV lithography and electrodeposition. Such a manufacturing protocol is expensive and slow. In comparison, replication of secondary HARMS from primary microscale mold inserts by compression molding is fast and simple.\textsuperscript{5, 6} We are therefore investigating its potential as a low-cost, high-throughput, production technique for metal-based microdevices.

Prior to 2003, replication by compression molding had only been achieved in polymer-based materials.\textsuperscript{7} Since 2003, we have demonstrated successful molding replication in Pb,\textsuperscript{8} Zn,\textsuperscript{9} Al,\textsuperscript{10} and Cu.\textsuperscript{11} We have shown that one critical element to successful micromolding of reactive metals, such as Al, is to engineer the near-surface chemical and mechanical properties of the mold insert, and that conformal deposition of a suitably chosen thin ceramic coating over the microscale mold insert is an effective means for accomplishing such surface engineering.\textsuperscript{9, 10, 12, 13}
To mold metals with higher melting temperatures ($T_m$), the bulk of the mold insert needs to be constructed out of materials with sufficient yield strength at the relevant molding temperature ($T$). We have fabricated high-temperature compatible, geometrically complex, microscale mold inserts out of refractory metals and alloys by combining UV lithography with parallel micro electrical discharge machining (µEDM), followed by conformal ceramic coating deposition.$^{14}$ Successful micromolding of Cu at reduced molding temperatures $T_r \sim 0.5$ ($T_r = T/T_m$) was achieved with such bulk and surface engineered mold inserts.$^{11}$

Molding replication of metal-based HARMS entails extensive plastic deformation within the molded metal. Understanding the mechanics of micromolding is important for accurately assessing the capabilities and limitations of this technique. In a previous paper,$^{15}$ we have addressed the issue of how the mechanical properties of the molded metal influence the contact stresses exerted on the mold insert during molding. The molding response, in terms of the total force on the insert versus the insert displacement, was measured in Pb as a function of temperature. Companion uniaxial tensile testing of Pb was conducted, and a simple model of the mechanics of micromolding was developed. According to this model, the molding response is predominantly governed by two parameters: the yield strength of the material at the molding temperature, and the friction between the insert/material contact surfaces. Although the experimental results on Pb are consistent with this model, further confirmation in terms of detailed finite element analysis (FEA) and experiments on other material systems were not given at the time.

The chapter extends our previous studies on Pb molding to the case of Al, and describes results of instrumented microscale compression molding of Al as a function of temperature. Our goal is to further check our model and understand the mechanics involved. In what follows, the
experimental procedures and results on the molding response of Al are given in Section 4.2, along with typical morphologies of molded Al features. Section 4.3 describes the essential elements of our mechanics-based model for metal micromolding, and provides preliminary FEA results that lend further support to this model. Section 4.4 gives the procedures and results of companion high-temperature mechanical testing of Al. Section 4.5 then attempts to understand these experimental results in view of our model and testing results. Section 4.6 closes the chapter with some concluding remarks.

4.2 Microscale Compression Molding of Al at Elevated Temperatures

Microscale Ta mold inserts were fabricated by µEDM. By sequentially cutting a flat Ta blank with a square active area of ~9500µm×9500mm (Figure 2.1) using a Mo rectangular blade electrode, the fabricated Ta mold insert consisted of a parallel array of nominally identical, microscale rectangular protrusions from a Ta base. As-cut Ta insert was subjected to electrochemical polishing (ECP) to remove an altered surface layer due to µEDM. A Ti-containing amorphous hydrogenated carbon (Ti-C:H) coating was conformally deposited over the electrochemically polished insert. The chemistry between the coating material and the molded metal influences the molding performance, such as friction and adhesion. Further details on the insert fabrication process and the effect of coating chemistry on molding performance were reported elsewhere.\textsuperscript{11, 16}

To be consistent, all Al molding force versus insert displacement data were acquired with one Ti-C:H coated Ta insert, an overview of which is shown in Figure 4.1 (see next page). Each microprotrusion was ~400µm in height, ~150µm in width, and ~9500µm in length. The insert contained a total of 13 microprotrusions, with a protrusion center-to-center spacing of ~780µm. Micron scale surface roughness exists on the sidewalls of the microprotrusions, resulting from
the combined actions of µEDM and ECP. Circular Al (1100H14, 99at.%+Al) disks, 35mm in diameter and 6.4mm in thickness, were molded at different temperatures using this insert. Al disks were mechanically pre-polished on both sides with SiC abrasive papers from 400 to 800 in grit size. The disk top surface was mechanically polished with SiC abrasive papers down to 1200 grit size and final polished with 3µm diamond slurry prior to being molded.

Figure 4.1 The Ti-C:H coated microscale Ta insert used to mold Al

Instrumented micromolding was carried out on a MTS858 single-axis testing system interfaced to a high-vacuum molding chamber with an ultimate base pressure of ~1×10⁻⁸ Torr (Figure 2.5). Pressures during high-temperature molding were ≤1×10⁻⁴ Torr. Two heating stations were installed within the vacuum chamber. The Al disks to be molded were fixed on the lower heating station, mechanically attached to the bottom of the molding chamber. The Ta mold insert was mechanically attached to the upper heating station, which was connected to the linear actuator through a bellow-sealed Z-motion feedthrough. All parts used for mechanical attachment were paralleled for good alignment, i.e., the axis of motion of the linear actuator is perpendicular to the surfaces of the Al disk. The temperatures of the two heating stations, heated
independently by separate resistive heating cartridges, were controlled to be within 10°C of each other. The Ta insert attached to the linear actuator could be programmed to move according to prescribed load forces in the force-controlled mode or prescribed actuator displacements in the displacement-controlled mode. The total axial force on the insert and the total axial displacement of the actuator, onto which the Ta insert was mounted, were continuously measured with a 5kN load cell with a resolution of ~5mN and a linear variable displacement transducer (LVDT) with a resolution of ~0.5 µm, both located outside the high-vacuum molding chamber. Further details on the instrumented micromolding apparatus and the molding procedure were described elsewhere.\textsuperscript{8,15}

Micromolding of Al was performed at 360°C, 400°C, 450°C, and 500°C, corresponding to a reduced temperature range of 0.67 ≤ T_r ≤ 0.83. Multiple molding runs were performed at each temperature. Demolding occurred isothermally at the molding temperature. Molding occurred under force-control with a constant loading rate of 100N/min.

The molding process involves the indentation of a flat Al plate by a series of parallel rectangular microprotrusions on the insert, and transfers the array of microprotrusions on the insert into an array of parallel rectangular microchannels on the Al plate. Scanning electron microscopy (SEM) examinations of molded features in Al were carried out on a Hitachi S-3600N microscope. Figure 4.2(a) (see next page) shows a cross-sectional view of an Al plate after being compression molded by the Ti-C:H coated Ta insert at 360°C, or T_r=0.68. It is evident that the sidewalls of the microchannels are close to vertical. The top surfaces of the molded Al plate are rounded, due to indentation-induced pileup. Figure 4.2(b) shows the top view of the same molded Al structure. Figure 4.2(c) shows a magnified view of the sidewall of a typical microchannel. The mottled sidewall surface morphology is representative of that created by the
micromolding process.\textsuperscript{15, 16} Similar morphologies of molded Al structures were observed at other molding temperatures.

(a) A cross-sectional view of a parallel array of microchannels in Al

(b) The corresponding top view of the microchannel array

Figure 4.2 Morphologies of Al structures molded at 360\textdegree C

(Figure con’d.)
Because of the existence of various parts between the Ta insert and the LVDT, measured total displacement includes a system stiffness contribution. To obtain the true molding response in terms of the relationship between the axial force on the insert and the actual depth of penetration of the rectangular microprotrusions on the insert into the molded Al, this system stiffness has to be measured and subtracted. The stiffness of the molding apparatus was obtained by multiple measurements of the total force – total displacement curve of a flat Ta blank, with the same active area of 9500µm×9500mm as the actual insert but without the microprotrusions, compressing on Al disks at room temperature. To simulate the actual experiments, the Ta blank was remounted onto the top of the upper heating station and a new Al disk was reattached to the bottom heating station several times in between stiffness calibration runs. The stiffness calibration runs occurred over similar force range as that used in actual molding experiments. Results of such stiffness calibration runs are shown in Figure 4.3 (see next page), in which data from 7 separate runs were superimposed. It is evident from Figure 4.3 that a variation in the system stiffness exists due to operational variabilities in Ta insert and Al disk attachments and
other factors. From a linear fit to the data shown in Figure 4.3, an average system stiffness of 19kN/mm is obtained. The actual system stiffness ranges from 16kN/mm to 22kN/mm, judging from the two extremes of the stiffness calibration data. The true molding response was obtained by subtracting the average system stiffness of 19kN/mm from all measured total force – total displacement curves. Because of the variation in system stiffness, a displacement uncertainty of ±10µm exists at the load of 1000N.

Figure 4.3 Measurements of the stiffness of the instrumented molding apparatus

Figure 4.4 shows a series of Al molding response curves measured at 360°C, 400°C, 450°C, and 500°C. By subtracting the system stiffness contribution, the total compressive force on the insert, $P$, is given as a function of the indentation depth, $d$, of the microprotrusions into the molded Al plate. Each individual curve represents the result from a separate molding run. The data scatter exhibited by the $P$-$d$ curves measured at the same temperature, about ±10% or less of force variation at the same molding depth, shows the typical variability of the micromolding process. All molding response curves are qualitatively similar. At each temperature, measured $P$-$d$ curves show an initially stiff response, in which $P$ increases rapidly with increasing $d$. 
Thereafter the \( P-d \) curves exhibit a sudden bend-over, after which \( P \) increases approximately linearly with further increasing \( d \). The maximum molding force, observed at the maximum molding depth of \(~220\mu m\), decreases monotonically with increasing molding temperature.

![Figure 4.4 Measured Al molding response: total molding force \( P \) as a function of the indentation depth \( d \) at four different temperatures](image)

### 4.3 Companion Simple Model

In an earlier paper,\(^{15}\) a model for the average contact pressure, \( p \), versus indentation depth, \( d \), for an isolated rigid punch pressed into an elasto-plastic half space is developed. As shown in Figure 4.5 (see next page), the punch is a right circular cylinder of radius \( r \), having a flat end with rounded corners of relatively small radii \( r_o \) \((r_o/r \sim 0.05)\). It appears that few analytical results for the elasto-plastic response of such an axisymmetric configuration exist in the literature. Accordingly, the model draws on corresponding results for spherical indentation.

For spherical indentation, the idea of self-similar growth of the yield region once a “fully plastic state” has been reached is advanced and confirmed experimentally by Tabor.\(^ {17}\) In this fully plastic state, the contact pressure normalized by an appropriately defined flow stress, \( \sigma_f \), of the indented material approaches a constant. The idea of self-similar growth of the yield region
was further supported with FEA.\textsuperscript{18} In this geometrically self-similar state, the shape of the yield region becomes approximately invariant, while the size the yield region scales linearly with the contact size.\textsuperscript{18} In the case of spherical indentation, the contact size increases as the extent of indentation progresses. In contrast, the size of contact during indentation by a cylindrical punch remains a constant as indentation progresses.

![Figure 4.5 Geometry for the isolated punch model](image)

For the cylindrical punch case, a self-similar yield region, of constant width $w_Y$ and constant depth $d_Y$, is therefore assumed, as indicated by the broken line in Figure 4.5. In the steady state, this constant-shaped yield region forms in front of the punch and advances into the material together with the punch as indentation progresses. With this assumption, resistance as indentation progresses stems from two sources: an approximately constant set of tractions acting on the boundary of the yield region, and a frictional traction $\tau_f$ acting on the sides of the indenter (Figure 4.5). It is further assumed that characteristic stresses surrounding the boundary of the
yield region in front of the punch as well as surrounding the punch sidewall scale linearly with \( \sigma_f \) of the material at the temperature of indentation. It follows that the total reaction force from the boundary of the yield region in front of the punch should scale linearly with \( \sigma_f \). Further on defining an effective friction coefficient \( f \) such that \( \tau_f = f\sigma_f \), the frictional force also scales linearly with \( \sigma_f \). This configuration thus leads to

\[
\frac{p}{\sigma_f} = C + f\left(\frac{d}{r/2}\right),
\]  

where \( p = P/\pi r^2 \) is the contact pressure, and \( C \) is a constant dependent on the detailed stress distribution around the boundary of the yield region in front of the punch. In the earlier paper,\(^{15}\) \( C \) was estimated to be 8.8 while \( \sigma_f \) was simply taken to be the yield strength \( \sigma_Y \) in lieu of any obviously superior choice at the time of reporting.

4.4 Consequent Tensile Testing of Al at Elevated Temperatures

To further test the validity of this simple model, uniaxial tensile testing of macroscale Al specimens was conducted at the temperatures of the molding experiments, namely 360°C, 400°C, 450°C, 500°C, and 550°C, to measure the appropriate \( \sigma_f \). (Figure 4.6) One additional factor of complication exists for the Al molding experiments. In the reduced temperature range

(a) Al tensile specimen

Figure 4.6 Al High temperature tension test setup
of $0.67 \leq T_r \leq 0.83$, results of tensile testing are expected to be dependent on strain rate. Because it is unclear what the exact strain rate corresponding to the Al molding experiments is, uniaxial tensile tests were conducted at several different strain rates. This high temperature tension test was carried out at High Temperature Materials Laboratory in Oak Ridge National Laboratory.
Cylindrical Al (1100O, 99at.% Al) rod specimens, with a gauge section of 6.35mm in diameter and 31.75mm in length (Figure 4.6(a), and dimensions are shown in Appendix A), were custom made and tested with an Instron4507 single-axis system in a 45cm long, three-zone furnace with three independent temperature controls (Figure 4.6(c)). Specimen temperatures at the two ends and the mid-point of the specimen gauge section were controlled to be within 3°C of each other. Specimen extension at high temperatures was determined using a custom-built extension stage (Figure 4.6(b)). The two ends of the extension stage were rigidly fastened to the two ends of the Al rod specimen. The relative displacement between the two ends was measured by two separate LVDTs whose outputs were averaged to calculate the mean specimen extension and axial strain. At each temperature, tensile tests were conducted at three different longitudinal strain rates \( \frac{d\varepsilon}{dt} \) of \( 3 \times 10^{-4}/\text{sec} \), \( 1 \times 10^{-3}/\text{sec} \), and \( 5 \times 10^{-3}/\text{sec} \), respectively.

![Uniaxial stress-strain curves of Al measured at 360°C at three different strain rates](image)

Figure 4.7 Uniaxial stress-strain curves of Al measured at 360°C at three different strain rates

Figure 4.7 shows true longitudinal stress \( (\sigma) \) versus true longitudinal strain \( (\varepsilon) \) curves measured at 360°C. At all strain rates tested, measured \( \sigma - \varepsilon \) curves approximate that of an elastic – ideally plastic solid at \( \varepsilon < 10\% \). Similar \( \sigma - \varepsilon \) curves were obtained at other temperatures. The
yield strength, \( \sigma_Y \), is defined here simply as the maximum stress observed on the \( \sigma - \varepsilon \) curve. At a given temperature and a given strain rate, the scatter in measured \( \sigma_Y \) values is within \( \pm 10\% \). Because measured \( \sigma - \varepsilon \) curves exhibit little or no strain hardening, in our present problem we simply have \( \sigma_f = \sigma_Y \).

In high-temperature creep, \( \frac{d\varepsilon}{dt} \) can be related to stress and temperature by

\[
\frac{d\varepsilon}{dt} = A \frac{\sigma^n}{kT} \exp\left(-\frac{Q}{kT}\right),
\]

where \( A \) is a constant, \( n \) is the power-law exponent, and \( Q \) is an activation energy.\(^{19}\)

![Figure 4.8](image-url)

Figure 4.8 Analysis of uniaxial tensile testing data for Al according to the mechanism of power-law creep: \( \frac{d\varepsilon}{dt} T \exp(\frac{Q}{kT}) \) versus \( \sigma_f \) in a log-log plot.

Figure 4.8 summarizes results of uniaxial tensile testing on Al, in the form of \( \frac{d\varepsilon}{dt} T \exp(\frac{Q}{kT}) \) versus \( \sigma_f \) in a log-log plot. The value of \( Q \) is taken to be 1.47eV, that corresponding to substitutional self diffusion in pure Al.\(^{20}\) Data taken at all temperatures and all
strain rates fall approximately on one single straight line, conforming to expectations of power-law creep as expressed in Equation (4.2) and consistent with previous studies of creep in polycrystalline Al. From linear least-squares fitting to the data shown in Figure 4.8, the power-law exponent is determined to be $n=5.6\pm0.3$, in agreement with the range of power-law exponents, 4.4 to 6.4, observed in previous high-temperature creep studies on Al.

4.5 Rationalization of Molding Response Using the Model

The contact pressure $p$ is calculated by dividing the total molding force $P$, shown in Figure 4.5, by the total nominal contact area. To normalize measured contact pressures at different temperatures according to Equation (4.1), values of $\sigma_f$ are needed. These are strain rate dependent, as shown in Figures 4.7 and 4.8. Contrary to uniaxial tensile testing, where strain and strain rate are uniform throughout the specimen gauge section, in the micromolding experiment, strain and strain rate within the indented material vary with both location and time. Spatially, the plastic strain is high immediately around the corner of the indenter and decays rapidly at locations farther away from the indenter. Temporally, the rate of increase in plastic strain with increasing indentation depth $d$ is small at first and accelerates as massive plastic flow occurs around the corner of the indenter. The total plastic strain can reach a few hundred percent or more as the indenter is pushed into the material. Thus rather than an exact strain and an exact strain rate, only an effective strain and an effective strain rate can be defined at a particular $d$. Regardless of the exact strain field distribution within the indented material, the concept of a geometrically self-similar plastic zone suggests that the effective strain and effective strain rate at $d$ should be the same if the indenter took the same amount of time to reach $d$, independent of the value of $\sigma_f$ (i.e., the molding temperature). For the present molding experiments executed under a constant loading rate of 100N/min, the time the indenter took to reach $d = 220\mu m$ ranges
from ~13min at 360°C to ~5min at 500°C, and is not exactly a constant. Ignoring this variation, we chose to normalize $p$ with values of $\sigma_f$ corresponding to one constant strain rate.

Figure 4.9 shows the normalized molding response, as expressed by $p/\sigma_f$ versus the normalized indentation depth, $d/(w/2)$. Values of $\sigma_f$ at different temperatures were taken at the same strain rate of $1 \times 10^3$/sec. The scatter resulting from such a normalization, as judged by the scatter in the value of $p/\sigma_f$ shown in Figure 4.9, is about ±10% or less at the maximum value of $d/(w/2)$. This level of scatter is comparable to that observed in the raw $P$-$d$ curves and to the uncertainty in the $\sigma_f$ measurements. Thus within the measurement uncertainties, such a normalization combines measured molding responses at different temperatures into a universal molding response curve, in agreement with Equation (4.1). Furthermore, beyond the initial stiff response at small indentation depths, $p/\sigma_f$ appears to be linearly proportional to $d/(w/2)$ with a constant slope, again in agreement with Equation (4.1). A linear least-squares fit to the data
shown in Figure 4.9, in the range of $1 < d/(w/2) < 3$, yields an average slope of 0.8. This value is somewhat higher than the previous value of $f \sim 0.5$ determined through previous Pb micromolding experiments, but is still plausible as a friction coefficient. The successful normalization of measured molding force – indentation depth curves for Al into a universal molding response curve lends further support to our mechanics-based model.

Similar molding response normalizations were carried out using values of $\sigma_f$ taken at strain rates of $3 \times 10^{-4}/sec$ and $5 \times 10^{-3}/sec$, respectively. The scatters in the $p/\sigma_f - d/(w/2)$ curves are slightly larger for these two cases, about $\pm 15\%$ or less. The quality of this normalization thus appears to depend somewhat on the particular strain rate at which values of $\sigma_f$ are evaluated, but is not extremely sensitive within the current range of strain rates tested. As compared to spherical indentation, much larger plastic strains are involved in the present molding experiments. A definition of an effective strain and an effective strain rate appropriate for the micromolding experiments remains to be established.

4.6 Concluding Remarks

We have carried out detailed measurements of the molding response of Al in the temperature range of 360°C to 500°C. Companion uniaxial tensile testing of Al was conducted at the molding temperatures. Our results, as summarized in Figure 4.9, are consistent with the simple model of microscale compression molding presented in section 4.3. Together with our previous molding experiments on Pb, the present experiments on Al suggest that this simple model captures the essentials of the molding process. Several comments regarding microscale compression molding can be offered based on the present data.

First, a characteristic contact pressure is required to reach the fully plastic state. For the present rectangular strip punch geometry, this characteristic $p$ is about $3.5 \sigma_y$ at $d/(w/2) \sim 2$, and
varies somewhat depending on the value of $f$. The important conclusion is that the contact pressure is mainly limited by the yield strength of the indented material, provided that the indenter does not deform in a plastic manner or fracture in a brittle manner during indentation. A particular material can be used to make a microscale mold insert if the bulk of the indenter made of this material can withstand the contact pressures incurred during indentation without plastic deformation or fracture. At sufficiently high temperatures, the yield strength of the molded metal can be made sufficiently low, e.g., $\sim 20$MPa. The choice of materials capable of serving as a mold insert for metals is therefore wider than what is commonly appreciated, and is in particular not limited to refractory ceramics such as SiC, WC, diamond, etc.

Second, no distinction was made between the flow stress and the yield strength in the present case since Al exhibits little strain hardening at the molding temperatures of 360$^\circ$C to 500$^\circ$C. A better definition of $\sigma_f$ may lead to a better normalization of the molding response in cases where the indented material exhibits significant strain hardening, and remains a topic of future study.

Third, our simple model is based entirely on continuum mechanics and contains no characteristic length scale. The satisfactory combination of microscale compression molding experiments and macroscale tensile testing experiments on Al, as evident from Figure 4.9, suggests that continuum mechanics gives a satisfactory description of the molding experiments when the length scale of characteristic features is about 150µm. Possible deviations of the molding response from continuum mechanics description as the characteristic size of molded features decreases down to µm or sub-µm scale is both interesting scientifically and relevant to fabrication of nanoscale metal-based structures, and remain to be examined.
4.7 References


4. J. D. Williams, W. Wang, Microfabrication of an electromagnetic power relay using SU-8 based UV-LIGA technology, Microsystem Technologies, 10(10), 699-705, December, 2004


10. D. M. Cao, W. J. Meng, Microscale compression molding of Al with surface engineered LiGA inserts, Microsystem Technologies, 10(8-9), 662-670, November, 2004


14 D. M. Cao, J. Jiang, R. Yang, W. J. Meng, Fabrication of high-aspect-ratio microscale mold inserts by parallel μEDM, Microsystem Technologies, 12(9), 839-845, August, 2006


16 D. M. Cao, J. Jiang, W. J. Meng, Metal micromolding with surface engineered inserts, Material Research Society Symposium Proceedings, 890, 0890-Y03-03.1, 99, 2006


20 D. A. Porter, K. E. Easterling, Phase transformations in metals and alloys, Stanley Thornes, UK, 2000


22 H. J. Frost, M. F. Ashby, Deformation mechanism maps, the plasticity and creep of metals and ceramics, Pergamon Press, Oxford, 1982
Chapter 5. Finite Element Analysis of an Al Slab Indented by a Periodic Array of Elastic Strip Punches

5.1 Introduction

Solid bodies in elastic contact have been of interest to scientists and engineers for over a century. The contact of two solid bodies was investigated by Hertz, who provided contact stresses for elastic, frictionless indentation of two non-conforming bodies. Later, Huber gave the complete stress and strain distributions within two elastic spheres in contact. Spence provided an analytical solution for elastic contact with friction. A long history of scientific study regarding plasticity-inducing contacts exists as well. In his pioneering work, Tabor found the well-known scaling relationship between the hardness and the yield/flow stress of the indented metal, and invoked the concept of geometrical similarity in scaling indentation experiments with different sized indenters. Johnson considered the influence of elasticity on indentation. Numerically, Hardy was the first to study spherical indentation of elasto-perfectly plastic solids and gave the stress distribution and its evolution. Sinclair et al. studied spherical indentation for work hardening materials with finite element analysis (FEA). Their results tracked with better accuracy the load force – indentation depth relationship, and showed that the plastic yield region propagates in a geometrically self-similar manner as indentation progresses. Mesarovic and Fleck investigated the effects of elasticity and work-hardening rate of the indented material and friction on spherical indentation. They carried the simulation to larger indentation depth and observed the limitation of Tabor’s work. Biwa and Storakers studied Brinnel indentation with a focus on fully plastic behavior of rigid-plastic materials with power-law hardening, and observed geometrical self-similarity in yield region propagation. Guyot et al. presented frictionless spherical indentation of deformable substrates indented by a deformable sphere. They compared cases of work hardening and perfect
plasticity and showed good agreements with works of Johnson and Sinclair.\textsuperscript{8, 13} Numerous additional papers have been published on the topic of analyzing elasto-plastic indentation with FEA.\textsuperscript{14, 15, 16}

From the very beginning, solving engineering problems has provided motivations for conducting studies on contact and indentation. The investigation of Hertz on contact of two solid bodies was motivated by the engineering need to understand deformation of precision optical surfaces. The work of Tabor provided the scientific foundation for hardness testing on metals and alloys.

However, a detailed study of elasto-plastic strip indentation is not available in the literature. The only related research is the elastic analysis of strip indentation conducted by Shtaerman.\textsuperscript{17}

In this chapter, we will discuss the response of an elasto-perfectly plastic substrate indented by long strip punches without friction through FEA. We first check the model and meshes by comparing FEA outputs with available analytical solutions for elastic contact. Then the calibrated meshes are used to carry the indentation simulation to the elasto-plastic regime and beyond. Next, we discuss the FEA results and compare them to results of instrumented molding experiments. Finally, the concept of self-similar propagation of the yield region is confirmed by observing the yield region propagation in FEA.

In what follows, the formulation for elasto-plastic simulation is given in Section 5.2, with particular attention being paid to the selection of a representative stress-strain curve for the indented material. Next, in Section 5.3, the approach used in the FEA model is described together with verification via test problems and convergence checks. Thereafter, in Section 5.4, results for applied pressure versus indentation depth are compared to those from corresponding
molding experiments. Contact and interior stresses, as well as yield regions, are given as the simulation proceeds through an elasto-plastic regime into a fully plastic one. The chapter closes in Section 5.5 with some concluding remarks in the light of results found.

5.2 Formulation of a Model Problem

![Figure 5.1 Cross-section of an elasto-plastic slab indented by a periodic array of strip punches](image)

The configuration chosen to model the application entails a uniformly spaced array of elastic strip punches, modeling the tantalum (Ta) insert, pressed into an elasto-plastic slab in a state of plane strain, modeling the Al substrate (Figure 5.1). Each punch has a flat section of width of $2a$, edge radii $r_e$, and a total width $2b = 2(a + r_e)$. Each punch is pressed slowly into the elasto-plastic slab by a pressure $p$, and makes contact with the slab over a strip of width $2l$ ($l > a$). This region is largely framed in rectangular Cartesian coordinates $(x, y)$, supplemented with cylindrical polar coordinates $(r, \theta)$ for the indenter near the edge of contact. The coordinate systems are related by
\[ x = a + r \sin \theta, \quad y = -r_e + r \cos \theta, \quad (5.1) \]

for \(-\pi < \theta \leq \pi\). We use periodic symmetry to confine attention to the region to the right of the \(y\)-axis and the left of the middle line between two punches. Thus the regions for analysis are the indenter region, \(R_I\), and the substrate region, \(R_S\), and these are given by

\[
R_I = \{(x, y) \mid 0 < x \leq a, -h < y < 0, \text{ and } a < x < b, -h < y < \sqrt{r_e^2 - (x - a)^2} - r_e\},
\]

\[
R_S = \{(x, y) \mid 0 < x < W, 0 < y < H\}, \quad (5.2)
\]

where \(h\) is the height of \(R_I\), and \(W\) is the width, \(H\) the height of \(R_S\). With these geometric preliminaries in place, we can formally state this model problem as follows.

In general, we seek the accumulated plane strain stresses \(\sigma_x, \sigma_y\) and \(\tau_{xy}\), or \(\sigma_r, \sigma_\theta\) and \(\tau_{r\theta}\), and their associated displacements \(u\) and \(v\), or \(u_r\) and \(u_\theta\), as functions of \(x\) and \(y\) throughout \(R_I\) and \(R_S\), together with the contact extent \(l\), for all time \(t > 0\). The stresses are accumulated from corresponding rates in accordance with

\[
\sigma_x = \int_0^t \dot{\sigma}_x \, dt, \quad (5.3)
\]

where \(t\) is time, with like expressions for the other components in terms of their respective rates \(\dot{\sigma}_y, \dot{\tau}_{xy}, \dot{\sigma}_r, \dot{\sigma}_\theta\) and \(\dot{\tau}_{r\theta}\). The displacements are accumulated from corresponding rates in accordance with

\[
u = \int_0^t \dot{u} \, dt, \quad (5.4)
\]

with like expressions for the other components in terms of the rates \(\dot{v}, \dot{u}_r\) and \(\dot{u}_\theta\). The stress rates and displacement rates throughout \(R_I\) and \(R_S\) satisfy field equations, boundary conditions, and contact inequalities, as follows.
The field equations are: the two-dimensional stress rate equations of equilibrium, and the stress rate versus displacement rate relations for a linear elastic, homogeneous and isotropic, solid in a state of plane strain for \( R_i \); the two-dimensional stress rate equations of equilibrium, and the flow rule for a homogeneous and isotropic, elastic and incompressible-plastic solid complying with von Mises’ yield criterion with isotropic hardening in a state of plane strain for \( R_S \).

The boundary conditions are: the applied pressure conditions,
\[
\dot{\sigma}_y = -\dot{p}, \quad \dot{\tau}_{xy} = 0, \quad (5.5)
\]
on \( y = -h \) for \( 0 < x < b \), where \( \dot{p} \) is the incremental pressure rate that accumulates to the pressure \( p \); the contact conditions that insist on continuity of tractions and the normal displacement within the contact region
\[
\dot{\sigma}_y |_{y=0^-} = \dot{\sigma}_y |_{y=0^+}, \quad \dot{\tau}_{xy} |_{y=0^-} = \dot{\tau}_{xy} |_{y=0^+}, \quad \dot{v} |_{y=0^-} = \dot{v} |_{y=0^+}, \quad (5.6)
\]
on \( y = 0 \) for \( 0 < x < a \),
\[
\dot{\sigma}_r |_{r=r_c^-} = \dot{\sigma}_r |_{r=r_c^+}, \quad \dot{\tau}_{r\theta} |_{r=r_c^-} = \dot{\tau}_{r\theta} |_{r=r_c^+}, \quad \dot{u}_r |_{r=r_c^-} = \dot{u}_r |_{r=r_c^+}, \quad (5.7)
\]
on \( r = r_c \) for \( 0 < \theta < \theta_c \), where \( \theta_c = \sin^{-1}[\left(l-a\right) / r_c] \), as well as applying Amonton’s law for friction
\[
\dot{u} |_{y=0^-} = \dot{u} |_{y=0^+} \quad \text{if} \quad \dot{\tau}_{xy} \leq f\dot{\sigma}_y, \quad (5.8)
\]
on \( y = 0 \) for \( 0 < x < a \), where \( f \) is the coefficient of friction,
\[
\dot{u}_\theta |_{r=r_c^-} = \dot{u}_\theta |_{r=r_c^+} \quad \text{if} \quad \dot{\tau}_{r\theta} \leq f\dot{\sigma}_r, \quad (5.9)
\]
on \( r = r_e \) for \( 0 < \theta < \theta_e \); the stress-free conditions, outside of contact,

\[
\dot{\sigma}_x = 0, \quad \dot{t}_{xy} = 0,
\] (5.10)

on \( x = b \) for \(-h < y < -r_e\),

\[
\dot{\sigma}_r = 0, \quad \dot{t}_{r\theta} = 0,
\] (5.11)

on \( r = r_e \) for \( \theta_e < \theta < \pi/2 \),

\[
\dot{\sigma}_y = 0, \quad \dot{t}_{xy} = 0,
\] (5.12)

on \( y = 0 \) for \( l < x < W \); the periodic symmetry conditions,

\[
\dot{u} = 0, \quad \dot{t}_{xy} = 0,
\] (5.13)

on \( x = 0 \) for \(-h < y < H\), and \( x = W \) for \( 0 < y < H \); and the far-field roller restraint conditions,

\[
\dot{v} = 0, \quad \dot{t}_{xy} = 0,
\] (5.14)

on \( y = H \) for \( 0 < x < W \).

The contact inequalities insist that: the normal contact stress be nowhere tensile within
the contact region,

\[
\dot{\sigma}_y \leq 0, \quad (5.15)
\]

on \( y = 0, \quad 0 \leq x < a \) and

\[
\dot{\sigma}_r \leq 0, \quad (5.16)
\]

on \( r = r_e, \quad 0 < \theta < \theta_e \); and there is no contact outside of the contact region,

\[
\left. v \right|_{r=r_e} \leq \left. v \right|_{y=0} + r_e - \sqrt{r_e^2 -(x-a)^2}, \quad (5.17)
\]

for \( x \geq l \), where the displacement on the left-hand side of (5.17) is on \( R_l \), the displacement on the
right-hand side on \( R_S \).
The foregoing formulation only applies for small deflections. In fact we seek to track response for indentation depths \( d \) that are of the order of ten percent of \( W \). For such an increase in displacement, we take the coordinate system to be attached to the indenter. This leaves (5.5)-(5.11) as is, while (5.12) maintains stress-free conditions but now on a deformed surface, (5.13) applies on a vertical range modified by the accumulated displacement at the respective \( x \) locations, and (5.14) applies at \( y = H - d \). We also need to invoke a finite strain formulation using a logarithmic or Hencky strain as in ANSYS.\(^{18}\)

Several specifications are required for the FEA of this problem. First, \( r_c / b, W / b, h / b, \) \( H / b \) are taken to be 1/25, 5.2, 8/3 and 64/3, respectively, so as to be in accord with the geometry in the molding application.\(^{19}\) Second, the friction coefficient is taken to be \( f = 0.7 \), as estimated in Jiang et al.\(^{19}\) Because this is but an estimate, we also take \( f = 0 \) to gauge the effects of friction and to assess how critical is the accuracy of this estimate. Third, Young’s modulus and Poisson’s ratio of the Ta indenter are taken to be \( E_I = 186 \) GPa and \( \nu_I = 0.35 \) from the ASM Metals Handbook.\(^{20}\) The Ta insert is assumed to behave as a linear elastic material throughout the whole indentation process. This assumption is confirmed by experimental observations of no permanent indenter deformation after numerous indentation runs. Finally, the stress-strain curve of the Al slab is selected from curves obtained from Al uniaxial tensile tests at 360\(^\circ\)C, the temperature which the molding experiments are conducted.

Figure 5.2(a) (see next page) shows the uniaxial true stress \( \sigma \) versus uniaxial true strain \( \varepsilon \) curve for Al obtained at a longitudinal strain rate of \( \dot{\varepsilon} = 3 \times 10^{-4} / s \) at 360\(^\circ\)C. This curve is converted from raw experimental data of force versus extension, assuming uniform gauge deformation in the range \( 0 < \varepsilon < 0.1 \) since in situ observation of gauge deformation is not possible for the current experimental setup. It exhibits a response which closely approximates
that of an elastic-perfectly-plastic solid. The yield stress $\sigma_y$ is 15MPa. Since the uniaxial tensile test did not have sufficient resolution to capture the initial elastic response accurately, values of the Young’s modulus $E$ and Poisson’s ratio $\nu$ of Al are obtained from a previous determination of elastic moduli at the temperature of interest by Varshni.\textsuperscript{21} The values taken from Varshni for Al at 360°C are $E = 56$ GPa and $\nu = 0.33$.

![Figure 5.2 Aluminum stress strain curves at 360°C](image)

(a) $\dot{\varepsilon} = 0.0003 / \text{s}$

(b) $\dot{\varepsilon} = 0.001 / \text{s}$ and $\dot{\varepsilon} = 0.005 / \text{s}$

The uniaxial tensile testing is conducted at high molding temperature ($T / T_m = 0.68$, where $T$ is the temperature at which the molding is performed in K, $T_m$ is the melting temperature of Al in K). Because of this high molding temperature, the deformation may involve creep. Creep manifests itself as a dependence of the stress strain curve on strain rate. To explore the possibility of such effects being significant, we did the uniaxial tensile tests at two more strain rates, $\dot{\varepsilon} = 1 \times 10^{-3} / \text{s}$ and $\dot{\varepsilon} = 5 \times 10^{-3} / \text{s}$ (Figure 5.2(b)). Clearly the curves in Figure 5.2(a)(b) show there are marked strain rate effects with $\sigma_y$ varying from 15 to 21MPa. Consequently, we need to make a judgment as to the most appropriate strain rate for the FEA.

To estimate an appropriate average strain rate corresponding to the molding experiment, we recognize that, in our problem, the plastic strain dominates the total strain. We therefore
adopt as our estimate of a representative strain rate, \( \dot{\varepsilon}_r \), the average strain in the plastic region at the conclusion of the indentation process divided by the time to complete indentation.

Figure 5.3 Approximation of deformation in aluminum after indentation

Figure 5.3 shows a schematic of the material under the indenter. Because of symmetry, only the region \( R_S \) needs to be considered. A region under the indenter yields plastically due to the indentation. At a particular molding depth \( d \), the boundary of the yield region is shown schematically as the dash-dotted line. Where the plastic yield region intersects the side boundaries of \( R_S \), two yield region depths are defined: \( d_y \) and \( d'_y \) at \( x = 0 \) and \( x = W \), respectively. Because of the penetration of the indenter into the indented material, an elevation of the original surface outside the indenter occurs. We simply approximate this elevation as an uniform rise of the surface level by an amount of \( d_u \). The value of \( d_u \) then follows from the assumption of incompressible plastic flow and is
We further simplify the shape of the plastic yield region to that shown by the dotted line in Figure 5.3. This simplified yield region has depths \( d_y \) in the range \( 0 < x < b \), and \( \bar{d}_y \) in the range \( b < x < W \). The value of this representative depth \( \bar{d}_y \) is taken to be the average of \( d_y \) and \( d'_y \). The magnitudes of the strains in the ranges \( 0 < x < b \) and \( b < x < W \) are then given simply as \( d / d_y \) and \( d_a / \bar{d}_y \), respectively. An estimate of an average strain magnitude for the entire material under the indenter can then be obtained as an weighted average of these two strain values. Then using (5.18) we have, as our representative strain rate,

\[
\dot{\varepsilon}_r = \frac{bd}{Wt} \left( \frac{1}{d_y} + \frac{1}{\bar{d}_y} \right),
\]

where \( t \) is the time to complete the indentation process.

In using (5.19) to estimate a representative strain rate, the values of \( b \) and \( W \) are known from the indentation configuration we are modeling, while \( d \) and \( t \) are available from experimental measurements of the indentation process. On the other hand, \( d_y \) and \( d'_y \), hence \( \bar{d}_y \), are not directly available from measurements. Instead they are outcomes of modeling the experiment and FEA. Thus we need to run our FEA simulation for an assumed strain rate and companion yield stress to obtain \( d_y \) and \( d'_y \), and so a value of the representative strain rate of (5.19). Then if the two strain rates are in fair agreement, the assumed strain rate is in fact appropriate; If not, another rate needs to be assumed and the estimation process repeated.

So proceeding to evaluate \( \dot{\varepsilon}_r \) of (5.19), we have, from Jiang et al., \(^{19} \) \( W / b = 5.2 \) as previously, and from a typical molding experiment \( d = 220 \mu \text{m} \) when \( t = 13 \text{min} \). Then for an assumed strain rate of \( \dot{\varepsilon} = 3 \times 10^{-4} / \text{s} \) with consequent \( \sigma_y = 15 \text{MPa} \), FEA leads to estimates of
\( d_y = 730 \mu m \) and \( d_y' = 560 \mu m \). Hence from (5.19), \( \dot{\varepsilon}_r = 1.6 \times 10^{-4} / s \), a factor of two less than the assumed rate. We judge this value to be in fair accord with the assumed value, and accordingly use the assumed strain rate and the stress-strain curve Figure 5.2(a) in the remainder of this study.

### 5.3 Finite Element Analysis and Verification

The commercial code ANSYS, Version 11.0 with a university license\(^\dagger\), is used for the FEA. For discretization, we use four-node quadrilateral elements (PLANE182).\(^\dagger\) We also use surface-to-surface contact elements in conjunction with the Lagrange multiplier on contact normal and penalty on tangent method for the contact algorithm (TARGE169 and CONTA171). This combination of elements and algorithm is demonstrated to be arguably the most effective means for dealing with the present type of contact problems using ANSYS by Sezer and Sinclair.\(^\dagger\)

After some tuning to capture the elastic stress gradients present, the initial coarse mesh (Mesh 1) features significant element size variation from the region near the edge of contact to the far-field (Figure 5.4(a) (see next page), in which rollers indicate symmetry conditions). Within the region of the smallest elements, element sizes are essentially uniform. Also in the far-field, element sizes are nearly uniform. Not shown in Figure 5.4(a) is the mesh for the indenter. Element nodes in the contact region on the indenter are taken so that they are aligned with those in the contact region of the indented material.

To check for convergence, we systematically refine the coarse mesh by halving element sides for the indenter and indented material to produce three additional meshes (Meshes 2, 3, 4).

\(^*\) From further FEA simulations, we find these estimates of \( d_y, d_y' \) to be fairly insensitive to the assumed value of \( \dot{\varepsilon}_r \), and so find \( \dot{\varepsilon} = 3 \times 10^{-4} / s \) in fact to be the value with the best agreement with \( \dot{\varepsilon}_r \).

\(^\dagger\) The university license limits the total number of the elements to be less than 128,000.
This results in numbers of host elements approximately quadrupling while numbers of contact elements double (Table 5.1).

![Figure 5.4 Coarse meshes](image)

(a) indentation application  
(b) single punch test problem

Table 5.1 Element numbers in meshes

<table>
<thead>
<tr>
<th>Mesh number</th>
<th>PLANE182</th>
<th>TARGE169</th>
<th>CONTA171</th>
</tr>
</thead>
<tbody>
<tr>
<td>1(h)</td>
<td>842 (1,074)</td>
<td>73</td>
<td>62</td>
</tr>
<tr>
<td>2(h)</td>
<td>3,313 (4,283)</td>
<td>146</td>
<td>123</td>
</tr>
<tr>
<td>3(h)</td>
<td>13,743 (17,466)</td>
<td>292</td>
<td>245</td>
</tr>
<tr>
<td>4(h)</td>
<td>53,754 (67,763)</td>
<td>582</td>
<td>489</td>
</tr>
</tbody>
</table>

Before running the elasto-plastic indentation simulation, two elastic test problems are carried out to verify the mesh effectiveness. The first problem is that of frictionless indentation of an elastic half-space by one rigid, long, strip punch with small edge radii. The second problem is that of frictionless indentation of an elastic half-space by a periodic array of long strip punches with sharp corners. For the single punch case, an exact solution for the contact stress distribution
is available in [17]. For the periodic punch case, an analytical solution for the interior stresses is derived and included in the Appendix B. For these two elastic test problems, we use the same element type, contact elements, and contact algorithm as those used ultimately in elasto-plastic FEA. Comparison with corresponding exact solutions then enables an assessment of the initial accuracy of our finite element meshes both on the contact surface and within the indented material.

The coordinate system for the single punch test problem and the indenter geometry are the same as those shown in Figure 5.1. From [17], the analytical solution for the contact stress, \( \sigma_c = -\sigma_y \big|_{y=0} \) on \( 0 \leq x \leq l \), is

\[
\sigma_c = \frac{E l}{2 \pi r_c (1 - \nu^2)} \left[ 2\varphi_0 \sin \varphi + \ln \left( \frac{\sin(\varphi_0 + \varphi)}{\sin(\varphi_0 - \varphi)} \right) \right],
\]

where \( E \) and \( \nu \) are now the Young’s modulus and Poisson’s ratio of the elastic half-space, respectively,

\[
\cos \varphi = x/l, \quad \cos \varphi_0 = a/l,
\]

and the contact extent is determined from the numerical solution of

\[
p = \frac{E l^2}{8(1 - \nu^2) r_c b} [2\varphi_0 - \sin 2\varphi_0].
\]

The Shtaerman solution exhibits large contact stresses near the edge of contact \( (x \approx l) \) when \( r_c/b \) is very small. Capturing of such contact stress concentrations provides a stringent test for the FEA mesh.

The configuration for the FEA of the single punch test problem is also similar to that for the elasto-plastic indentation shown in Figure 5.1. One punch together with a finite region within the indented material is considered. Due to central symmetry, only the regions \( R_I \) of the indenter
and $R_S$ of the indented material are considered. For convenience, stress-free conditions are taken for the right most boundary of $R_S$ ($x = W$).\footnote{The actual tractions that act at this location are not available in Shtaerman (1949), nor are they readily derived.} To effectively simulate the response of an elastic half space, several choices of the size of $R_S$ (i.e., the values of $W$ and $H$) are considered. The final choice of $W$ and $H$ are made by noting that further increases in their values bring little change ($< 0.2\%$) in the contact stress distribution.

The specifications for the FEA then are as follows. First, the values of $r_c/b$, $W/b$, $h/b$, $H/b$ are taken to be $1/25$, $64/3$, $8/3$ and $64/3$, respectively. Second, $E$ and $\nu$ are taken to be the same as those adopted for the indented Al previously in Section 5.2. Third, to reflect the high rigidity of the indenter as compared to that of the elastic half space, the Young’s modulus of the indenter is set to be $2 \times 10^6 E$ while its Poisson’s ratio is taken to be $0.3$. The value chosen for the indenter modulus is larger than that used in [22]: Therein contact stress results are shown to be insensitive to the choice of indenter modulus once it exceeds $10^6 E$. Finally, computations are conducted at two pressures applied on the top of the indenter, $p/E = 2.679 \times 10^{-6}$ and $p/E = 4.464 \times 10^{-6}$. The latter applied pressure corresponds to the case where initial plastic yielding will be induced in the indented Al slab simulation: The former provides a check on response at an intermediate load which actually has a higher stress concentration.

Figure 5.4(b) (see p85) shows the initial coarse mesh (Mesh 1h) for FEA of the single punch test problem. The mesh used for the elasto-plastic indentation case, shown in Figure 5.4(a), is used in the unmeshed blank region shown in Figure 5.4(b). To check for convergence, we systematically refine the coarse mesh by halving element sides to produce three further meshes (Meshes 2h, 3h, 4h). The resulting numbers of elements for Mesh 1h through 4h are
included in Table 1 in parentheses for PLANE182 elements (numbers remain the same for TARGE169 and CONTA171 elements).

For the ultimate applied pressure \( p/E = 4.464 \times 10^{-6} \), the corresponding contact stress distributions for all four meshes are compared with Shtaerman’s analytical solution (5.20) in Figure 5.5. (see next page) Except for Mesh 1h, the FEA results closely follow the exact solution over most of the contact region \( 0 \leq x/l \leq 0.9999 \). Agreement is not as good for the peak contact stress and at the edge of contact. However, the FEA results appear to be converging in both locations.

Table 5.2 Normalized peak contact stresses, \( \sigma_{\text{max}} \), for the two load levels

<table>
<thead>
<tr>
<th>Mesh number</th>
<th>( p/E = 2.679 \times 10^{-6} )</th>
<th>( p/E = 4.464 \times 10^{-6} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1h</td>
<td>123.307</td>
<td>108.404</td>
</tr>
<tr>
<td>2h</td>
<td>133.940</td>
<td>115.120</td>
</tr>
<tr>
<td>3h</td>
<td>137.813</td>
<td>117.100</td>
</tr>
<tr>
<td>4h</td>
<td>139.293</td>
<td>117.672</td>
</tr>
<tr>
<td>Exact value</td>
<td>139.821</td>
<td>117.941</td>
</tr>
</tbody>
</table>

To examine convergence of the key peak contact stress, Table 5.2 shows corresponding FEA results at both load levels. In Table 5.2, the normalized peak contact stress, \( \bar{\sigma}_{\text{max}} \), is the maximum value of normalized contact stress, \( \sigma_c/p \), on the interval \( 0 \leq x/l \leq 1 \). Peak contact stresses are uniformly converging throughout the present mesh sequence, with a convergence rate that approaches quadratic. Errors for the ultimate load are 8.1, 2.4, 0.7 and 0.2% on Meshes 1h, 2h, 3h and 4h. Corresponding errors for the intermediate load are 11.8, 4.2, 1.4 and 0.4%. These are larger because of the larger stress concentration present. However, when normalized
by the ultimate load, these errors are 7.1, 2.5, 0.9 and 0.2%. Viewed in this light, Mesh 3h has maximum local errors of less than one percent. So as to keep computational effort down, we accept this error level and judge Mesh 3 to have sufficient initial surface resolution to take the elasto-plastic simulation forward.

![Figure 5.5 Comparison of FEA and exact contact stresses for the single punch test problem](image)

Figure 5.5 Comparison of FEA and exact contact stresses for the single punch test problem

To evaluate the appropriateness of the FEA meshes for initial interior response in the actual, periodic, elasto-plastic application, the periodic punch test problem treats the frictionless indentation of an elastic half space by a periodic array of long, rigid, strip punches with sharp corners. The contact stresses in this punch problem are as in [23] for a single flat-ended punch. While these stresses thus deviate from that in the single punch test problem and our actual application near the edge of contact (as illustrated in Figure 5.5), over 99% of the contact region they agree to within 0.1%. Thus we can expect interior response away from the edge of contact to be close between this test problem and our actual application.
This geometry for the periodic test problem is similar to in Figure 5.1 with \( r_c = 0 \), thus \( l = b \), while the coordinate system remains the same as in Figure 5.1. An analytical solution to the periodic punch test problem is derived and given in the Appendix B.

Although the analytical solution developed applies to the case of punches with sharp corners, we continue to use the mesh of Figure 5.4(a) and refined versions thereof with their small radius of curvature near the edge of contact. From the foregoing discussion of the closeness of the contact stresses with \( r_c \) small or zero away from the edge of contact, we do not expect any significant effect from this discrepancy in the interior away from the edge of contact. Too, this continued use enables us to test exactly the same meshes as ultimately used in the actual elasto-plastic simulation. Material properties for the indenter and the molded material used in the single punch test problem are also adopted for the periodic test problem.

![Comparison of FEA and analytical interior stresses for the periodic test problem on the y-axis](image)

(a) \( \sigma_y / p \)  
(b) \( \sigma_x / p \)

Figure 5.6 Comparison of FEA and analytical interior stresses for the periodic test problem on the y-axis

To assess the accuracy of the present FEA, values of \( \sigma_x \) and \( \sigma_y \) along the y-axis from Meshes 1-4 are plotted in Figure 5.6 and compared with the analytical solution (from (B8) of the Appendix B). For \( y / b \geq 2 \), results for Meshes 2-4 have converged to the analytical solution and
are virtually indistinguishable from it on the scale in Figure 5.6. For $y/b < 2$, both $\sigma_x$ and $\sigma_y$ are converging on Meshes 2-4, and have converged to within 0.4% on Mesh 3. A similar comparison holds for $\sigma_x$ and $\sigma_y$ along the symmetry line at $x = W$.

In the near field ($y/b < 2$), there are small but persistent differences between converged FEA stresses and analytical stresses (maximum difference $\approx 1\%$). This is because of differences in geometry near the edge of contact between the problem subjected to FEA and the problem solved analytically: The former has a geometry as in the close up in Figure 5.1, the latter a geometry for a sharp-edged punch. Given this explanation, then, we accept Mesh 3 as having sufficient initial interior resolution to take the elasto-plastic simulation forward. In what follows, therefore, we use Mesh 3. To provide some confirmation of this choice, we also use Mesh 1.

In taking the elasto-plastic simulation forward with Mesh 3, we need to select the load steps to be used. In the elasto-plastic simulation, the “load” is actually applied via the displacement of the indenter, $d$, and the pressure required, $p$, then backed out: With elastic perfectly-plastic material response, such an inverse approach tends to provide greater stability in the simulation. At the outset we do this for as large an increment in $d$, $\Delta d$, as possible so as to reduce computation. After some trial and error we find that $\Delta d = 0.1\mu m$ enables the code to converge for $d \geq 2\mu m$ but not for $d < 2\mu m$: For $d < 2\mu m$, we find that $\Delta d = 0.01\mu m$ suffices (except for the first load step for which $\Delta d = 0.002\mu m$, corresponding to the initiation of yield in the Al). With these initial choices, we check for convergence of the pressure $p$ with load step by successively halving $\Delta d$. Table 5.3 (see next page) gives values of $p$ for the resulting load increments and for varying depths throughout the range treated in the simulation (maximum $d = \ldots$)

---

§ That this is so can be confirmed by subjecting the actual problem solved analytically to FEA with the same Meshes 1-4 except for some local modifications near $x = l$. When this is done, differences between converged FEA results and the analytical solution are removed.
37µm). Based on differences between pressure values at a given depth, the values of $p$ are consistent with numerical convergence with load step, albeit oscillatory convergence, and appear to have converged to within 0.1%. We thus use $\Delta d = 0.0025\mu m$ before $d = 2\mu m$ and $\Delta d = 0.025\mu m$ after $d = 2\mu m$ in the simulation.

Table 5.3 Load step convergence check of pressure, $p$ (MPa)

<table>
<thead>
<tr>
<th>Molding depth, $d$ ($\mu m$)</th>
<th>$\Delta d$</th>
<th>$\Delta d/2$</th>
<th>$\Delta d/4$</th>
<th>$\Delta d/4$(DR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>43.926</td>
<td>44.056</td>
<td>44.013</td>
<td>44.021</td>
</tr>
<tr>
<td>10</td>
<td>45.290</td>
<td>45.285</td>
<td>45.285</td>
<td>45.289</td>
</tr>
<tr>
<td>20</td>
<td>46.360</td>
<td>46.337</td>
<td>46.353</td>
<td>46.359</td>
</tr>
<tr>
<td>30</td>
<td>47.241</td>
<td>47.294</td>
<td>47.257</td>
<td>47.277</td>
</tr>
<tr>
<td>35</td>
<td>47.693</td>
<td>47.763</td>
<td>47.745</td>
<td>47.725</td>
</tr>
</tbody>
</table>

During the elasto-plastic simulation, appreciable deformation can accumulate. To avoid corresponding mesh distortion, rezoning is applied (REZONE$^{18}$). Before $d = 2\mu m$, we simply rezone whenever required to keep the elasto-plastic simulation continuing. That is, for example, when the code does not converge at load step 40, we back up to load step 36, rezone, and continue. This results in rezoning at load steps 36, 60, 76, 180, 280, and 800. After $d = 2\mu m$, we find that the simulation can proceed with rezoning every ten load steps (because $\Delta d$ is now ten times bigger, this corresponds to 100 of our initial $\Delta d$). To check for convergence, we then double the number of rezones. Table 5.3 includes results for this double rezoning and our smallest load steps (headed by $\Delta d/4$ (DR)): These appear to have converged to within 0.05%.

Ultimately, at $d = 37\mu m$, we cannot get the version of ANSYS used to converge for further load steps.$^{18}$ This lack of convergence persists for either Mesh 3 or Mesh 1, for yet
smaller $\Delta d$, and for double rezoning. Possibly a more refined mesh than allowed by the university license limits would enable the simulation to progress further. Here, though, the results that follow are only up to this modest ultimate depth of 37µm (cf. experimental molding depths of 220µm).

5.4 Finite Element Results and Validation

The molding response, as determined by the FEA, is displayed in Figure 5.7 in terms of the molding pressure $p$ normalized by the yield strength $\sigma_y$ versus the molding depth $d$ normalized by the punch half width $b$. An initially stiff response is observed, with $p/\sigma_y$ rising rapidly with increasing $d/b$, followed by a much more compliant response, with $p/\sigma_y$ increasing approximately linearly with $d/b$. A fairly sharp knee separates these two regimes of predominantly elastic response and plastic flow.

![Figure 5.7 Molding response: normalized molding pressure $p/\sigma_y$ vs normalized molding depth $d/b$ with different meshes](image)

The mesh dependence of the FEA output is also illustrated in Figure 5.7, which shows two molding response curves obtained with Mesh 1 and 3. On the scale of Figure 5.7, no
perceptible difference can be discerned in the initial stiff elastic portion of the response curve. In
the more compliant plastic regime, the difference in the values of $p/\sigma_Y$, as calculated from Mesh
1 and 3, is on average 1.5% and at most 3.1% within the range $0.05 \leq d/b \leq 0.5$. Thus the
normalized molding pressure is not that sensitive to the mesh employed, and either mesh could
suffice to compute this response. We comment further on underlying reasons for the good
agreement between molding pressures for the two meshes subsequently when we discuss contact
stresses, though certainly one factor is that $p/\sigma_Y$ vs. $d/b$ represents an averaged response
integrated over the entire punch, hence is less sensitive to the detailed determination of the stress
distribution underneath the indenter.

Figure 5.8 Comparison of molding response: FEA and experiment

Figure 5.8 shows experimentally measured molding response of Al at 360°C, together
with the corresponding FEA results. The experimental data points shown in Figure 5.8 represent
the collection of data from five separate molding runs under the same nominal conditions. At
small molding depths of $d/b < 0.02$, the data scatter is the most evident. The actual molding
depth, i.e., the depth of penetration of the punch into the molded material, was obtained by
subtracting the estimated system stiffness contribution from the directly measured total displacement. The system stiffness was estimated from separate calibration runs, and was $19 \pm 3\text{kN/mm}$. This uncertainty in system stiffness represents a displacement uncertainty of $\pm 10\mu\text{m}$ at a load of 1kN. The apparent negative molding depth shown in Figure 5.8 simply reflects the uncertainty in the estimated system stiffness. Also shown in Figure 5.8 is the FEA output for the normalized molding response of Al, using the corresponding $\sigma$-$\varepsilon$ relationship shown in Figure 5.2(a) for Al as input. It is noted that both the experimental data and the FEA output show a first regime with a stiff response followed by second regime with a much less stiff response. In the second regime, pressure increases approximately linearly with increasing molding depth. A relatively sharp turning point separates the two regimes. From the experimental data, the turning point was located at $p/\sigma_y \approx 2.4$. The FEA results yielded a turning point value of $p/\sigma_y \approx 3.0$, and differ from the experimental values by about 25%. It should be noted that punch indentation, in contrast to uniaxial tension, is not an iso-strain problem. Therefore an uncertainty exists in the selection of the $\sigma$-$\varepsilon$ relationship obtained from tension experiments conducted at one single strain rate and applying it to the FEA of the indentation problem. Considering this uncertainty, the 25% difference represents a reasonable agreement.

The FEA generated molding response in the second less-stiff regime is of interest. It is somewhat counter intuitive to see in Figure 5.8 that the FEA generates an increasing $p/\sigma_y$ vs. $d/b$ curve from an elasto-perfectly plastic $\sigma$-$\varepsilon$ curve (Figure 5.2(a)) and without any friction effects. We offer a qualitative argument for this response by considering conservation of energy as indentation proceeds.
Ignoring elastic strains and any temperature increases, the work done by the indenter must equal the plastic strain energy produced. Thus for a perfectly-plastic substrate occupying the periodically representative region $R_3$, conservation of energy has

$$pbd = \sigma_r \varepsilon_r A_r.$$  \hspace{1cm} (5.23)

In (5.23), $A_r$ is the area of the yield region within $R_3$ and $\varepsilon_r$ continues as a representative strain throughout $A_r$. Increasing the molding pressure to $p + \Delta p$ produces in $d$ to $d + \Delta d$, $\varepsilon_r$ to $\varepsilon_r + \Delta \varepsilon_r$ and $A_r$ to $A_r + \Delta A_r$, while $\sigma_r$ remains constant because the substrate is perfectly-plastic. Then the incremental work done is balanced by the incremental increase in the plastic strain energy, that is,

$$pb\Delta d = \sigma_r (A_r \Delta \varepsilon_r + \varepsilon_r \Delta A_r).$$  \hspace{1cm} (5.24)

Introducing the dimensionless variables $\delta = d / b$ and $\alpha_r = A_r / b^2$ then taking the limit $\Delta \delta \to 0$ gives

$$\frac{p}{\sigma_r} = \frac{d}{d\delta} (\varepsilon_r, \alpha_r).$$  \hspace{1cm} (5.25)

Hence on differentiating

$$\frac{d}{d\delta} \left( \frac{p}{\sigma_r} \right) = \frac{d^2}{d\delta^2} (\varepsilon_r, \alpha_r),$$  \hspace{1cm} (5.26)

and the molding response stiffens even when the substrate is perfectly-plastic if the second derivative of the total plastic strain, $\varepsilon_r \alpha_r$, is positive.

Although a careful calculation of the second derivative in (5.26) is difficult, an estimate can be obtained following along the lines of the estimate found for the representative strain rate in Section 5.2. Specifically, estimates of $\varepsilon_r$ and $A_r$ can be obtained from (5.19) and the
simplified description of the yield region as shown in Figure 5.3. These estimates involve \( d_y \) and \( \overline{d}_y \). Fitting FEA values for these depths gives

\[
d_y / b = 2.05\delta + 3.72
\]

\[
\overline{d}_y / b = 2.41\delta + 1.55,
\]

(5.27)

for \( \delta \geq 0.226 \) (cf. Figure 5.13(a): the \( d_y \) need for \( \overline{d}_y \) is not clearly defined for \( \delta < 0.226 \)). Substituting (5.27) into the estimates, assembling the product term in (5.26), differentiating, then evaluating, in fact gives a right-hand side that is positive but decreasing to zero as \( O(\delta^{-3}) \) as \( \delta \) increases. Thus following the sharp turning point, the preceding energy argument offers an explanation of how the molding pressure can increase with depth despite the substrate being perfectly-plastic. Ultimately, however, this source of stiffening can be expected to be negligible.

In the light of the foregoing, an additional source of the apparent stiffening of the \( p/\sigma_Y \) vs. \( d/b \) curve needs to be identified. This is the friction between the indenter and the indented material once contact is established on the indenter sidewall. Experimental examinations of the sidewall morphologies of molded metal microchannels, one typical example of which is shown in Figure 4.2(c), indicate the existence of sidewall contact, and consequently, a frictional contribution to the total molding force. The plane-strain FEA, however, did not show indenter sidewall contact up to the indentation depth of \( d/b \approx 0.5 \). We believe this to be a consequence of over-constraining due to plane-strain conditions. Therefore, additional FEA of the same indentation geometry using plane-stress condition was carried out. The plane-stress simulation indeed revealed sidewall contact from the beginning of the indentation process (but yielded a turning point at \( p/\sigma_Y \) of about 1). The plane-strain and plane-stress conditions represent respectively an over-constraining and an under-constraining as compared to the physical indentation problem, a more accurate representation of which may require a three-dimensional
FEA. It is apparent from Figure 5.8 that the experimentally measured molding response shows a steeper slope than the corresponding FEA output, consistent with the fact that the plane-strain FEA did not include the frictional contribution.

![Figure 5.9 Molding response regimes](image)

Figure 5.9 Molding response regimes

Figure 5.9 shows the initial molding response according to the FEA. In order to capture the early response in more detail, a log-scale is used on the $d/b$ axis. Then the molding response can be categorized into three regimes. An elastic contact regime encompasses a very small range of indentation depth, $d/b < 3 \times 10^{-4}$. An elasto-plastic regime follows when a yield region initiates underneath the corner of the indenter. As $d/b$ increases from $3 \times 10^{-4}$ to $8 \times 10^{-3}$, $p/\sigma_Y$ increases rapidly. This increase appears exponential due to the nature of the log plot. In actuality, the increase of $p/\sigma_Y$ with increasing $d/b$ maintains an approximate linear character with a slightly reduced slope as compared to that in the elastic regime. At $d/b \approx 8 \times 10^{-3}$, a sharp knee in the $p/\sigma_Y$ vs. $d/b$ curve is observed. At this knee, there is an apparent discontinuity in the slope of the $p/\sigma_Y$ vs. $d/b$ curve. Such a clear transition is absent from spherical indentation problems, but has been
observed in a previous plane-strain simulation of cylindrical indentation.\textsuperscript{24, 25} This knee sets the beginning of what we choose to term a fully plastic regime.

To track the change in contact stress distribution as indentation progresses from the elastic regime to elasto-plastic regime and beyond, a contact coordinate $s$ is defined first, which runs along the edge of the punch. The values of the normalized contact coordinate, $s/b$, of 0 ($s = 0$) and 0.96 ($s = a$) correspond to locations at the punch center and at the end of the flat punch bottom, respectively. As the punch sinks into the indented material, $s/b$ can become larger than 1. In this case where zero friction is assumed, the contact stress $\sigma_c$ is defined as the stress component normal to the contacting surfaces. The so-defined $\sigma_c$ is consistent with the contact stress defined in the elastic contact regime, as shown on page 12. Figure 5.10(a) shows the distribution of $\sigma_c$ along the edge of the punch obtained with Mesh 1. The two $\sigma_c$ profiles shown in Figure 5.10(a) correspond to $p/\sigma_y = 0.5$ and 1.0, respectively.

![Contact stress distributions at different normalized pressures in the elasto-plastic regime](image)

**Figure 5.10** Contact stress distributions at different normalized pressures in the elasto-plastic regime

Figure 5.10(a) shows that a significant contact stress concentration, $K_c \equiv \sigma_c^{\text{max}} / p$, persists into the elasto-plastic contact regime. Even though $K_c$ has decreased from about 115 at
the initiation of plastic yielding, its values are still about 6 and 3 at \( p / \sigma_y = 0.5 \) and 1.0, respectively. Figure 5.10(b) shows the same \( \sigma_c \) distributions obtained with Mesh 3. Comparison between Figures 5.10(a) and (b) indicates a mesh dependence of the \( \sigma_c \) distribution, especially near the corner of the punch. In order to capture finer details of the \( \sigma_c \) distribution, a mesh finer than Mesh 3 may be needed. Figure 5.11 shows the \( \sigma_c \) distribution corresponding to \( p / \sigma_y = 3.2 \), obtained with Mesh 3. At this load, \( d/b \approx 0.47 \) and the indentation is in the fully plastic regime. It is apparent from Figure 5.11 that the contact stress concentration is almost completely abated, with \( K_c \approx 1.03 \). Without the sharp contact stress variation, little mesh dependence is observed. Figure 5.11 shows that, in the fully plastic regime, the punch is subjected to approximately an uniform compression, with little contact stress variation across the punch bottom.

![Figure 5.11 Contact stress distributions in the fully plastic regime](image)

Figure 5.11 Contact stress distributions in the fully plastic regime

Figure 5.12 (see next page) shows \( \sigma_x \) and \( \sigma_y \) distributions along the y-axis within the interior of the indented material, obtained with Mesh 1 and Mesh 3. These interior stress
distributions show little mesh dependence. While equal values of $\sigma_x$ and $\sigma_y$ are expected at $y = 0$ in the single punch problem, the current configuration of periodic punches makes these values unequal at the surface.

Figure 5.12 FEA interior stresses on the y-axis

Figure 5.13 Yield region propagation
Contours of von Mises stress $\sigma_{VM}$ distribution in the indented material are shown in Figure 5.13(a). As the indentation depth increases, the boundary between yielded and unyielded regions propagates deeper into the indented material. The geometry of the yield region is characterized by the yield region depth at $x = 0$ ($d_y$) and $x = W$ ($d'_y$), as described in Figure 5.3. With this description, the propagation of the yield region as indentation progresses can be viewed by tracking $d_y$ and $d'_y$ as a function of the indentation depth $d$, the normalized plots of which are shown in Figure 5.13(b).

Therein, as $d/b$ increases beyond 0.01 (for $d_y$) and 0.25 (for $d'_y$), $d_y$ and $d'_y$ are approximately linear with respect to $d$. This means that, in this state, the boundary between yielded and unyielded regions translates linearly with the indenter into the indented material. This phenomenon is analogous to how the yielded region propagates into the indented material during spherical indentation. A previous FEA showed that, in the case of spherical indentation, the vertical and lateral size of the yielded region scale linearly with the contact width $2a$.\textsuperscript{7, 8} Furthermore, the geometric shape of the yielded region remains approximately constant when scaled by $2a$, and is therefore self-similar. The concept of geometrically self-similar propagation of the yield region, in the present situation where the indenter width or the contact width is a constant, is consistent with our present FEA results showing linear translation of the boundary between yielded and unyielded regions with the indenter.

## 5.5 Concluding Remarks

An initial FEA of the elasto-plastic indentation of Al by a periodic array of frictionless flat punches with rounded corners has been performed. The FEA mesh used was calibrated in the elastic contact regime against a known analytic solution for the same geometry. The contact stresses obtained with this mesh are shown to be accurate to within about 1% in the elastic range,
in the vicinity of the greatest stress concentration which is on the order of 100 times the average pressure. With this calibrated mesh, the indentation process is tracked by the FEA into the elasto-plastic regime. The indentation response, as expressed by the applied pressure normalized by the yield stress of Al as a function of the indentation depth normalized by the punch half-width, is shown to consist of two stages. In the initial stage, the indentation response is observed to be stiff. Following a sharp transition, the normalized pressure is approximately linear with respect to the normalized molding depth in the late stage. The propagation of the yield region in the latter stage appears to be consistent with geometric self-similarity as the extent of indentation progresses.

5.6 References

2. H. T. Huber, Stresses in the contact of two elastic spheres. Annals Physics 14, 153, 1904


13 K. L. Johnson, Contact mechanics, Cambridge University Press, 1985


23 M. A. Sadowsky, Two-dimensional problems of elasticity theory (in German). Zeitschrift für Angewandte Mathematik und Mechanik 8, 107-121, 1928

104

Chapter 6. Direct Microscale Imprinting of Al at Room Temperature with Si Inserts

6.1 Introduction

Metal-based high-aspect-ratio micro-/nano-scale structures (HARMS/HARNS) enable functions not achievable with polymeric micro/nano electromechanical system devices. Metallic HARMS can be used in heat exchangers and electromagnetic relays for high power applications.\(^1\),\(^2\) Metallic HARNS can function as components in subwavelength photonic devices.\(^3\) Metallic HARMS/HARNS can be formed by physical and chemical deposition processes on a substrate followed by a lift-off process. An alternative method for producing such metallic HARMS/HARNS at low cost and with high throughput is to directly mold a metallic substrate using a mold insert. Replication of HARMS in various metals by compression molding at elevated temperatures has been demonstrated.\(^4\),\(^5\) Pang et al. have shown direct molding replication at room temperature in Al with SiC inserts fabricated by electron beam lithography (EBL) and reactive ion etching (RIE), creating HARNS with lateral dimensions \(\sim 40\) nm.\(^6\) The reason given for the choice of SiC inserts was that inserts made of Si are easily broken during the molding process.\(^6\) This impression is echoed by additional studies of direct metal nanoimprinting with inserts made of refractory ceramics, such as diamond and SiC.\(^7\)

However, the possibility of using Si inserts for compression molding of metals should not be dismissed too easily. As compared to SiC, diamond, and other refractory ceramics, Si-based inserts incur less material cost and are more easily fabricated. Protocols for creating micro-/nanoscale complex patterns in Si with appropriate aspect ratio and sidewall roughness by photolithography (PL)/EBL and RIE are well established.\(^8\) Successful use of Si inserts in molding
replication of metallic HARMS/ HARNs without damage will improve the versatility of micro-
/nano- scale molding replication technology.

In this chapter, we first show experimental results that demonstrate successful micron
scale molding of Al at room temperature with Si inserts without insert damage. We then
summarize a companion finite element analysis (FEA) undertaken to understand the
experimental observations.

6.2 Molding Experiments

For the molding experiments, arrays of parallel rectangular microprotrusions were
fabricated on 500 µm thick Si(100) wafers by PL and RIE. Patterned areas are 2mm×2mm.
Following PL and RIE, the Si wafers were surface etched in a pure Ar(99.999%) inductively
coupled plasma (ICP). After the ICP etch, a Cr interlayer was deposited onto the structured Si
wafer with magnetron sputtering and followed by deposition of a conformal amorphous silicon
nitride (a-Si:N) layer. The thicknesses of Cr and a-Si:N layers were both ~ 200nm. Further
details on coating deposition and utility in molding replication were reported elsewhere. After a-
Si:N deposition, the pitch of the protrusions was measured to be 11.7 µm. The height and width
of each microprotrusion were measured to be 20 µm and 6.7 µm, respectively. The Si wafers were
diced around the patterned areas, leaving pieces slightly larger than the 2mm×2mm patterned
areas as mold inserts.

As-received, pure Al foils (99.99at.%, annealed), 250 µm in thickness, were molded at
room temperature using the Si inserts. The molding experiments were carried out on a MTS 858
Mini Bionix® II single-axis testing system. A Ni alloy actuator rod was pressed directly on the
back of the Si insert, which was placed on top of the Al foil sitting on a flat Ni alloy platform.
The Si insert was indented into the Al foil at room temperature by loading the actuator rod at a
constant rate of 50N/min to a maximum force of 500N. This loading caused the Si insert to sink into the Al foil. After indentation, demolding occurred by mechanically separating the Si insert from the molded Al foil. Scanning electron microscopy (SEM) examinations of Si inserts and molded Al features were carried out on a Hitachi S-3600N microscope operated at 15kV.

6.3 Molding Experiment Results

Figure 6.1 Room temperature replication of microscale Al structures by compression molding (a) an intact Si insert after one Al molding (b) the corresponding replicated Al structure

Figure 6.1(a) shows the state of an a-Si:N coated Si insert after one room temperature Al compression molding run. It is clear from Figure 6.1(a) that the Si insert remained intact after the Al molding and insert-Al separation processes, and that little Al transfer occurred from the molded Al foil to the insert. Figure 6.1(b) shows the corresponding replicated Al structure. The parallel array of rectangular microprotrusions is replicated into a parallel array of rectangular microchannels in Al. The depth of replicated Al features was estimated from SEM images to be ~15µm, while the width and pitch of the Al microchannels were measured to be respectively ~6.7µm and ~11.7µm, demonstrating good replication fidelity.

It is apparent that no damage occurred to the Si insert shown in Figure 6.1 due to Al molding, as was the case for 4 more molding runs. We did observe Si insert breakages during
additional Al molding runs, occurring either during molding or demolding. Our observations of these breakages suggest that they were caused by bending stresses induced by insert misalignment during molding or uneven shear tractions during demolding. Nonetheless, the results shown in Figure 6.1 indicate that, absent such misalignment, Si inserts can be used for room temperature Al molding without damage.

### 6.4 Finite Element Analysis

To gain a better understanding of why the Si insert can remain intact, we next describe a finite element stress analysis of Al compression molding. For the FEA, the molding process was modeled as plane strain indentation of an elasto-plastic slab by a periodic array of long strip punches. Figure 6.2 shows the indentation geometry in cross section.

![Figure 6.2](image)

**Figure 6.2** Geometry for the FEA model of indentation of an elasto-plastic slab by a periodic array of rectangular punches

In Figure 6.2, the center-to-center distance between punches is \( l \). The individual punches are of width \( 2b \), with flat bottoms of width \( 2a \) and small edge radii \( r_0 \). From measurements on the present Si inserts, \( l, b, a, \text{ and } r_0 \) are, respectively, 11.7µm, 3.35µm, 2.95µm, and 0.4µm. Each
punch is long out of plane and is pressed into the slab with an applied pressure $p$, where $p = p_0(2b/l)$, $p_0$ being the pressure applied to the entire insert. The thickness of the slab is $h$ and it rests on a smooth rigid foundation. Planes of periodic symmetry occur halfway between punches and under the punch centers. By symmetry, the indentation response of the entire punch array is captured by FEA of two representative regions, $R$ within the indented material and $R'$ within the punch.

To facilitate the analysis, frictionless contact is assumed and friction effects simply inferred from known contact problems. The Si insert is taken to be a homogeneous and isotropic, linear elastic solid, with a Young’s modulus $E = 163$GPa and a Poisson’s ratio $\nu = 0.22$. The elastic anisotropy of Si is not considered important for present purposes. Because of their small thicknesses and elastic moduli similar to that of the Si insert, the coatings are expected to have little influence on contact stresses $\sigma_c$. Furthermore, no coating failure was observed experimentally. Therefore, although the FEA took the overall dimension of the insert, including the coating contribution, into account, the coatings were otherwise not considered in the FEA.

The stress strain curve of the elasto-plastic Al slab was determined through uniaxial tensile testing of cylindrical 1100O Al bar specimens (99%+ Al, annealed, gauge diameter 6.35 mm) rather than foils to facilitate testing. The measured true stress strain curve is shown in Figure 6.3. (see next page) This curve is linearly elastic, with $E = 69$GPa and $\nu = 0.33$, up to a yield stress $\sigma_y = 10$ MPa, and thereafter hardens to reach an ultimate stress $\sigma_U = 24$ MPa at a true strain of 20%. Further description of the FEA is given elsewhere. The actual FEA included choices of element type and contact algorithm, as well as meshing, verification with an elastic contact problem with a known analytical solution, convergence checks, and rezoning with plastic flow. Here only the pertinent results are summarized.
Figure 6.3 Al1100O stress strain curve (room temperature)

Figure 6.4 Progression of applied pressure as indentation progresses into the elasto-plastic regime

Figure 6.4 shows the progression of normalized applied pressure, $p/\sigma_y$, as a function of the normalized indentation depth, $d/b$. This indentation response shows an initial stiff section followed by a much more compliant section. As $d/b$ increases from 0 to 0.08, $p/\sigma_y$ increases
rapidly from 0 to ~6. In the more compliant section, \( p/\sigma_Y \) is approximately linearly proportional to \( d/b \) and exhibits a slower increase from 6.8 to 9.7 as \( d/b \) increases from 0.14 to 0.75.

The first inset in Figure 6.5 shows the distribution of contact stress, \( \sigma_c = -\sigma_z \), along the bottom of the punch. The values of the normalized contact coordinate, \( s/a \), of 0 and 1 correspond to the locations at the punch center and the end of the flat punch bottom, respectively.

![Progression of contact stress concentration](image)

Figure 6.5 Progression of contact stress concentration as indentation progresses into the elasto-plastic regime. The two insets show \( \sigma_c \) distributions at \( d/b \) values of \( 4 \times 10^{-5} \) and 0.85, respectively. The arrows link the insets to their corresponding \( K_c \) values.

This \( \sigma_c \) profile corresponds to \( d/b = 4 \times 10^{-5} \) and the point where plasticity is first initiated within the indented Al. At this depth, the maximum value of the contact stress \( \sigma_c^{\text{max}} \) is located very close to \( s/a = 1 \), due to the small edge radius. A large contact stress concentration, \( K_c = \sigma_c^{\text{max}} / p \sim 65 \), exists at this depth. As \( p \) increases, the indentation progresses into the elasto-plastic regime. The increase in \( \sigma_c^{\text{max}} \) near the punch edge \((s/a \approx 1)\) are less rapid than those in \( \sigma_c \) near the punch center \((s/a = 0)\), because the Al at the punch edge is rendered more compliant by virtue of having yielded there. As shown in Figure 6.5, this leads to a rapid decrease in \( K_c \) even
though $d$ remains relatively small ($K_c \sim 1.3$ at $d/b \sim 0.05$). At larger indentation depths of $d/b > 0.075$, $K_c$ remains approximately constant at 1.2, reflecting a small but persistent elevation of $\sigma_c$ around the punch corner. As illustrated in the second inset of Figure 6.5, $\sigma_c$ is approximately constant across much of the punch bottom as the indentation goes well into the elasto-plastic regime, with $0.9 < \sigma_c / p < 1.2$. In this regime, the indented material makes contact with the punch sidewall. This sidewall contact pressure is again approximately a constant, with $\sigma_c / p \sim 0.2$, corresponding to a sidewall contact pressure of $\sim 2\sigma_y$. During insert withdrawal, this sidewall contact pressure decreases rapidly.

### 6.5 Discussion

The FEA results illustrate several important characteristics of the mechanics of metal micromolding. First, the magnitude of the applied pressure $p$ is limited by plastic yielding within the indented material. The value of $p/\sigma_Y$ increases from 6.8 to 9.7 as $d/b$ increases from 0.14 to 0.75, and is linearly extrapolated to be $\sim 25$ at $d/b \sim 4$. Thus, in correspondence to the present experiments, $p$ is limited to be less than $\sim 25\sigma_Y$ at an aspect ratio of molded features of two or less, or $p$ is less than 250MPa.

Second, the implications of these FEA results are that fracture and fragmentation at edges of contact are not likely for the Si micro-punches during loading. This is because the directions of relative motion between the punch and the indented material do not produce any tensile stresses in the punch. In addition, the reduction in $K_c$ must produce a like reduction in frictional shear, resulting in shear stresses within the punch below the shear strength of Si. More specifically, peak shear stress is of the order of $f\sigma_c^{\text{max}}$, with $f$ being the coefficient of friction. Taking a high value of $f = 1$, this gives a peak shear stress of $\sim 30\sigma_Y$ at $d/b \sim 4$, or $\sim 300$MPa. Shear stresses within the punch bulk are less than one half of this value.
Ideal tensile and shear strengths of Si are reported to be 22 and 6.8GPa, respectively.\textsuperscript{16} Experimentally measured fracture strengths of Si depend critically on the presence of strength-limiting defects within the specimen.\textsuperscript{17} Tensile testing of microscale Si specimens with characteristic dimension of $\sim 20\mu$m yielded tensile strength values of $\sim 1.2GPa$\textsuperscript{18} placing their compressive and shear strengths at above 1.2GPa and 600MPa, respectively, and well in excess of estimated peak shear stresses within the punch during molding.

Experimentally, fracture and fragmentation of Si micropunches at edges of contact were not observed during room temperature Al molding runs, consistent with the shear stress estimation presented above. When insert breakage occurred, arrays of micro-punches were observed to separate from the flat foundation of the insert at corners (e.g., location C in Figure 6.2), and to be buried within the molded Al. Without having carried out detailed analysis, we can nevertheless see two scenarios leading to such breakages. First, a slight misalignment during molding can put one edge of the punch in contact with the molded Al first (e.g., at $z = 0$ and $x = a$ in Figure 6.2). Such edge contact can generate tensile stresses at C due to bending. Second, misalignment during demolding can lead to uneven frictional tractions on the two sides of the punch sidewall, which can also lead to tensile stresses at C due to bending. Any such tensile stresses would be greatly amplified by the sharp corner at C, and hence could lead to fracture. On the other hand, the present experimental observations and FEA results suggest that, absent such misalignments during molding or demolding, Si inserts can be used to mold Al without damage.

It should be noted that the present FEA is based on continuum mechanics, and so contains no dependence on the characteristic size of molded features. Possible size dependence of materials’ response in the context of micro-/nano-scale molding replication has not been explored and remains a topic of future study. However, because the Si fracture strength can be
expected to increase with decreasing specimen size, Si inserts can be expected to perform as well as in the present case or better for direct nanoscale imprinting of metals.

### 6.6 Summary

In summary, room temperature micron scale compression molding of Al foils was demonstrated experimentally without damaging the Si inserts. The mechanics of the molding process was assessed through FEA, the results of which offer some justification of the present experimental observations.

### 6.7 References

2. J. D. Williams, W. Wang, Microfabrication of an electromagnetic power relay using SU-8 based UV-LIGA technology, Microsystem Technologies, 10(10), 699, 2004
4. D. M. Cao, W. J. Meng, Microscale compression molding of Al with surface engineered LiGA inserts, Microsystem Technologies, 10(8-9), 662, 2004
10 W. A. Brantley, Calculated elastic constants for stress problems associated with semiconductor devices, Journal of Applied Physics, 44, 534. The Si elastic constants, $E$ and $\nu$, were taken to be the average of Voigt and Reuss values.


Appendix A.  High Temperature Tensile Test Specimen Drawing

NOTES:
1. ALL SPECIMENS AND MATERIAL MUST BE IDENTIFIED AT ALL TIMES WITH HEAT NUMBER.
2. NO DRILLING MATERIAL MUST NOT BE GROUND AT ANY TIME DURING THE FABRICATION OF SPECIMENS.
3. ALL DIAMETERS MUST BE CONCENTRIC WITHIN 0.0001" TIR.
4. REDUCED DIAMETER D1 MUST BE WITHIN 1/4" OF GAGE LENGTH CENTER LINE.
5. ALL RADIUS MUST BLEND WITHOUT UNDERCUTS OR STEPS.
6. RETURN ALL EXCESS MATERIAL TO REQUESTER.
7. THREADS MUST BE TURNED CONCENTRIC TO CENTER LINE WITHIN 0.001" TIR
   AND CLASS 2 FIT WITH SPECIMEN GRIP (SUPPLIED BY REQUESTER).
8. SPECIMEN GAGE LG. TO HAVE UNIFORM SLOPE FROM MAX DIA. D2 TO MIN DIA. D1,
   MAX UNIFORM SLOPE NOT TO EXCEED 0.002" CHANGE IN DIA. PER 1 INCH OF
   CHANGE IN LENGTH.

SPECIMEN
TENSILE SPECIMEN -- H/HV

DIAMETERS

- D1 = 0.250" ± 0.001"
- D2 = FROM 0.0005" TO 0.0015"
  GREATER THAN D1.

REFERENCE DRAWINGS

U. S. NATIONAL LABORATORY
UPSKILLED BY
UNION CARBIDE CORPORATION
OAK RIDGE, TENNESSEE
METALS & CERAMICS DIVISION MC-4800-5

SPECIMEN
TENSILE SPECIMEN -- H/HV

DIAMETERS

- D1 = 0.250" ± 0.001"
- D2 = FROM 0.0005" TO 0.0015"
  GREATER THAN D1.

REFERENCE DRAWINGS

U. S. NATIONAL LABORATORY
UPSKILLED BY
UNION CARBIDE CORPORATION
OAK RIDGE, TENNESSEE
METALS & CERAMICS DIVISION MC-4800-5

SPECIMEN
TENSILE SPECIMEN -- H/HV

DIAMETERS

- D1 = 0.250" ± 0.001"
- D2 = FROM 0.0005" TO 0.0015"
  GREATER THAN D1.

REFERENCE DRAWINGS

U. S. NATIONAL LABORATORY
UPSKILLED BY
UNION CARBIDE CORPORATION
OAK RIDGE, TENNESSEE
METALS & CERAMICS DIVISION MC-4800-5

SPECIMEN
TENSILE SPECIMEN -- H/HV

DIAMETERS

- D1 = 0.250" ± 0.001"
- D2 = FROM 0.0005" TO 0.0015"
  GREATER THAN D1.
Appendix B. Stress Analysis for Periodic Test Problem

The inverse problem that serves as our periodic punch test problem may be stated as follows. In general, we seek the plane strain stresses $\sigma_x$, $\sigma_y$ and $\tau_{xy}$, and their associated displacements $u$ and $v$, as functions of $x$ and $y$ throughout $R_S$, satisfying the appropriate field equations and boundary conditions. The field equations are: the two-dimensional stress equations of equilibrium, and the stress versus displacement relations for a linear elastic, homogeneous and isotropic, solid in a state of plane strain for $R_S$. The boundary conditions are: the applied stress conditions

$$\tau_{xy} = 0, \quad (B1)$$
onumber

on $0 < x < l$, where $p$ is now the average pressure on this interval, together with stress-free, periodic symmetry, and far-field roller conditions as in (5.12), (5.13) and (5.14) sans dots. In particular, we are interested in the interior stresses on the $x$-axis.

The solution of the foregoing problem follows from the superposition of Airy stress functions $\phi_0$ and $\phi_n$ given by

$$\phi_0 = -\frac{pl}{2W}(l^2 - x^2)^{1/2}, \quad (B2)$$

$$\phi_n = [\text{ch}\alpha_n(H - y) + \beta_n(H - y)\text{sh}\alpha_n(H - y)]\cos\alpha_n x,$$

where $\alpha_n$ and $\beta_n$ are constants. Because these are both biharmonic functions, stresses generated by them using the usual relations are not only in equilibrium but also compatible, thereby ensuring companion displacements exist. For $\phi_0$, such stresses and ensuing displacements capture average stress effects in $R_S$ and satisfy (5.13) and (5.14).
sans dots. For \( \phi_n \), stress and displacements satisfy like versions of (5.13) and (5.14) provided

\[
\alpha_n = n\pi / W ,
\]  

(B3)

for \( n = 1, 2, \ldots \). The shear-free conditions in (B1) and (5.12) sans dots can then be met by taking

\[
\beta_n = -\frac{\alpha_n \theta \alpha_n H}{\alpha_n H + \theta \alpha_n H} .
\]  

(B4)

With (B3) and (B4) holding in (B2), our complete Airy stress function \( \phi \) is then given by

\[
\phi = \phi_0 + \sum_{n=1}^{\infty} a_n \phi_n ,
\]  

(B5)

wherein \( a_n \) are the coefficients to be adjusted to meet the one remaining boundary condition, \( \sigma_y \) of (B1) and (5.13) sans dots. Substituting (B5) into these conditions leads to

\[
a_n = \frac{4pl}{\pi D_n} \int_0^1 \cos \alpha_n x \, dx ,
\]

\[
D_n = \alpha_n^2 W (\cosh \alpha_n H + \beta_n \alpha_n H) .
\]  

(B6)

On making the change of variable \( x = l \sin \tau \) and using the integral representation for the zeroth-order Bessel function, \( a_n \) of (B6) becomes

\[
a_n = 2plJ_0(\alpha_n l) / D_n .
\]  

(B7)

In effect, \( a_n \) of (B7) completes the solution of the problem.

In particular, the solution for the stresses on the \( x \)-axis has
$$\sigma_s \big|_{y=0} = -\frac{pl}{W} \left[ \frac{\nu}{1-\nu} - \sum_{n=1}^{\infty} \frac{2n\pi J_0(\alpha_n l)}{D_n} \left( (\alpha_n + 2\beta_n) \text{ch} \alpha_n (H-y) + \alpha_n \beta_n (H-y) \text{sh} \alpha_n (H-y) \right) \right],$$

$$\sigma_y \big|_{x=0} = -\frac{pl}{W} \left[ 1 + \sum_{n=1}^{\infty} \frac{2n\pi \alpha_n J_0(\alpha_n l)}{D_n} \left( \text{ch} \alpha_n (H-y) + \beta_n (H-y) \text{sh} \alpha_n (H-y) \right) \right]. \quad (B8)$$

These series solutions are only conditionally convergent for $y = 0$. However, for $y = 0$, we know that $\sigma_s = \sigma_y = -2p/\pi$ from (B1) and elasticity theory. For $y > 0$, the series converge rapidly because of exponential decay with increasing $n$. These are the series sums used in Figure 5.6.

To pose the foregoing inverse problem as a contact problem, we simply need to prescribe the solution for the vertical displacement on $0 < x < l$ as the profile of a rigid frictionless punch with sharp edges. The required displacement solution is

$$v \big|_{y=0} = \frac{pl(1+\nu)}{E} \left[ \frac{1-2\nu}{1-\nu} \frac{H}{W} - \sum_{n=1}^{\infty} \frac{4(1-\nu) J_0(\alpha_n l)}{D_n} \beta_n \text{sh} \alpha_n H \cos \alpha_n x \right]. \quad (B9)$$

This series converges slowly near $x = l$ because of Gibbs phenomenon, but nonetheless can be summed even at this location to within 0.1% with 3200 terms.
Appendix C. Letters of Copyright Permission

From: "Permissions Europe/NL" <Permissions.Dordrecht@springer.com>
To: "Jing Jiang" <jjiang3@lsu.edu>
CC:
Subject: RE: Ask for Reusing of Published Materials
Date: Thursday, March 27, 2008 6:40:33 AM

Dear Sir,

With reference to your request (copy herewith) to reprint material on which Springer Science and Business Media controls the copyright, our permission is granted, free of charge, for the use indicated in your enquiry.

This permission
- allows you non-exclusive reproduction rights throughout the World.
- permission includes use in an electronic form, provided that content is
  * password protected;
  * at intranet;
- excludes use in any other electronic form. Should you have a specific project in mind, please reapply for permission.
- requires a full credit (Springer/Kluwer Academic Publishers book/journal title, volume, year of publication, page, chapter/article title, name(s) of author(s), figure number(s), original copyright notice) to the publication in which the material was originally published, by adding: with kind permission of Springer Science and Business Media.

The material can only be used for the purpose of defending your dissertation, and with a maximum of 100 extra copies in paper.

Permission free of charge on this occasion does not prejudice any rights we might have to charge for reproduction of our copyrighted material in the future.

Best wishes,

Nel van der Werf (Ms)

Rights and Permissions/Springer
Van Godewijckstraat 30 | P.O. Box 17
3300 AA Dordrecht | The Netherlands
tel +31 (0) 78 6576 298
fax +31 (0)78 65 76-300
Nel.vanderwerf@springer.com
www.springeronline.com

----------------------------------------------------------------------------
To Whom It May Concern:

I am Jing Jiang/J. Jiang, the author of the following three papers:

3. “Direct microscale imprinting of Al at room temperature with Si inserts”, MICROSYSTEM TECHNOLOGIES, DOI 10.1007/s00542-008-0567-6

I would like to put some contents in these three papers into my dissertation. So I need to get your permission. If you can email to me the permission as soon as possible before April 1, it would be great. Thanks a lot.

My contact information is as follows:

Jing Jiang
Patrick F. Taylor Hall 2508
Mechanical Engineering Department
Louisiana State University
Baton Rouge, LA, 70803
(O) 225-5785947
(F) 225-5789195
jjiang3@lsu.edu

Lorraine Wolf
122
To Whom It May Concern:

I am Jing Jiang/J. Jiang, the author of the following paper:

I would like to put some contents in these three papers into my dissertation. So I need to get your permission. If you can email to me the permission as soon as possible before April 1, it would be great. Thanks a lot.

My contact information is as follows:

Jing Jiang
Patrick F. Taylor Hall 2508
Mechanical Engineering Department
Louisiana State University
Baton Rouge, LA, 70803
(O) 225-5785947
(F) 225-5789195
jjiang3@lsu.edu
Vita

Jing Jiang was born in 70’s, in Chongqing, People’s Republic of China. He received his bachelor’s degree in theoretical and applied mechanics at University of Science and Technology of China in 1999, and a master’s degree in solid mechanics at the same university in 2002. In 2003, he started his graduate study in the Department of Mechanical Engineering at Louisiana State University in the United States of America. He has authored and coauthored eleven journal papers and five conference proceeding publications by 2008. Now he is a candidate for the degree of Doctor of Philosophy in the Department of Mechanical Engineering of LSU.