2009

Fabrication, bonding, assembly, and testing of metal-based microchannel devices

Fanghua Mei
Louisiana State University and Agricultural and Mechanical College, fmei1@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/1905

This Dissertation is brought to you for free and open access by the Graduate School at LSU Digital Commons. It has been accepted for inclusion in LSU Doctoral Dissertations by an authorized graduate school editor of LSU Digital Commons. For more information, please contact gradetd@lsu.edu.
FABRICATION, BONDING, ASSEMBLY, AND TESTING OF METAL-BASED MICROCHANNEL DEVICES

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

in
The Department of Mechanical Engineering

By

Fanghua Mei
B.S., Shanghai Jiao Tong University, Shanghai, China, 2002
M.S., Shanghai Jiao Tong University, Shanghai, China, 2005
August, 2009
To my family
ACKNOWLEDGEMENTS

In the journey of my dissertation research, I would like to express my most sincere gratitude to my major professor, Dr. Wen Jin Meng, for his dedication and support throughout my research. The valuable research experience gained during these four years would not have been possible without his encouragement and support.

I feel so honored to have Dr. Dorel Moldovan, Dr. Shengmin Guo, Dr. Sunggook Park, Dr. Pratul K. Ajmera, and Dr. Hak-Chul Shin to be my dissertation committee members. I thank you all for your continued support and guidance. I appreciate your positive comments and personal encouragement.

I would like to thank Dr. Srinath Ekkad, Dr. Dean Miller, Mr. Jon Hiller, Dr. Dongmei Cao, Dr. Varshni Singh, and Dr. Jing Jiang for their collaboration and technical assistances.

I also want to thank my lab mates, Bin Lu, Ke Chen, Yang Mu, Pritish Parida, and Wynn Phillips for their valuable suggestions and help.

Very special thanks go to my dear wife, Yun Hu, for her understanding and strong support during these years. I would also like to thank my parents and sister who always understand me and encourage me in pursuing my goal.

# TABLE OF CONTENTS

DEDICATION ........................................................................................................... ii

ACKNOWLEDGMENTS ........................................................................................... iii

ABSTRACT .............................................................................................................. vii

CHAPTER 1. INTRODUCTION OF CURRENT MICROFABRICATION
TECHNOLOGIES FOR METAL-BASED MICROSTRUCTURES AND
DEVICES .............................................................................................................. 1

1.1 Introduction ..................................................................................................... 1

1.2 Techniques for Fabricating Metal-Based HARMS ........................................... 1
  1.2.1 Micromilling ............................................................................................ 2
  1.2.2 Laser Micromachining ............................................................................. 3
  1.2.3 Micro Electrical Discharge Machining (µEDM) ......................................... 3
  1.2.4 The LiGA Microfabrication Protocol ...................................................... 4

1.3 Replication of Metallic HARMS by Microscale Compression Molding ......... 6
  1.3.1 Surface Engineering of Mold Insert ......................................................... 6
  1.3.2 Mechanical Properties of Mold Insert ..................................................... 8

1.4 Bonding of Metal-Based HARMS .................................................................. 11

1.5 Testing of Metal-Based Microchannel Heat Exchangers ............................... 12

1.6 Summary ........................................................................................................ 13

1.7 References ..................................................................................................... 13

CHAPTER 2. EUTECTIC BONDING OF Al-BASED HIGH ASPECT RATIO
MICROSCALE STRUCTURES USING Al-Ge NANOCOMPOSITE THIN
FILM INTERMEDIATE LAYERS .............................................................................. 21

2.1 Introduction ..................................................................................................... 21

2.2 Experimental Procedures .............................................................................. 22

2.3 Results and Discussion ................................................................................... 24

2.4 Conclusion ..................................................................................................... 30

2.5 References ..................................................................................................... 30

CHAPTER 3. EVALUATION OF EUTECTIC BOND STRENGTH AND ASSEMBLY
OF Al-BASED MICROFLUIDIC STRUCTURES ......................................................... 33

3.1 Introduction ..................................................................................................... 33

3.2 Experimental Procedures .............................................................................. 34

3.3 Results and Discussion ................................................................................... 36

3.4 Conclusion ..................................................................................................... 42

3.5 References ..................................................................................................... 43

CHAPTER 4. STRUCTURE OF VAPOR-PHASE DEPOSITED Al-Ge THIN FILMS
AND MECHANISM OF Al-Ge INTERMEDIATE LAYER BONDING OF
Al-BASED MICROCHANNEL STRUCTURES .......................................................... 45

4.1 Introduction ..................................................................................................... 45
4.2 Experimental Procedures ................................................................. 46
  4.2.1 Vapor-Phase Deposition of Al-Ge Thin Films ................................. 46
  4.2.2 Compositional and Structural Characterization of Vapor-Phase Deposited
      Al-Ge Thin Films ........................................................................ 47
  4.2.3 Bonding of Al-based Microscale Structures .................................... 48
  4.2.4 FIB Characterization of Bonded Al MHE Structures ....................... 50
4.3 Results and Discussion .................................................................... 50
4.4 Conclusion ........................................................................................ 59
4.5 References ....................................................................................... 59

CHAPTER 5. EVALUATION OF BOND QUALITY AND ASSEMBLY OF Cu-BASED
            MICROFLUIDIC STRUCTURES WITH AI THIN FOIL
            INTERMEDIATE LAYERS ............................................................. 62
  5.1 Introduction .................................................................................... 62
  5.2 Experimental Procedures ............................................................... 63
      5.2.1 Molding Replication of Cu-Based Microchannels ......................... 63
      5.2.2 Bonding of Cu specimens with Al Thin Foil Intermediate Layers .... 63
      5.2.3 Evaluation of Bond Quality ..................................................... 64
      5.2.4 Assembly of Cu-Based Microchannel Devices and MHE Prototypes .. 64
      5.2.5 Thermal Testing of Cu-Based MHE Prototypes ............................... 65
  5.3 Results and Discussion ................................................................... 65
  5.4 Conclusion ...................................................................................... 72
  5.5 References ..................................................................................... 72

CHAPTER 6. BONDING OF Cu-BASED HIGH ASPECT RATIO MICROSCALE
            STRUCTURES WITH Sn INTERMEDIATE LAYERS ....................... 74
  6.1 Introduction .................................................................................... 74
  6.2 Experimental Procedures ............................................................... 75
      6.2.1 Bonding of Cu Specimens with Sn Thin Foil Intermediate Layers .... 75
      6.2.2 Evaluation of Bond Quality ..................................................... 75
      6.2.3 Bonding of Cu-based Microchannel Device Using Sn Thin Foil
            Intermediate Layer ..................................................................... 76
  6.3 Results and Discussion ................................................................... 76
  6.4 Conclusion ...................................................................................... 82
  6.5 References ..................................................................................... 82

CHAPTER 7. FABRICATION, BONDING, ASSEMBLY, AND HEAT TRANSFER
            TESTING OF Al- AND Cu-BASED MICROCHANNEL DEVICES ....... 85
  7.1 Introduction .................................................................................... 85
  7.2 Fabrication and Assembly ............................................................... 86
      7.2.1 Insert Fabrication ....................................................................... 86
      7.2.2 Molding Replication ............................................................... 87
      7.2.3 Assembly and Bonding ............................................................ 88
      7.2.4 Measurement of MHE Geometry .............................................. 91
  7.3 Flow and Heat Transfer Testing ....................................................... 92
      7.3.1 Experimental Setup .................................................................. 92
ABSTRACT

Microsystem technologies are believed to be an important part of the contemporary technological foundation and are becoming a commercially significant specialty area in manufacturing. The design and fabrication of microscale engineering structures has the potential of generating revolutionary changes in many products over a wide range of industrial sectors. Metal-based microchannel heat exchangers (MHEs) promise high heat transfer coefficients together with mechanical robustness, and are of interest for a wide range of applications. Fabrication technologies capable of creating high-aspect-ratio microscale structures (HARMSs) in metals such as Cu at low cost and high throughput are of particular interest. Likewise, simple and reliable bonding and assembly techniques are critical for building functional metal-based microfluidic devices.

This dissertation focuses on various aspects of fabrication, bonding, assembly, and testing of metal-based microdevices. In chapter 1, existing techniques for fabricating metal-based HARMSs are reviewed briefly and compared with each other. A new technique for fabricating metal-based HARMSs, high temperature compression molding, is introduced. Two related issues, bonding and assembly of metal-based HARMSs and testing of assembled metal-based microdevices are discussed respectively. In chapters 2-6, Cu- and Al-based HARMSs were successfully bonded using Al or Sn thin foil intermediate layers and co-deposited Al-Ge thin film intermediate layers, respectively. Quantitative evaluation of bond strengths was carried out as a function of various bonding parameters. Tensile bond strengths are shown to be ~30MPa for bonded Cu pieces and to exceed 75MPa, reaching as high as 165MPa, for bonded Al pieces. Detailed characterizations of the micro-/nano-scale structure of buried bonding interfaces were conducted to rationalize results of mechanical testing. Chapters 7&8 talk about systematic experimentation of fabrication, bonding, and testing of Cu- and Al-based MHEs, and detailed results and discussion on flow and heat transfer performance of these MHEs under two different testing configurations, constant heat flux and constant wall temperature. The results show the increase of surface roughness in the replicated microchannels can cause significant improvements to microchannel heat exchanger performance. Finally, chapter 9 summarizes this dissertation research with main results and achievements. Future work is also discussed in this chapter.
CHAPTER 1. INTRODUCTION OF CURRENT MICROFABRICATION TECHNOLOGIES FOR METAL-BASED MICROSTRUCTURES AND DEVICES

1.1 Introduction

Microsystem technologies are believed to be an important part of the technological foundation upon which the world can continue to improve the standard of living in the twenty-first century. Microelectromechanical systems (MEMS) have been studied intensely for over two decades. Si-based MEMS, fabricated by surface and bulk micromachining techniques derived from IC processing technology, dominate the current MEMS market. Although metal-based microdevices and Microsystems have not received as much attention in comparison, early examples of metal-based microdevices exist in the literature, including microchannel heat exchangers (MHEs) and magnetically driven microactuators. As these early examples show, the physical properties of metals afford metal-based microdevices functionalities not achievable with Si-based counterparts, be it increased thermal conductivities in the case of MHEs or magnetism in the case of magnetic actuators.

Metal-based high-aspect-ratio microscale structures (HARMS) are basic building blocks for metallic microdevices. One important factor responsible for the present paucity of metal-based microdevices and Microsystems is the current lack of suitable fabrication techniques for metal-based HARMS. In order for metal-based microdevices to be competitive in the market place, fabrication of metal-based HARMS should be simple and fast, and assembly of metallic HARMS into metal-based microdevices should be simple, fast, and reliable. The lack of such fabrication and assembly techniques for metal-based microdevices, coupled with the ready availability of technology for fabrication and assembly of Si-based MEMS, is largely responsible for the absence of metal-based microdevices and Microsystems on the current MEMS market.

This chapter provides a succinct description of competing techniques for fabricating metal-based HARMS, such as micromilling, laser micromachining, micro electrical discharge machining (µEDM), and LiGA and LiGA related techniques, including electrodeposition onto lost plastic micromolds, microcasting, and micro metal injection molding. In addition to these traditional techniques, a new technique for fabricating metal-based HARMS – high temperature compression molding replication is briefly introduced and the related issues are discussed, including surface engineering of the mold insert and the mechanical properties of mold insert bulk at elevated temperature. Finally, the issues concerning bonding and assembly of metal-based HARMS and testing of assembled metal-based MHEs are briefly pointed out. These issues will be further studied and discussed in this dissertation.

1.2 Techniques for Fabricating Metal-Based HARMS

Early microfabrication techniques originated from technologies developed for producing integrated circuits (ICs), and commonly combine some or all of the four basic fabrication steps of lithography, etching, deposition, and replication. Lithography involves the transfer of a pattern, defined by a mask, to a photosensitive resist by exposure to a radiation source. Exposure of the resist to radiation causes a change in its chemical properties, enabling selective resist removal in regions that were either exposed to the radiation, in the case of positive resists, or unexposed to...
the radiation, in the case of negative resists. Both UV and X-ray radiations are used in microfabrication, depending on the height and aspect ratio of the desired microscale structures. Etching involves the removal of material in regions uncovered through removal of the resist. One method of material removal is wet chemical etching (WCE), where uncovered material is dissolved away when immersed in a chemical solution. The other is reactive ion etching (RIE), where uncovered material is removed when placed in contact with a plasma, where material removal occurs either through physical sputtering or surface chemical reactions that generate volatile species. Deposition involves many forms, including the deposition of thin film materials conformally over microscale features and the filling of deep recesses, usually created through a combination of lithography and etching, with materials. Replication involves the use of microscale structures produced by a combination of the above methods as a mold insert to facilitate rapid and inexpensive reproduction of micro-components in substrates including polymers, metals, and ceramics. More extensive discussions on microfabrication processes are found elsewhere.7

Fabrication processes for Si-based microscale structures are mature and widely available. Direct application of Si microfabrication protocols to the production of metallic microparts is, in most cases, not feasible. For example, WCE of polycrystalline metals tends to be problematic, due to issues of preferential etching or lack of etch anisotropy. The plasma chemistry of RIE for metals has not been worked out in most cases, although the special circumstance involving deep anisotropic etching of Ti has been reported.8 Compared to Si microfabrication, processes and protocols for fabricating metal-based HARMS are diverse and not as standardized. A succinct description of relevant processes is given next.

1.2.1 Micromilling

For metal machining at the macroscale, milling is a well established industrial process. It is versatile and capable of creating complex three-dimensional features and structures. Adaptation of the milling process to the microscale, hence micromilling,9 has been used to fabricate micromolds and masks to aid in the development of micro components,10 diffraction grating on tiny parts,11 and millimeter long micro-trenches in metallic alloys.12 Micromilling tools are available commercially in diameters larger than 50µm. Custom-built tools as small as 22µm have been fabricated by the focused ion beam (FIB) machining process.13 Controlled machining over small feature sizes is made possible through the use of computer numerically controlled (CNC) ultraprecision machines. CNC ultraprecision machines have been used to create microscale features while maintaining submicron tolerance over a span of millimeters. The bottom of machined trenches has a typical root-mean-square (rms) roughness of ~100nm.14

Several issues exist regarding micromilling as a mass fabrication technique. One issue is tool wear, which has a strong effect on the quality of the machined parts. Progressive wearing of the cutting edge influences the geometry and the surface roughness of the machined parts. High temperature and high pressure during cutting promote the formation of cutting edge wear or build-up, and cause eventual tool breakage. Burr occurrence during the micromilling is another issue, and is difficult to avoid. It may be minimized by select smaller milling tools, at the expense of increased machining time and tooling costs. The sharpness of interior corners in machined parts is limited by the diameter of the milling tool, and is therefore limited to tens of microns. For these reasons, micromilling is more suitable for machining metal parts with small and medium lot sizes, with minimum dimensions about tens of microns.
1.2.2 Laser Micromachining

Pulsed lasers with wavelengths in different regions of the electromagnetic spectrum have been used for material removal in a non-contact fashion, including excimer lasers operating in the ultraviolet (UV)\textsuperscript{15, 16} and femtosecond pulsed Nd:YAG laser operating in the near infrared (IR)\textsuperscript{17}. Molecular dynamics (MD) simulations have been used to study the non-thermal\textsuperscript{18} and thermal\textsuperscript{19} mechanisms of material removal by interaction with a fast laser pulse. Recent studies include the use of nanosecond laser for nanoscale ablation of thin metal films\textsuperscript{20} and nanoscale patterning of Au nanoparticle films\textsuperscript{21}.

For microscale machining of metals, the material removal rates are observed to be on the order of 1µm per pulse, with laser beam diameter on the order of 10µm\textsuperscript{22}. Serial scanning of the laser beam is therefore necessary for forming extended microscale features in metals with depths on the order of a few hundred microns. At typical pulse repetition rates of 1kHz or less, creation of metallic microscale structures covering practical dimensions will be time consuming. Laser beam induced material removal creates elevated surface roughness on as-machined metal structures\textsuperscript{23, 24}. Additional surface treatments may therefore be required depending on the nature of the intended application. Laser micromachining, similar to micromilling, is therefore more suitable for shaping microscale features on specialized metal parts.

1.2.3 Micro Electrical Discharge Machining (µEDM)

EDM is a process of electrically removing material from any conductive workpiece. This is achieved by applying high-frequency voltage pulses between an electrode and the workpiece, usually immersed in a dielectric fluid such as water or oil. The electrode is not in physical contact with the workpiece, but causes an electrical discharge through the dielectric fluid across a small gap between them. Material removal from the workpiece accompanies the electrical discharge. Material removal mechanisms are complex\textsuperscript{25, 26}.

While the EDM technique was introduced over fifty years ago\textsuperscript{27}, adaptation of EDM to microscale machining, i.e., µEDM, has received more recent attention\textsuperscript{28}. Both sinker and wire forms of µEDM have been used\textsuperscript{29}. While typical µEDM operations yield features with dimensions on the order of tens of microns, creation of sub-micron features by µEDM has recently been attempted\textsuperscript{30, 31}. The µEDM process achieves material removal without contact with the electrode, and is advantageous for creating microscale features in hard and brittle materials such as tool steels, refractory alloys, and conducting ceramics.

Conventional sinker and wire µEDM operations use cylindrical, rectangular, and long-wire electrodes, and are serial subtractive machining techniques where material removal occurs one cut at a time. Creating complex microscale shapes may therefore involve a large number of sequential cuts. Because electrical discharge induced material removal inevitably leads to electrode wear, performing a large number of sequential cuts may necessitate electrode changing operations, which lead to increased machining time and alignment difficulties. The µEDM process creates increased surface roughness, as well as possible cracking and materials modification within the near-surface layer\textsuperscript{32, 33, 34}. Depending on the application, these near-surface defects may need to be removed through additional processing steps, such as chemical etching or polishing. Therefore, similar to micromilling and laser micromachining, µEDM may be more suited to fabrication of specialized metallic microparts, such as microscale moulds and dies\textsuperscript{35}.
1.2.4 The LiGA Microfabrication Protocol

The LiGA (Lithographie, Galvanof ormung, Abformung) microfabrication protocol represents an important approach toward producing metal-based HARMS. In conventional LIGA, lithography is performed on polymeric resists such as poly methyl methacrylate (PMMA) by exposure to X-ray radiation (Lithographie), usually from a synchrotron source, followed by chemical dissolution of the exposed portion of the resist (i.e., PMMA is a positive resist) [Figs. 1(a) and 1(b)]. Metals are electrodeposited into lithographically defined resist recesses (Galvanof ormung). After recess filling, electrodeposition is continued onto the top of the resist surface to form the structural backing. Chemical dissolution of the remaining resist then defines an all-metal structure fabricated by lithography and electrodeposition (LiG) [Figs. 1(c) and 1(d)]. Such primary metal-based HARMS can serve as either the final products or as the mold inserts from which secondary HARMS are replicated in materials amenable to compression molding (Abformung) [Figs. 1(e) and 1(f)]. This technique is a forming process in which a plastic material or metal is placed directly into a heated metal mold, then is softened by the heat, and forced to conform to the shape of the mold as the mold closes. The high energy X-ray radiation enables deep lithography in polymeric resists to depths of 1000µm or more, making LiG a common choice for fabricating tall microscale structures in metals.

X-ray lithography can create deep microscale patterns in resists, has a large depth of focus, and good reproducibility. Its biggest drawback is cost. To alleviate the cost issue, more sensitive resists, such as the negative resist SU-8, are used with X-ray radiation in combination with electrodeposition, to produce tall microscale structures in metals. Further drawbacks of the LiG process are the slow speed of electrodeposition and the limitation on materials that can be electrodeposited, both in terms of composition and structure. Readily electroplatable materials have been limited to Au, Cu, Ni and a few Ni alloys. Non-optimized structure of electrodeposited materials manifests itself, for example, in significant mechanical property degradations at elevated temperatures. Although the more sensitive SU-8 resist allows UV radiation to be used in the lithographic process in place of X-ray, it is difficult to remove once cross linked. To combat this problem, studies of SU-8 removal by lasers and plasmas have been conducted.
The high cost of LiG fabricated metallic HARMS stems from the combination of high lithography cost and slow speed of electrodeposition. Molding replication is the key to low-cost fabrication of metallic HARMS en masse. From the inception of LiGA in the early 1980s to early 2000s, various techniques of replication have been utilized to produce polymeric HARMS using LiG fabricated mold inserts, including compression molding or hot embossing, as well as injection molding. Efforts have been spent exploring techniques for improving the efficiency of metallic HARMS fabrication. A series of LiGA derivative techniques have been investigated, as described below.

A Electrodeposition onto Lost Plastic Micromolds

Here the idea is to produce conductive microscale structures by an inexpensive molding technique; and subsequently converting them into metallic HARMS by electrodeposition. For example, conductively filled thermoplastics were injection molded into micromolds which served as substrates for subsequent electrodeposition. Electrodeposition into high aspect ratio cavities provided by such conductive plastic microscale structures can lead to formation of voids, due to the combined effect of current density distribution between close-spaced plastic structures and diffusion of metal ions in the electrolyte. Electrodeposition begins at the base and walls of the plastic structures, and closes before gaps between the plastic structures are completely filled. To alleviate this problem, selective metallization of only the bottom of injection molded insulating plastic microscale structures was carried out. Subsequent electrodeposition proceeds from the bottom, and completely fills the plastic structure without void formation. However, such a remedy works only when the structure to be electrodeposited has one continuous bottom layer. Electrodeposition will not occur on electrically isolated islands.

It should also be pointed out that all methods by which metallic HARMS are produced by electrodeposition are restricted in their choice of electrolytes which allow microscale structures with high aspect ratios to be deposited. At present, this limits the choice of materials to Au, Cu, Ni, and a few Ni alloys. In using such methods for production, one has also to contend with the slow speed of electrodeposition.

B Microcasting

Like investment casting, microcasting is a lost-wax, lost-mold process. In this technique, injection molded plastic microscale structures are used as lost molds and embedded in a ceramic investment material. The embedded plastic microscale structures undergo pyrolysis during heating of the investment material, thereby forming a hollow ceramic mold. The heated hollow ceramic mold, whose inner walls carry the image of the plastic microscale structures, is subsequently filled with molten metal. Complete mold filling is achieved either by centrifugal casting or by vacuum pressure casting. After metal solidification and ceramic mold cool down, the metallic micro component is removed. Metallic microscale structures of Au-based alloys and aluminum bronze have been produced by microcasting. The microcasting technique lends itself particularly well to the fabrication of fully three-dimensional structures.

As compared to conventional LiG fabricated structures, surfaces of parts made by microcasting possess increased roughness, primarily as a result of the investment material. Not all alloys developed and proven for macroscale applications, such as Al-Ti alloys for high-temperature applications or hard metals for components subject to wear, are amenable to microcasting at the present time. Moreover, the complex processing steps are problematic as far as fabrication cost and efficiency are concerned.
In the micro metal injection molding (µMIM) process, also called powder metallurgical injection molding, feedstock consisting of fine metal powders mixed with an organic binder is injected into a mold which contains a LiG fabricated mold insert. In order to achieve good insert filling, injection molds which can be evacuated and heated are required along with highly fluid feedstock. After injection molding, the shaped compact is separated from the mold insert and heated to remove the binder system and generate contact between metal particles. Subsequently, in a defined atmosphere, the compact is further sintered into a solid microscale structure of close-to-theoretical density.51, 54

Incomplete mold filling can occur during the µMIM process, especially near the edges of the mold. Pores and cracks may exist on the surface as well as in the center of the metal structures. The µMIM technique requires multiple heat treatment steps, accompanied by part shrinkage. This makes the control of final part dimensions difficult.55 Microparts fabricated by µMIM suffer the usual shortcomings of parts made by powder metal processes, such as the degradation of mechanical and physical properties due to the presence of pores and cracks.

1.3 Replication of Metallic HARMS by Microscale Compression Molding

For each of the fabrication methods described in Section 1.2, some disadvantage exists regarding production of metallic HARMS en masse. However, LiGA process provides a concept of molding replication, which is an efficient fabrication technique for mass production. Prior to 2003, molding replication of HARMS from LiGA fabricated metallic mold inserts was achieved only in polymer-based materials.47 Extension of the technology of direct molding replication from polymeric materials to metals and alloys is desirable. The compelling advantage of the molding replication technique is that a large number of replicas can be produced repeatedly, once a durable mold insert is fabricated, thus offering the possibility of low-cost, high-throughput production of metallic HARMS appropriate for commercial applications.

Since 2003, we have advanced the technology regarding molding replication of metal-based HARMS by focusing our research on two technical issues central to successful metal micromolding, which in general occurs at higher temperature as compare to molding polymers. The first issue is controlling the near-surface chemical/mechanical interactions between the mold insert and the molded metal at high molding temperature. 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 parts. Another issue is improving the mechanical properties of the mold insert bulk at the relevant molding temperatures. At the molding temperature, the yield strength of the mold insert has to be sufficiently high to endure the stresses needed for molding metal parts. These issues form the challenges for molding replication of metallic HARMS by microscale compression molding.

1.3.1 Surface Engineering of Mold Insert

During compression molding of a given metal at high temperature, the chemical reactivity between the mold insert and the molded metal substrate have proven to be important problem. Chemical bonds can form between the insert and the substrate that can break the insert and damage the molded structure as it is withdrawn from the substrate. Figure 1.2 illustrates what can happen when microscale metallic inserts are used to mold reactive metals without
controlling the chemical/mechanical interactions between them. In this example, a LiG fabricated Ni insert, in the form of a 2-dimensional array of cylindrical microposts protruding from a flat base, was used to mold Al at ~460°C. Figure 1.2(a) shows the state of the molded Al plate after insert withdrawal. Although a microhole array was generated on the molded Al plate due to molding, some Al was torn away from one side of each microhole during demolding. Corresponding Al pickup occurred on each Ni micropost after demolding, as exemplified by the high magnification view of one typical Ni micropost in Fig. 1.2(b). The asymmetric damage on the Al plate and asymmetric Al pickup on the Ni microposts are believed to result from a small misalignment between the insert and the Al plate, leading to preferential insert/Al contact on one side of the micropost. As shown in Fig. 1.2(a), some Ni microposts were broken from the insert base during demolding and trapped within the Al microhole. The process of molding creates extensive plastic flow within the molded Al plate, leading to contact and adhesion between Ni on the insert and pristine Al in the molded plate. This chemical/mechanical interaction arises from the strong thermodynamic driving force for the mold insert material and the molded material to mix, and needs to be controlled for successful molding replication.

Fig. 1.2 Al molding with uncoated Ni insert at 460 °C: (a) overview of molded Al plat; (b) a typical uncoated Ni insert after molding

Fig. 1.3 Surface engineering of a Ni microscale mold insert with an array of Ni microposts ~400µm in height by conformal deposition of a Ti-C:H nanocomposite coating: (a) cross-section perpendicular to a Ni micropost; (b) cross-section parallel to a Ni micropost; (c) an array of molding replicated microholes in an Al plate with depths ~400µm; (d) the surface engineered mold insert consisting of an array of Ti-C:H coated Ni microposts after multiple molding replication runs at ~460°C.
Our approach to controlling the near-surface chemical/mechanical interactions between the insert and the molded metal is to **surface engineer** the microscale mold insert by conformally depositing a suitably chosen nanostructured ceramic coating. With an inductively coupled plasma (ICP) assisted hybrid chemical/physical vapor deposition (CVD/PVD) technique, conformal deposition of nanostructured ceramic coatings over LiGA fabricated Ni HARMS was demonstrated. Conformally deposited Ti-containing hydrogenated carbon (Ti-C:H) coatings over Ni HARMS were characterized by combining scanning electron microscopy (SEM) and scanning Auger electron spectroscopy (SAES) with sputter depth profiling. Relationships between the nanoscale structure, mechanical properties, and tribological characteristics were studied in detail in amorphous hydrogenated carbon (a-C:H) based and amorphous silicon nitride (a-Si:N) based two-phase nanocomposite coating systems.

With such surface engineered Ni mold inserts, we successfully demonstrated molding replication in Al at ~450°C with ~100% pattern transfer, without significant damage to the insert after multiple molding runs [Figure 1.3]. In contrast, significant bonding occurred between an uncoated Ni insert and Al during molding at the same temperature, leading to extensive damage to both the insert and the replicated microfeatures during demolding. These results show that controlling the near-surface chemical/mechanical interactions between the insert and the molded material is critical to successful high temperature micromolding of reactive metals, and that conformal deposition of suitable nanostructured ceramic coatings over insert surfaces is an effective means to achieve such control.

### 1.3.2 Mechanical Properties of Mold Insert

The quality of the replica depends upon the mechanical yield strength of the mold insert at elevated molding temperatures. One important problem confronting compression molding technology for metallic microscale structures is the lack of microstructure mold insert materials that retain high mechanical yield strengths at molding temperatures.

![Fig. 1.4](image.png)

**Fig. 1.4** Electrodeposited Ni-Fe mold insert before and after Cu molding at 500°C: (a) Ni-Fe mold insert before molding; (b) Ni-Fe mold insert after molding

Electrodeposition of Ni typically leads to nanocrystal formation, 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 \( \sim 400^\circ\text{C} \).\textsuperscript{71} Even though electrodeposited Ni inserts performed adequately for Al micromolding at \( \sim 450^\circ\text{C} \), additional Al molding runs at \( \sim 500^\circ\text{C} \) indeed led to permanent bending of Ni microposts. This undesirable plastic deformation of the insert decreases the faithfulness in replication as well as insert life. Attempts to electrodeposit alloys with higher high temperature strength have been made. Examples include Ni-Fe,\textsuperscript{72} Ni-Mo,\textsuperscript{73} and Ni-Co-Fe.\textsuperscript{74} These codepositions involve complex electrochemical reactions which limit the achievable composition range. Electrodeposited alloys also may not possess the optimal micro-/nano- scale structure for high temperature use. Figure 1.4 shows an example of using an electrodeposited Ni-Fe mold insert to mold Cu at \( \sim 500^\circ\text{C} \). Figure 1.4(a) shows a typical microprotrusion on the mold insert with small size features before molding. The sidewall and small features in the mold insert are very clear. Figure 1.4(b) shows a typical microprotrusion from the same insert after molding Cu at \( \sim 500^\circ\text{C} \). The microprotrusion on the insert was flattened and totally damaged. This case indicates that the materials of mold insert have to possess sufficiently strong mechanical properties at high molding temperature.

Our approach to improving the mechanical properties of the insert bulk at high temperatures is to fabricate mold inserts with microscale structures out of refractory metals and alloys with techniques other than electrodeposition. These metals and alloys possess high mechanical properties even at high temperature. To fabricate mold inserts with microscale features using these refractory metals and alloys, however, traditional machining techniques may encounter difficulties, such as tool damage/wear and time consuming. To solve this problem, \( \mu \text{EDM} \) technique is used to create microscale recesses in blank mold insert from refractory metals and alloys. The \( \mu \text{EDM} \) technique, a derivative of the conventional EDM process,\textsuperscript{75} has been used to fabricate HARMS out of metals\textsuperscript{76} and electrically conducting ceramics.\textsuperscript{77} This subtractive micromachining technique offers an alternative to electrodeposition for fabricating mold inserts out of high temperature materials. Using \( \mu \text{EDM} \), we have successfully fabricated HARMS inserts with simple geometries out of refractory metals and alloys, such as elemental Ta and Ni-based superalloys.

![Fig. 1.5 Hybrid fabrication and application of microscale Ta mold inserts: (a) morphology of as-\( \mu \text{EDMed} \) Ta surface showing the existence of the ASL with nodules and cracks; (b) cross-sectional electron diffraction pattern showing that the structure of ASL is B1-TaC rather than A2-Ta; (c) overview of surface engineered Ta HARMS insert fabricated by \( \mu \text{EDM} \) followed by ECP and conformal Ti-C:H deposition.](image-url)
Subtleties in using µEDMed HARMS as mold inserts were revealed by detailed morphological, chemical, and structural characterization of as-µEDMed surfaces by combining SEM with X-ray photoelectron spectroscopy (XPS) and transmission electron microscopy (TEM). In the case of Ta, an altered surface layer (ASL) of TaC forms due to reaction of Ta with the hydrocarbon-based dielectric fluid during µEDM, as shown in Figs. 1.5(a) and 1.5(b). Numerous nodules and surface cracks are present within the ASL, necessitating further surface engineering of as-µEDMed Ta HARMS before they can be used as mold inserts. This was accomplished by electrochemical polishing (ECP) removal of the ASL followed by conformal deposition of a Ti-C:H coating. Figure 1.5(c) shows an SEM overview of surface engineered Ta HARMS insert, consisting of an array of rectangular microprotrusions fabricated by µEDM followed by ECP and conformal Ti-C:H deposition.

A process similar to that described above for fabricating Ta mold insert with rectangular microprotrusions by µEDM followed by ECP and conformal a-Si:N deposition was employed to fabricate other refractory metals and alloys, such as Ni-based superalloys. Using these fabricated mold inserts, successful HARMS replication has been demonstrated in different metals or alloys.

Fig. 1.6 Molding on Al: (a) an overview of molded microchannels; (b) a cross-section view of molded microchannels.

Fig. 1.7 Molding on Cu: (a) an overview of molded microchannels; (b) a close-up view of a typical molded microchannel.
such as soft metal Al
harder metal Cu,
and even harder metal or alloys Ni and Ni-Ti.
Figures 1.6-1.9 show some SEM images of molded microstructures in different metals/alloys. Among these metals or alloys, Cu and Al possess higher bulk thermal conductivities and are easy to mold, and are therefore worthy of further exploration as the bare material for various microdevices.

1.4 Bonding of Metal-Based HARMS

Bonding and assembly are vital for fabricating functional microchannel devices, such as MHEs. Once a microchannel has been fabricated in a substrate, whether Si or metal, a leak-tight cover plate must be affixed before it can be used as a heat exchanger. Several bonding methods have been reported for Si-based microsystems, including anodic bonding, direct bonding, and intermediate layer bonding.

Anodic bonding is also called field-assisted thermal bonding or electrostatic bonding and is usually established between a sodium glass and silicon for MEMS. Direct bonding is also called silicon fusion bonding, which is based on chemical reaction between OH groups present at
the surface of native silicon or grown oxides covering the wafers. Direct bonding usually require three steps: surface preparation, contacting, and thermal annealing. Intermediate layer bonding uses an additional material layer to join the components being bonded. The use of a chemically/structurally distinct intermediate layer at the bonding interface can lower the bonding temperature, eliminate bulk melting of the parts to be joined, promote interdiffusion, and increase the bond quality. Applying thin film intermediate layers on one or both component surfaces may also serve to minimize feature distortions when microscale structures are bonded. Among the various intermediate layer bonding strategies, employing eutectic intermediate bonding layers has been investigated, including Au-Si eutectic bonding and Al-Ge eutectic bonding. The eutectic temperature, $T_E$, of the Al-Ge system is $424^\circ$C, significantly below the melting temperature of Al, $T_m=660^\circ$C. It is therefore possible to use Al-Ge intermediate layers to bond Al parts. For Cu-based alloy system, Al-Cu and Sn-Cu systems also have eutectic reactions and are possible to use Al or Sn intermediate layers to bond Cu pieces.

Simple and reliable bonding of metal-based microchannel structures is becoming the most critical element for metal-based MHEs after molding replication on different metals has been achieved. Without bonding, metal-based microchannel cannot be assembled into functional devices. Systematic studies on bonding method, bonding quality, and bonding mechanism for metal-based microchannel, especially for Cu- and Al- based, are quite necessary and urgent. Once the successful bonding of metal-based microchannel is achieved, the prototypes of metal-based MHEs are becoming feasible through proper assembly.

1.5 Testing of Metal-Based Microchannel Heat Exchangers

To better understand the performances of metal-based MHEs, flow and heat transfer testing is necessary. Extensive studies of microchannel fluid flow and heat transfer have been carried out over the past two decades. Design of heat exchangers taking advantage of heat transfer enhancement in microchannels has been contemplated previously. Heat transfer characteristics of Ni-based microchannel devices have been reported in the literature. These devices were fabricated following LiG-related protocols and not easily adapted to large scale production. Flow performance and heat transfer coefficient characteristics were also reported for Al-based minichannels. These minichannels were fabricated by multiport extrusion, which limits the microchannel dimension to larger than 700$\mu$m. Heat transfer characteristics have been measured previously by Lee et al. in Cu microchannel devices fabricated by mechanical cutting of Cu plates followed by sealing with a polymeric top plate. They examined a variety of rectangular Cu microchannels ranging from 194$\mu$m to 534$\mu$m in width, with the same channel aspect ratio ($H/W$) of 5. These devices were sealed by polymeric top plates, which limit their applications as heat exchangers.

Flow and heat transfer performances in microchannels depend significantly on the channel configurations, such as surface roughness and dimension, as well as the type of liquid flow. Most studies on heat transfer characteristics in microchannel are focusing on smooth or small surface roughness ($<1\mu$m). Increasing surface roughness and decreasing channel dimension will lead to increase in heat transfer performance. Therefore, systematic studies on the effects of surface roughness and microchannel dimension in bonded and assembled metal-based MHEs are needed. In addition, further characteristics on the effect of liquid flow used to test are also required. These issues are becoming important concerns for the research of metal-based MHEs.
1.6 Summary

Current microfabrication techniques for fabricating metal-based HARMS have been reviewed and discussed. A new technique for fabricating metal-based HARMS – high temperature compression molding replication has been introduced and the related issues have been discussed in detail. Examples of molded microstructures in different metals have been presented. Further issues concerning bonding and assembly of metal-based HARMS and testing of assembled metal-based microdevices are discussed. These issues form the main research interests of this dissertation.

1.7 References


V. K. Varadan, K. J. Vinoy, K. A. Jose, RF MEMS and their applications, John Wiley and Sons Ltd, UK (2003).


CHAPTER 2. EUTECTIC BONDING OF Al-BASED HIGH ASPECT RATIO MICROSCALE STRUCTURES USING Al-Ge NANOCOMPOSITE THIN FILM INTERMEDIATE LAYERS *

2.1 Introduction

Metal-based microdevices can possess functionalities not achievable with Si-based microelectromechanical systems (MEMS), examples include microscale electrostatic actuators, microscale electromagnetic actuators, and micro heat exchangers. Realization of metal-based microdevices often requires the fabrication of metal-based high-aspect-ratio microscale structures (HARMS). An important method for fabricating metal-based HARMS is the LiGA technique, combining X-ray/UV lithography (Lithographie) with metal electrodeposition (Galvaniformung) and replication by compression molding (Abformtechnik). Primary metal-based HARMS fabricated by lithography and electrodeposition (LiG) are expensive and slow to make. Among the three steps of the LiGA protocol, replication of secondary HARMS from primary microscale mold inserts by compression molding is the key to low-cost mass production. Typical mold inserts possess one active surface, consisting of two-dimensional (2D) microscale features protruding out of a base plane, forming a so-called two-and-one-half dimensional (2.5D) structure.

Prior to 2003, HARMS replication by compression molding had only been achieved in polymer-based materials. Since 2003, we have demonstrated successful HARMS replication in Pb, Zn, Al, and Cu by compression molding. Microscale mold inserts were fabricated out of refractory metals with micro electrical discharge machining (µEDM). Refractory metal mold inserts with complex 2D microscale patterns were fabricated by combining primary HARMS fabrication by LiG with pattern transfer to refractory metals by parallel µEDM. Modification of the near-surface chemical and mechanical properties of the mold insert was critical to successful molding of reactive metals, and was achieved by conformal deposition of suitably chosen thin ceramic coatings.

Al-based HARMS are of particular engineering interest. Due to the low density, high thermal conductivity, and ready availability of Al, Al-based HARMS are leading material candidates for building light-weight micro heat exchangers. Because Al cannot be electrodeposited, Al-based HARMS cannot be fabricated using the standard LiG protocol. Replication by compression molding from a microscale mold insert is perhaps the only method through which numerous microscale features can be generated in Al in parallel.

Replicated metallic HARMS consist of microscale 2D depressions into a flat metal plate, forming a negative 2.5D structure of the mold insert. To form any functional metal-based microdevice from such replicated metallic HARMS, proper assembly and packaging are required. At the very least, the bonding of a cap layer to form enclosed microscale features is required for many microdevice applications, notably those involving fluid flow. Current research on assembly and packaging technologies are primarily focused on Si-based microdevices. Bonding of metal-based HARMS, on the other hand, has not been dealt with in the literature to any degree of detail.

Several bonding methods, such as anodic bonding, direct bonding, and intermediate layer bonding, have been reported for the joining and sealing of Si-based microdevices. Among these methods, applying thin film intermediate layers on one or both component surfaces...
can help lower the bonding temperature, which may serve to minimize feature distortions when microscale structures are bonded. Among the various intermediate layer bonding strategies, employing eutectic intermediate bonding layers has been investigated, including Au-Si eutectic bonding\textsuperscript{21} and Al-Ge eutectic bonding.\textsuperscript{22} The eutectic temperature, $T_E$, of the Al-Ge system is 424°C, significantly below the melting temperature of Al, $T_m$=660°C.\textsuperscript{23} It is therefore possible to use Al-Ge intermediate layers to bond Al parts.

Different forms of Al-Ge intermediate layer can be used for bonding purposes. Vu et al. bonded two Si pieces by depositing alternating layers of pure Al and pure Ge on the surface of each of the two Si pieces.\textsuperscript{22} In this chapter, we report vapor phase codeposition of a series of Al-Ge composite thin films and the use of Al-Ge composite films as intermediate bonding layers to bond a flat Al coupon to replicated Al HARMS. The micro-/nano- scale structures of the Al-Ge composite films were characterized. Morphologies of bonded Al-based HARMS, including that of separated surfaces were examined.

### 2.2 Experimental Procedures

Al-Ge composite thin films were deposited with a radio frequency (rf) inductively coupled plasma (ICP) assisted hybrid physical/chemical vapor deposition tool, a schematic of which is shown in Fig. 2.1. The deposition chamber is spherical in geometry and contains two facing induction coils, together with two direct current (dc) balanced-magnetron sputtering guns. The induction coils are connected to 13.56MHz, 1.25kW generators through tuning networks. When energized, the induction ignite ICP through dielectric windows, filling the entire deposition chamber. Further details on the hybrid tool design have been reported previously.\textsuperscript{24, 25}

![Fig. 2.1 A schematic of the deposition system (top view). Specimen is placed at the center of the chamber. A and B are ICP sources; C and D are balanced magnetron sputtering guns.](image)

One Al (99.99\%) cathode and one Ge (99.99\%) cathode, both 75mm in diameter, were placed facing each other and sputtered in the current controlled mode. The distance between the Al and Ge cathodes was about 250mm. Substrates for Al-Ge deposition included Si wafers with a diameter of 50mm, polished Al coupons (Al1100, 99\%+) with a diameter of 35mm, and replicated Al HARMS (Al1100, 99\%+). The substrates were ultrasonically cleaned in acetone
and methanol before being mounted on a rotatable holder situated in the middle of the two targets. The ultimate base pressure of the deposition system is less than $1.0 \times 10^{-8}$Torr. Typical background pressures prior to deposition runs were about $1 \times 10^{-7}$Torr.

The entire deposition sequence was carried out in an Ar (99.999%) atmosphere, with a total pressure of about 1.3mTorr. The deposition sequence consisted of a substrate surface etch followed by codeposition of Al and Ge. Substrate etching occurred in a pure Ar ICP with a total input rf power of 1000W, a substrate bias of -200V, and an etch duration of 3min. Sputtering of Al and Ge cathodes commenced immediately after substrate surface etch. One series of Al-Ge composite films were deposited on Si(100) substrates. For this series of specimens, a fixed Al cathode current of 0.5A was used, and the Ge cathode current was varied from 0.1A to 0.5A to alter the Ge composition within the film. The deposition duration was fixed at 30min for this series of specimens. Additional depositions on flat Al coupons and replicated Al HARMS were carried out with a fixed Al cathode current of 1.0A and a fixed Ge cathode current of 0.45A for 60min. The substrate bias was fixed at -100V for all depositions. Substrates were rotated continuously in the center of the deposition zone at about 12rpm during both etching and deposition. No intentional substrate heating or cooling was applied during the entire deposition process.

![Image](image_url)

**Fig. 2.2** The custom-built, high-vacuum, high-temperature compression-molding/bonding system: (a) an optical overview of the system layout; (b) a schematic of the system.

Bonding experiments were carried out using a single-axis MTS858 Mini-Bionix II testing system interfaced to a custom-built high-vacuum chamber. As shown in Fig. 2.2, the testing system included a hydraulically powered, cross-head-mounted, linear actuator, and a TestStar© digital controller capable of controlling either the load force or the actuator displacement. The linear actuator could be programmed to move according to prescribed load forces in the force-controlled mode or actuator displacements in the displacement-controlled mode. The axial force was measured by a 25kN full-scale load cell with a resolution of $\sim$5mN, and the axial displacement was measured by a linear variable displacement transducer (LVDT) with a.
resolution of \( \sim 0.5 \mu \text{m} \). The entire molding/demolding process was programmed through the TestStar software and controlled through the TestStar digital controller. Two heating stations were installed within the vacuum chamber. The lower heating station was mechanically attached to the bottom of the vacuum chamber. The upper heating station was connected to the linear actuator through a bellow-sealed motion feedthrough. The two heating stations were heated separately by resistive heating cartridges and the temperatures were measured by two separate K-type thermocouples. Careful machining of the heating stations was performed to ensure that the surfaces of the two heating stations were parallel to each other and perpendicular to the actuator axis.

Pumped by a turbomolecular pump system, the ultimate base pressure of the molding chamber is about \( 2 \times 10^{-9} \text{Torr} \). Typical background pressures during bonding experiments were about \( 1 \times 10^{-6} \text{Torr} \). Flat Al coupons and Al HARMS with Al-Ge composite films deposited on the bonding surfaces were placed face-to-face on the lower heating station. The chamber was evacuated, and both heating stations were heated to about 400°C. The upper heating station was then contacted with the Al assembly to be bonded, and an increasing compression force was applied to the assembly at a constant loading rate of 100N/min. A constant force hold was executed for 12min after the compression force reached the desired level. Temperatures of both heating stations were increased during the compression force increase, so that the specimen temperature was increased to 500°C when the constant force hold occurred. After the constant force hold, the linear actuator was withdrawn from the Al assembly and the system was cooled down.

A JEOL2010 transmission electron microscope (TEM) was employed to characterize the micro-/nano-scale structure of the Al-Ge composite films. Cross-sectional TEM specimens of Al-Ge films deposited on Si(100) substrates were prepared with standard face-to-face gluing, mechanical thinning followed by dimple grinding, and final ion milling using 4kV Ar\(^+\) ions at a 4° take-off angle on a Gatan Precision system. Compositional analysis of Al-Ge composite thin films were performed by energy dispersive X-ray spectroscopy (EDS) and X-ray photoelectron spectroscopy (XPS). EDS measurements were conducted using a EDAX system equipped with an ultra-thin window detector, attached to a Hitachi S3600N scanning electron microscope (SEM). EDS spectra were collected at an electron beam energy of 15keV and a detector take-off angle of 36°. XPS measurements were conducted on a Kratos AXIS165 system. Surface etching prior to XPS measurements on Al-Ge films was carried out with ion beam sputtering with 5kV Ar\(^+\) ions for 30min. SEM examinations of bonded Al assemblies were conducted on the Hitachi S3600N SEM.

### 2.3 Results and Discussion

Figure 2.3(a) shows an overview of a microscale Ta mold insert, which consists of one single rectangular microprotrusion, with a width of 105µm and a height of 400µm, meandering through the entire active area of the insert, 1cm×1cm. The 2.5D pattern on the Ta insert was fabricated by parallel µEDM using a primary Ni HARMS electrode made by LiG.\(^{11}\) The center-to-center distance between two adjacent microprotrusions is 400µm. The parallel µEDM process created microfeatures in Ta with vertical sidewalls and well defined top-to-sidewall transitions. Following the parallel µEDM process, surfaces on the as-machined Ta insert was etched electrochemically. A Ti-containing amorphous hydrogenated carbon (Ti-C:H) film was then conformally deposited over the entire insert using an ICP assisted vapor phase deposition process.\(^{12,13}\)
Figure 2.3(b) shows an overview of an Al plate after being compression molded by the Ti-C:H coated Ta insert shown in Fig. 2.3(a). The molding process transferred the meandering rectangular microprotrusion on the insert into a meandering rectangular microchannel on the Al plate. The continuous microchannel, with a height of 300μm, has vertical sidewalls and sharp sidewall-to-bottom transitions. In Figure 2.3(c), a higher magnification view of a section of the microchannel in Al shows the typical sidewall roughness, due in part to the sidewall roughness of the rectangular microprotrusion on the Ta insert. The top surface between two parallel sections of the microchannel is convex upward due to material pile-up during compression molding of Al. Figure 2.3(c) also shows that the top surface, sidewall, and bottom of the microchannel are quite clean. This kind of molded Al plate can therefore serve as the building block for Al-based microfluidic devices after a cap layer is bonded to it.

![Figure 2.3 SEM examination of a Ta mold insert and molded features in an Al disk: (a) overview of Ta mold insert; (b) overview of molded Al plate; (c) high magnification view of a section of the molded microchannel.](image)

To employ eutectic bonding with an Al-Ge interlayer, the composition of Al-Ge films should be measured and correlated to the deposition conditions. In addition, the micro-/nano-scale structure of the Al-Ge films should be characterized. Figure 2.4(a) shows an EDS spectrum collected from one Al-Ge film, deposited at Al and Ge cathode currents of 0.5A and 0.4A, respectively. Similar EDS spectra were collected from Al-Ge films deposited at different Al and Ge cathode currents, at the same spectrometer settings, the same magnification, and from an identical area of about 10μm×10μm in size. Information present within these spectra therefore
Fig. 2.4 Chemical composition characterization: (a) EDS spectrum of Al-Ge film deposited at Al 0.5A and Ge 0.4A; (b) Ge/Al composition ratio for a series of Al-Ge films vs. Ge cathode current with a fixed Al current of 0.5A.

Fig. 2.5 Surface morphology and impurity characterization: (a) SEM image of Al-Ge film deposited at Al 0.5A and Ge 0.1A; (b) a typical XPS spectrum of Al-Ge film deposited at Al 0.5A and Ge 0.4A.

reflects the average film composition. The factory-supplied EDS data analysis software, EDAX Genesis (version 3.6), was used for automatic background fitting and removal. A standardless quantification routine employing ZAF corrections was included as a part of the data analysis software, and was used to obtain atomic percentages from raw EDS spectra. Spectra collected from Al-Ge films contained signal from Al and Ge within the film, and signal from the Si substrate. No oxygen signal is present above the noise level. Because of the presence of the Si substrate signal, only the ratio of Ge at.% composition to Al at.% composition is considered. Figure 2.4(b) shows the Ge to Al composition ratio for a series of Al-Ge films, deposited at a fixed Al cathode current of 0.5A, as a function of Ge cathode current. The thickness of this series of Al-Ge films, as measured by cross-sectional SEM, is about 360nm. The deposition rate for this series of Al-Ge films is therefore about 12nm/min. As expected, the average Ge to Al composition ratio increases monotonically with the Ge cathode current. The Ge to Al
composition ratio for the Al-Ge film deposited at an Al cathode current of 0.5A and a Ge cathode current of 0.2A is close to that corresponding to the Al-Ge eutectic composition of Al₇₀Ge₃₀.²³

Figure 2.5(a) shows a SEM micrograph of the surface of one film from the series of Al-Ge films, whose Ge to Al composition ratios are shown in Fig. 2.4(b). This film was deposited at Al and Ge cathode currents of 0.5A and 0.1A, respectively. The surface shown in Fig. 2.5 contains about 400nm lateral corrugations, resulting in a rough surface. Despite this surface roughness, this film appears to be fully dense. The surface morphology shown in Fig. 2.5(a) is typical of all Al-Ge films in the series. Figure 2.5(b) shows a typical XPS spectrum collected from one Al-Ge film from the same series of Al-Ge films. This film was deposited at Al and Ge cathode currents of 0.5A and 0.4A, respectively. The O1s signal is hardly discernable from the background after surface sputter etching with Ar⁺ ions. Quantification of the XPS spectrum using factory-supplied sensitivity factors indicated that the oxygen content within the Al-Ge film is less than 1at.%. Because of the presence of the surface roughness on the Al-Ge films, removal of surface contamination by sputter etching tends to be less effective due to shadowing effects, resulting in possible exaggeration of the oxygen content within the bulk of the film. XPS measurements on other Al-Ge films revealed similar impurity content.

Fig. 2.6 Cross-sectional TEM characterization of Al-Ge film deposited at Al 0.5A and Ge 0.3A: (a) bright-field (BF) micrograph; (b) companion dark-field (DF) micrograph.

Figure 2.6(a) shows a cross-sectional TEM bright-field (BF) micrograph obtained from one film from the series of Al-Ge films, whose Ge to Al composition ratios are shown in Fig. 2.4(b). This film was deposited at Al and Ge cathode currents of 0.5A and 0.3A, respectively. Figure 2.6(a) shows the nanoscale structure of this Al-Ge film, from regions close to the Si(100) substrate to regions close to the top surface of the film. Additional high-resolution imaging showed that the region immediately next to the Si(100) substrate with the dark contrast is crystalline Ge, while regions with light contrast above the Ge layer is crystalline Al. These imaging results were also confirmed with a series of EDS spectra collected from these different regions. Thus Fig. 2.6(a) presents clear evidence of phase separation within the co-deposited Al-Ge film. The companion dark-field (DF) micrograph is shown in Fig. 2.6(b). In the DF image, the Ge regions exhibit light contrast while the Al regions exhibit dark contrast. Figure 2.6(b) again shows that a crystalline Ge layer forms next to the Si(100) substrate. Immediately above this Ge layer, lateral separation occurs between crystalline Ge and crystalline Al grains in the
plane parallel to the substrate surface. As the Al-Ge film deposition continues, some Al grains begin to spread laterally and cover the Ge region underneath, showing evidence of transverse separation between crystalline Ge and crystalline Al grains in the direction perpendicular to the substrate surface. Thus, Fig. 2.6 shows evidence of both lateral as well as transverse separation between Ge and Al within the co-deposited Al-Ge film.

The separation between crystalline Ge and crystalline Al phases within codeposited Al-Ge films is expected on thermodynamic grounds. The equilibrium Al-Ge binary phase diagram shows the presence of a simple eutectic and very limited terminal solubilities. Consistent with this expectation, both lateral and transverse phase separation between Al and Ge have been observed in electron beam coevaporated Al-Ge films.27,28 For the purpose of using Al-Ge films for intermediate layer bonding, Fig. 2.6 shows that the presently co-deposited Al-Ge films afford mixing of Al and Ge regions on the order of 100nm. This intimate mixing promotes eutectic melting of the entire Al-Ge film once \( T_E \) is reached, and therefore is beneficial for bonding purposes. As compared to Al-Ge eutectic bonding using Al/Ge multilayers, codeposition of Al-Ge films is easier since intimate mixing of Al and Ge is achieved naturally through the process of phase separation and only the average film composition needs to be controlled.

Fig. 2.7 Cross-sectional SEM examination of bonded Al HARMS/Al coupon with tilt 15deg: (a) overview of the bonded piece; (b) high magnification view of a bonded channel.

Composite Al-Ge intermediate layers were codeposited onto one replicated Al HARMS, consisting of a parallel array of straight microchannels, and one flat Al coupon. To test the feasibility of simultaneous bonding of different features, the microchannel width was made to vary between 65\( \mu \)m and 265\( \mu \)m. The Al-Ge intermediate layer was deposited at a fixed Al cathode current of 1.0A and a fixed Ge cathode current of 0.45A, making its average composition close to the eutectic composition. After deposition of the Al-Ge intermediate layers, the replicated Al HARMS and the flat Al coupon were bonded at a final holding temperature of 500°C. The applied pressure during the final hold stage of bonding was about 1.5MPa. The cross-sectional overview of the bonded piece at a tilt angle of 15deg is shown in Fig. 2.7(a). The Al microchannels are well enveloped by the flat coupon. On the replicated Al HARMS, top surfaces between parallel microchannels are convex upward due to material pile-up during molding. The highest portion of the pile-up protrudes slightly higher than the original surface level, as shown for example in Fig. 2.3(c). These convex surfaces contact the counter surface on the flat Al coupon first, and were flattened during the bonding process under the applied pressure. This flattening resulted in a narrowing of the microchannel near the top. This channel narrowing
Fig. 2.8 Surface morphology of separated surface: (a) high magnification view; (b) low magnification view near the sidewall.

is shown at a higher magnification in Fig. 2.7(b), and is more pronounced for microchannels with smaller width, as is evident in Fig. 2.7(a). Figure 2.7 shows that the replicated Al HARMS and the flat Al coupon are bonded tightly together, with no obvious crevices observed.

The bonded Al HARMS/Al coupon assembly could not be separated by simple pulling and dropping. The presence of the microchannels and the observed channel narrowing during bonding gives rise to complications for quantitative bonding strength measurement, because of the difficulty in making an accurate measurement of the actual bonding area. Quantitative bonding strength measurements remain to be performed in the future. The bond quality was, however, assessed in a qualitative manner by mechanically separating the bonded Al HARMS/Al coupon assembly and observing the morphology of the separated surfaces. With a tensile load, the bonded Al HARMS/Al coupon assembly fractured from the original interface containing the Al-Ge intermediate layers. The morphology of the separated surface is illustrated in Fig. 2.8. Figure 2.8(a) shows a high magnification view of the separated surface on the replicated Al HARMS. The separated surface possesses a cellular-like structure containing numerous micron- and submicron- scale dimples, suggesting that separation of the bonded surfaces occurred via ductile fracture, and that the Al-based HARMS and Al coupon are well bonded. Figure 2.8(b) shows a lower magnification view of the separated surface on the replicated Al HARMS. This micrograph covers an area close to one microchannel, a small fraction of the microchannel sidewall is visible on the extreme right hand side of the micrograph. Two distinct morphologies are observed over the top separated surface: the first ~40µm from the microchannel sidewall appears smooth [region A in Fig. 2.8(b)], the rest shows the cellular-like structure with micron- and submicron- scale dimples [region B in Fig. 2.8(b)]. This suggests that region A close to the microchannel was not bonded. This is because this part of the surface did not come into contact with the flat Al coupon during bonding, due to the upward convexity of the original surface as shown in Fig. 2.3(c). In addition, it appears that more Al-Ge has accumulated near the transition between regions A and B, where the two Al surfaces first came into contact. This is possibly due to the migration of the molten Al-Ge during bonding at above the eutectic temperature from the center of the contact area to the edge under the applied pressure. The non-bonding of region A implies that small crevices exist near the top of the microchannels. The implications of these crevices to fluid flow within microchannels or the elimination of these crevices through improved bonding procedures remain to be investigated.
2.4 Conclusion

A series of Al-Ge composite thin films were vapor-phase codeposited and their composition and micro-/nano-scale structures were characterized. Eutectic bonding of a replicated Al HARMS and a flat Al coupon was carried out using an Al-Ge intermediate layer. Successful bonding was demonstrated at 500°C and an applied pressure of 1.5MPa, using Al-Ge intermediate layers with an average composition close to the eutectic composition. Examination of the bonded Al HARMS/Al coupon assembly, including the morphology of separated surfaces, showed that good bonding quality was achieved. Bonded metallic HARMS serve as platforms for constructing metal-based functional microsystems.

2.5 References


EDAX Inc., Genesis Spectrum SEM Quant ZAF Version 3.60, 03-NOV-2003, Copyright@2003.


CHAPTER 3. EVALUATION OF EUTECTIC BOND STRENGTH AND ASSEMBLY OF AL-BASED MICROFLUIDIC STRUCTURES *

3.1 Introduction

Since Tuckerman and Pease pointed out in 1981 that miniaturizing cooling channel dimensions will lead to increases in heat transfer efficiency, research and development on fabrication of and heat transfer within microfluidic devices have proliferated. Typical microchannel dimensions within microscale heat exchangers (MHEs) are in the 50-500µm range. Experimental heat transfer investigations have been conducted on Si-based microchannel devices and metallic microchannels fabricated by mechanical cutting. Although the original Tuckerman and Pease study was conducted in Si-based microchannels, the combination of mechanical properties and thermal conductivity of Si suggests that the overall performance of Si-based MHEs may not be as favorable as Al- or Cu-based ones in many applications. Fabrication of metallic microchannels by microscale mechanical cutting is a serial and slow process, which becomes more difficult as the geometrical complexity of the microchannels increases.

The LiGA technology, combining X-ray/UV based deep lithography (Lithographie), metal electrodeposition (Galvaniformung), and molding replication (Abformung), is an important approach to fabricating polymer-based and metal-based high-aspect-ratio microscale structures (HARMS). The high cost of X-ray/UV lithography and low speed of electrodeposition conspire to make primary metal-based HARMS fabricated by lithography and electrodeposition (LiG) expensive. The process of molding replication therefore holds the key to low-cost mass production of two-and-one-half dimensional (2.5D) metallic HARMS with arbitrary planar micropatterns.

Polymethylmethacrylate (PMMA) MHE prototypes were built with LiGA technology by molding PMMA sheets with electrodeposited microscale Ni mold inserts followed by gluing with polymer-based adhesives. Ni MHE prototypes were built with a variation of the LiG process, by Ni electrodeposition onto metal-seeded polymer mandrels followed by dissolution of the polymer. From the perspective of materials’ selection, neither PMMA nor Ni is the ideal material for MHEs because of the combination of their densities, thermomechanical properties, and thermal conductivities. From the perspective of fabrication, the process of electrodeposition onto “lost-molds” is not an optimal technique for low-cost mass production due to the multiple fabrication steps, low manufacturing throughput, and limitations on materials. We have previously demonstrated successful 2.5D HARMS fabrication by direct molding replication in Al and Cu with surface engineered microscale mold inserts. The ability of generating arbitrary planar micropatterns on mold inserts made of refractory metals was demonstrated by combining primary HARMS fabrication by LiG with parallel pattern transfer by micro electrical discharge machining.

Bonding and assembly are vital for fabricating functional MHEs. Several bonding methods, such as anodic bonding, direct bonding, and intermediate layer bonding, have been reported for Si-based microsystems. Recently, we have reported successful bonding of Al-based HARMS with Al-Ge eutectic intermediate layers. Vapor phase deposited Al-Ge nanocomposite thin films were utilized to bond Al-based microchannels without excessive distortions of microscale features. In this chapter, we report further experiments on Al-Ge eutectic thin film bonding of Al structures. Tensile strength of eutectic bonds was measured as a

* Reprinted by permission of “Microsystem Technologies”
function of bonding parameters by tensile testing. Morphological and chemical examinations of fractured bonding surfaces helped to further elucidate bonding mechanisms. With the Al-Ge eutectic thin film bonding strategy, successful assembly of Al-based two-layer microchannel devices was demonstrated.

3.2 Experimental Procedures

Al-Ge composite thin films were deposited with a radio frequency (rf) inductively coupled plasma (ICP) assisted hybrid tool. Al-Ge films were deposited by codeposition from two separate sputter sources, one for pure Al(99.99%) and the other for pure Ge(99.99%). Further details on the ICP assisted hybrid deposition system, Al-Ge sputter codeposition, and structural characterization of Al-Ge nanocomposite thin films have been reported in Chapter 2 and our previous paper.

Substrates for Al-Ge thin film deposition included cuboid Al coupons (Al6061, ~22mm × ~22mm × ~18mm) with surfaces mechanically polished to less than 1µm in roughness, polished Al foils (Al1100, 99%+) ~800µm in thickness, and replicated Al HARMS (Al1100, 99%+). The substrates were ultrasonically cleaned in acetone and methanol before being mounted on a rotatable holder situated in the middle of the deposition zone. The ultimate base pressure of the deposition system is less than 1.0×10^{-8}Torr. Typical background pressures prior to deposition runs were about 1×10^{-7}Torr.

The entire deposition sequence was carried out in pure Ar (99.999%), with a total pressure of ~1.3mTorr. The deposition sequence consisted of a substrate surface etch followed by codeposition of Al and Ge. Substrate etching occurred in a pure Ar ICP with a total rf input power of 1000W, a substrate bias of -100V, and an etch duration of 15min. Sputtering of Al and Ge cathodes commenced immediately after substrate surface etch. The substrate bias was fixed at -50V during all Al-Ge depositions. All Al-Ge depositions were carried out with a fixed Al cathode current of 1.0A and a fixed Ge cathode current of 0.5A, respectively. This deposition condition resulted in a Ge to Al composition ratio close to that corresponding to the Al-Ge eutectic composition of Al_{70}Ge_{30}. One series of Al-Ge composite films were deposited on cuboid Al coupons, which were used to prepare tensile testing specimens for the evaluation of bond strength. For this series of Al-Ge films, the deposition duration was varied from 15 to 60min, corresponding to film thickness ranging from ~0.5 to ~2.0µm. Additional Al-Ge depositions on polished Al foils and replicated Al HARMS were carried out for 60min, corresponding to a film thickness of ~2.0µm.

Bonding experiments were carried out using a MTS858 single-axis testing system interfaced to a custom-built high-vacuum chamber. One heating station was mechanically attached to the bottom of the vacuum chamber. A second heating station was mechanically attached to the top linear actuator of the MTS858 testing system through a bellows-sealed motion feedthrough. The linear actuator could be programmed to move according to prescribed load forces in the force-controlled mode or actuator displacements in the displacement-controlled mode. The total axial force was measured by a 25kN load cell, and the total axial displacement of the actuator was measured by a linear variable displacement transducer. Further details on this high-vacuum, high-temperature, bonding system also have been given in Chapter 2 and our previous paper.

To create tensile testing specimens, Al-Ge composite films were deposited onto the bonding surfaces of two cuboid Al coupons, ~22mm × ~22mm in area. The two Al coupons were placed face-to-face on the bottom heating station, forming an assembly of ~36mm in length along the axial direction, which is perpendicular to the bonding interface. A small hole was
drilled at the corner of the bottom Al coupon close to the bonding interface. A K-type thermocouple was inserted into the hole to measure the temperature of the interface during bonding. After the chamber was evacuated, both heating stations were heated to temperatures about 10 degrees higher than the target bonding temperatures of 450, 500, and 550°C. During the heating process, the top heating station was close to the top surface of the Al coupon assembly but not in contact with it. The temperature of the bonding interface during this heating process, as measured by the K-type thermocouple, was always less than 390°C and below the Al-Ge eutectic temperature of 424°C. Once the bottom and top heating stations reached steady state temperatures, the upper heating station was then put into contact with the Al coupon assembly using the linear actuator. An linearly increasing compression force was applied to the Al coupon assembly such that loading force levels of 250, 500, and 750N were reached after a constant duration of 8min. These loading forces correspond to applied pressures of ~0.5, ~1.0, and ~1.5MPa, respectively. A constant force hold was executed for 10min after the compression force reached the desired levels. During the compression force increase, the temperature of the bonding interface increased to close to the target temperatures of 450, 500, and 550°C. Further temperature increase was registered on the K-type thermocouple during the constant force hold. For all bonding runs, the total temperature change measured by the K-type thermocouple during the 10min constant force hold was less than 12°C. The nominal bonding temperature was taken to be the average value of the K-type thermocouple reading during the 10min constant force hold. After this hold period, the heaters were turned off to cool the bonded specimen, with the same force still applied. After the temperature of bonding interface decreased to less than 400°C, the linear actuator was withdrawn from the Al assembly and the system was cooled down to room temperature.

A series of two-Al-coupon assemblies were bonded at different temperatures and applied pressures, and with different thicknesses of Al-Ge intermediate layers. Using a Struers Accutom5 cutting machine, the four outermost sections along the axial direction, ~3-4mm in thickness, were removed from the bonded Al specimen. From the remaining specimen of ~14mm × ~14mm × ~36mm in dimension, four tensile testing specimens were obtained by performing two perpendicular bisecting cuts parallel to the axial direction, resulting in a final tensile specimen dimension of ~7mm × ~7mm × ~36mm. After cutting, the four surfaces of the tensile specimen parallel to the axial direction were mechanically polished with 600 grit silicon carbide papers to remove damages. Tensile testing along the specimen axial direction was performed to evaluate the tensile strength of the bonding interface, and was carried out on a MTS810 system with hydraulic grips. Surface morphologies of fractured bonding surfaces were conducted on a Hitachi S3600N scanning electron microscope (SEM) combined with an X-ray energy dispersive spectroscopy (EDS) system (EDAX) equipped with an ultra-thin window detector.

A bonding process similar to that described above for bonding two cuboid Al coupons was employed to assemble a two-layer microchannel device. Al-Ge composite thin films were deposited on both sides of a polished Al foil and the feature surfaces of two replicated Al HARMS, with a nominal thickness of ~2µm. After Al-Ge film deposition, the two Al HARMS were placed face to face on the bottom heating station, with the Al foil inserted in the middle. Both heating stations were heated to slightly above 500°C, and the upper heating station was then put into contact with the assembly. An increasing compression force was applied to the assembly at a constant loading rate of 300N/min. A constant force hold was executed for 10min after the compression force reached the desired level of ~1000N. This force level corresponds to an applied pressure of ~1.5MPa. After the constant force hold, the linear actuator was withdrawn
from the Al assembly and the system was cooled down. SEM examinations of the bonded Al assembly were also conducted on the Hitachi S3600N SEM.

### 3.3 Results and Discussion

A typical, as-prepared, tensile specimen is shown in Fig. 3.1(a). During tensile testing, the two ends of the specimen were gripped by the hydraulic grips. The gripped sections were ~10mm in length, leaving an un-gripped section of ~16mm in length with the bonding interface in the middle. The bonding interface, whose location was approximately indicated by the arrow in the figure, is not readily perceptible.

![Fig. 3.1](image)

**Fig. 3.1 Tensile testing of bonded Al6061 specimens:** (a) images of one typical as-prepared specimen and one typical tested specimen. The locations of the bonding interface are approximately indicated by the arrows. Numbers on the ruler denote mm; (b) a typical tensile testing data set, resulting in a clean break at the bonding interface; (c) a typical tensile testing data set, resulting in severe plastic deformation of the gripped specimen sections without breaking the bonding interface.

Figure 3.1(b) shows one set of tensile testing data, plotted as the tensile stress vs. the specimen extension. As the extension increases, the tensile stress increases to a maximum of ~143MPa. At this moment, a clean break occurred at the bonding interface, leading to the sharp drop in stress shown in Fig. 3.1(b). This tensile test therefore yields a measurement of the tensile
bond strength. A different outcome of tensile testing is illustrated in the set of tensile testing data shown in Fig. 3.1(c). In this case, the tensile stress increases with increasing specimen extension to a maximum of ~167MPa, after which a relatively quick decrease in stress is observed. The tested specimen is also shown in Figure 3.1(a), from which it is evident that the observed decrease in stress is not due to breakage at the bonding interface. Instead, severe plastic deformations at the gripped sections resulted in the observed stress decrease. In this situation, the tensile testing shows that the strength of the bonding interface is higher than 167MPa, and yields only a lower limit on the bond strength.

Figure 3.2 summarizes results of bond strength measurements as a function of bonding temperature. For this series of measurements, all bonding runs were performed at an applied pressure of ~1.5MPa and a thickness of ~2µm of Al-Ge intermediate layer deposited on each bonding surface. The total thickness of the Al-Ge intermediate layers at the bonding interface is therefore ~4µm. Out of all 15 tensile tests, the four performed on specimens corresponding to the bonding temperature of ~450°C all resulted in clean breaks at the bonding interface (denoted “break” in Fig. 3.2). Out of the seven tests performed on specimens corresponding to the bonding temperature of ~500°C, only one resulted in breaking at the bonding interface. The other six tests resulted in deformations at gripped sections without any break at the bonding interface (denoted “no break” in Fig. 3.2). All four tests performed on specimens corresponding to the bonding temperature of ~550°C resulted in deformations at gripped sections only (denoted “no break” in Fig. 3.2). It is evident from Fig. 2 that measured values of bond strength exhibit large scatter. At the bonding temperature of ~450°C, measured bond strength varies between 85 to 156MPa. Similar comments apply to measurements on specimens corresponding to the bonding temperature of ~500°C, where measured bond strength varies between 77 to greater than 167MPa.

![Fig. 3.2 Bond strength as a function of bonding temperature.](image)

The fracture surfaces of the specimen bonded at ~500°C, which exhibited a clean break at the bonding interface during tensile testing, were examined by SEM. Even at the low magnification of Fig. 3.3, it is apparent that two kinds of regions exist on this fracture surface, heuristically termed the “good” and the “bad” regions. Differences in surface morphologies of
these two kinds of regions result in different observed contrast at this low magnification. The typical morphology of the “good” region is exemplified by the SEM micrograph shown in Figure 3.4(a). Within the “good” region, faceted regions, ~1-2µm in size, are interdispersed with regions containing numerous micron and submicron sized dimples. The chemical makeup of the faceted regions and the dimpled regions were probed by X-ray intensity mapping with the EDS system. Figures 3.4(b) and 3.4(c) show respectively Al-K and Ge-L X-ray intensity maps corresponding to Fig. 3.4(a). Faceted regions, e.g., those labeled by A, B, and C in Fig. 3.4, yield low Al-K and high Ge-L characteristic X-ray emissions under electron beam excitation, and contain Ge crystallites at the surface. Dimpled regions show little Ge-L X-ray count, and consist entirely of the Al matrix. Exceptions can be found, e.g., in the region labeled D in Fig. 3.4. Although facets are observed in the SEM micrograph at D, the corresponding X-ray maps show no deficiency in Al-K counts and no enhancement in Ge-L counts at D. Thus it appears that the Ge crystallite originally at D has detached from the Al matrix during fracture.

Fig. 3.3 A low-magnification view of the fracture surface of the specimen bonded at ~500°C. The two areas with different morphologies are labeled “G” and “B”, denoting respectively “good” and “bad” regions.

Fig. 3.4 Morphological and chemical characterizations of the “good” region on the fracture surface: (a) an SEM micrograph; (b) the corresponding Al-K X-ray map; (c) the corresponding Ge-L X-ray map. Identical magnifications apply to (a), (b), and (c).

The typical morphology of the “bad” region is exemplified by the SEM micrograph shown in Figure 3.5(a). Within the “bad” region, faceted regions, e.g., those labeled by A and B
in Fig. 3.5, are again observed. These faceted regions, however, are \( \sim 5 \mu m \) in size and larger than that observed in Fig. 3.4(a). Outside these faceted regions, the presence of dimples are less pronounced as compared to that observed in Fig. 3.4(a). Al-K and Ge-L X-ray intensity maps corresponding to Fig. 3.5(a), shown respectively in Figs. 3.5(b) and 3.5(c), again confirm that faceted regions contain Ge crystallites at the surface and that dimpled regions consist entirely of the Al matrix.

Fig. 3.5 Morphological and chemical characterizations of the “bad” region on the fracture surface: (a) an SEM micrograph; (b) the corresponding Al-K X-ray map; (c) the corresponding Ge-L X-ray map. Identical magnifications apply to (a), (b), and (c).

Observations shown in Figs. 3.3, 3.4, and 3.5 have several implications. First, these observations show that, within the entire bonding interface, Ge and Al are phase separated. This is expected thermodynamically, because the Al-Ge system forms a simple eutectic with limited terminal solubilities.\(^{21}\) Our previous experiments showed that phase separation occurred within as-deposited Al-Ge films prepared by sputter codeposition. The Ge grains within as-deposited Al-Ge films were \( \sim 100 \text{nm} \) in size.\(^{18}\) During the bonding process, as the temperature increases above the Al-Ge eutectic temperature of 424°C, the Al-Ge nanocomposite films first melt into a homogeneous liquid phase, sandwiched in between the two Al solid bulks. As the temperature of the bonding interface cools below the eutectic temperature, eutectic crystallization occurs, forming separate regions consisting of only Ge crystallites and only crystalline Al.

Second, the presence of faceted Ge crystallites on the fracture surface suggests that separations occur at interfaces between Ge crystallites and the Al matrix and possibly across Ge crystallites in a brittle manner. On the other hand, the presence of the characteristic dimples on the fracture surface indicates that separation of the Al regions involves ductile fracture.\(^{22}\)

Third, we speculate that the two major differences observed between the “good” and “bad” regions, namely the size of the Ge crystallites and the density and severity of dimples in the Al regions, arise from local variations in applied pressure during the bonding process. The local applied pressure may be higher and lower in the “good” and “bad” regions, respectively. This local variation in applied pressure could be due to non-conformal profiles of the mating surfaces, surface undulation and roughness, and system misalignment. In regions with higher applied pressure, the more intimate mechanical contact suppresses lateral coarsening of the Ge crystallites and promotes diffusional mixing of the Al regions perpendicular to the bonding interface, resulting in smaller Ge crystallite sizes and increased density of dimples in the Al regions during fracture. Lower applied pressure results in less intimate mechanical contact, which leads to increased lateral coarsening of the Ge crystallites and diminished diffusional mixing of the Al regions perpendicular to the bonding interface. This diminished mixing results
in decreased density of dimples in the Al regions during fracture and consequently lower bond strength.

Further fracture surface examinations were performed on specimens bonded at ~450°C. In general, the morphology of these fracture surfaces is similar to that observed on specimens bonded at ~500°C, and consists of Ge crystallite regions and Al regions with dimples. Results of SEM examinations can be summarized into the qualitative statement that increased bond strength is correlated with higher density and severity of dimples observed in the Al region. We therefore render the additional speculation that the overall bond strength is dominated by ductile fracture across Al regions within the bonding interface. Higher strength interfaces induce more plastic deformation of the Al regions during fracture, and therefore increased density and severity of dimples. The observed presence of “good” and “bad” regions on the fracture surfaces therefore provides a rationalization for the observed large scatter in bond strength values: the measured overall bond strength represents an average over the entire bonding interface and is dependent on the relative dominance of “good” and “bad” regions within this interface. The “intrinsic” bond strength can be higher than 167MPa, according to data presented in Fig. 3.2. Improved bonding procedures are needed to minimize bonding variability, and will be pursued in the future. One additional factor, which can potentially influence the outcome of bond strength measurements, is misalignment during tensile testing. Excessive axial misalignment leads to changes in the mode of fracture at the bonding interface from tensile separation to peeling. Care was taken during tensile testing to minimize misalignment effects.

Figure 3.6 summarizes results of bond strength measurements as a function of applied pressure during bonding. For this series of measurements, all bonding runs were performed at a bonding temperature of ~500°C and a thickness of ~2μm of Al-Ge intermediate layer deposited on each bonding surface. At the lowest applied pressure of ~0.5MPa, all four tensile tests resulted in clean breaks at the bonding interface, yielding measured values of bond strength ranging from 84 to 140MPa. Out of a total of 11 tensile tests performed on specimens bonded at applied pressures of ~1.0 or ~1.5MPa, only two tests resulted in breaks at the bonding interface. The rest of the specimens failed at the gripped sections during testing. Out of these tests, the
highest strength values obtained are higher than 175 and 167MPa at applied pressures of ~1.0 and ~1.5MPa, respectively. Thus while the measured bond strength at the applied pressure of ~0.5MPa appears adequate, this series of tests suggests that more reliable bonds are formed at applied pressures of ~1.0MPa or higher.

Figure 3.7 summarizes results of bond strength measurements as a function of the thickness of Al-Ge intermediate layer deposited on each bonding surface. For this series of measurements, all bonding runs were performed at a bonding temperature of ~500°C and an applied pressure of ~1.5MPa. At the lowest Al-Ge film thickness of ~0.5µm, measured bond strength ranged from 89 to over 152MPa. No gross difference can be discerned from tests performed on specimens bonded at Al-Ge film thicknesses of ~1.0 or ~2.0µm, with the highest measured bond strength exceeding 167MPa in both cases. The maximum strength value was obtained at the Al-Ge film thickness of ~1.0µm, which exceeds 188MPa. At all three Al-Ge film thicknesses of ~0.5, ~1.0, and ~2.0µm, similar scatter in bond strength value is observed.

Two Al HARMS pieces were used to assemble a two-layer microchannel structure. In each Al piece, a set of parallel rectangular microchannels, ~1cm in total length and ~330µm in depth, was replicated into the Al bulk from surface engineered Inconel X750 inserts by compression molding. Two plena were machined into the Al bulk and connected to the two ends of the microchannel array. To test the feasibility of simultaneously bonding microfeatures of different sizes, the widths of microchannels were made to vary between less than 80µm to more than 250µm. The polished Al foil with Al-Ge films deposited on both sides was inserted in between the two Al HARMS pieces, and the bonding process described above yielded a three-piece Al assembly containing two layers of parallel microchannels. Threaded holes were machined into the plena and served as external fluid connections. Figure 3.8(a) shows a cross-sectional view of a portion of the assembled two-layer microchannel structure, obtained by mechanical cutting perpendicular to the microchannel arrays. The entire structure contains 20 microchannels in each layer. Figure 3.8(b) shows a close-up view of a typical bonded...
Fig. 3.8 Assembly of a two-layer, Al-based, microchannel device: (a) a low magnification overview of a portion of the assembled device; (b) a higher magnification view of one typical bonded microchannel; (c) a still shot of the exit water jets from the microchannel device cross-section.

The bonding interface is not discernable in this higher magnification view, indicative of the quality of bonding achieved. As shown in Fig. 3.8(c), water was fed into one plenum and flew out of the cut cross-section as individual jets. Multilayered microfluidic structures such as that shown in Fig. 8 can serve as a platform for Al-based microfluidic devices, including MHEs.

3.4 Conclusion

Successful eutectic bonding of Al6061 pieces with Al-Ge intermediate thin film layers is demonstrated. We have conducted detailed measurements of tensile bond strength as a function of bonding parameters. At all bonding conditions tested, our results indicated that the tensile strength across the bonding interface, ~22mm × ~22mm in dimension, is at least 77MPa and can be higher than 188MPa. Observed scatter in bond strength values is rationalized with
morphological and chemical characterizations of fractured bonding interfaces. With the Al-Ge eutectic bonding strategy, successful assembly of a two-layer microchannel device is demonstrated. Assembly of functional, metal-based, microfluidic devices is planned for the future.

3.5 References


CHAPTER 4.  STRUCTURE OF VAPOR-PHASE DEPOSITED Al-Ge THIN FILMS AND MECHANISM OF Al-Ge INTERMEDIATE LAYER BONDING OF Al-BASED MICROCHANNEL STRUCTURES *

4.1 Introduction

Micro/nano electro-mechanical systems (MEMS/NEMS) technologies aim to integrate structures and materials at the micro- and nano- scales to achieve expanded functionalities and increased performance for wide ranging applications. Proper bonding and packaging are vital to ensure the structural integrity and efficient working of MEMS/NEMS devices. Early MEMS applications utilize Si-based structures, well known examples of which include MEMS cavities in ink jet printers and MEMS sensors in automobiles.1 Si-based MEMS enjoy well developed manufacturing protocols, originating from the mature Si integrated circuit (IC) fabrication technologies. Various bonding schemes for Si-based and Si-related microsystems have been investigated,2 including direct bonding,3 anodic bonding,4 intermediate layer bonding,5 and surface activated bonding.6,7 Three-dimensional (3D) packaging schemes for Si-based MEMS have been contemplated.8

Metal-based MEMS devices can offer functions not achievable with Si-based devices, an example of which is magnetically actuated micro relays.9 Other metal-based MEMS devices can have performance improvements over similar Si-based devices, especially in harsh environments. Metallic microchannel heat exchangers (MHEs) made of Al or Cu offer one such example.10 The higher bulk thermal conductivities of Al and Cu as compared to that of Si lead to improved thermal performance of Al- or Cu- based MHEs over similar Si-based devices, while the much increased ductility of Al and Cu as compared to that of Si results in improved mechanical integrity of Al- or Cu- based MHEs.

The basic building block for metal-based MEMS is metallic high-aspect-ratio microscale structures (HARMS). One important fabrication strategy for metal-based HARMS is the LiGA (Lithographie, Galvanoformung, Abformung) technique.11 One common approach is to fabricate metal-based HARMS by combining deep X-ray/UV lithography on polymeric resists followed by electrodeposition into developed resist recesses (LiG). Examples of LiG fabrication include Ni-based microchannel gas chromatography columns.12 While metallic structures with high aspect ratios and smooth sidewalls can be fabricated by LiG, the high cost of deep lithography and slow speed of electrodeposition conspire to make such structures expensive and less suitable for mass production.

Recently, direct replication of metallic HARMS by compression molding using surface engineered microscale mold inserts has been demonstrated in a wide range of metals, from softer metals such as Pb13 and Al14 to harder metals such as Cu and Ni.15 As compared to LiG fabrication, HARMS replication by molding is simple and fast once a durable mold insert is made, and offers the potential for low-cost, high-throughput production of metal-based HARMS. Typical mold inserts consist of a microscale 2D pattern protruding out of a basal plane. Compression molding using such inserts forms the so-called 2.5D structures on the surface of the molded metal. This molded structure is then bonded to a mating component to complete the assembly. Proper bonding techniques are critical for building functional metal-based MEMS from such replicated 2.5D HARMS.

* Reprinted by permission of “Journal of Materials Research”
Techniques for bonding metals at the macroscale are well established. Bonding of metal-based HARMS, on the other hand, is much less studied. Fusion welding involves bulk melting of the parts to be joined and is not suitable for bonding of metallic HARMS. One particular difficulty related to bonding of Al pieces stems from the propensity of Al surface to form a native oxide layer, which makes direct Al-Al diffusion bonding more difficult. Among the different diffusional bonding techniques, the use of a chemically/structurally distinct intermediate layer at the bonding interface can lower the bonding temperature, eliminate bulk melting of the parts to be joined, promote interdiffusion, and increase the bond quality. The bonding process would be facilitated if the process of depositing a chemically distinct intermediate layer can at the same time serve to remove the native oxide layer on surfaces of the Al pieces. When the intermediate layer is an eutectic layer, the process of melting also facilitates the breakup of any residual oxide.

Examples of bonding using eutectic intermediate layers exist in the literature. Utilizing the Au-Si eutectic at 363°C, Tiensuu et al. vapor-phase deposited Au thin films onto surfaces of Si micro-components with different geometries, and conducted bonding experiments at ~520°C. Utilizing the Al-Ge eutectic at 424°C, Vu and Zavracky vapor-phase deposited alternating thin film layers of Al and Ge and used them as the intermediate layer for bonding and sealing of Si-based MEMS structures at temperatures above 525°C. In one example of successful bonding of metal-based HARMS, Mei et al. demonstrated bonding of Al-based microchannel structures using sputter co-deposited Al-Ge thin film intermediate layers. The average tensile bond strengths of Al pieces bonded with co-deposited Al-Ge intermediate layers were measured experimentally as a function of bonding temperature and pressure and shown to exceed 75MPa.

Apart from the technological interest in Al-Ge thin films as intermediate layers for bonding of metallic HARMS, Al-Ge thin films are of scientific interest as a model system for studying the process of phase separation during deposition. Al and Ge form a simple eutectic system with limited terminal solid solubilities at room temperature, with the eutectic temperature $T_E$ at 424°C and the eutectic composition about 70at% Al - 30at% Ge. In this chapter, the structure of sputter co-deposited Al-Ge thin films was characterized using transmission electron microscopy (TEM). Interesting nanoscale features were revealed by phase contrast imaging, and expose an interesting aspect of the Al-Ge phase separation process during thin film deposition that is not previously observed. Also in this paper, HARMS-containing Al coupons were bonded to flat Al plates with sputter co-deposited Al-Ge thin film intermediate layers. The structure around the original bonding interface was investigated by combining scanning electron microscopy (SEM) with focused ion beam (FIB) sectioning, and shed light on the metallurgy of bonding and mechanisms through which relatively high bond strengths are achieved.

4.2 Experimental Procedures

4.2.1 Vapor-Phase Deposition of Al-Ge Thin Films

Al-Ge composite thin films were prepared using a radio frequency (rf) inductively coupled plasma (ICP) assisted hybrid deposition tool, which combines a 13.56MHz ICP with direct current (dc) balanced magnetron sputtering. Further details on the hybrid tool design have been reported previously. Al-Ge films were co-deposited from two separate sputter sources, one for a pure Al (99.99%) cathode and another for a pure Ge (99.99%) cathode, both 75mm in diameter. The cathodes were placed facing each other at a distance of 32.5cm and sputtered in
the current controlled mode. The ultimate base pressure of the deposition system is \(<1.3 \times 10^{-6}\) Pa (1\(\times\)10^{-8}Torr). Typical background pressures prior to deposition runs were \(~1.3 \times 10^{-5}\) Pa (1\(\times\)10^{-7}Torr). The entire deposition was carried out in an Ar (99.999\%) atmosphere, with a total pressure of \(~0.17\) Pa (1.3\(\times\)10^{-3}Torr).

Substrates were mounted on a rotatable holder situated in the middle of the two cathodes, and rotated continuously in the center of the deposition zone at \(~12\) rpm during both etching and deposition. The deposition sequence consisted of a substrate surface etch followed by Al and Ge co-deposition. Substrate surface etching occurred in a pure Ar ICP with a total input rf power of 1000W, a substrate bias of -200V, and an etch duration of 3min. Sputtering of Al and Ge cathodes commenced immediately after substrate surface etch. No intentional substrate heating or cooling was applied during the entire deposition process. Previous temperature measurements on Si wafer substrates during deposition in the ICP assisted deposition tool showed substrate temperatures \(~140^\circ\)C. Additional measurements showed that the substrate temperature exhibited little dependence on the cathode power, an observation rationalizable by the relatively large distance between the substrate and the sputter cathodes.

Al-Ge thin films were co-deposited onto flat Al plates, Al coupons containing molded microchannels, and Si(100) wafers. To investigate the internal structure of Al-Ge films, one series of Al-Ge films were deposited onto 50mm diameter Si(100) substrates. Prior to deposition, the Si wafers were cleaned in acetone and methanol. The substrate bias during deposition was fixed at -100V. For this series of specimens, a fixed Al cathode current of 0.5A was used, and the Ge cathode current was varied from 0.1A to 0.5A to alter the Ge composition within the film. The deposition duration was fixed at 30min for this series of specimens and the film thickness was about 400nm. To increase the Al-Ge film deposition rate onto various Al substrates without changing the average film composition, Al and Ge cathode currents were increased while keeping their ratio fixed, resulting in a final Al-Ge film thickness of \(~2\) \(\mu\)m.

### 4.2.2 Compositional and Structural Characterization of Vapor-Phase Deposited Al-Ge Thin Films

Impurity content within the co-deposited Al-Ge thin films was analyzed by X-ray photoelectron spectroscopy (XPS). XPS measurements were conducted on a Kratos AXIS165 system. Surface etching prior to XPS measurements on Al-Ge films was carried out with ion beam sputtering with 5kV Ar\(^+\) ions for 30min. Compositional quantitation from raw XPS data made use of factory-supplied sensitivity factors. The oxygen content within sputter co-deposited Al-Ge films was determined to be 1at.\% or less. Quantitative compositional analysis of Al-Ge composite thin films were performed with energy dispersive X-ray spectroscopy (EDS). EDS measurements were conducted using a EDAX Genesis system equipped with a liquid-nitrogen cooled, 10nm\(^2\), Super Ultra-Thin Window Si(Li) detector, attached to a Hitachi S3600N scanning electron microscope. EDS spectra were collected at an incident electron beam energy of 15keV and a detector take-off angle of 36°. The factory-supplied EDS data analysis software was used for automatic background fitting and removal. A standardless quantification routine employing ZAF corrections was used to obtain the average ratio of Ge at.\% composition to Al at.\% composition.

Cross-sectional TEM was used to characterize the micro-/nano- scale structures of the Al-Ge composite films. Cross section TEM specimens of Al-Ge films deposited on Si(100) substrates were prepared following the standard procedures of face-to-face gluing, mechanical thinning followed by dimple grinding, and final ion milling using 4kV Ar\(^+\) ions at a 4° take-off.
angle on a Gatan 691 Precision Ion Polishing System. Imaging was carried out on a JEOL 2010 microscope operated at 200kV. Another EDAX Genesis system equipped with a liquid-nitrogen cooled, 30mm², Super Ultra-Thin Window Si(Li) detector was attached to the TEM, and was used to obtain compositional information from regions examined in the TEM.

4.2.3 Bonding of Al-based Microscale Structures

An array of rectangular microchannels was replicated into one Al coupon by compression molding with inserts made of Ni-based superalloy Inconel X750®. One fluid supply channel and one fluid drain plenum were created using micro electrical discharge machining (µEDM) onto the molded Al coupon to provide fluidic connections to the replicated microchannel array. Figure 4.1(a) shows an overview of such an Al coupon containing a microchannel array with 19 parallel rectangular microchannels, together with a fluid supply channel and a fluid drain plenum. Mechanical drilling was used to make two through-holes connecting to the supply channel and three through-holes within the drain plenum. Figure 4.1(b) provides a more detailed view of the fluidic supply connections on the Al coupon, from one through-hole to the supply channel which connects to the replicated array of rectangular microchannels. The supply channel has the mottled surface morphology typical of structures cut by µEDM, and provides connections to the microchannel arrays without obstructions at the microchannel entrances.

![Fig. 4.1 A typical Al-based microchannel structure: (a) an overview of one Al coupon containing an array of replicated rectangular microchannels; (b) a SE image of the fluidic supply connections to the microchannel array.](image)

To form all-Al, enclosed, microchannel structures, Al coupons containing replicated microchannels together with fluid supply channel and drain plenum, as exemplified in Fig. 4.1, were bonded to flat Al plates. The bonding process utilized co-deposited Al-Ge thin films with average compositions close to the eutectic composition, ~Al₇₀Ge₃₀, as intermediate layers. The thickness of the Al coupons and flat Al plates was 6.4mm. Bonding surfaces of Al coupons and flat Al plates were mechanically polished with SiC abrasive papers and finished with 1µm diamond particle suspension before loading into the vacuum system. They were surface-etched in a pure Ar ICP with a total input rf power of 1000W, a substrate bias of -100V, and an etch duration of 15min. The Ar ICP surface-etch removes the native oxide layer existing on Al surfaces. Immediately after etching, Al-Ge thin films were co-deposited onto the Al coupons and flat plates at a fixed Al cathode current of 1.0A and a fixed Ge cathode current of 0.5A,
respectively. The substrate bias during Al-Ge co-deposition was fixed at -50V. These deposition conditions resulted in an average Ge to Al composition ratio within the Al-Ge film close to that corresponding to the Al-Ge eutectic composition of Al\textsubscript{70}Ge\textsubscript{30}. The deposition duration was 60 min and the total Al-Ge film thickness was \(\sim 2\mu m\). Due to substrate rotation during deposition, the present sputter co-deposition process is more conformal in nature. In the case of Al coupons containing replicated microchannels, the process of co-sputtering deposits the Al-Ge film over the surfaces of the microchannel sidewall and bottom in addition to the bonding surfaces.

Bonding experiments were carried out using a MTS858 single-axis testing system interfaced to a high-vacuum chamber containing two heating stations. One was mechanically attached to the bottom of the vacuum chamber and another was mechanically attached to the top linear actuator of the MTS858 testing system through a bellows-sealed motion feedthrough. The two heating stations were heated separately by resistive heating cartridges. Careful machining of the heating stations was performed to ensure that the surfaces of the two heating stations were parallel to each other and perpendicular to the actuator axis. The temperatures of the heating stations were measured by two separate K-type thermocouples inserted into the bodies of the heat stations, close to their top surfaces.

Al-Ge films with average composition close to the eutectic composition and a total thickness of \(\sim 2\mu m\) were deposited onto one Al coupon containing microscale features and one flat Al plate. After Al-Ge film deposition, the Al coupon and the Al plate were placed face-to-face on the lower heating station. The chamber was evacuated, and both heating stations were heated. A compression force was applied to the Al pieces until the average applied pressure reached \(\sim 1.5\)MPa. At this time the temperatures of both heating stations reached \(\sim 500^\circ C\). Because of the small thickness of Al plates and the high thermal conductivity of Al, this temperature was taken as the bonding temperature. The total time the specimen was held at the bonding temperature under 1.5MPa pressure was \(\sim 40\)min. Typical temperature fluctuation during this time is \(\pm 5^\circ C\).

Initial examination of bonded Al microchannel structures began by mechanically cutting them in directions perpendicular to the microchannel array. To avoid damage to the bonded microchannel structure, the cut was placed within the drain plenum on the Al coupon. To fully reveal the cross sectional configurations of the bonded microchannels, the cut piece containing the microchannel array was mechanically polished back close to the microchannel exits. Figure 4.2 A cross-sectional view of a portion of one bonded microchannel structure in Al.
4.2 show a cross-sectional view of a portion of the bonded microchannel structures in Al. The top horizontal line delineates the location of the bonding interface between the Al coupon and the flat Al plate. The bottom wavy line reveals the bottom of the drain plenum made by μEDM. Additional evaluation of bond quality was performed through measurements of the average tensile strengths of such eutectically bonded interfaces as a function of bonding parameters.21

4.2.4 FIB Characterization of Bonded Al MHE Structures

A Zeiss 1540XB FIB system was employed to characterize the structure of Al-Al interfaces bonded with Al-Ge composite thin film intermediate layers, as well as the corner profile of bonded Al microchannels. One additional mechanical cut was performed on the bonded Al microchannel structure. The cut was made using a Struers Accutom5 precision cutting machine, in the direction perpendicular to the microchannel array and bisecting it. Prior to cutting, the microchannel structure was heated to ~150°C and the entire microchannel array was filled with molten thermoplastic polymer Crystalbond™ through the fluid supply channel. After cool down to room temperature, the solidified Crystalbond™ within the microchannels helped to avoid damage to the microchannel structure due to mechanical cutting. After cutting, two pieces containing the microchannel array on the cut cross-section surfaces were obtained. The cut cross-section surfaces were further mechanically polished with SiC abrasive papers and finished with 1μm diamond particle suspension, after which the Crystalbond™ was dissolved with acetone. One of these two cut pieces was used to characterize the micro-/nano- structures of bonded Al-Al interface by FIB polishing on the cross-section surface. Another cut piece was further thinned from the side of the flat Al plate, until its top surface is within 1mm to the microchannel array. Then, this thinned top surface was polished with a tilting angle of ~10° until a portion of the flat Al plate was totally removed, exposing the microchannel array underneath while leaving the rest of the microchannel array still buried. With this final tilted polishing, a very thin layer of the bonded flat Al plate covers the microchannel array near the transition from exposed to buried microchannels. FIB sectioning through this thin top layer exposed bonded microchannels without mechanical contact, and was used to characterize the corner profile of bonded microchannels. Morphological examination and compositional analysis of FIB polished and sectioned surfaces was performed using secondary electron (SE) imaging and EDS attached to the Zeiss 1540XB FIB system. In some instances, it was beneficial to lightly etch the mechanically polished surface with the ion beam. This was accomplished by imaging the surface with a low current ion beam at normal incidence for a short time.

4.3 Results and Discussion

Figure 4.3 shows a cross-sectional TEM dark-field (DF) image obtained from one specimen of the Al-Ge film series co-deposited onto Si(100) substrates. This film was co-deposited at the Al and Ge cathode currents of 0.5A and 0.3A, respectively. The average ratio of Ge at.% composition to Al at.% composition of this film was determined via EDS to be ~0.7 and higher than 0.43, the value corresponding to the Ge to Al ratio at the eutectic point. The DF image was obtained under a specimen tilt such that the Si substrate was in the <110> zone-axis orientation and the electron beam is in parallel with the film/substrate interface.

The overall structure of this Al-Ge film is apparent from Fig. 4.3. The DF imaging reveals Ge regions in light contrast and Al-rich regions in dark contrast. The Si substrate interface is readily visible, next to which is a crystalline Ge layer of ~50nm in thickness. Above
this Ge layer, lateral separation occurs between Ge regions and Al-rich regions in the plane parallel to the substrate surface. As the Al-Ge film thickness increases, some Al-rich regions begin to spread laterally and cover the Ge region underneath, showing evidence of transverse separation between Ge regions and Al-rich regions in the direction perpendicular to the substrate surface. The existence of phase separation was confirmed with EDS spectra, which showed predominantly Ge signal when the electron beam was placed in the regions showing light contrast and predominantly Al signal when the electron beam was placed in the regions showing dark contrast. Due to the small sizes of these regions, EDS spectra collected from Al-rich regions always contained signals from Ge, and it is difficult to obtain quantitative composition measurements from these regions due to possible signal overlap from the neighboring Ge regions. The observation of both lateral and transverse separation between Ge and Al-rich regions within the co-deposited Al-Ge film is consistent with previous observations made in electron beam co-evaporated Al-Ge films. Based on the limited room temperature terminal solid solubility in the Al-Ge system, phase separation within co-deposited Al-Ge films is expected on thermodynamic grounds. Phase contrast imaging of the crystalline Ge layer next to the Si substrate reveals that they are epitaxially oriented. Whether this epitaxy caused Al atoms to initially move away from the Si interface and retards Al-Ge lateral phase separation needs further experimental confirmation.

Phase contrast imaging of the specimen shown in Fig. 4.3 was conducted. Figure 4.4(a) shows a high-resolution image of the region marked A in Fig. 4.3, close to the transition between Ge and Al-rich regions. Figure 4.4(a) reveals that the Ge region consists of single crystal Ge grains, grain boundaries, as well as twin boundaries. More interestingly, Fig. 4.4(a) reveals that the Al-rich region next to the Ge region is amorphous in structure. The entire amorphous region shown in Fig. 4.4(a), above the crystalline Ge region, is within the Al-rich region shown in Fig. 4.3. Considering the tendency for elemental Al to rapidly crystallize in the fcc structure, Fig. 4.4(a) therefore suggests that the amorphous region bordering Ge contains both Al and Ge atoms. This suggestion is consistent with the presence of Ge signal in EDS spectra collected from the Al-rich region. Figure 4.4(b) shows a high-resolution image of the region marked B in Fig. 4.3,
again close to the transition between Ge and Al-rich regions. Here the Ge region consists of one single crystal Ge grain together with twins on Ge(111). The Al-rich region bordering Ge is again amorphous in structure, and is suggested to contain both Al and Ge atoms.

Figure 4.4 (a) Phase contrast image of the region marked A in Fig. 4.3; (b) phase contrast image of the region marked B in Fig. 4.3.

Figure 4.5 shows additional phase contrast images from another specimen of the Al-Ge film series co-deposited onto Si(100) substrates. This film was co-deposited at the Al and Ge cathode currents of 0.5A and 0.1A, respectively. The average ratio of Ge at.% composition to Al at.% composition of this film was determined via EDS to be ~0.2 and lower than 0.43, the value corresponding to the Ge to Al ratio at the eutectic point. Figure 4.5(a) captures an interesting moment during the process of Al-Ge phase separation within this specimen. A single crystalline Ge region completely surrounds an Al-rich region. Ge(111) lattice fringes are clearly visible within the Ge region. Lattice fringes are also clearly visible in the center of the Al-rich region. Taking the Ge(111) lattice fringe spacing to be 3.27Å, that corresponding to the (111) plane spacing of bulk Ge, the lattice fringe spacing in the center of the Al-rich region is measured to be 2.02Å, in good agreement with that corresponding to the (200) plane spacing of bulk Al. Therefore, the lattice fringes in the center of the Al-rich region indicate the presence of a single crystal fcc Al grain without significant Ge incorporation. An amorphous region exists on either side of the fcc Al grain, suggesting that these regions are compositionally mixed and contain both Al and Ge atoms. Figure 4.5(b) shows a high-resolution image around a boundary between another Ge region and its neighboring Al region, and illustrates a different stage in the process of Al-Ge phase separation within the same specimen. Ge(111) lattice fringes are clearly visible within the Ge region. Lattice fringes are also clearly visible within the Al region. Taking again the Ge(111) lattice fringe spacing to be 3.27Å, the lattice fringe spacing in the Al region is measured to be 2.33Å, in good agreement with that corresponding to the (111) plane spacing of bulk Al. Figure 4.5(b) shows that the Ge(111) lattice fringes within the Ge grain appear to be parallel to Al(111) lattice fringes within the Al grain. The division between the Ge and Al grains appears to be an atomically sharp grain boundary, without any sign of an amorphous phase. The process of Al-Ge phase separation therefore appears to have been completed at this location, without any remnant of a compositionally mixed Al-Ge region.
Fig. 4.5 Phase contrast images from an Al-Ge film with an average Ge to Al atomic ratio of ~0.2: (a) an Al-rich region completely surrounded by crystalline Ge. White lines delineate Ge(111) and Al(200) lattice fringes; (b) a boundary between a crystalline Ge region and its neighboring Al region. White lines delineate Ge(111) and Al(111) lattice fringes.

Figure 4.6 shows a phase contrast image from another specimen of the Al-Ge film series co-deposited onto Si(100) substrates. This film was co-deposited at the Al and Ge cathode currents of 0.5A and 0.2A, respectively. The average ratio of Ge at.% composition to Al at.% composition of this film was determined via EDS to be ~0.45 and close to the value corresponding to the Ge to Al ratio at the eutectic point. Figure 4.6 shows nanoscale dispersion of crystalline Ge and crystalline Al regions. Taking the Ge(111) lattice fringe spacing in the Ge region to be 3.27Å, the lattice fringe spacings in two Al regions are measured to be 2.34Å and 2.04Å, in good agreement with those corresponding to the (111) and (200) plane spacings of bulk fcc Al, respectively. Additional regions appear to be amorphous in structure, indicating incomplete Al-Ge compositional segregation at these locations.
Two points regarding the structure of and the process of Al-Ge phase separation within co-deposited Al-Ge films can be made. First, images presented in Figs. 4.4, 4.5, and 4.6 suggest that, independent of the average film composition, not all the Al atoms within the co-deposited Al-Ge film initially condense onto the substrate in an elementally-pure fcc phase. A fraction of the Al atoms condense instead into a metastable Al-Ge binary amorphous mixture. The process of Al-Ge phase separation then proceeds through a compositional segregation, during which Ge and Al atoms from the amorphous mixture diffuse and attach respectively to crystalline Ge and Al grains. Formation of a metastable amorphous Al-Ge phase by sputter co-deposition is consistent with many previous examples of amorphous phase formation by sputter deposition. The present mode of Al-Ge phase separation, as illustrated in Figs. 4.4, 4.5, and 4.6, has to our knowledge not been reported in the literature. Second, close to the eutectic composition, a nanoscale mixing of Ge and Al-rich domains is achieved within the co-deposited Al-Ge film through the process of Al-Ge phase separation. For the purpose of using Al-Ge films for intermediate layer bonding, such intimate mixing promotes eutectic melting of the entire Al-Ge film once $T_E$ is reached, and is therefore beneficial for bonding purposes. As compared to Al-Ge eutectic bonding using multilayers of elemental Al and elemental Ge, codeposition of Al-Ge films is more easily implemented since nanoscale mixing of Al and Ge regions is achieved naturally through the process of phase separation and only the average Al-Ge film composition needs to be controlled.

The structure of Al-Al interfaces within the bonded Al microchannel structure was examined by combining SEM with FIB sectioning. Figure 4.7(a) shows a SE image obtained from the cross section of the bonded Al microchannel structure using the Zeiss FIB system. The imaged area, approximately 250µm×190µm, covers a portion of the entire cross section specimen and is close to one corner of one microchannel. Prior to SE image and EDS spectrum acquisition, the entire imaged area was lightly etched with the Ga$^+$ ion beam. The SE image shows a band with speckled contrast around the location of the original bonding surfaces, with a
width of ~100µm. The speckled contrast in Fig. 4.7(a) arises from Ge precipitates, as confirmed by the corresponding Ge-L X-ray intensity map shown in Fig. 4.7(b), in which Ge precipitates are indicated by excesses in Ge-L intensity. The Ge precipitates range in size from <1 to ~4µm. It is clear from Fig. 4.7(b) that a band of Ge precipitates also surrounds the sidewall of the Al microchannel, where the mating Al plate is not present. The presence of Ge precipitates around the sidewall of the Al microchannel is reasonable because the Al-Ge thin film was deposited onto the microchannel sidewall during sputter-codeposition.

Fig. 4.7 Cross-sectional examination of Al-Al interface structure in a bonded Al microchannel device: (a) a SE image close to one corner of one microchannel; (b) the corresponding EDS Ge-L intensity map, with the same magnification as in (a).

Fig. 4.8 FIB sectioning into the mechanical cross-section of the bonded Al-Al interface: (a) a SE image of the FIB section; (b) and (c) are respectively the corresponding Al-K and Ge-L X-ray intensity maps, with the same magnification as in (a).

To ascertain that the presence of the Ge precipitate band is not an artifact of the mechanical polishing process, one perpendicular cut, ~38µm in length and ~20µm in depth, was made with the Ga⁺ ion beam into the cross section surface. This cut, clearly visible in Fig. 4.7(a), straddles the location of the original bonding surfaces. Figure 4.8(a) shows a SE image of the FIB cut into the cross-section, in which Al grains with sizes of 10µm and larger are clearly visible. Ge precipitates, with sizes ranging from ~0.7µm to ~4µm, are dispersed within Al grains in an apparently random fashion. The presence of Ge precipitates is confirmed by corresponding Al-K and Ge-L X-ray intensity maps, shown respectively in Figs. 4.8(b) and 4.8(c). Locations of
Ge precipitates are marked by deficiencies in Al-K intensity in Fig. 4.8(b) and excesses of Ge-L intensity in Fig. 4.8(c). Figure 4.8 shows that, aside from the presence of the random Ge precipitates, no clear demarcation exists around the original bonding surfaces of the two Al pieces. Observations shown in Fig. 4.8 are consistent with those shown in Fig. 4.7, and indicate that Ge precipitates indeed distribute themselves in an approximately uniform manner across an extended interface region ~100µm in width. Similar SE images, together with Al-K and Ge-L X-ray intensity maps, were obtained from around the original bonding surfaces midway in between two microchannels. These data again showed the presence of a band of Ge precipitates surrounding the location of the original bonding surfaces, with a width of ~100µm.

Fig. 4.9 A typical SE image of the tensile fracture surface of an Al-Al specimen bonded at ~500°C.

The information obtained from SEM on FIB cross sections of the bonded Al-Al interface, shown in Figs. 4.7 and 4.8, is consistent with SEM examination of tensile fracture surfaces of bonded Al-Al specimens. Figure 4.9 shows a typical SE image of a tensile fracture surface of a specimen bonded at ~500°C. Faceted regions with sizes ranging from <1 to ~4µm are interdispersed with regions containing numerous micron and submicron sized dimples. The chemical compositions of the faceted and dimpled regions were probed by EDS mapping. Faceted regions yield low Al-K and high Ge-L counts, and represent exposed Ge crystallites. Dimpled regions show low Ge-L counts, and show the Al matrix. Thus SEM fractography confirms the Al-Ge phase separation within the bonding interface region, as well as the sizes of Ge precipitates as observed by SEM on FIB cross sections. Further, the presence of faceted Ge crystallites on the fracture surface suggests that separations occurred either at interfaces between Ge crystallites and the Al matrix or across Ge crystallites in a brittle manner, while the observation of micro/nano scale dimples on the fracture surface suggests that separation of the Al regions involves ductile fracture.28

Taken together, observations shown in Figs. 4.7, 4.8, and 4.9 offer a plausible explanation for the high average tensile strength measured for the bonded Al-Al interface: the Al-Ge intermediate layer bonding process has effectively joined the Al coupon with the flat Al plate across an extended interface region without a clear demarcation left at the location of the original bonding surfaces. The average tensile bond strength is dominated by ductile fracture of the Al matrix within this extended interface region. The approximately uniform distribution of Ge precipitates, <4µm in size, within this extended interface region with width of ~100µm...
means that the area of Ge precipitates across any cross section is lower than that would be in the case where all Ge atoms are concentrated within a narrow interface region. This increases the area fraction of Al-Al bonds and consequently the average tensile strength. The observation of extended bonding interface region formation offers some metallurgical guidance for bonding of other metal-based HARMS.

It is then appropriate to discuss mechanisms through which such an extended bonding interface region is achieved. During the bonding process, the two solid Al pieces are in contact with a layer of Al-Ge eutectic liquid formed from the vapor-phase deposited Al-Ge intermediate layer thin films, with a total thickness of ~4µm. Because bonding occurred at ~500°C, above T_E = 424°C, liquid layer broadening by dissolution of solid Al into the Al-Ge eutectic liquid is considered first. The appropriate phase diagram coordinates to consider are the eutectic point, T_E = 424°C and Al_{70}Ge_{30}, and the liquidus coordinate in equilibrium with the Al solidus at the bonding temperature, T = 500°C and Al_{80}Ge_{20}, respectively.\(^{18}\) As a rough estimate, taking the densities of liquid Al and liquid Ge to be their respective solid values, \(\rho_{Al} = 3.27 \text{ g/cm}^3\) and \(\rho_{Ge} = 5.32 \text{ g/cm}^3\), assuming that the density of Al-Ge liquid obeys Vegard’s law, and ignoring the temperature dependence of densities, a simple calculation was made of the change in the liquid layer thickness due to additional Al incorporation into the Al-Ge eutectic liquid, as the liquid composition shifts from Al_{70}Ge_{30} to Al_{80}Ge_{20}. This calculation shows that, resulting from this liquidus composition shift, the Al-Ge liquid layer broadens from ~4 to less than 6µm.

The liquidus layer with composition Al_{80}Ge_{20} further reacts with solid Al to form an Al solidus with ~2 at.% Ge.\(^{29}\) A much wider Al solidus layer may result due to its lower Ge composition. For example, assuming that all Ge atoms within the original Al_{70}Ge_{30} eutectic liquid layer, ~4µm in thickness, get dispersed into a solidus layer of composition Al_{98}Ge_{2}, the same calculation as outlined in the previous paragraph leads to an estimated solidus layer width of ~50µm. Because of the absence of information on the partitioning of Ge atoms between the liquidus and solidus, the actual thickness of the Al solidus layer is unknown. Nonetheless, the above estimate illustrates the potential of the solidus reaction in dispersing Ge atoms to a much wider region around the original bonding interface. Additional cross-sectional metallographic observations around the bonding interface region showed Al grains spanning the entire bonding interface region, suggesting the possibility of epitaxial re-growth from un-melted Al grains.\(^{29}\)

Because the bonding temperature of 500°C is 83% of the melting temperature of Al, diffusion of Ge within solid Al also needs to be considered. Measurements of the dependence of Ge diffusion coefficient in Al on temperature and pressure by Thurer et al. gave

\[
D_{Ge}(T, p) = D_0 \exp\left(-\frac{Q + p\Delta V}{kT}\right),
\]

where \(D_0\), \(Q\), and \(\Delta V\) are respectively the diffusion pre-exponential factor, activation energy, and activation volume. The measured values of \(D_0\), \(Q\), and \(\Delta V\) are respectively \(3.39 \times 10^{-5} \text{ m}^2/\text{s}\), 1.24eV, and 1.2Ω where Ω denotes the atomic volume.\(^{30}\) Furthermore, Ge has been observed to be the dominant diffusing species in Al/Ge bilayer interdiffusion experiments.\(^{31}\) Taking \(p\) to be 10MPa, the magnitude of \(p\Delta V\) is about 1meV. Therefore it can be concluded that pressure effects on Ge diffusion in the present context of bonding should be negligible.

Ignoring the activation volume term in Eq. (1), \(D_{Ge}(T = 500^\circ C)\) is ~2.9×10^{-13} \text{ m}^2/\text{s}. As an estimate of the extent of Ge diffusional spreading, the Al liquidus and solidus reactions discussed above are ignored, and Ge diffusion into solid Al from locations of the vapor-deposited thin film on the original bonding surface and the microchannel sidewall surface is
considered. The extent of this diffusion can be estimated from available solutions to Fick’s diffusion law.\textsuperscript{32} The solution appropriate for these two situations is that for a strip source diffusing either into an infinite or a semi-infinite solid,

\[ \frac{C(x,t)}{C_0} = \frac{1}{2} \left\{ \text{erf} \left( \frac{h - x}{\sqrt{4Dt}} \right) + \text{erf} \left( \frac{h + x}{\sqrt{4Dt}} \right) \right\}. \quad (2) \]

In Eq. (2), \( C(x,t) \) is the concentration of the diffusing species at location \( x \) and time \( t \), and \( D \) is the diffusion coefficient. For the infinite solid case (diffusion from the original bonding surface), the initial distribution of the diffusing species is concentration \( C_0 \) at \(-h < x < +h\), and diffusion occurs in both \(+x\) and \(-x\) directions. For the semi-infinite solid case (diffusion from the microchannel sidewall surface), the initial distribution of the diffusing species is concentration \( C_0 \) at \( 0 < x < +h \), and diffusion occurs only in the \(+x\) direction. The value of \( h \) is taken to be \( 2\mu m \), the thickness of the original Al-Ge thin film layer. A characteristic diffusion time can be defined as \( h^2/D \), which in the present case of Ge diffusing into Al at \(~500^\circ C\) is \(~14s\). At this characteristic time, the maximum concentration of the diffusing species has decreased to \(~0.52\) of its initial value. At \( t = 40\text{min} \), \( Dt/h^2 \) is \(~174\) and the maximum concentration of the diffusing species has decreased to \(~0.043\) of its initial value. For the infinite solid case, the full-width-at-half-maximum of the corresponding concentration profile is \(~44h\), or \(88\mu m\), at \( t = 40\text{min} \). Accordingly, Ge diffusion within solid Al should also contribute significantly to the observed broadening of the bonding interface. Summarizing the discussion above, it is believed that the observed interfacial broadening results from a combination of liquidus and solidus reactions together with Ge diffusion within solid Al.

![Fig. 4.10 A SE image of a cross-sectional view of one bonded Al microchannel after FIB sectioning through the thin top Al layer. The arrow points to the location of the exposed microchannel corner.](image)

Solidification of the Al liquidus will lead to the formation of some Ge precipitates. On the other hand, Ge precipitates have been observed to form from solid state interdiffusion of Al/Ge bilayer thin films\textsuperscript{31} and from Al\textsubscript{98.4}Ge\textsubscript{1.6} alloys homogenized at 425\(^\circ C\) followed by quenching, through defect mediated processes.\textsuperscript{33} Details of Ge precipitate nucleation and growth during the bonding process have not been clarified, and remain a topic for future study.
The quality of bonding at the corner where the microchannel sidewall and the top flat plate meet is of concern. Failure to bond well at these corner locations may leave sharp crevices, which can act as initiators for subsequent mechanical failure due to the presence of stress concentration at these locations. Traditional metallographic polishing induces excessive deformation, which can mask the as-bonded morphology. The non-contact material removal by FIB is well suited to such examinations. Figure 4.10 shows a SE image of a cross-sectional view of one bonded Al microchannel after FIB sectioning through the thin top Al layer, exposing bonded microchannels without mechanical contact. Because the rate of material removal by the ion beam is low, only a small portion of the microchannel around one of its top corners is revealed. It is evident from Fig. 4.10 that the corner of the microchannel is well bonded, without extra crevices forming at the location of the original bonding surfaces. Observations such as those shown in Fig. 4.10 further attest to the quality of the Al-Ge thin film intermediate layer bonding of Al-based HARMS. It is also interesting to point out that several small holes exist close the microchannel surface. The origin of these holes is unclear at present, and needs to be investigated further.

4.4 Conclusion

The structure of sputter co-deposited Al-Ge thin films were studied by high-resolution TEM as a function of the film composition. It is shown, for the first time to our knowledge, that at least a portion of the Al atoms condense into a metastable Al-Ge amorphous phase. The process of Al-Ge phase separation at these locations proceeds via a compositional segregation, during which Ge and Al atoms from the amorphous mixture diffuse and attach respectively to crystalline Ge and Al grains. The structure of the interface between Al-based HARMS bonded to flat Al plates is studied by combining FIB sectioning with SEM. An extended bonding interface region, ~100µm in width, is observed and suggested to result from a combination of Al liquidus and solidus reactions and diffusion of Ge within solid Al at the bonding temperature. The extended bonding interface region is suggested to be beneficial to Al-Al bonding via Al-Ge intermediate layers, the formation of which offers some metallurgical guidance for bonding of other metal-based HARMS.

4.5 References


A. Bhushan, D. Yemane, E. B. Overton, J. Goettert, M. C. Murphy, Fabrication and preliminary results for LiGA fabricated nickel micro gas chromatograph columns, JMEMS 16(2), 383-393 (2007)


CHAPTER 5. EVALUATION OF BOND QUALITY AND ASSEMBLY OF Cu-BASED MICROFLUIDIC STRUCTURES WITH Al THIN FOIL INTERMEDIATE LAYERS *

5.1 Introduction

Technologies capable of improving heat transfer and energy efficiency can impact diverse market sectors. The nearly exponential growth in module-level heat flux is responsible for the intense current interest in advanced cooling technologies for microelectronics.¹ In 1981, Tuckerman and Pease demonstrated that decreasing liquid cooling channel dimensions to the micron scale would lead to orders of magnitude increases in heat transfer rates.² Since then, intense research on microchannel heat exchangers (MHEs) has ensued.³ Extensive studies of solid-fluid heat transfer in microchannel devices have documented their ability to provide high heat transfer coefficients,⁴ concentrating predominantly on Si-based microchannels.⁵, ⁶, ⁷

The choice of Si-based microchannels does not result from the fact that Si is the optimal material for cooling devices, but rather that fabrication technology for Si-based high-aspect-ratio microscale structures (HARMS) is the most mature and most widely available. Si possesses a lower bulk thermal conductivity than those of Cu and Al.⁸ In addition, the brittle nature of Si diminishes the mechanical robustness of Si-based MHEs and precludes their use in applications where mechanical loading/shock is a consideration.

At the present time, the lack of suitable fabrication techniques for metal-based MHEs has been the main bottleneck preventing serious contemplation of using them for various applications. High thermal conductivity metal-, such as Cu- and Al-, based MHEs promise on-par or better heat transfer performance as compared to Si-based devices, together with much increased mechanical robustness. Developing technologies capable of fabrication and assembly of Cu- and Al-based MHEs at low-cost and high-throughput will enable their application to a much wider range of market sectors.

Molding replication represents an important strategy toward efficient fabrication of metal-based HARMS. The LiGA microfabrication protocol provides one important example, combining deep X-ray/UV lithography (Lithographie) on polymeric resists, metal electrodeposition (Galvanoformung) into developed resist recesses to form primary HARMS, and replication (Abformung) of secondary HARMS from the primary HARMS (mold inserts).⁹ Primary HARMS made by lithography and electrodeposition (LiG) are expensive due to high cost of lithography and low speed of electrodeposition. The replication process produces HARMS in different engineering materials without repeating the lithography-development-deposition steps, and is thus an intrinsically low-cost and high-throughput fabrication technique. Since 2003, successful HARMS replication has been demonstrated in Pb, Zn, and Al with LiG-fabricated Ni inserts,¹⁰, ¹¹ and in Cu and Ni with inserts made of Ta or Inconel X750.¹², ¹³, ¹⁴, ¹⁵

Replicated metallic HARMS need to be bonded and assembled to form MHE devices. Different bonding methods have been reported for Si-based microsystems.¹⁶, ¹⁷, ¹⁸ In comparison, bonding of metal-based HARMS is much less studied. Recently, we have reported successful bonding of Al microchannels to other Al microchannels and Al flat plates through the use of Al-Ge eutectic thin film intermediate layers.¹⁹ Measured tensile strengths of these eutectic bonds exceeded 75MPa and reached as high as 165MPa. Multilayered Al microchannel arrays were successfully assembled.²⁰

* Reprinted by permission of “Journal of Vacuum Science & Technology A”
In this chapter, we report successful bonding of Cu-based HARMS. Bond strengths were measured and bonding interfaces were examined. Cu pieces containing replicated microchannels were assembled with other machined Cu pieces to form integrated MHE prototypes, on which preliminary heat transfer tests were conducted.

5.2 Experimental Procedures

5.2.1 Molding Replication of Cu-Based Microchannels

Cu 110 (99.9+wt.% Cu) coupons, ~35.5mm×~35.5mm square by ~6.4mm in thickness, were molded with an amorphous silicon nitride (a-Si:N) coated Inconel X750® insert. The insert consisted of an array of 19 parallel rectangular microprotrusions on the active area, with dimensions of ~15mm×~15mm. Compression molding was carried out on a MTS858 single-axis testing system interfaced to a high-vacuum chamber, which houses two heating stations. One heating station was mechanically attached to the bottom of the vacuum chamber. A second heating station was mechanically attached to the top linear actuator of the MTS858 system through a bellows-sealed motion feed-through. The two heating stations were heated separately by resistive heating cartridges and their temperatures were measured by two separate K-type thermocouples. Further details on the molding system have been reported in Chapter 2. The coupon to be molded was fastened to the top surface of the bottom heating station, while the insert was mechanically attached to the top surface of the heating station attached to the linear actuator. Total axial force on and axial displacement of the insert were measured continuously during the entire molding and demolding process. Molding of Cu coupons occurred with both the coupon and the insert heated to ~500°C. The molding process replicated patterns on the insert into an array of 19 parallel rectangular microchannels on the Cu coupon, with an average width of ~150µm and depth of >400µm. Further details on insert fabrication and molding replication of Cu-based HARMS have been reported elsewhere.12, 13, 14, 15, 19

5.2.2 Bonding of Cu Specimens with Al Thin Foil Intermediate Layers

Bonding experiments were also carried out on the MTS858 system interfaced to the vacuum chamber. Two rectangular Cu coupons (~25mm×~16mm×~16mm), with surfaces mechanically polished to less than 1µm in roughness, were bonded with one Al thin foil intermediate layer. The two Cu coupons to be bonded were placed face to face on the bottom heating station with a thin Al foil (Al1100, 99%+) inserted in the middle, forming an assembly of ~32mm in length along the axial direction perpendicular to the bonding interface. A small hole was drilled at the corner of the bottom Cu coupon close to the bonding interface, into which a K-type thermocouple was inserted to measure the interface temperature. After the chamber was evacuated, both heating stations were heated. The top heating station was close to the top surface of the Cu coupon assembly but not in contact with it. After the bottom and top heating stations reached 500°C, the upper heating station was then placed in contact with the Cu coupon assembly using the linear actuator. A linearly increasing compression force was applied to the Cu coupon assembly such that loading force levels of 425, 850, and 1700N were reached after a constant duration of 10min. These compressive loading forces correspond to average applied pressures of ~1, ~2, and ~4MPa, respectively. A constant force hold was executed after the compression force reached the desired level. During the compression force increase, the temperatures of the bottom and top heaters were raised, leading to further temperature increase.
of the bonding interface. The interface temperature was recorded continuously during the constant force hold, which was executed for 12 min after the interface temperature reached ~550°C, corresponding to the Al-Cu eutectic temperature of 548°C. During this 12 min hold, the interface temperature increased further and reached steady state. This steady state temperature, as measured by the K-type thermocouple, was taken as the nominal bonding temperature. After this 12 min hold, the heaters were turned off with the same force still applied. After the temperature of the bonding interface decreased to <450°C, the linear actuator was withdrawn from the top surface of the Cu assembly and the system was cooled down to room temperature. In order to take advantage of the Al-Cu eutectic, the bonding temperatures were chosen to be above 550°C. The bonding temperatures were also chosen to be below 600°C, in order to avoid bulk melting of the Al foil at 660°C.

5.2.3 Evaluation of Bond Quality

A series of Cu two-coupon assemblies were bonded at different applied pressures and with different thicknesses of Al foils. Using a Struers Accutom5 precision cutting machine, the four outermost sections along the axial direction, ~2-3 mm in thickness, were removed from the bonded Cu specimen. From the remaining specimen of ~20 mm×~12 mm×~32 mm in dimension, four tensile testing specimens were obtained by performing two perpendicular bisecting cuts parallel to the axial direction, resulting in a final tensile specimen dimension of ~10 mm×~6 mm×~32 mm. After cutting, the four surfaces of the tensile specimen parallel to the axial direction were mechanically polished with 600-grit silicon carbide papers to remove possible damages. Tensile testing along the specimen axial direction was performed to evaluate the tensile strength of the bonding interface, and was carried out on a MTS810 system with hydraulic grips. Examination of fractured bonding surfaces was conducted on a Hitachi S3600N scanning electron microscope (SEM) equipped with an EDAX X-ray energy dispersive spectroscopy (EDS) system. X-ray diffraction (XRD) patterns from fractured bonding surfaces were collected on a Rigaku MiniFlex X-ray diffractometer using Cu Kα radiation.

5.2.4 Assembly of Cu-Based Microchannel Devices and MHE Prototypes

A process similar to that described above for bonding two rectangular Cu coupons was employed to assemble Cu-based two-layer MHE prototypes and single-layer microchannel devices. To form MHE prototypes, Cu blocks containing both holes for accommodating cylindrical cartridge heaters and holes/plena for fluidic connections were designed and fabricated with conventional machining. Arrays of parallel microchannels were replicated into Cu coupons by molding. Surfaces of Cu blocks and coupons were polished with 1200-grit silicon carbide papers prior to bonding. The MHE assembly consisted of one such Cu block placed between two Cu coupons, each containing a parallel array of replicated microchannels. One 10 μm-thick Al foil was inserted in between each coupon/block interface. The entire coupon/block/coupon assembly was placed on top of the bottom heating station. After evacuation, both heating stations were heated to above 500°C, and the upper heating station was then put into contact with the assembly. An increasing compression force was applied to the assembly at a constant loading rate of 300 N/min. A constant force hold was executed for 15 min after the compression force reached the desired level of ~3000 N, corresponding to an average applied pressure of ~3 MPa. After commencing the constant force hold, the assembly temperature further increased from ~560°C to ~580°C over 5 min. The final bonding conditions for the coupon/block/coupon
assemblies are therefore ~580°C in temperature, ~3MPa in applied pressure, and ~10min in duration, respectively. After the constant force hold, the linear actuator was withdrawn from the assembly and the system was cooled down.

To form single-layer microchannel devices, one Cu coupon was used, which contained a parallel array of replicated microchannels. Two fluidic plena connecting to the two ends of the parallel microchannel array were fabricated into the Cu coupon by micro electrical discharge machining (EDM). This Cu coupon, containing both the microchannel array and the fluidic plena, and one blank Cu coupon were placed face to face on the bottom heating station, with a 10µm thick Al foil inserted in the middle, and bonded at ~580°C and an applied pressure of ~3MPa.

### 5.2.5 Thermal Testing of Cu-Based MHE Prototypes

A testing apparatus was designed and built to evaluate the heat transfer characteristics of assembled Cu MHE prototypes. The apparatus consisted of three sections: water supply section, test section, and data acquisition section. The water supply section consisted of a pressure-regulated water storage tank, which supplied water to the specimen at a constant pressure and provided a smooth and stable flow through the microchannels at low flow rates. A valve placed downstream of the tank exit was used to make fine adjustments to the flow rate. For data acquisition, an Instrunet data acquisition system interfaced to a PC was used to collect thermocouple readings. Further details on this experimental setup have been reported elsewhere.²²

### 5.3 Results and Discussion

Tensile testing was performed on bonded Cu two-coupon assemblies. During tensile testing, the two ends of the specimen were gripped by the hydraulic grips. The gripped sections were ~10mm in length, leaving an un-gripped section of ~12mm in length with the bonding interface in the middle. As the specimen extension increases, the tensile stress increases to a maximum, after which breakage occurred at the bonding interface leading to a quick drop in stress. The maximum tensile stress observed on the stress-extension curve yields a measurement of the tensile bond strength.

Figure 5.1 shows measured bond strength as a function of the applied bonding pressure. For this series of measurements, all bonding runs were performed at ~580°C and an Al foil thickness of ~25µm. In all cases, clean breaks occurred at the bonding interface. At the lowest pressure of ~1MPa, measured bond strength ranges from 33 to 45MPa. No significant difference can be discerned from tests performed on specimens bonded at pressures of ~2 or ~4MPa. The maximum strength measured reached 48MPa. Figure 5.2 shows measured bond strength as a function of the thickness of Al foil at the bonding interface. For this series of measurements, all bonding runs were performed at ~580°C and an applied pressure of ~4MPa. At the lowest Al foil thickness of ~10µm, measured bond strength ranges from 39 to 52MPa. Again, no gross difference can be discerned from tests performed on specimens bonded with Al foils with thicknesses of ~25 or ~38µm. Measured bond strength values ranges from 33 to 46MPa. Data shown in Figs. 5.1 and 5.2 indicate little variation in bond quality within the range of applied pressure and Al foil thickness tested. As stated above, our desire to take advantage of the Al-Cu eutectic and to avoid bulk melting of the Al foil limits the bonding temperature within the range of 550-600°C. Within this small temperature range, the bonding temperature is believed not to have a major influence on the bond strength.
Fig. 5.1 Tensile bond strength as a function of the average applied pressure.

Fig. 5.2 Tensile bond strength as a function of the thickness of the Al foil intermediate layer.

Structural, morphological, and chemical characterizations of fracture surfaces of bonded Cu coupon specimens were conducted to elucidate bonding mechanisms. Figure 5.3 shows three XRD patterns obtained from fracture surfaces bonded respectively at Al foil thicknesses of 10, 25, and 38µm. Major diffraction peaks within all three patterns can be indexed to belong to either a fcc Cu phase or a fcc Al phase, indicating their presence in the interface region after bonding. The lattice parameters for the Cu phase are 3.663Å, 3.658Å, and 3.667Å at Al foil thicknesses of 10, 25, and 38µm, respectively. In comparison, the lattice parameter of the starting Cu coupon is measured at 3.618Å. The corresponding lattice parameters for the Al phase are respectively 4.102Å, 4.103Å, and 4.111Å. In comparison, the lattice parameter of elemental Al is 4.05Å. The respective lattice parameter increases over those of elemental Cu and Al suggest possible dissolution of Al into Cu and vice versa in the interface region. Besides those indexed to
fcc Cu and fcc Al phases, other diffraction peaks remain in all three XRD patterns, indicating the formation of additional Al-Cu compounds within the interface region during bonding. According to the Al-Cu phase diagram,\textsuperscript{21} a single eutectic exists between the fcc Al phase and the $\theta$-Al$_2$Cu phase. If bonding occurred solely via the eutectic mechanism, only the $\theta$-Al$_2$Cu phase would be expected to be present in the interface region. Not all the additional diffraction peaks present within the three XRD patterns can be indexed to $\theta$-Al$_2$Cu, thus indicating the presence of Al-Cu compounds other than $\theta$-Al$_2$Cu in the interface region and suggesting that bonding may have occurred via a combination of eutectic and diffusional mechanisms.

![X-ray diffraction patterns](image)

Fig. 5.3 Three X-ray diffraction patterns obtained respectively from three fractured interfaces bonded with Al intermediate layers 10, 25, and 38$\mu$m in thickness.

![SEM micrographs and X-ray maps](image)

Fig. 5.4 Morphological and chemical characterizations of a typical fractured interface bonded with a 25$\mu$m-thick Al foil: (a) an SEM micrograph; (b) the corresponding Al-K X-ray map; (c) the corresponding Cu-L X-ray map. Identical magnifications apply to (a), (b), and (c).

The morphology and chemical makeup of the fractured surface of one specimen bonded with 25$\mu$m-thick Al foils were examined through SEM and EDS mapping. A typical morphology is shown in Fig. 5.4(a). Some regions bear resemblance to remnants of the Al foil, e.g., that labeled as region A. Other regions appear clean from these remnants, e.g., that labeled as region B.
B. Such observed morphology suggests that the original Al foil region at the bonding interface was torn during the fracture process and attached randomly to one of the two fracture surfaces. The distribution of Al and Cu within the fracture surface area corresponding to Fig. 5.4(a) is probed by the Al-K and Cu-L X-ray intensity maps, shown respectively in Figs. 5.4(b) and 5.4(c). Dark areas without either Al-K or Cu-L intensities are visible in both Figs. 5.4(b) and 5.4(c). This is because some parts of the remaining Al foil region bent upward after the fracture process and blocked the electron beam during the mapping scans. Figure 5.4(c) shows an apparently uniform Cu-L intensity from the entire area and little contrast between regions A and B. Figure 5.4(b) shows that the Al-K intensity level in region A appears higher than that in region B, indicating a higher Al content in region A. Data shown in Figs. 5.4(b) and 5.4(c) are therefore consistent with the interpretation that the original Al foil region at the bonding interface was retained in region A on the fracture surface during the fracture process. The presence of Al-K intensity in region B suggests that Al diffused into the Cu bulk during bonding.

A cross-sectional view of a typical bonded interface, after mechanical polishing with 1200-grit SiC papers, is shown in Fig. 5.5(a). The interface region is visible with a distinct contrast. The corresponding Al-K X-ray intensity map is shown in Fig. 5.5(b). Data in Fig. 5.5(b) clearly show the localization of Al to the interface region. Analogous Cu-L map shows the presence of Cu throughout the same interface region. The thickness of this interface region is >20µm, and significantly exceeds the initial Al-foil thickness of 10µm. Thus, data shown in Figs. 5.4 and 5.5 are consistent with the occurrence of Al-Cu interdiffusion during bonding, and suggest that bonding occurred via a combination of eutectic and diffusional mechanisms. More detailed microstructural examinations of the bonding interface region await further study.

![Fig. 5.5 Cross-sectional characterization of a typical fractured interface bonded with a 10µm-thick Al foil: (a) an SEM micrograph; (b) the corresponding Al-K X-ray map. Identical magnifications apply to (a) and (b).](image)

One Cu coupon, containing replicated microchannels and two electrical discharge machined fluidic plena, and another blank Cu coupon, with the same overall dimensions of 35.5mm×35.5mm×6.4mm, were bonded together to form a single-layer microchannel device. The one Cu coupon contains a set of 19 parallel rectangular microchannels, ~15mm in total length, ~150µm in width, and ~400µm in depth. A 10µm-thick Al foil was inserted in between the two Cu coupons, and bonding occurred at ~580°C and an applied pressure of ~3MPa to form one array of enclosed microchannels. A cross section of the bonded structure was obtained by mechanical cutting in one of the fluidic plena, perpendicular to the microchannel array. The cut structure was mechanically polished back to close to the microchannel exit. Figure 5.6 shows
such a cross-sectional view of the microchannel exit area. The upper straight line in Fig. 5.6 shows the location of the original Al foil. The rippled line below the microchannels delineates the bottom of the fluidic plenum, which due to the electrical discharge machining process is not flat. Closer examination of the bonded structure showed no evidence of deformation of the Cu structure.

Fig. 5.6 A cross-sectional SEM micrograph of a portion of an array of 19 bonded Cu microchannels.

Fig. 5.7 Assembly of Cu-based MHE prototype: (a) an optical image of two Cu coupons with replicated microchannel arrays and one Cu heater block. Holes A and B are respectively internal and external water connections. Holes C house cylindrical cartridge heaters; (b) an optical image of the assembled Cu MHE prototype with external fluid connectors, four heaters, and one extra heater on the outside. Numbers on the rulers in (a) and (b) are in mm.

Two Cu coupons, each containing one set of parallel rectangular microchannels ~15mm in length, ~150µm in width, ~400µm in depth, and covering a total area of ~15mm×15mm, are shown in Fig. 5.7(a) together with one specially-designed Cu heater block, with overall dimensions of 43mm×43mm×15mm. On the top heater block surface visible in Fig. 5.7(a), two plena were machined to connect to the two ends of the microchannel array on the Cu coupon. The distance between two plena is ~12.8mm. Identical arrangements exist on the opposing bottom heater block surface, not visible in Fig. 5.7(a). One hole within each plenum, ~7.5mm in
diameter and labeled as A in Fig. 5.7(a), was machined through the entire heater block to connect the two opposing plena on the top and bottom heater block surfaces. One additional hole on each side of the heater block was machined perpendicularly to hole A, labeled as B in Fig. 5.7(a), and formed the external water connections. Four parallel holes, labeled as C in Fig. 5.7(a), with diameter of ~6.4mm and length of ~41mm, were drilled parallel to hole B and used to house four cylindrical cartridge heaters. A 10µm-thick Al foil was inserted in between each Cu coupon/heater block interface. The three-piece coupon/heater block/coupon assembly was bonded at ~580°C and an applied pressure of ~3MPa to yield one Cu MHE prototype containing two parallel layers of microchannels connected to one single inlet and exit connection. Figure 5.7(b) shows an overview of this Cu MHE prototype assembled with external fluid connections and four cylindrical cartridge heaters, with 6.4mm in diameter and ~180W of maximum power each, inserted into the heater block. The entire prototype volume is dominated by that of the heater block.

![Fig. 5.8 Water flow rate through the Cu MHE prototype as a function of the associated pressure drop.](image)

Water flow through the assembled Cu MHE prototype and heat transfer from the cartridge heaters to water was tested. The total pressure drop across the inlet and exit fluid connections was measured with a Dywer digital manometer with a minimum reading of 690Pa (0.1psi). Figure 5.8 shows the rates of water flow through the microchannel arrays in the Cu MHE prototype as a function of the associated pressure drop, measured at a water temperature of 28±1°C. The flow rate increases monotonously with increasing pressure drop across the MHE, and reaches >1.5 liter/min at a pressure drop of ~0.48MPa (70psi).

To test the heat transfer performance of the Cu MHE prototype, the four cylindrical cartridge heaters were used to provide the heat source. Two thermocouples were inserted respectively into the inlet and outlet tubes with the help of T-fittings and then sealed with epoxy. Additional thermocouples were placed on the top and bottom surface of the MHE, as well as the side surface close to the heaters. The entire assembly was then encased within PVC insulation, with holes drilled into the PVC to allow for the fluid inlet, outlet, and pressure meter tube connections. Figure 5.9 shows the outlet-inlet water temperature difference as a function of water pressure drop across the MHE. Data were taken at two different levels of total heater input power.
At the higher input power of ~710W, the water temperature increases >7°C at the flow rate of ~1.4liter/min and >25°C at the flow rate of ~0.4liter/min. The efficiency of heat transfer, as defined by the ratio of power gained by water to total heater input power, is shown in Fig. 5.10 as a function of the water flow rate. The efficiency increases with increasing flow rate and reaches a maximum value of 99.7%. At higher flow rates, the lower MHE body temperature results in reduced heat loss to the PVC insulation and therefore higher overall heat transfer efficiency. The lowest efficiency measured exceeds 97.5%.
5.4 Conclusion

Successful bonding of Cu 110 coupons, without and with microscale features, is achieved with thin Al foil intermediate layers. Measurements of tensile bond strength were conducted as a function of various bonding parameters. At all bonding conditions tested, measured strength across the bonding interface, ~25mm×~16mm in dimension, is at least 33MPa and can reach as high as 52MPa. Morphological and chemical characterizations of fractured bonding interfaces suggest that bonding occurred via a combination of eutectic and diffusional mechanisms. Cu-based, one-layer microchannels and two-layer MHE prototypes were successfully assembled. High overall heat transfer efficiency was demonstrated on such Cu-based MHE prototypes.

5.5 References


CHAPTER 6.  BONDING OF Cu-BASED HIGH ASPECT RATIO MICROSCALE STRUCTURES WITH Sn INTERMEDIATE LAYERS *

6.1 Introduction

Technologies for efficient removal of high heat flux are useful in diverse applications, e.g., cooling of next-generation microelectronic devices and solid-state lighting modules. High rates of heat transfer between fluid flowing inside microchannels and the solid body of the microchannel device were demonstrated in the early 1980s.\(^1\) Since then, microchannel heat exchangers (MHEs) have become a leading contender for solving high heat flux removal problems.\(^2\) Although most experimental heat transfer investigations have been conducted on Si-based microchannel devices,\(^3\) metal-based MHEs, with higher thermal conductivities and increased mechanical robustness, offer potential performance improvements over Si-based counterparts.\(^4\) Elemental Cu, with room-temperature thermal conductivity lower than only diamond and Ag, is one of the most important materials for heat exchange applications.

Efficient fabrication techniques for metal-based HARMSs are critical for transitioning metal-based microdevices from laboratory curiosities to practical commodities. Serial subtractive techniques, including micromilling,\(^5\) micro electrical discharge machining (\(\mu\)EDM),\(^6\) and laser-beam direct writing\(^7\) have been used to fabricate metal-based HARMS. These techniques tend to be time consuming, and may suffer from tool wear and limitations when fabricating closely spaced features.

Replication of microscale parts by compression molding represents an important strategy for efficient fabrication of metal-based HARMSs. From a HARMS mold insert, secondary HARMSs are replicated quickly and in parallel.\(^8\) We have demonstrated successful HARMS replication by compression molding in Pb,\(^9\) Zn,\(^10\) Al,\(^11\) Cu,\(^12\) Ni,\(^13\) and NiTi.\(^14\) Molding replication of Cu-based HARMSs, in particular, paves the way for economical manufacturing of Cu-based MHEs.

To form any functional Cu-based MHEs from replicated Cu HARMSs, techniques for simple and efficient bonding and assembly are critical. Current research on assembly and packaging technologies are primarily focused on Si-based microdevices,\(^15\) with much less attention given to bonding of metal-based HARMSs. Bonding of metal parts at elevated temperatures using chemically/structurally distinct intermediate layer(s) at the bonding interface can lower the bonding temperature, eliminate bulk melting of the parts to be joined, and increase the bond quality. Examples of bonding using eutectic intermediate layers include Si bonding with the Au-Si eutectic\(^16\) and Al bonding with the Al-Ge eutectic.\(^17,\)\(^18,\)\(^19\) We have previously explored bonding strategies for Cu-based HARMSs. Cu-based MHEs have been successfully assembled through intermediate layer bonding with vapor-phase deposited Al-Ge thin films\(^20\) and thin foils of pure Al.\(^21\) The desire to use non-vacuum-based techniques and lower bonding temperatures supplied the motivation for searching of additional protocols for bonding Cu-based HARMSs.

In this chapter, we report results of Cu-to-Cu bonding with thin intermediate layers of elemental Sn. Bonding quality is assessed through quantitative evaluation of tensile bond strength as a function of bonding temperature and applied pressure, and rationalized through structural and chemical examination of tensile fracture interfaces. Structural examination of the Cu/Sn/Cu interface region is accomplished by combining focused ion beam (FIB) sectioning and

* Reprinted by permission of “Microsystem Technologies”
imaging with ion-induced secondary electrons (IISEs). Successful bonding of Cu-based HARMSs to flat Cu plates with Sn intermediate layers is demonstrated, leading to assembly of all-Cu MHE prototypes.

6.2 Experimental Procedures

6.2.1 Bonding of Cu Specimens with Sn Thin Foil Intermediate Layers

Bonding experiments were carried out on a MTS858 single-axis testing system interfaced to a high-vacuum chamber, which houses two heating stations. One heating station was mechanically attached to the bottom of the vacuum chamber. A second heating station was mechanically attached to the top linear actuator of the MTS858 system through a bellow-sealed motion feed-through. The two heating stations were heated separately by resistive heating cartridges and their temperatures were measured by two separate K-type thermocouples. The two Cu coupons (99.9+wt.% Cu), ~25mm×~16mm×~16mm in dimension, with surfaces mechanically polished to less than 1µm in roughness, were placed face to face on the bottom heating station with a thin elemental Sn foil (99.9%+wt.% Sn, ~25 µm in thickness) inserted in the middle, forming an assembly of ~32mm in length along the axial direction perpendicular to the bonding interface. After the chamber was evacuated, both heating stations were heated. The top heating station was close to the top surface of the Cu coupon assembly but not in contact with it. After the bottom and top heating stations reached respectively the target temperatures of 290, 360, and 450°C, the upper heating station was then placed in contact with the Cu coupon assembly using the linear actuator. A linearly increasing compression force was applied to the Cu coupon assembly with a constant loading rate of ~300N/min. A constant force hold was executed for 5min after the compression force reached the desired levels of 105, 850, 1700, 2550, and 3400N. These compressive loading forces correspond to average applied pressures of ~0.25, ~2, ~4, ~6, and ~8MPa, respectively. After this 5min hold, the heaters were turned off with the same force still applied. After the temperature of the heating stations decreased to <200°C, the linear actuator was withdrawn from the top surface of the bonded assembly and the system was cooled down to room temperature. The temperature of the bonding interface was taken to be the average of the heat station thermocouple readings.

6.2.2 Evaluation of Bond Quality

A series of Cu coupon-coupon assemblies were bonded at different temperatures and different applied pressures. Using a Struers Accutom5 precision cutting machine, the four outermost sections along the axial direction, ~2-3mm in thickness, were removed from the bonded Cu specimen. From the remaining specimen of ~20mm×~12mm×~32mm in dimension, three tensile testing specimens were obtained by performing two cuts parallel to the axial direction. After cutting, mechanical drilling was used to make two through holes, ~5mm in diameter, close to the two ends of each tensile specimen. The two holes were used to hold the specimen by two custom-built specimen holding blocks designed for tensile testing. After drilling, the four surfaces of the tensile specimen parallel to the axial direction were mechanically polished with 600-grit silicon carbide papers to remove possible damages, resulting in a final specimen dimension of ~10mm×~6mm×~32mm. Tensile testing along the specimen axial direction was performed until specimen fracture to evaluate the average tensile strength of the bonding interface, and was carried out on a MTS810 system with hydraulic grips.
Examination of tensile fracture surfaces was conducted on a Hitachi S3600N scanning electron microscope (SEM) equipped with an EDAX X-ray energy dispersive spectroscopy (EDS) system. In addition to conventional SEM imaging with electron-induced secondary electrons (EISEs), intensity maps of electron-induced characteristic X-rays were used to examine the chemistry of tensile fracture surfaces. Further characterization of the interface region of Cu coupon-coupon assemblies bonded with elemental Sn intermediate layers was carried out on a FEI Quanta 3D FEG Dual-Beam™ FIB instrument. Surfaces of as-bonded specimens were mechanically polished with SiC abrasive papers and finished with 1µm diamond particle suspension. Polished surfaces containing the bonding interface region were lightly etched with a 30kV Ga⁺ ion beam by imaging the surface at normal incidence for a short time. Higher resolution examination of the interface region was carried out through a cross-sectional cut perpendicular to the polished surface using 30kV Ga⁺ ion beam. Ion channeling contrast from differently oriented polycrystals was obtained from IISE images.22

6.2.3 Bonding of Cu-Based Microchannel Device Using Sn Thin Foil Intermediate Layer

Cu coupons, ~35.5mm×~35.5mm square by ~6.4mm in thickness, were molded with an amorphous silicon nitride (a-Si:N) coated Inconel X750® insert. The mold insert consisted of an array of 19 parallel rectangular microprotrusions on the active surface, with dimensions of ~15mm×~15mm. Compression molding was carried out on the MTS858 system interfaced to the vacuum chamber. The coupon to be molded was fastened to the top surface of the bottom heating station, while the insert was mechanically attached to the top surface of the heating station attached to the linear actuator. Total axial force on and axial displacement of the insert were measured continuously during the entire molding and demolding process. Molding of Cu coupons occurred with both the coupon and the insert heated to ~500°C. The molding process replicated the parallel rectangular microprotrusions on the insert into an array of 19 parallel rectangular microchannels on the Cu coupon, with an average width of ~150µm and depth of >400µm. Further details on insert fabrication and molding replication of Cu-based HARMSs have been reported elsewhere.11, 13, 14

A process similar to that described above for bonding Cu coupon-coupon assemblies was employed to bond Cu-based, single-layer, microchannel devices. One Cu coupon, which contained a parallel array of replicated microchannels, and one blank Cu plate were bonded together. Two fluidic plena connecting to the two ends of the parallel microchannel array were cut into the first Cu coupon by μ-EDM. Correspondingly, two fluidic plena were also cut into the blank Cu coupon. Surfaces of both coupons were polished with 1200-grit silicon carbide papers prior to bonding. One elemental Sn foil was inserted in the middle of two coupons. The entire coupon/foil/coupon assembly was placed on top of the bottom heating station. After evacuation, both heating stations were heated to the desired temperature, and the upper heating station was then put into contact with the assembly. An increasing compression force was applied to the assembly at a constant loading rate of 300N/min. Bonding occurred during a constant force hold, executed for 5min, after the compression force reached the desired level.

6.3 Results and Discussion

Figure 6.1(a) shows the two custom-built specimen holding blocks designed for evaluating the average tensile bond strengths of Cu coupon-coupon assemblies. A screw passes through two opposing holes on one C-shaped steel holding block, as well as the bonded coupon-
coupon assembly through the hole drilled into one end of it. The diameter of the screw is less than the inner diameter of the hole on the specimen, ensuring that the specimen receives no sideways loading. The end of the screw is fixed by a nut. During tensile testing, the two C-shaped holding blocks were held in a facing pair of hydraulic grips. In Fig. 6.1(a), an as-bonded Cu coupon-coupon assembly is shown to be held in between the two steel holding blocks. As-bonded coupon-coupon assemblies were pulled in tension until fracture occurred from the original bonding interface region, one example of which is also shown in Fig. 6.1(a). The result of a typical tensile test is shown in Fig. 6.1(b). The total tensile force was converted into a tensile stress by normalizing with the cross-sectional area of the original bonding interface. Because diameters of the holding screws are less than the inner diameters of the holes on the specimen, initial tensile extension produced no force. Figure 6.1(b) shows that, once the holding screws are engaged, the tensile stress increases monotonically until fracture occurs abruptly at the original bonding interface region. The maximum tensile stress observed is taken as the average tensile bonding strength.

A series of bonding runs were executed, with the same elemental Sn foil thickness of ~25µm while varying other bonding conditions. Figure 6.2 shows measured average tensile bonding strength as a function of the bonding temperature, with the applied pressure fixed at ~4MPa. Figure 6.2 shows tight clustering of average tensile bonding strength values at ~370°C and ~450°C, while significant scatter in strength values, 10-25MPa, is observed at ~295°C. The tensile bonding strength exhibits a moderate increase with increasing bonding temperature, and is ~30MPa at a bonding temperature of ~450°C. Figure 6.3 shows measured average tensile bonding strength as a function of the applied pressure, with the bonding temperature fixed at ~370°C. Figure 6.3 shows that the average tensile bonding strength increases significantly as the applied pressure increases from ~0.25MPa to ~4MPa. At applied pressures exceeding 4MPa, the average tensile bonding strength shows little further increase. Figure 6.3 shows a maximum tensile bonding strength value of ~30MPa, achieved at applied pressures of 6-8MPa.

Fig. 6.1 Tensile testing of bonded Cu coupon-coupon assemblies: (a) images of one typical as-prepared specimen with two custom-built specimen holding blocks and one specimen tested to fracture; (b) a typical tensile testing data set.
Examination of the tensile fracture surfaces, exemplified by images shown in Fig. 6.4, provides clues to observed variations in the average tensile bonding strength. Figure 6.4(a) shows a typical EISE or SEM image of the tensile fracture surface of a Cu coupon-coupon assembly bonded at ~370°C with an applied pressure of ~4MPa. The fracture surface presents two kinds of intertwined regions: one with a brighter contrast and another with a darker contrast. Figures 6.4(b) and 6.4(c) show respectively Sn-L and Cu-K X-ray intensity maps corresponding to Fig. 6.4(a). Regions with the brighter SEM contrast yield higher Sn-L and lower Cu-K X-ray counts, and are richer in Sn at the fracture surface. Regions with the darker SEM contrast yield lower Sn-L and higher Cu-K X-ray counts, and are richer in Cu at the fracture surface. Figures 6.5(a) and 6.5(b) show typical SEM images of tensile fracture surfaces from two Cu coupon-
Fig. 6.4 Morphological and chemical characterizations of a typical fractured interface bonded with a 25µm-thick Sn foil: (a) an SEM image; (b) the corresponding Sn-L X-ray map; (c) the corresponding Cu-K X-ray map. Identical magnifications apply to (a), (b), and (c).

Fig. 6.5 Typical SEM images of tensile fracture surfaces from two Cu coupon-coupon assemblies bonded at ~370°C with applied pressures of (a) ~4MPa; (b) ~0.25MPa.

Fig. 6.6 High-magnification views of Sn-rich regions on fracture surfaces bonded at (a) ~4MPa; (b) ~0.25MPa.

coupon assemblies bonded at applied pressures of ~4 and ~0.25MPa, respectively. In both cases, bonding was conducted at ~370°C. At the higher bonding pressure of ~4MPa, the Sn-rich regions
cover the fracture surface more uniformly. At the lower bonding pressure of ~0.25MPa, Cu-rich regions cover large contiguous areas on the fracture surface, with apparently little Sn interaction with them. Further morphological differences between the Sn-rich regions on fracture surfaces bonded at ~4 and ~0.25MPa are shown in Figs. 6.6(a) and 6.6(b). At the higher bonding pressure of ~4MPa, a mottled appearance is observed from the fractured Sn-rich regions. Micron and submicron sized nodules and holes are apparent on the fracture surface, suggesting that some plastic deformation took place during fracture. In contrast, the fractured Sn-rich regions at the lower bonding pressure of ~0.25MPa presents large Sn grains with smooth top surfaces, indicating clean separation with little plastic deformation induced.

Data presented in Figs. 6.4, 6.5, and 6.6 suggest that the average tensile bonding strength depends on interaction between the Sn intermediate layer and surfaces of the mating Cu coupons. Metallurgical interactions occurred across only a part of the interface region. Fracture across such regions gives rise to the observed Sn-rich fracture regions with mottled surface morphologies and indications of fracture-induced plastic deformation. Across the other part of the interface region, little metallurgical interaction between Sn and Cu took place. Fracture across such regions leaves either a Cu-rich surface, with Sn attached to the fractured counterpart, or a smooth top surface on Sn grains, with little plastic deformation. At a fixed bonding temperature, the application of increasing bonding pressure appears to promote the metallurgical interaction between the Sn intermediate layer and surfaces of the Cu coupons, thereby increasing the average tensile bond strength. At a fixed applied pressure, increasing the bonding temperature has apparently similar effects.

Fig. 6.7 A typical IISE image of the bonded interface region after cross-sectional cutting by FIB.

Further examination of the bonding interface region was carried out through FIB. One as-bonded Cu coupon-coupon assembly, bonded at ~375°C under an applied pressure of ~4MPa was mechanically polished on one surface. A cross-sectional cut, perpendicular to the polished surface, was made into the specimen with 30kV Ga⁺ ion beam. This cut, made with a 65nA ion current and of ~50µm in length and ~20µm in depth, straddles the bonding interface region. To
protect the original specimen surface from ion beam damage, a thin Pt strip was overlaid via ionbeam induced deposition of a gaseous, Pt-containing, metalorganic precursor prior to Ga⁺ sectioning. Fine polishing of the rough-cut cross section was accomplished with 30kV Ga⁺ beams of progressively reduced currents. Figure 6.7 shows a typical IISE image, showing the mechanically polished specimen top surface, the remnant of the thin Pt strip, and the polished ion beam section into the top surface. On the ion beam section, the IISE image exhibits clear ion channeling contrast, due to differences in grain orientation with respect to the incident Ga⁺ beam. Figure 6.7 clearly shows the large polycrystalline Cu grains, with grain size exceeding 10µm, located on both sides of the bonding interface region. An elemental or compound Sn layer, ~10µm in total thickness, is located at the bonding interface. Within the layer, individual grains, ~5µm in size, are clearly visible. Much smaller grains, with sizes of ~2µm or less, are seen from Fig. 6.7 to be located at the interfaces between this layer and elemental Cu (denoted e.g., by the solid arrow in Fig. 6.7). These smaller grains may be additional Cu-Sn compound(s) formed during bonding. Figure 6.7 also reveals that this formation does not appear to be occurring uniformly across the entire interface, as exemplified by the region highlighted by the broken arrow in Fig. 6.7. Such a lack of interfacial reaction may result from an unbroken surface oxide layer. Thus Fig. 6.7 shows that the exact nature of tensile fracture surfaces of Sn intermediate layer bonded Cu coupon-coupon assemblies may be somewhat complex: the fracture may occur from within the elemental or compound Sn layer, or at various interfaces delineated in Fig. 6.7. The chemistry and morphology of such fracture failures remain to be characterized in detail and correlated to bonding conditions.

Fig. 6.8 Cross-sectional SEM images of a bonded Cu microchannel structure: (a) a cross-sectional overview of a portion of the bonded microchannel structure; (b) a higher magnification view of one typical bonded microchannel.

Figure 6.8(a) shows a portion of a typical Sn intermediate layer bonded, enclosed, all-Cu, microchannel structure. The structure, consisting of a flat Cu plate and a mating Cu coupon with an array of parallel microchannels replicated onto its surface, was bonded at ~370°C under an applied pressure of ~4MPa. Some excess Sn had flown out of the bonding interface region and are visible on the side of the Cu plate. Each microchannel remains unobstructed, as shown in Fig. 6.8(b). Such all-Cu, enclosed, microchannel structures can serve as building blocks for metal-based microchannel fluidic devices, such as MHEs.⁴, ¹⁰
Fig. 6.9 An assembled all-Cu microfluidic prototype: (a) a pre-bonded, all-Cu, enclosed microchannel structure together with two Cu fluidic adaptor pieces; (b) overview of the assembled prototype, with stainless steel fluidic connectors attached to the Cu adaptors.

Sn intermediate layer bonding can be utilized to achieve bonding in non-planar configurations. Figure 6.9(a) shows three parts to be assembled: a pre-bonded, all-Cu, enclosed microchannel structure together with two Cu fluidic adaptor pieces. A Sn wire, 0.8mm in diameter, was laid within each mating hole on the adaptor piece. The three parts were then assembled mechanically, and bonded at ~400°C under an applied force of 100N. Figure 6.9(b) shows the assembled final prototype, with stainless steel fluidic connectors attached to the Cu adaptors. Such all-metal microchannel assemblies can endure high internal pressures, and are potentially useful in a range of applications.

6.4 Conclusion

Bonding of Cu-based HARMSs with thin intermediate layers of elemental Sn was accomplished. The quality of resulting bonds was assessed through tensile testing. Average tensile bond strength was measured quantitatively as a function of bonding temperature and applied pressure. Structural and chemical examinations of tensile fracture surfaces were carried out with SEM and X-ray mapping. Further examination of the bonding interface region was accomplished through FIB sectioning across the bonding interface together with IISE imaging. Successful assembly of Cu-based microchannel structures and complete microchannel fluidic prototypes was demonstrated. The present results offer one additional fabrication protocol for manufacturing of Cu-based microsystems.

6.5 References


CHAPTER 7. FABRICATION, BONDING, ASSEMBLY, AND HEAT TRANSFER TESTING OF AL- AND Cu- BASED MICROCHANNEL DEVICES *

7.1 Introduction

Science and technology related to new and more environmentally friendly ways of producing and distributing energy are being intensely investigated. New technologies capable of increasing the energy efficiency and decreasing the size and weight of transportation, housing, and appliance systems, if successfully commercialized, will reduce energy consumption and positively impact the global environment. In 1981, Tuckerman and Pease pointed out that decreasing liquid cooling channel dimensions to the micron scale will lead to increases in heat transfer rates, and demonstrated experimentally a forty-fold improvement in heat-sinking capability in Si-based microchannels anodically bonded to Pyrex cover plates. Since then, intense research on microchannel heat exchangers (MHEs) has ensued. Many heat transfer investigations have been conducted on Si-based microchannels. Si is not the optimal material for cooling devices in terms of its thermal and mechanical properties. The choice of Si-based microchannels is largely due to the fact that fabrication technology for Si-based high-aspect-ratio microscale structures (HARMS) is the most mature and most widely available.

Metals such as Cu and Al possess higher bulk thermal conductivities than that of Si. Metal-based MHEs also promise increased mechanical robustness over similar Si-based devices. Various techniques for fabricating metal-based HARMS have been studied. Serial subtractive techniques, include micromilling, micro electrical discharge machining (µEDM), and laser-beam direct writing, are time consuming and may encounter difficulties when fabricating microscale features in geometrical proximity. Micro powder injection molding requires heat treatment steps that involve part shrinkage and may suffer from incomplete mold filling. Micro casting also requires multiple heat treatment steps, and is an inefficient “double lost mold” technique. Cross flow MHEs made of Cu and stainless steel were fabricated from precision-cut metal foils, stacked and bonded together. Such fabrication techniques are still serial in nature, and less suitable for large-scale production of metal-based MHEs.

Molding replication offers an important alternative toward efficient fabrication of metal-based HARMS. The process of replication involves the use of primary HARMS, produced by a combination of lithography, etching, deposition, and other techniques, as a mold insert to create the negative of the insert pattern in metals by direct compression molding. One important example of molding replication is contained within the LiGA protocol for HARMS fabrication, combining deep X-ray/UV lithography (Lithographie) on polymeric resists, metal electrodeposition (Galvanoformung) into developed resist recesses, and molding replication (Abformung) of secondary HARMS. Primary HARMS made by lithography and electrodeposition (LiG) are expensive due to the high equipment and process costs of lithography and the low speed of electrodeposition. The importance of the molding replication step is that secondary HARMS can be produced in different engineering materials without repeating the lithography/developing/deposition steps, and thus at low cost and high throughput.

Since 2003, successful HARMS replication by direct compression molding has been demonstrated in Pb and Zn, Al, and Cu. Two critical elements were needed for successful molding replication of reactive metals such as Cu and Al. The first one is to control the near-
surface chemical/mechanical interactions between the mold insert and the molded metal. This was achieved via conformal deposition of suitable ceramic coatings over HARMS mold inserts. The second one is to improve the mechanical properties of the mold insert bulk at elevated molding temperatures. This was achieved by fabricating mold inserts out of refractory metals and alloys with µEDM. Efficient fabrication of refractory mold inserts containing geometrically complex microfeatures were achieved through parallel µEDM, in which complex micropatterns were transferred simultaneously onto surfaces of refractory metals/alloys using LiG-fabricated electrodes.

Using these surface-engineered, refractory, mold inserts, microchannels with complex geometries have been replicated successfully in metals such as Cu and Al by compression molding. In addition to fabricating metallic microchannels by molding replication, Al-based microchannels were successfully bonded to other Al microchannels and flat plates by eutectic bonding with vapor-phase co-deposited Al-Ge thin film intermediate layers. Multilayered microchannel arrays were successfully assembled to form metal-based MHE prototypes. Measured tensile bond strengths exceeded 75MPa and reaching as high as 165MPa.

To demonstrate the feasibility of metal-based, high-efficiency, compact MHEs, heat transfer characteristics of device prototypes need to be measured. Extensive studies of microchannel fluid flow and heat transfer have been carried out over the past two decades. Design of heat exchangers taking advantage of heat transfer enhancement in microchannels has been contemplated previously. Heat transfer characteristics of Ni-based microchannel devices have been reported in the literature. These devices were fabricated following LiG-related protocols and not easily adapted to large scale production. Heat transfer characteristics were also reported for Al-based minichannels with \( D_h \) of ~2mm. These minichannels were fabricated by multiport extrusion, which limits \( D_h \) to ~1mm.

Heat transfer characteristics have been measured previously by Lee et al. in Cu microchannel devices fabricated by mechanical cutting of Cu plates followed by sealing with a polymeric top plate. They examined a variety of rectangular Cu microchannels ranging from 194µm to 534µm in width, with the same channel aspect ratio \( (H/W) \) of 5. In this chapter, results of fabrication, assembly, and heat transfer testing of simple, entirely Al- and Cu-based, MHE prototypes containing replicated microchannel arrays with \( D_h \) of ~200µm are described. A constant heat flux was supplied via cartridge heaters mounted on one side of the MHE devices. Data reported by Lee et al. have been used as a benchmark against present heat transfer rate measurements. The potential influence of microchannel surface profile on heat transfer rates is discussed in light of the current results.

7.2 Fabrication and Assembly

7.2.1 Insert Fabrication

Microscale mold inserts were fabricated from Ni-based superalloy Inconel X750® plates. As-received Inconel plates were machined to square insert blanks, with an active area of ~15000µm×~15000µm, ~3200µm in height. The top surface of the blank was mechanically polished with SiC abrasive papers down to 1200 grit size. Insert fabrication from the blanks involved three main steps: µEDM of the active area, electrochemical polishing (ECP) of as-machined microscale Inconel features, and deposition of a conformal amorphous silicon nitride (a-Si:N) coating over electrochemically polished microscale features. A SARIX high precision micro erosion machine (Model SR-HPM-B) was used for insert µEDM. Flat molybdenum (Mo)
sheets with a thickness of 500µm were used as blade electrodes. A series of parallel cuts were made on the insert blank sequentially. Erosion of the insert blank through the µEDM process resulted in the formation of a trench under the Mo electrode. Sequential cuts led to the formation of an array of trenches, or an array of parallel rectangular microprotrusions in between the trenches. As-machined Inconel blanks were electrochemically polished for a total of 10min in the current-controlled mode in a mixed acid solution of HClO₄ (70%) and CH₃COOH (80%) at a volume ratio of 1:1. Following ECP, a conformal a-Si:N coating was deposited over the Inconel inserts in a radio frequency (rf) inductively coupled plasma (ICP) assisted hybrid chemical/physical vapor deposition system. Further details on coating deposition and a-Si:N coatings have been reported previously. 

**Fig. 7.1** SEM examination of the a-Si:N coated Inconel mold insert: (a) an overview; (b) a high magnification view of a typical rectangular microprotrusion on the mold insert.

Surface morphology examinations were performed on a Hitachi S3600N scanning electron microscope (SEM) and a Veeko Wyko3100 optical profilometer (OP). A SEM overview of one such a-Si:N coated Inconel insert is shown in Fig. 7.1(a). The thickness of the a-Si:N coating is ~600nm. The active surface of this insert contains 19 parallel rectangular microprotrusions, with a center-to-center spacing of ~750µm. The sequential cutting process led to variations in the widths of the microprotrusions. The average width of all microprotrusions, as measured from SEM micrographs, is 154µm. Actual width values for the 19 microprotrusions ranged from 140 to 175µm, reflecting imprecision in electrode alignment/translation during the µEDM process. As evident from Fig. 7.1(a), the process of µEDM with Mo blade electrodes led to the formation of rounded trench bottoms. The height of the straight section of the microprotrusions is ~400µm. Figure 7.1(b) shows a close-up SEM image of a typical microprotrusion. The numerous micron scaled features on the microprotrusion surface resulted from preferential etching during the ECP process, and delineated the grain structure of the underlying Inconel substrate. The a-Si:N coating follows the substrate morphology conformally without significantly modifying it.

### 7.2.2 Molding Replication

Cu 110 (99.9+ wt.% Cu) and Al 6061 (1.0 wt.% Mg, 0.6 wt.% Si, 0.27 wt.% Cu, 0.2 wt.% Cr, balance Al) coupons, with the same geometry of 35.5mm×35.5mm square and 6.4mm
The molding process replicated the array of 19 parallel rectangular microprotrusions on the insert into an array of 19 parallel rectangular microchannels on the molded Cu and Al coupons. The average width of microchannels on the Cu coupon was measured to be 150µm, while the actual microchannel width ranged from 134 to 170µm. The average width of microchannels on the Al coupon was measured to be 148µm, while the actual microchannel width ranged from 137 to 172µm. The measured variation in the width of microchannels results largely from microprotrusion width variation on the mold insert. The final width of the molded microchannel tends to be somewhat smaller than that of the corresponding microprotrusion on the mold insert, by ~3% and ~4% for Cu and Al, respectively. Depths of molded microchannels were greater than 400µm.

**7.2.3 Assembly and Bonding**

One supply channel and one drain plenum were further machined using μEDM onto molded Cu and Al coupons to provide fluidic connections to replicated microchannel arrays. Flat stainless steel sheets with a thickness of 1100µm were used as blade electrodes for μEDM. One single cut with the steel blade electrode was made to create the fluid supply channel, while six consecutive cuts with partial overlap were performed to create the fluid drain plenum. Depth of cuts were kept the same for all cuts on the supply and drain sides.

As shown in Fig. 7.2(a) for one Cu coupon, mechanical drilling was used to make two through holes connecting to the supply channel and three through holes within the drain plenum. The through holes within the drain plenum were placed symmetrically with respect to the microchannel array. All through holes were mechanically tapped on the coupon back side away from the microchannel array to allow external fluid connections using plastic adaptors. The choice of the 6.4mm coupon thickness accommodates the placement of taps. Figure 7.2(b) provides a more detailed view of fluidic supply connections on the Cu coupon from one tapped hole to the supply channel, which connects to the replicated array of rectangular microchannels. The supply channels have the mottled surface morphology typical of structures cut by μEDM,
and provide connections to the microchannel arrays without obstructions at the entrances. Figure 7.2(c) shows a high magnification view of replicated Cu microchannels. The microchannels have vertical sidewalls and sharp sidewall-to-bottom transitions. Micron scale surface roughness is clearly visible on the sidewall and bottom of all microchannels. Figure 7.2(d) shows a typical OP image obtained from the bottom of a replicated Cu microchannel.

Fig. 7.2 Fabrication of the Cu-based MHE specimen: (a) an overview of the molded Cu coupon containing the array of rectangular microchannels, fluid supply channel, and fluid drain plenum. Numbers shown on the ruler are in mm; (b) a detailed view of the fluid supply channel; (c) a detailed view of the replicated microchannels; (d) a typical OP image of the bottom of one replicated microchannel.

A nominally identical configuration was created on one Al coupon, from which data analogous to those shown in Fig. 7.2 were obtained. Further work is needed to optimize the fluid supply and drain configurations in terms of fluid flow.

A quantitative evaluation of the surface roughness within molded microchannels is expressed in the value of the peak-to-valley roughness $R_z$. Four independent OP images, analogous to that shown in Fig. 7.2(d), were obtained from the bottom surfaces of four different microchannels in the Cu and the Al coupon. Values of $R_z$ obtained from the four OP images were averaged. The average $R_z$ values are 11.8µm and 8.2µm for the bottom surfaces of Cu and Al microchannels, respectively. In this experiment, values of $R_z$ for the microchannel sidewalls were not obtained. From qualitative SEM observations, it is surmised that surface roughness of
the microchannel sidewalls is smaller. Further examination of surface roughness on microchannel sidewalls shows the value is \(~5\mu m\).

As expected from the replication process, surface roughness on the microchannel bottoms mimic the roughness present on the top surface of the insert microprotrusions shown in Fig. 7.1(b). As stated above, such surface roughness on the mold insert resulted from preferential etching during the electrochemical polishing step of insert fabrication. Furthermore, the molding process involves large strain plastic deformation and may create different surface roughness for the microchannel sidewalls. The observed \(R_z\) values, \(~10\mu m\), are larger than what is typically obtained from micromilling.

To form entirely Cu- and Al- based MHEs, Cu and Al coupons containing replicated microchannels and fluidic connections are bonded respectively to flat Cu and Al plates. The thickness of the flat plates is 6.4mm, the same as that of the Cu and Al coupons. Bonding surfaces of coupons and flat plates were mechanically polished with SiC abrasive papers and finished with 1\(\mu m\) diamond particle suspension. Al-Ge composite thin film intermediate layers were deposited onto polished surfaces via sputter co-deposition in a pure Ar (99.999\%) atmosphere, with a total pressure of \(~0.17\)Pa. Two separate sputter targets, one for pure Al(99.99\%) and the other for pure Ge(99.99\%), were used. The polished Cu or Al coupons and plates were ultrasonically cleaned in acetone and methanol before being mounted on a rotatable holder situated in the middle of the deposition zone. The deposition sequence consisted of a radio frequency (rf) inductively coupled plasma (ICP) substrate surface etch followed by Al and Ge co-deposition. Substrate etching occurred in a pure Ar ICP with a total rf input power of 1000W, a substrate bias of \(~100V\), and an etch duration of 20min. Sputtering of Al and Ge targets commenced immediately after substrate surface etch. Substrates were rotated continuously at \(~12\)rpm during both etching and deposition. All Al-Ge depositions were carried out at fixed target currents of 1.0A for Al and 0.45A for Ge, respectively. The substrates bias during deposition was fixed at \(-50V\). These deposition parameters resulted in a Ge to Al composition ratio close to that of the Al-Ge eutectic at Al\(_{70}\)Ge\(_{30}\).\(^{36}\) The Al-Ge film thickness was \(~2\mu m\). Further details on Al-Ge thin film co-deposition and structure, as well as evaluation of Al-Ge eutectic bond strength, have been reported previously.\(^{25, 26}\)

![Fig. 7.3 An overview of the assembled Cu and Al MHE specimens.](image)

Bonding experiments were carried out using the MTS858 single-axis testing system interfaced to a high-vacuum chamber containing two heating stations. Cu or Al coupons
containing microscale features and flat Cu or Al plates, with Al-Ge composite films deposited on the bonding surfaces, were placed face-to-face on the lower heating station. The chamber was evacuated, and both heating stations were heated. Bonding of Al coupon and plate was carried out at a temperature of ~500°C with an applied pressure of ~1.5MPa, while bonding of Cu coupon and plate occurred at a temperature of ~540°C with an applied pressure of ~3MPa. Further details on the eutectic bonding protocol and evaluation of bond quality have been given elsewhere.25, 26 Figure 7.3 shows an optical overview of bonded Cu and Al MHE prototypes, with plastic external fluid adaptors attached.

7.2.4 Measurement of MHE Geometry

Final examination of assembled Cu and Al MHE prototypes was accomplished by mechanically cutting them in directions perpendicular to the microchannel arrays after heat transfer measurements. The cuts were placed within the drain plena. To fully reveal the cross sectional configurations of the bonded microchannels, the cut pieces containing the microchannel arrays were mechanically polished back close to the microchannel exits. Figure 7.4(a) shows a cross-sectional view of a portion of the bonded Cu microchannel structure. Similar cross-sectional images were obtained from the cut Al prototype. Water was passed from the fluid supply channel through the microchannel arrays. Microscale water jets exiting the microchannels were observed on the drain plenum side to ensure that each microchannel has remained intact during the bonding process. The replicated Cu and Al microchannel arrays each contain 19 rectangular microchannels. Our observations showed exit water jets from all 19 microchannels in the assembled Cu MHE, while exit water jets were observed from 18 microchannels in the assembled Al MHE. Therefore it appears that one microchannel in the assembled Al MHE was blocked during the bonding process, and only 18 through microchannels remained. The exact reason behind the blockage of one microchannel out of 19 in the Al MHE is not understood.

The blockage of one of 19 microchannels in the assembled Al MHE implies that the bonding process may also change the dimensions of the microchannels from their as-replicated state. Figure 7.4(b) shows a higher magnification view of two bonded microchannels in the Cu device. Similar images were again obtained from the Al device.

Fig. 7.4 Destructive examination of the assembled Cu MHE specimen: (a) a cross-sectional view of a portion of the bonded microchannel structures; (b) a higher magnification view of two bonded microchannels.
Table 7.1. Average microchannel dimensions of assembled Cu and Al MHE specimens.

<table>
<thead>
<tr>
<th>Device</th>
<th># of channels</th>
<th>Average dimensions of bonded microchannels</th>
<th>Dimensions of fluid supply channel</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>H (µm)</td>
<td>W (µm)</td>
</tr>
<tr>
<td>Cu</td>
<td>19</td>
<td>320±6</td>
<td>150±5</td>
</tr>
<tr>
<td>Al</td>
<td>18</td>
<td>291±6</td>
<td>115±5</td>
</tr>
</tbody>
</table>

To obtain the exact dimensions of bonded Cu and Al microchannels, measurements of microchannel width and height were performed from micrographs similar to Fig. 7.4(b) on each microchannel for both Cu and Al MHE specimens. For the Cu and Al MHE specimens, measured microchannel widths and heights were averaged and shown in Table 7.1. The average hydraulic diameter for the microchannel array, \( D_h = \frac{4A}{p} = \frac{2WH}{(W+H)} \), was calculated from the average microchannel dimensions and shown also in Table 7.1. Results indicate that the average width of microchannels in the Cu device exhibits little change from that on the as-replicated Cu coupon, while the average microchannel width of the Al device decreased significantly from that on the as-replicated Al coupon. Considering that Al bonding occurred at about 83% of the Al melting temperature, the observed narrowing of the microchannels is speculated to result from plastic deformation during the bonding process, which decreased the microchannel height and resulted in the observed width reduction. The final configurations of the Cu and Al MHE specimens contain relatively wide fins between microchannels, ~600µm in width and ~300µm in height. Optimization of metal-based MHE geometry design will be considered in future work.

7.3 Flow and Heat Transfer Testing

7.3.1 Experimental Setup

For the present experiments, one Cu and one Al MHE specimen were used as cooling devices with water as the coolant. Figure 7.5(a) shows a schematic of the testing apparatus designed and built to evaluate the heat transfer characteristics of the Cu and Al specimens, the average dimensions of microchannels within which are given in Table 7.1. The apparatus consisted of three sections: water supply section, test section, and data acquisition section. The water supply section consisted of a water holding tank connected to the exit of a pressure-regulated air bottle, and supplied water to the MHE specimen at a constant pressure. Regular water was used without deionization or degassing. This setup provided a smooth and stable flow through the microchannels at low flow rates. A valve placed downstream of the tank exit was used to make fine adjustments to the flow rate. A pressure relief valve was also connected to the tank for safety measures.

Figure 7.5(b) shows a schematic of the test section, which consisted of either the Cu or Al MHE specimen enclosed in a rigid PVC foam insulation with two heaters placed in contact with the specimen top face. Each rectangular heater was fabricated by inserting a ~200watt cylindrical cartridge heater into a machined Cu block, 16mm×16mm×30mm in dimension with mechanically polished external surfaces. The two Cu blocks were bonded together, and then attached to the MHE top surface with conducting silver glue, which provided a uniform contact and eliminated air-gaps between contacting surfaces. The two cartridge heaters were powered by one dc power supply in the constant-voltage mode. A maximum input heat flux of ~39watt/cm² was obtained from the combined output of the two cartridge heaters, based on the total base area.
of the copper heater blocks of 32mm×30mm. It should be noted that this base area is much larger than the footprint of the microchannels, ~13mm×15mm. Holes were drilled into the PVC insulation for the fluid inlet and outlet tube connections.

Fig. 7.5 Experimental setup for heat transfer measurements: (a) a schematic of the testing apparatus; (b) a schematic of the test section, in which black dots indicate locations of thermocouples; (c) a schematic of the thermocouple fixture; (d) plan-view of thermocouple locations (shown as black dots).
Thermocouples, 36-gauge and K-type, were used to measure the inlet and outlet water temperatures. Two thermocouples were inserted through the two inlet tubes into the two tapped holes connecting to the fluid supply channel. Thermocouple insertion into the inlet tube was accomplished with the help of a T-fitting and then sealed with epoxy, as shown schematically in Fig. 7.5(c). With such a thermocouple placement, the inlet water temperature was measured before the water came into contact with heated surfaces. Three additional thermocouples were inserted directly through the three outlet tubes into the three tapped holes connecting to the fluid drain plenum. Because none of the thermocouples were placed within the microchannels, whether the water flow within the microchannels is laminar or turbulent should not affect the temperature measurements.

Another thermocouple was placed on the bottom surface of the MHE specimen, which was then placed in the PVC insulation. Four additional thermocouples were placed on the top surface of the MHE specimen and two additional thermocouples were placed on top of the Cu heater block. Thermocouple locations are indicated schematically in Figs. 7.5(b) and 7.5(d). In the data acquisition section, an Instrunet data acquisition system interfaced to a PC was used to collect the thermocouple readings. All thermocouples were calibrated with a two-point calibration, using a mixed ice/water bath and boiling water as the reference points. Temperature deviations at the two calibration temperatures were ~±0.1°C in all cases. The differential pressure across the water inlet and outlet was measured with a Dywer digital manometer with a minimum reading of 0.1psi (690Pa).

To further understand the cooling behavior of assembled Cu and Al MHE specimens, the transient cooling response of Cu and Al MHE specimens was characterized using infrared (IR) camera. Figure 7.6 shows a schematic of testing section for infrared thermography. This set-up is similar to the one used for heat transfer testing. Cu or Al MHE specimen was enclosed in a rigid PVC foam insulation with the copper heater block placed in contact with the surface without plastic connectors. The other surface, with plastic connectors, was facing the IR camera. The emissivity of this surface was determined by comparing temperature reading from the IR camera to the reading from a calibrated thermocouple bonded onto the surface. The MHE surface was painted black to increase the emissivity value up to 0.96. The MHE specimens were initially
heated to a set temperature before water was introduced into the microchannels. Infrared thermography of the surface was captured at 60Hz with a FLIR SC500 IR camera to determine the external surface temperature of the MHE specimens so as to assess the overall cooling rate.

### 7.3.2 Measurements

Heat transfer measurements were performed on both the Cu and Al MHE specimens at similar input heat fluxes, differential water pressures, and volumetric water flow rates, which ranged respectively from 15 to 39 watt/cm², 6.9×10³ to 4.5×10⁵ Pa (1 to 65 psi), and 0.05 to 0.8 liter/min. The difference in outlet and inlet water temperatures ranged from 7 to 34°C. The steady state power gain by the water flowing through the MHE can be expressed as

\[
q_{gain} = \rho \cdot c_p \cdot Q \cdot (T_{o, \text{min}} - T_{i, \text{avg}}),
\]

where the volumetric flow rate \( Q \) was obtained by measuring the volume of water collected at the exit over a fixed period of time. The average inlet water temperature was obtained from readings of the two thermocouples placed within the fluid supply channel. The minimum outlet water temperature was obtained by taking the minimum reading from the three thermocouples placed within the drain plenum. Therefore, \( q_{gain} \) computed from Eq. (1) represents a conservative calculation of the water power gain. For the two Cu and Al MHE specimens measured, 88 to 98% of the output power from the cartridge heaters was transferred to the water.

The overall averaged heat transfer coefficient for the microchannel array is computed using

\[
h = \frac{q_{gain}}{A_{total} \cdot (T_w - T_m)};
\]

where \( A_{total} \) is the total area available for convection within the test specimen. It is evident from Figs. 5.5(b) and 5.5(d) that the inlet water temperature was measured from thermocouples placed at locations before the water enters the fluid supply channel, while the outlet water temperature was measured from thermocouples placed at locations very close to the microchannel exits. As shown in Fig. 7.2(a), the supply channel is an integral part of the heated MHE body. Therefore \( A_{total} \) was taken to be the sum of contributions from the fluid supply channel and the microchannel array. Because the hydraulic diameter of the supply channel greatly exceeds that of the microchannels, including the supply channel area in \( A_{total} \) presents a conservative estimate of \( h \). The mean water temperature, \( T_m \), is taken to be \( (T_{o, \text{avg}} + T_{i, \text{avg}}) / 2 \). In all measurements on both Cu and Al MHE specimens, \( T_m \) ranged from 25 to 50°C. The temperature difference between the top and bottom surfaces of the Cu MHE ranged from 0.3 to 4°C. Within the Cu MHE specimen, the microchannels were located half way between the top and bottom surfaces. Because it is difficult to measure the exact wall temperature in the current experimental configuration, \( T_w \) was estimated by taking the average between the mean reading of the four thermocouples placed on the MHE top surface and the reading of a single thermocouple placed on the MHE bottom surface [see Fig. 7.5(d)]. The same methodology was applied to the Al MHE specimen. In this case, due to the lower thermal conductivity of Al, the temperature difference between the top and bottom surfaces of the Al MHE ranged from 6 to 18°C. The values of \( T_w \) so estimated ranged from 37 to 66°C and from 49 to 65°C for the Cu and Al MHE specimens, respectively. While this linear temperature interpolation scheme should yield a reasonable estimate of \( T_w \) in the case of the Cu MHE specimen, the estimated \( T_w \) is less reliable in the case of the Al MHE, due to the larger temperature difference between the top and bottom specimen surfaces.
The experimental uncertainty for each measured parameter was estimated. The uncertainties of microchannel dimension measurements, $\delta H$, $\delta W$, and $\delta L$, are 6µm, 5µm, and 50µm, respectively. The uncertainties of the supply channel dimension measurements, $\delta H_s$, $\delta W_s$, and $\delta L_s$, are 6µm, 10µm, and 200µm, respectively. Flow rate was measured by collecting a volume of water over a fixed time duration. The total volume of water collected was >300ml in all cases and the uncertainty of volume reading is 2.5ml. The resulting uncertainty in measurements of volumetric flow rate $\dot{Q}$ is better than 0.8%. The uncertainty for each thermocouple reading is 0.25 oC or less. This uncertainty in temperature measurements was used to estimate uncertainties of quantities such as $T_o-T_i$ and $T_w-T_m$. The pressure drop across the water inlet and outlet was obtained from the Dywer digital manometer and the uncertainty for this pressure reading is 0.05psi (345Pa) or less.

7.3.3 Data Reduction

The Reynolds number corresponding to water flowing through the microchannels is related to the average water velocity $\overline{V}$ or volumetric flow rate $\dot{Q}$ through the microchannels by

$$Re = \frac{\rho \overline{V} D_h}{\mu} = \frac{4 \rho \dot{Q}}{\mu}$$

(3)

where both water density and viscosity depend on temperature.\(^{37}\) The differential pressure across the microchannel inlet and outlet needed to generate the desired water flow is expressed through the Darcy friction factor\(^{37, 38}\)

$$f = \frac{\Delta P D_h}{\frac{1}{2} \rho \overline{V}^2 L}$$

(4)

Measured heat transfer coefficients were converted to dimensionless Nusselt numbers based on the average hydraulic diameter of the microchannels\(^{33, 37}\)

$$Nu = \frac{h D_h}{K_f}$$

(5)

where values of $D_h$ for the Cu and Al MHE specimens are given in Table 5.1, and $K_f$ is the thermal conductivity of water at $T_m$.\(^{37}\)

The experimental uncertainties in the measurements were estimated using\(^{39, 40}\):

$$\frac{\delta Re}{Re} = \left[ \left( \frac{\delta \dot{Q}}{\dot{Q}} \right)^2 + \left( \frac{\delta H}{H} \right)^2 + \left( \frac{\delta W}{W} \right)^2 \right]^{1/2}$$

(6)

$$\frac{\delta f}{f} = \left[ \left( \frac{2 \delta \dot{Q}}{\dot{Q}} \right)^2 + \left( \frac{3 \delta H}{H} \right)^2 + \left( \frac{3 \delta W}{W} \right)^2 + \left( \frac{\delta P}{P} \right)^2 + \left( \frac{\delta L}{L} \right)^2 \right]^{1/2}$$

(7)

$$\frac{\delta Nu}{Nu} = \left[ \left( \frac{\delta \dot{Q}}{\dot{Q}} \right)^2 + \left( \frac{2 \delta H}{H} \right)^2 + \left( \frac{2 \delta W}{W} \right)^2 + \left( \frac{\delta L}{L} \right)^2 + \left( \frac{\delta H}{H_1} \right)^2 + \left( \frac{\delta W_1}{W_1} \right)^2 \right]^{1/2}$$

$$+ \left( \frac{\delta T_o - T_i}{L_1} \right)^2 + \left( \frac{\delta \left( T_o - T_i \right)}{T_o - T_i} \right)^2 + \left( \frac{\delta \left( T_{wall} - T_{water} \right)}{T_{wall} - T_{water}} \right)^2$$

(8)
where the values of in the terms above are based on the uncertainties mentioned in measurement section and the collected data during measurement.

### 7.3.4 Results and Discussion

Figure 7.7(a) shows the collection of data regarding the rates of water flow through the microchannel array in the Cu MHE specimen and the associated pressure drops, expressed in the form of Darcy friction factor versus Reynolds number. The data were collected during heat transfer testing and, as stated above, in a range of mean water temperatures of $25^\circ C < T_m < 50^\circ C$ instead of at a constant $T_m$. Considering the experimental uncertainties in the measurements of $\Delta P$, $L$, $Q$, $W$, $H$, the uncertainties for $Re$ and $f$ values extracted for the Cu MHE specimen were about 4% and 13%, respectively, assuming independent and uncorrelated measurements on all quantities. The data shown in Fig. 7.7(a) exhibit a rapid drop from greater than 0.25 to ~0.15, as $Re$ increases from 250 to 500. As $Re$ increases further from 500 to 2000, a less rapid decrease in the friction factor from ~0.15 to ~0.08 is observed. As $Re$ increases beyond 2000, the decrease is even less rapid.

![Fig. 7.7 Data on water flow through and pressure drop across microchannel arrays: (a) Darcy friction factor versus Reynolds number for the Cu MHE specimen; (b) Darcy friction factor versus Reynolds number for the Al MHE specimen. In both graphs, calculations based on fully developed and entrance length effects included laminar flows are shown as $f_{fd}$ and $f_{app}$, respectively. Values for the Blasius correlation for turbulent flow through smooth pipes and for the Haaland correlation for turbulent flow through rough pipes are also included in both graphs.](image)

Included in Fig. 7.7(a) are results of friction factor calculations for fully developed laminar flow in a rectangular channel, $f_{fd} = 63.1/Re$, with $W$ and $H$ adopting average values for the microchannel array within the Cu MHE specimen. $Re$ Entrance length considerations in the laminar flow regime have been discussed previously. Defining a dimensionless entrance length as $x^+ = L/(D_h \, Re)$, an expression for an apparent friction factor $f_{app}$, which takes entrance length effects into account, is given by
\[ f_{\text{app}} \frac{\text{Re}}{2} = \left[ \left( \frac{3.2}{(R + 0.57)} \right)^2 + \left( f_{\text{fd}} \frac{\text{Re}}{2} \right)^2 \right]^{1/2}. \] (9)

Figure 7.7(a) plots \( f_{\text{fd}} \) and \( f_{\text{app}} \) as a function of \( \text{Re} \). It is evident that the difference between \( f_{\text{fd}} \) and \( f_{\text{app}} \) is minimal in the range \( 200 < \text{Re} < 900 \). Friction factor correlations for turbulent flow at \( \text{Re} > 2000 \) are also plotted in Fig. 7.7(a). Friction factor values were calculated from the Blasius correlation for fully developed turbulent flow in smooth pipes,

\[ f = 0.316 / \text{Re}^{0.25}, \] (10)

and the Haaland correlation for fully developed turbulent flow in rough pipes,

\[ \frac{1}{\sqrt{f}} = -1.8 \log \left( \frac{\varepsilon / D_h}{3.7} \right)^{1.11} + \frac{6.9}{\text{Re}}. \] (11)

The roughness parameter \( \varepsilon \) in Eq. (11) was taken as 11.8\( \mu \)m, the average value of \( R_z \) measured from the bottom surface of the microchannels in the Cu MHE specimen. As mentioned in Section IIC, surface roughness on the microchannel sidewalls is smaller than that for the microchannel bottom surface. The calculated values for the Haaland correlation are therefore somewhat uncertain.

It is evident that measured friction factors show reasonable agreement with laminar flow calculations at \( \text{Re} < 500 \). At high Reynolds numbers (\( \text{Re} > 2000 \)), measured \( f \) values are significantly higher than those calculated from the Blasius correlation for turbulent flow in smooth pipes, but are much closer to those calculated from the Haaland correlation for turbulent flow in rough pipes.

Figure 7.7(b) shows Darcy friction factor plotted versus Reynolds number for water flowing through the microchannel array in the Al MHE specimen. The data were again collected during heat transfer testing, and in the temperature range of \( 25^\circ\text{C} < T_m < 50^\circ\text{C} \). The uncertainties for \( \text{Re} \) and \( f \) values extracted for the Al MHE specimen were about 5% and 14%, respectively, again assuming independent and uncorrelated measurements on all quantities. The variation of \( f \) with \( \text{Re} \) appears similar to that observed for the Cu MHE specimen. Figure 7.7(b) also displays results of friction factor calculations for fully developed laminar flow in a rectangular channel, \( f = 65.7 / \text{Re} \), with \( W \) and \( H \) adopting average values for the microchannel array within the Al MHE specimen.\(^{41, 42}\) Apparent friction factor \( f_{\text{app}} \) values, taken entrance length effects into account, were likewise calculated and displayed in Fig. 7.7(b). Friction factor correlations for fully developed turbulent flows in smooth and rough pipes were also calculated in a way similar to that described for the Cu MHE specimen and plotted in Fig. 7.7(b). Overall, friction factors measured from the Al MHE specimen show a dependence on \( \text{Re} \) similar to that for the Cu MHE specimen. At high Reynolds numbers (\( \text{Re} > 2000 \)), measured \( f \) values again exceed significantly those calculated from the Blasius correlation.

Liu and Garimella investigated water flow in smooth rectangular microchannels with hydraulic diameter ranging from 244 to 974\( \mu \)m, combining friction factor measurements with flow visualization. In long channels with \( L/D_h \) ranging from 42 to 127, their measured friction factors agreed with laminar flow calculations up to \( \text{Re} \sim 1600 \). Judging from significant friction factor deviation from laminar flow calculations combined with flow visualization, they concluded that turbulent flow occurred at \( \text{Re} > 2000 \), with the transition from laminar to turbulent flow occurring over \( \sim 1600 < \text{Re} < 2000 \).\(^{40}\) Mala and Li observed water flow through
microtubes with diameters ranging from 50 to 254µm, and divided their friction factor versus Reynolds number data into three regions: at \( Re < 500 \) where observations agreed with laminar flow calculations; at \( Re > 1500 \) where observations followed the Blasius equation for turbulent flow; and a transition region with \( 500 < Re < 1500 \) where measured friction factors are higher than those from laminar flow calculations.\(^{38}\)

The range of presently measured \( f \) values, from \( \sim 0.06 \) to \( \sim 0.25 \), is in agreement with that from Liu and Garimella\(^{40}\) as well as with that from Mala and Li.\(^{38}\) Pressure loss corrections exist due to flow contraction at the junction between the fluid supply channel and the microchannel array and flow expansion at the junction between the microchannel array and the drain plenum. Formulae for pressure loss corrections at standard, sharp-edged entrance and exit junctions are available in both the laminar and turbulent flow regimes.\(^{40}\) Based on these formulae, the pressure loss was estimated and found to result in a 20 to 30% decrease in the measured pressure values, and therefore a like decrease in the \( f \) values. In the present situation, the fluidic connections, as shown in Figs 5.2 and 5.3, do not quite match the standard configuration. Applying these pressure loss formulae thus leads to increased uncertainties. We chose therefore to plot the as-measured friction factors in Figs. 5.7(a) and 5.7(b). The general trend of \( f \) vs. \( Re \) is unaltered by the pressure loss correction: it shows reasonable agreement with laminar flow calculations at low Reynolds numbers (\( Re < 500 \)), and lies in between the Blasius and Haaland correlation values at high Reynolds numbers (\( Re > 2000 \)).

![Graph](image)

Fig. 7.8 Data on heat transfer characteristics: Nusselt number versus Reynolds number. Red and blue symbols are data from the Cu and Al MHE specimens, respectively. The green symbols are data of Lee et al. [33]. Conventional correlations based on fully developed (FD) and simultaneously developing (Sieder-Tate, SD) laminar flows and the Gnielinski correlation for turbulent flows are included in the graph.

Figure 7.8 shows the collection of data regarding overall averaged heat transfer coefficients in the Cu and Al MHE specimens and the associated water flow through the corresponding microchannel arrays, expressed in the form of Nusselt number versus Reynolds
number. Data collection was limited to \( Re \sim 3000 \) due to pressure limitation of the plastic tube connections. The uncertainties for \( Nu \) values extracted for the Cu and Al MHE specimens were less than 12% and 11%, respectively, assuming independent and uncorrelated measurements on all quantities involved in the Nusselt number definition [Eq. (5)].

Figure 7.8 shows that, in the range of 500 < \( Re < 2250 \), good agreement exists between Nusselt numbers measured respectively from the Cu and the Al MHE specimens. At \( Re > 2500 \), data from the Cu and the Al MHE specimens start to diverge, with \( Nu \) values from the Cu specimen exceeding those from the Al specimen. Also shown in Fig. 7.8 for comparison are the data of Lee et al., obtained from machined Cu microchannels. This particular data set were taken from microchannels with \( D_h = 318 \mu m \), the smallest \( D_h \) value studied by Lee et al., and extend over a smaller range of \( Re \), from \( \sim 500 \) to \( \sim 2500 \). As evident from Fig. 7.8, the presently measured \( Nu \) values are higher than those of Lee et al. over much of the range of 500 < \( Re < 2500 \). Nusselt number values of Lee et al. approach the present data at \( Re \sim 500 \) and \( Re \sim 2500 \). Also plotted in Fig. 7.8 are several conventional Nusselt number correlations. The correlation for fully developed laminar flow and the Sieder-Tate correlation for simultaneously developing laminar flow were calculated using dimensions relevant to the Cu MHE specimen. The Gnielinski correlation for transitional and fully developed turbulent flows is also plotted. Data shown in Fig. 7.8, obtained from the Cu and Al MHE specimens, exhibit a general trend similar to that depicted by the various conventional correlations.

The present data obtained from the Cu and Al MHE specimens, as well as the data of Lee et al., show Nusselt number increases with increasing Reynolds number. At the same \( Re \) values, the present data show higher \( Nu \) values as compared to those of Lee et al. Visualization of flow within the microchannel arrays is not possible for the assembled, non-transparent, Cu and Al MHE specimens. Nonetheless, we speculate that the presence of significant surface roughness within the microchannel arrays resulting from the molding replication process is responsible for the observed higher \( Nu \) values. The increased surface roughness within the microchannel arrays may lead to increased cross-wise flow mixing, resulting in higher heat transfer as compared to smoother channels. The divergence between \( Nu \) values measured from the Cu and Al MHE specimens at \( Re > 2500 \) is not understood at the present time. The present data on the Cu and Al MHE specimens suggest that controlling surface roughness of or engineering profiles into microchannels may be beneficial for heat transfer. The efficacy of such an approach awaits future investigation.

Figure 7.9 shows the transient cooling response of Cu MHE from the initial state to the steady state. Figure 7.10(a) shows the temperature curve for the entire cooling response and Fig. 7.9(b) shows the temperature images corresponding to the instants marked in Fig. 7.9(a). Initially, the heater and water were both off and the surface temperature distribution at this state is shown in image 1. At instant 1, the heaters were switched on to provide an input power of 370 W. The surface temperature was constantly monitored with help of thermocouples placed on the surface was allowed to rise. As soon as the surface temperature reached 100°C (instant 2), the valve was opened to push the water with a pressure of 40psi through the microchannels. The temperature of the specimen was cooled down rapidly (instant 3) and within a minute the system reached steady state (instant 4). Images 2 and 3 show the temperature distributions of the specimen surface right before and after the water on. In image 3, it is apparent that the temperature in the region of microchannel array was lower, meaning this microchannel area was cooled down first. The time difference between instant 2 and instant 3 is only about one second. Image 4 shows the temperature distribution of the specimen at steady state. Cool water comes into the two inlet
tubes and hot water goes out from the three outlet tubes. It can also be observed from image 4 that the insulation is slightly heated up, indicating that there is some heat absorbed by the surrounding insulation.

The potential of Cu- and Al- based MHEs is illustrated in Fig. 7.10. Initially, the heaters attached to the Cu MHE specimen was turned on and the specimen was allowed to reach a set temperature of ~100°C. Keeping the same power input (~20W) into the heaters, water was suddenly introduced into the Cu MHE by fixing a $\Delta P$ across the outlet and inlet. The specimen

Fig. 7.9 Transient cooling response of Cu MHE specimen: (a) cooling curve from initial state to steady state; (b) infrared thermal field images from instants 1-4, corresponding to the instants shown in (a).
Fig. 7.10 Overall cooling characteristics of the Cu and Al MHE specimens: (a) Cu MHE surface temperature versus time as water is flown through the microchannel array; (b) a fit to the data shown in (a) assuming exponential decay with two time constants; (c) the fast time constant obtained through data fitting as a function of the water pressure drop across the MHE device. Black, red, and blue symbols denote time constants obtained with starting temperatures of 70, 85, and 100°C, respectively.

Surface temperature was monitored in real time using the IR camera. Figure 7.10(a) shows the change in surface temperature of the Cu MHE as a function of time after water was introduced into the specimen at a fixed $\Delta P$. The MHE surface temperature drops more than 50°C within the first 5s, followed by a more gradual decrease to ~30°C in the next 60s. The first and faster drop in temperature occurs when water draws heat away from the Cu bulk. The second and more gradual drop in temperature occurs when heat is drawn away from the surrounding PVC insulation. The insulation has a higher heat capacity, resulting in a more gradual decrease in temperature. Figure 7.10(b) shows a fit to the measured surface temperature versus time data,
assuming a compound exponential decay with two distinct time constants $\tau_1$ and $\tau_2$. It is evident that an excellent fit to the data was obtained using such a two-time-constant fit. Consistent with expectation, $\tau_1$ is on the order of a few seconds while $\tau_2$ is on the order of a few tens of seconds. Figure 7.10(c) shows values of fitted time constant $\tau_1$ for both the Cu and Al MHE specimen, obtained at different values of $\Delta P$ and initial specimen temperature $T_1$. As $T_1$ varies from 70 to 100°C, fitted values of $\tau_1$ show no significant variation. As expected, $\tau_1$ decreases with increasing $\Delta P$ or water flow rate, and is in the range of 1 to 2s for the Cu MHE and 2 to 2.5s for the Al MHE.

7.4 Conclusion

Fabrication of Cu- and Al- based microchannels through molding replication with surface engineered Inconel mold inserts has been demonstrated. Cu- and Al- based microchannel arrays were assembled through eutectic bonding to form entirely Cu- and Al- based MHE prototypes. Heat transfer tests were conducted to determine the average rate of heat transfer from heater blocks placed outside the MHEs to water flowing within the microchannel arrays. At the same Reynolds number, the presently measured Nusselt number values are higher than those previously obtained from smoother Cu microchannels. It is speculated that the presence of significant surface roughness within the microchannel arrays resulted in increased flow mixing and thus the observed higher heat transfer rate. Enhancement in heat transfer was demonstrated for both the Cu and Al MHE specimens. Rapid cooling of the MHE specimen bulk can be achieved by running water through the microchannels.

Our present results on fabrication, assembly, and heat transfer characteristics of Cu- and Al- based MHE prototypes illustrate the potential of metal-based MHEs in many heat transfer applications, the fully realization of which awaits future exploration.

7.5 Nomenclature

$A$ - Area, m$^2$
$c_p$ - Specific heat, kJ/kg·K
$D_h$ - Microchannel hydraulic diameter, µm
$f$ - Darcy friction factor
$h$ - Heat transfer coefficient, W/m$^2$·K
$H$ - Channel height, µm
$K$ - Thermal conductivity, W/m·K
$L$ - Channel length, µm
$Nu$ - Nusselt number
$\Delta P$ - Differential pressure, Pa
$p$ - Microchannel perimeter, µm
$\dot{Q}$ - Volumetric flow rate, m$^3$/s
$q$ - Power, W
$Re$ - Reynolds number
$Rz$ - Peak-to-valley roughness, µm
$T$ - Temperature, °C
$\bar{V}$ - Average velocity of water, m/s
$W$ - Channel width, µm
$x^+$ - Dimensionless entrance length
Greek symbols
\( \varepsilon \) - Roughness parameter, \( \mu m \)
\( \rho \) - Density, \( \text{kg/m}^3 \)
\( \tau \) - Time constant, s
\( \mu \) - Viscosity, N·s/m²

Subscripts
\( \text{app} \) - Apparent
\( \text{avg} \) - Average
\( f \) - Fluid
\( \text{fd} \) - Fully developed
\( i \) - Inlet
\( m \) - Mean
\( \text{max} \) - Maximum
\( \text{min} \) - Minimum
\( o \) - Outlet
\( s \) - Supply channel
\( w \) - Wall

7.6 References


CHAPTER 8.  FURTHER ANALYSIS OF FLOW AND HEAT TRANSFER CHARACTERISTICS IN Cu-BASED MICROCHANNEL DEVICES *

8.1 Introduction

In the previous chapters, fabrication, bonding, and assembly of Al- and Cu- based microchannel devices were introduced in detail. Microchannels created in Al and Cu plates by compression molding were bonded through solid-state bonding techniques to Al and Cu plates, respectively, to form all-Al and all-Cu, enclosed, microchannel devices 1, 2, 3, 4. Examination of bonding mechanisms was accomplished through focused ion beam sectioning and scanning electron microscopy 5. The effectiveness of metal-based MHEs in accomplishing solid-to-fluid heat transfer needs to be confirmed by experimentation. Preliminary heat transfer testing was performed on all-Al and all-Cu MHEs. In these experiments, a constant heat flux was supplied via cartridge heaters mounted on one side of the MHE device 6.

Accurate measurements of the solid-to-fluid heat transfer rate require an accurate estimate of the solid wall temperature of the microchannel array. Relatively large temperature gradients can be induced within the body of the metal-based MHEs during constant heat flux testing, making the estimate of solid wall temperature less reliable 6. In chapter, an alternative, constant solid surface temperature testing configuration was adopted for the measurement of heat transfer rates. The present testing configuration enabled more accurate heat transfer rates to be obtained. Fluid flow within long microchannels incurs large pressure drops, while entrance length effects may influence the overall heat transfer when shorter microchannels are employed. Designs of functional metal-based MHEs need to consider both fluid flow and heat transfer so microchannel geometries with optimized heat transfer performance and minimal fluid flow penalties can be reached. In the present paper, fabrication and assembly of all-Cu MHEs are reported together with results of fluid flow and heat transfer measurements. Entrance length effects were considered in analyzing fluid flow and heat transfer rate data obtained from all-Cu MHE prototypes. Effects of microchannel surface roughness on fluid flow and heat transfer were considered as well.

8.2 Experimental Procedures

8.2.1 Molding Replication of Cu-Based Microchannels

Microscale mold inserts were fabricated from Ni-based superalloy Inconel X750® plates 7. As-received Inconel plates were machined to square insert blanks, with an active area of ~20000µm×~20000µm, ~3200µm in height. Insert fabrication from the blanks involved three main steps: micro electrical discharge machining (µEDM) of the active area, electrochemical polishing (ECP) of as-machined microscale Inconel features, and deposition of a conformal amorphous silicon nitride (a-Si:N) coating over electrochemically polished microscale features. A scanning electron microscopy (SEM) overview of one such a-Si:N coated Inconel insert is shown in Fig. 8.1(a). The thickness of the a-Si:N coating is ~600nm. The active surface of this insert contains 26 parallel rectangular microprotrusions, with a center-to-center spacing of ~750µm. The average width of all microprotrusions, as measured from SEM micrographs, is

* Reprinted by permission of “Journal of Micromechanics and Microengineering”
~178µm. The height of the straight section of the microprotrusions is ~400µm. Figure 1(b) shows a close-up SEM image of a typical microprotrusion. The numerous, micron and submicron scale features on the microprotrusion surface are a typical result of the combined µEDM and ECP processing. The thin a-Si:N coating conforms to the insert surface. For example, the roughness of Al coupon surfaces was measured by non-contact optical profilometry (OP) with a Wyko NT3300 instrument at randomly chosen locations. The average values of Al coupon surface roughness, $R_z$, were obtained respectively through OP measurements as 3.0 and 2.9µm, before and after a-Si:N coating deposition. Consider a typical measurement scatter of ~1µm, such results indicate that the conformal coating does not alter the surface morphology significantly.

Fig. 8.1 SEM examination of the a-Si:N coated Inconel mold insert: (a) an overview; (b) a high magnification view of a typical rectangular microprotrusion on the mold insert.

Cu 110 (99.9+wt.% Cu) coupons, ~42mm×~42mm square by ~6.4mm in thickness, were polished with silicon carbide papers of various grit sizes, finished with 1µm diamond suspension, and molded with the a-Si:N coated Inconel insert shown in Fig. 1. Compression molding was carried out on a MTS858 single-axis testing system interfaced to a high-vacuum chamber. The vacuum chamber housed two heating stations, one for the Cu coupon and the other for the mold insert. Molding of Cu coupons occurred with both the coupon and the insert heated to ~500°C. The molding process replicated patterns on the insert into an array of 26 parallel rectangular microchannels on the Cu coupon, with an average width of ~178µm and depth of ~400µm. Further details on mold insert fabrication, coating deposition, and protocols for molding replication have been reported elsewhere $^6,8,9,10$. The present choice of channel dimensions is not optimal in terms of thermal performance. Future studies regarding the effects of altering the channel aspect ratio H/W needs to be carried out. Optimization of channel width and spacing to maximize overall heat transfer performance requires a balance between solid-fluid heat transfer and thermal conduction within the solid, and requires further study as well.

8.2.2 Bonding and Assembly of Cu-Based MHEs

One fluid supply plenum and one fluid drain plenum were further machined using µEDM onto a blank Cu coupon and a molded Cu coupon to provide fluidic connections to replicated
microchannel arrays. Flat stainless steel sheets with a thickness of 1100µm were used as blade electrodes for µEDM. Six consecutive cuts on each end of the microchannel array with partial overlap were performed to create the fluid supply and drain plena. Depth of cuts, ~1.5mm, were kept the same for all cuts on both the supply and drain sides. Surfaces of both the molded coupon and the blank coupon were lightly polished with 1µm diamond suspension after the µEDM operation. Figure 8.2(a) shows a typical pair of molded and blank Cu coupons, with the fluid supply and drain plena already machined into them.

![Fig. 8.2 Fabrication of the Cu-based MHE specimen: (a) an overview of a typical pair of Cu coupons, one molded and one blank, containing one fluid supply plenum and one fluid drain plenum. An array of parallel rectangular microchannels exists between the supply and drain plena on the molded coupon. Numbers shown on the ruler are in mm; (b) a detailed view of one fluidic plenum on the molded Cu coupon; (c) a high magnification view of one typical replicated Cu microchannel.](image)

As shown in Fig. 8.2(a), mechanical drilling was used to make two large through-holes and one small through-hole within each plenum. The through-holes were placed symmetrically with respect to the microchannel array. All large through-holes were mechanically tapped from the coupon back side, away from the microchannel array, to allow external fluid connections using plastic adaptors. The choice of the 6.4mm coupon thickness accommodated the placement of taps. The small through-hole within each plenum connected to a small stainless steel tube,
which in turn provided connection for either a digital manometer or a thermocouple. Figure 8.2(b) provides a more detailed view of one fluidic plenum on the Cu coupon. The plenum has the mottled surface morphology typical of structures cut by µEDM, and provided transitions from the plenum to the microchannels without obstructions at the entrances. Figure 8.2(c) shows a high magnification view of one typical replicated Cu microchannel. The microchannel has vertical sidewalls and sharp sidewall-to-bottom transitions. Micron scale surface roughness is clearly visible on the sidewall and bottom of the microchannel. The bottom roughness appears random, while the sidewall roughness exhibits clear streaking in the molding direction, resulting from the motion of the insert into the molded material. The roughness on the microchannel bottom was obtained via direct OP imaging. The roughness on the microchannel sidewall was obtained by mechanical cutting along the microchannel followed by mechanical polishing to reveal one side of the microchannel prior to OP imaging.

Fig. 8.3 Optical profilometry images obtained from typical replicated Cu microchannels: (a) bottom surface; (b) sidewall surface. The image has a ninety degree rotation such that the molding direction is from the left to the right.

Figures 8.3(a) and 8.3(b) show respectively OP images obtained from typical replicated Cu microchannel bottom and sidewall surfaces. A quantitative evaluation of the surface roughness is expressed in the value of the peak-to-valley roughness $R_z$. Three independent OP images, analogous to that shown in Fig. 8.3(a), were obtained from the bottom surfaces of three different microchannels in the replicated Cu coupon. Three analogous OP images were obtained from the microchannel sidewall surfaces. Values of $R_z$ obtained from the three OP images were averaged. The average $R_z$ values are $\sim 4.7 \mu m$ and $\sim 4.5 \mu m$ for the bottom and sidewall surfaces, respectively. As expected for the replication process, surface roughness on the microchannel bottoms mimics the roughness present on the top surface of the insert microprotrusions shown in Fig. 8.1(b), which resulted from preferential etching during the ECP step of insert fabrication. The large-strain plastic deformation within the molded metal may lead to different surface roughness for the microchannel sidewalls. Typical variation in microchannel surface roughness was assessed by OP measurements on different Cu coupons molded by the same Inconel insert. On each coupon, measurements of $R_z$ were performed at randomly chosen locations on the microchannel bottom and sidewall surfaces. Results of such measurements on several Cu coupons showed $R_z$ values from 3 to $5 \mu m$. 

111
Bonding experiments were also carried out on the MTS858 system interfaced to the vacuum chamber. Molded and blank Cu coupons, as shown in Fig. 8.2(a), were placed face to face on the bottom heating station with a thin Al foil (Al1100, 99%+, ~10 µm in thickness) inserted in the middle. After the chamber was evacuated, both heating stations were heated. Bonding of Cu coupons was carried out at a temperature of ~575°C with an applied pressure of ~3MPa. Further details on the Cu bonding protocol and evaluation of bond quality have been given elsewhere 3. Figure 8.4 shows an optical overview of one bonded Cu MHE prototype, with plastic external fluid adaptors attached.

8.3 Fluid Flow and Heat Transfer Measurements

8.3.1 Experimental Setup

Similar testing apparatus that introduced in chapter 7 was designed and built to evaluate the fluid flow and heat transfer characteristics of the assembled Cu MHE prototype. Figure 8.5(a) shows a schematic of the testing apparatus, which consists of three sections: water supply section, test section, and data acquisition section. The water supply section consisted of a pressure-regulated water storage tank. The tank was pressurized by high-pressure, pure (99.9%+, nitrogen gas, and supplied water to the MHE specimen at a constant pressure and provided a smooth and stable flow through the microchannels at low flow rates. A valve placed downstream of the tank exit was used to make fine adjustments to the flow rate. The placement of the water outlet tube deeper in the tank as compared to the height of the nitrogen inlet tube ensures that no gas bubble would enter the water supply to the testing section.

For the test section, the Cu MHE prototype was completely immersed in an external water bath contained in a 2000ml glass beaker placed on a hot plate. Two small stainless steel tubes were inserted directly into the small through-holes in the fluid supply and drain plena of the Cu MHE and sealed with epoxy. The differential pressure across the water inlet and outlet was measured with a Dywer digital manometer connected to the two stainless steel tubes, with a measurement sensitivity of 0.1 psi (690Pa). The total water flow across the microchannel array was obtained by measuring the volume of water collected at the exit end over a fixed period of
time. The characteristics of water flow across the microchannel array were quantified by measuring the total volumetric flow rate versus the pressure drop, with the external water bath maintained at room temperature.

![Experimental setup](image)

Fig. 8.5 Experimental setup for heat transfer measurements: (a) a schematic of the testing apparatus; (b) a schematic of the thermocouple fixture; (c) a schematic cross-sectional view of the test section, together with thermocouple locations (shown as black dots). Symbols A and B denote thermocouples inserted into the supply and drain plena through the external fluidic connecters and the stainless steel tubes, respectively.

During heat transfer testing, the external water bath was heated and continuously stirred, and provided a constant-temperature environment surrounding the entire Cu MHE specimen. Thermocouples, 36-gauge and K-type, were used to measure the inlet and outlet water temperatures. Within each plenum, two thermocouples were inserted through the two plastic connecters into the plenum. One additional thermocouple was inserted through the stainless steel tube into the small through-hole connecting to the plenum. Thermocouple insertion into the tube was accomplished with the help of a T-fitting and then sealed with epoxy, as shown schematically in Fig. 8.5(b). To measure the solid wall temperature of the Cu MHE, two small holes were drilled symmetrically from two sides of the Cu device into the body center of the Cu coupon close to the microchannel, perpendicular to the flow direction and parallel to the microchannel array. Two thermocouples were inserted into these two holes and sealed with...
epoxy. The distances between the thermocouple tips to the microchannel array were ~1.5mm. The schematic cross-sectional view of this testing section, together with thermocouple locations, is indicated in Fig. 8.5(c).

In the data acquisition section, an Instrunet data acquisition system interfaced to a PC was used to collect the thermocouple readings. All thermocouples were calibrated with a two-point calibration, using a mixed ice/water bath and boiling water as the reference points. Temperature deviations at the two calibration temperatures were ±0.1°C in all cases.

8.3.2 Measurements

Heat transfer measurements were performed with the Cu MHE specimen immersed in the external water bath. Measurements were performed at different water pressure drops and volumetric water flow rates, which ranged respectively from $3.5 \times 10^3$ to $2.4 \times 10^5$Pa (0.5 to 35psi) and 0.06 to 0.90liter/min. Depending on the water flow rate within the MHE specimen, the external water bath temperature varied, from 60 to 85°C, because a different amount of heat was being removed. For each experimental condition, the external water bath temperature was uniform and constant throughout the experiment.

Water within the supply and drain plena undergoes further heating from the plena surfaces. The two thermocouples inserted through the plastic connectors (A) are farther from the microchannel array, while the thermocouple inserted through the stainless steel tube (B) was placed the closest to the entrance/exit of the microchannel array. Consistently, we observe the readings from the B thermocouple to be 2-4°C higher than the readings from the A thermocouples on the supply side. Similarly, the readings from the A thermocouples was 0.5-1.0°C higher than the reading from the B thermocouple on the drain side. The readings from the B thermocouples were therefore taken as the water inlet and outlet temperatures. In so doing, the difference between water outlet and inlet temperatures arise approximately solely from heating from surfaces of the microchannel array, thus eliminating heating contributions from the supply and drain plena surfaces. For all the experiments, the difference between outlet and inlet water temperatures ranged from 6.4 to 28.3°C. At the maximum water flow rate of 0.90liter/min, the rate of heat removal due to water flow is ~420W. The mean water temperature, defined as the arithmetic average of the water outlet and inlet temperatures, $T_m = (T_o + T_i) / 2$, ranged from 30 to 44°C. Because it is difficult to measure the exact solid wall surface temperature in the current experimental configuration, $T_s$ was estimated by taking the average reading of the two thermocouples inserted into the MHE body. In all cases, difference in readings from these two thermocouples is less than 1.1°C. Thus the current, constant solid surface temperature testing configuration enables a more accurate estimate of $T_s$. For all the experiments, the values of $T_s$ so estimated ranged from 40 to 62°C.

Final examination of assembled Cu MHE prototype was accomplished by mechanically cutting them in directions perpendicular to the microchannel arrays after heat transfer measurements. The cuts were placed within the drain plenum. To fully reveal the cross sectional configurations of the bonded microchannels, the cut pieces containing the microchannel arrays were mechanically polished back close to the microchannel exits. Figure 8.6(a) shows a cross-sectional view of a portion of the bonded Cu microchannel structure. Water was passed from the fluid supply plenum through the microchannel arrays. Microscale water jets exiting the microchannels were observed on the drain plenum side to ensure that each microchannel has remained intact during the bonding process. The replicated Cu microchannel array contains 26
rectangular microchannels. Observations showed exit water jets from all 26 microchannels in the assembled Cu MHE. Figure 8.6(b) shows a higher magnification view of two bonded microchannels in the Cu device. To obtain the exact dimensions of bonded Cu microchannels, measurements of microchannel width $W$ and height $H$ were performed from micrographs similar to Fig. 8.6(b) on each and every microchannel for the Cu MHE specimen. Measured microchannel widths and heights were averaged and shown in Table 8.1. Measurements of microchannel length were performed from SEM images obtained from the molded Cu coupon, after the fluid supply and drain plena were created by µEDM but before bonding to the blank Cu coupon. The hydraulic diameter of the microchannel, $D_h$, is equal to $4A_c / p$ with $A_c = WH$ and $p = 2(W + H)$ being respectively the microchannel cross-sectional area and perimeter. The average hydraulic diameter for the microchannel array was calculated from the average microchannel dimensions and shown also in Table 8.1.

![Fig. 8.6 Destructive examination of one assembled Cu MHE specimen: (a) a cross-sectional view of a portion of the bonded microchannel structures; (b) a higher magnification view of two bonded microchannels.](image)

Table 8.1. Average microchannel dimensions of the assembled Cu MHE specimen, from which the heat transfer data was obtained.

<table>
<thead>
<tr>
<th># of channels</th>
<th>$H$ (µm)</th>
<th>$W$ (µm)</th>
<th>$L$ (µm)</th>
<th>$D_h$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>26</td>
<td>341±5</td>
<td>178±5</td>
<td>17320±50</td>
<td>234±5</td>
</tr>
</tbody>
</table>

The experimental uncertainty for each measured parameter was estimated. The uncertainties of microchannel dimension measurements, $δH$, $δW$, and $δL$, are 5µm, 5µm, and 50µm, respectively. Volumetric water flow rate was measured by collecting a specific volume of water over a fixed time duration. The total volume of water collected was >300ml in all cases and the uncertainty of volume reading is 2.5ml. The resulting uncertainty in measurements of the volumetric flow rate $\dot{Q}$ is better than 0.8%. The uncertainty for each thermocouple reading is 0.25°C or less. This uncertainty in temperature measurements was used to estimate uncertainties of quantities such as $T_o-T_i$, etc. The experimental uncertainties in the measurements were estimated using the same equations described in chapter 7.
8.4 Results and Discussion

8.4.1 Data Reduction

8.4.1.1 Flow Characteristics and Friction Factor

The average velocity of water flowing through the microchannels was determined from the volumetric flow rate of water, the total number of channels in the microchannel array, and the channel cross-sectional area,

\[ V_{avg} = \frac{\dot{Q}}{N_c A_c}. \]  

(1)

In the present case, \( N_c = 26 \). The Reynolds number was then calculated from

\[ \text{Re} = \frac{\rho V_{avg} D_h}{\mu}, \]  

(2)

where values for the dynamic viscosity of water, \( \mu \), and the density of water, \( \rho \), were taken respectively as those at 298K. The uncorrected Darcy friction factor was determined from

\[ f_{uncorr} = \frac{2D_h}{\rho L V_{avg}^2}, \]  

(3)

where \( \Delta P_{i-o} \) is the experimentally measured water pressure drop across the microchannel array. This measured pressure drop includes pressure losses when fluid enters the microchannels from the supply plenum and exits the microchannels into the drain plenum. A correction for this pressure drop is therefore necessary to obtain the true friction factor. However, the pressure loss coefficients associated with inlet into and exit from the microchannels depend significantly on the precise entrance/exit geometry, which is difficult to characterize and not measured in detail. The precise values of these pressure losses are therefore unknown. The choice was therefore made to present measured values of \( f_{uncorr} \), as calculated from Eq. (3), which quantifies the resistance to fluid flow through the microchannel array. All raw data points were included in the conversion from flow rate and pressure drop to Reynolds number and friction factor values.

8.4.1.2 Heat Transfer Characteristics and Nusselt Number

The rate of total heat transfer from the solid Cu body to the water flowing through the microchannel array within the Cu MHE was calculated from

\[ \dot{q}_{tot} = \dot{m} c_p (T_o - T_i). \]  

(4)

The inlet water temperature \( T_i \) and outlet water temperature \( T_o \) were obtained respectively from the B thermocouples inserted through the stainless steel tubes into the small through-holes connecting to the supply and drain plena. The temperature readings from these thermocouples best represent the actual water temperature entering and exiting the microchannels. In Eq. (4), the water mass flow rate \( \dot{m} \) was calculated from measured \( \dot{Q} \) values, taking respectively the values of \( c_p \) and \( \rho \) as those corresponding to \( T_m \) in the experiment. The average heat transfer coefficient for a single microchannel was calculated from

\[ h_{avg} = \frac{\dot{q}_{tot} / N_c}{A_c \Delta T_{lm}}, \]  

(5)
where $A_s$ is the surface area of one microchannel. In Eq. (5), the log-mean temperature difference is defined as

$$\Delta T_{\text{lm}} = \frac{\Delta T_o - \Delta T_i}{\ln \left( \frac{\Delta T_o}{\Delta T_i} \right)}$$

(6)

where $\Delta T_o$ and $\Delta T_i$ are respectively $T_o - T_i$ and $T_s - T_i$, i.e., the differences between the temperature of the solid wall surface and the water outlet and inlet temperatures. The complete immersion of the Cu MHE specimen into the external water bath creates a testing condition that approximates a constant surface temperature boundary condition, which leads to an exponentially decaying difference between the wall temperature and the mean fluid temperature. Under such a testing condition, the log-mean temperature difference is the appropriate one to use for the calculation of the average heat transfer coefficient $h_{avg}$

$${\text{If } T_s - T_m}$$

were used in Eq. (5) instead, $h_{avg}$ would be underestimated by 4-15% for the present test conditions. The average Nusselt number for a single microchannel was calculated from $h_{avg}$ as

$$Nu_{\text{avg}} = \frac{h_{avg}D_h}{K_f}$$

(7)

where the value of the water thermal conductivity $K_f$ is taken as that corresponding to $T_m$. The average Nusselt number quantifies the heat transfer efficiency between the solid MHE body and the working fluid flowing through the microchannel array. All raw data points were included in the conversion from temperature and flow rate measurements to Reynolds number and Nusselt number values.

### 8.4.2 Data Analysis and Discussion

#### 8.4.2.1 Flow Characteristics and Friction Factor

Values of $f_{uncorr}$ for the Cu MHE specimen, whose dimensions are shown in Table 1, are plotted against the Reynolds number in Fig. 8.7. The calculation of error bars on $f_{uncorr}$ considered measurement uncertainties in microchannel dimensions $W$, $H$, and $L$, water volumetric flow rate $\dot{Q}$, and water pressure drop $\Delta P_{i-o}$, assuming all uncertainties are independent and uncorrelated. Measured friction factor decreases monotonically with increasing $Re$. The maximum uncertainties for $Re$ and $f$ values extracted for the Cu MHE specimen were ~4% and ~14%, respectively.

For fully developed laminar flow in smooth rectangular channels, an approximation to an analytically derived expression for the friction factor is given as

$$f_{\text{fd}} \approx 96\left(1 - 1.3553\alpha + 1.9467\alpha^2 - 1.7012\alpha^3 + 0.9564\alpha^4 - 0.2537\alpha^5\right)$$

(8)

where $0 < \alpha = W / H < 1$ is the channel aspect ratio. For the particular Cu MHE specimen, whose friction factor data are shown in Fig. 8.7, $\alpha = 0.522$. Thus in this case $f_{\text{fd}} \approx 61.65$, according to Eq. (8).

Since the microchannels tested here may contain a considerable portion of their length that is in the hydrodynamically developing region for laminar flow, a correlation for an apparent friction factor that includes entrance effects in laminar flow was also used for comparison.
Defining a dimensionless entrance length as \( x^+ = L/(D_n \, Re) \), an expression for an apparent friction factor \( f_{\text{app}} \), which takes entrance length effects into account, is given by Shah and London as \(^{14}\)

\[
f_{\text{app}} \, Re = \left[ \left\{ \frac{3.2}{(x^+)^{0.57}} \right\}^2 + \left( f_{\text{lam,fd}} \, Re \right)^2 \right]^{1/2}.
\] (9)

In the limit of long channels with \( x^+ \) approaching infinity, \( f_{\text{app}} \) approaches \( f_{\text{lam,fd}} \) and is independent of \( L \). In the short channel limit, \( f_{\text{app}} \) is \( L \) dependent. Values of \( f_{\text{lam,fd}} \) and \( f_{\text{app}} \), as calculated from Eqs. (8) and (9) using parameters corresponding to the Cu MHE specimen in question, are shown in Fig. 8.7 as a function of the Reynolds number in the range 250 < \( Re < 1000 \). It is evident from Fig. 8.7 that, in this \( Re \) range, little difference exists between \( f_{\text{lam,fd}} \) and \( f_{\text{app}} \), implying that entrance length effects do not significantly alter the friction factor for the present channel geometry. It is also evident that reasonable agreement exists between the calculated laminar flow friction factors and values of \( f_{\text{uncorr}} \), with the inference that flow within the microchannels in this range of Reynolds numbers is described reasonably well by laminar flow calculations.

Fig. 8.7 Data on water flow through and pressure drop across microchannel arrays: Darcy friction factor versus Reynolds number for two Cu MHE specimens and one smooth, circular, stainless steel tube of 1mm in diameter. In this graph, calculations based on fully developed and entrance length effects included laminar flows are shown as \( f_{\text{fd}} \) and \( f_{\text{app}} \), respectively. Values calculated for the Haaland correlation for turbulent flow through both smooth and rough pipes are also included in this graph.

At \( Re \sim 2000 \), the value of \( f_{\text{uncorr}} \) is \( \sim 0.06 \). The Haaland correlations for the friction factor in fully developed turbulent flow within a tube \(^{11}\)
\[
\frac{1}{\sqrt{f}} = -1.8\log \left[ \left( \frac{\varepsilon / D_h}{3.7} \right)^{1.11} + \frac{6.9}{\text{Re}} \right],
\]

are also plotted in Fig. 8.7 for \( \text{Re} > 2000 \). In Eq. (10), \( \varepsilon \) is the roughness parameter. Two cases are plotted, corresponding respectively to values of \( \varepsilon \) being 0 and 4.6\( \mu \)m. The latter \( \varepsilon \) value corresponds to the average value of \( R_z \) measured from the microchannel bottom and sidewall surfaces of the Cu MHE specimen. In the turbulent flow regime, the hydrodynamic entrance length effects on friction factor are neglected in this analysis. Results of friction factor measurements obtained from one additional Cu MHE specimen, with dimensions similar to that shown in Table. 8.1, are also summarized in Fig. 8.7, again in the form of \( f_{\text{uncorr}} \) versus \( \text{Re} \). Using the same experimental setup, friction factor measurements were conducted on a smooth, circular, stainless steel tube with an inner diameter of 1\( \text{mm} \) and a length of 40cm, the results of which are also shown in Fig. 8.7. Measured friction factors for the circular tube show good agreement with the laminar flow expression of \( f = 64/\text{Re} \) at \( \text{Re} < 1000 \) and reasonable agreement with the Haaland correlation for smooth tubes at \( \text{Re} >2500 \). The observed deviations from the laminar flow expression and the Haaland correlation in the transition region, \( 1000< \text{Re} < 2500 \), is well documented in the literature. Results of flow measurement on the stainless steel tube lend confidence to the flow characteristic measurements in microchannels. Friction factor data measured from two separate Cu MHE specimens, shown in Fig. 8.7, illustrate typical experimental scatter for flow measurements on microchannel arrays, and show that independently fabricated Cu MHE devices with similar geometric dimensions exhibit similar flow characteristics. Values of \( f_{\text{uncorr}} \), obtained from the two Cu MHE devices, appear to be closer to the Haaland correlation values calculated for 4.6\( \mu \)m roughness and lie above those obtained from the smooth stainless steel tube. It is suggested that a major contributor to this friction factor increase is the surface roughness present within the microchannels.

### 8.4.2.2 Heat Transfer Characteristics and Nusselt Number

Values of the average Nusselt number, \( \text{Nu}_{\text{avg}} \), calculated from Eqs. (5-7), are plotted against the Reynolds number in Fig. 8.8. The calculation of error bars on \( \text{Nu}_{\text{avg}} \) considered measurement uncertainties on temperatures \( T_s, T_o, \) and \( T_i, \) water volumetric flow rate \( \dot{Q} \), and microchannel dimensions \( W, H, \) and \( L, \) again assuming all uncertainties are independent and uncorrelated. The relative error in \( \text{Nu} \) increases with increasing \( \text{Re} \). The maximum uncertainty for \( \text{Nu} \) values extracted for the Cu MHE specimen was \( \sim 16\% \). With increasing water flow rate, the difference in water outlet to inlet temperatures decreases, thereby increasing the relative error in \( \Delta T \) values and leading to larger relative errors for \( \text{Nu} \).

Known Nusselt number correlations are compared to the experimental data. For laminar flow, the Hausen correlation \(^{12}\), valid for a constant surface temperature and thermally developing flow, is given by

\[
\text{Nu}_{\text{Hausen}} = 3.66 + \frac{0.0668(D_h / L) \text{Re Pr}}{1 + 0.04[(D_h / L) \text{Re Pr}]^{2/3}},
\]

where the Prandtl number for water, \( \text{Pr} = c_p\mu / K_f \), characterizes the ratio of the momentum and thermal diffusivities. The Sieder-Tate correlation \(^{12}\), valid for a constant surface temperature and combined hydrodynamically and thermally developing flow, is given by
\[
Nu_{\text{Sieder-Tate}} = 1.86 \left( \frac{\text{Re Pr}}{L/D_h} \right)^{1/3} \left( \frac{\mu}{\mu_s} \right)^{0.14}.
\]

All quantities in Eqs. (11) and (12) are evaluated at the mean temperature \(T_m\) except the value of \(\mu_s\), which is evaluated at the solid wall temperature \(T_s\). Nusselt numbers calculated from the Hausen and Sieder-Tate correlations, as expressed in Eqs. (11) and (12), are plotted in Fig. 8.8 in the Reynolds number range of \(Re < 1500\). The difference between Hausen and Sieder-Tate correlations stems partly from the fact that the Sieder-Tate correlation was derived for experimental conditions where temperature differences are large \(^{12,16}\). It is evident from Fig. 8.8 that experimental \(\bar{Nu}_{\text{avg}}\) values significantly exceed the Hausen and Sieder-Tate correlation values at \(250 < Re < 1500\). Both the Hausen/Sieder-Tate correlations and the experimental \(\bar{Nu}_{\text{avg}}\) values increase with increasing \(Re\), with the experimental data increasing at a faster rate. Consistent with the present results, such a general trend was observed in the previous measurements on Cu- and Al-based MHEs using a constant heat flux testing methodology \(^6\).

For turbulent flow, the Dittus-Boelter correlation, with a recommended application range of \(2500 < Re < 1.24 \times 10^5\), is given by \(^{12,17,18}\)
\[
Nu_{\text{Dittus-Boelter}} = 0.023 \text{Re}^{4/5} \text{Pr}^{2/5}.
\]

Fig. 8.8 Data on heat transfer characteristics of the Cu MHE device: Nusselt number versus Reynolds number. Conventional correlations for smooth pipes based on simultaneously developing (Sieder-Tate) and thermally developing (Hausen) laminar flows and the Dittus-Boelter and Petukhov correlations for turbulent flows are shown in the graph. Corrected Dittus-Boelter and Petukhov correlations considering entrance length effect and surface roughness effect are also included in this graph.
The Petukhov correlation, with a recommended application range of $4000 < \text{Re} < 5 \times 10^6$, is given by

$$
\text{Nu}_{\text{Petukhov}} = \frac{(f/8) \text{Re} \text{Pr}}{C + 12.7(f/8)^{1/2} (\text{Pr}^{2/3} - 1)}.
$$

The values of $Re$ and $Pr$ in Eqs. (13) and (14) are evaluated at the mean temperature $T_m$. The parameter $C$ in Eq. (14) is given by $C = 1.07 + 900/\text{Re} - [0.63/(1 + 10 \text{Pr})]$. The Petukhov friction factor correlation, $f = (0.79 \ln \text{Re} - 1.64)^2$, is used to calculate values of $f$ in Eq. (14)\textsuperscript{15}. Nusselt numbers calculated from the Dittus-Boelter and Petukhov correlations, as expressed in Eqs. (13) and (14), are plotted in Fig. 8.8 in the Reynolds number range of $2000 < \text{Re} < 3000$. It is evident that these two correlations yield nearly identical $Nu$ values in this Reynolds number range. While the trend of monotonically increasing $Nu$ with increasing $Re$, as observed in the present measurements, is reasonably well captured by the Dittus-Boelter and Petukhov correlations, measured $\text{Nu}_{\text{avg}}$ values significantly exceed the Dittus-Boelter and Petukhov correlation values.

The conditions under which the Dittus-Boelter and Petukhov correlations were derived do not match the actual experimental conditions. Both correlations apply to fully developed flow. To account for thermal entrance length effects, which may be present in the microchannels, a correlation by Al-Arabi was used\textsuperscript{12,15,18}. This correlation is expressed as

$$
\frac{\text{Nu}_{\text{avg}}}{\text{Nu}_{\text{fd}}} = 1 + \frac{C}{L/D_h} = \beta_{\text{dev}},
$$

where $\text{Nu}_{\text{fd}}$ is the Nusselt number in the fully developed region and the parameter $C$ is given by $C = (L/D_h)^{0.11}(0.68 + 3000/\text{Re}^{0.81})/\text{Pr}^{1/6}$. This correlation allows the Dittus-Boelter and Petukhov correlations to be adjusted to include the effects of the thermal entrance region. As evident from Eq. (15), this adjustment yields higher values of $\text{Nu}_{\text{avg}}$ as compared to $\text{Nu}_{\text{fd}}$.

In addition to thermal entrance length effects, the influence of microchannel surface roughness on heat transfer needs to be considered. The inclusion of the friction factor in the Petukhov correlation for the Nusselt number offers the possibility of considering Nusselt number enhancement from surface roughness effects: increasing surface roughness would lead to increases in the friction factor and the Nusselt number. To render an explicit estimate for the influence of surface roughness, two sets of Petukhov correlation values were re-calculated using friction factor values obtained from the Haaland correlation, expressed in Eq. (10), assuming $\varepsilon$ values of 0 and $4.6 \mu m$, respectively. When $\varepsilon = 0$ is assumed, calculated Petukhov correlation values are very close to those of the original Petukhov correlation. When $\varepsilon = 4.6 \mu m$ is assumed, calculated Petukhov correlation values are higher due to increases in the friction factor.

To adjust the Dittus-Boelter equation for surface roughness effects, a correlation by Norris was used\textsuperscript{16}. This correlation is expressed as

$$
\frac{\text{Nu}_{\text{rough}}}{\text{Nu}_{\text{smooth}}} = \left( \frac{f_{\text{rough}}}{f_{\text{smooth}}} \right)^{0.68 \text{Pr}^{0.215}} = \beta_{\text{rough}}.
$$

Consistent with the estimate of surface roughness effects on the Petukhov correlation described above, values for the Haaland friction factor correlation, assuming respectively $\varepsilon$ values of 0 and $4.6 \mu m$, were used for the calculation of the Norris correlation. Because $f_{\text{rough}} > f_{\text{smooth}}$, $\beta_{\text{rough}}$ is greater than 1.
Taking both the effects of thermal entrance length and surface roughness into account, the corrected equations for the Petukhov correlation and the Dittus-Boelter correlation can be expressed respectively as

\[ \text{Nu}_{\text{Petukhov,corr}} = \frac{(f/8) \text{RePr}}{C + 12.7(f/8)^{1/2} (\text{Pr}^{2/3} - 1)} \beta_{\text{dev}} \]  \hspace{1cm} (17)

and

\[ \text{Nu}_{\text{Dittus-Boelter,corr}} = 0.023 \text{Re}^{4/5} \text{Pr}^{2/5} \beta_{\text{dev}} \beta_{\text{rough}} \]  \hspace{1cm} (18)

In Eq. (17), the \( f \) values correspond to those calculated from the Haaland correlation assuming \( \varepsilon = 4.6 \mu\text{m} \). Calculated values of \( \text{Nu}_{\text{Petukhov,corr}} \) and \( \text{Nu}_{\text{Dittus-Boelter,corr}} \) are shown in Fig. 8.8 as well. It is clear from Fig. 8.8 that corrected Petukhov and Dittus-Boelter correlations match the measured \( \text{Nu}_{\text{avg}} \) data much better than their uncorrected counterparts, with the corrected Dittus-Boelter correlation showing good agreement with the experimental data. While the uncertainties in using the Haaland friction factor correlation in the calculation of various corrections makes the exact magnitude of the agreement less meaningful, it is believed that the trends as depicted by these corrections are significant. These trends illustrate the need to consider surface roughness and entrance length effects in analyzing the current set of flow and heat transfer data, as well as the need to ensure that correlations used for comparison with microchannel heat transfer experiments are appropriate for the given test conditions.

Finally, it is of interest to comment on the overall heat transfer performance of the present Cu-based MHEs in comparison to similar Si-based devices. For the Cu MHE specimen in question, a total power removal of \( \sim 420\text{W} \) was achieved at a water flow rate of \( 0.90\text{liter/min} \). The heat absorption into water caused an outlet-inlet temperature rise of \( 6.4^\circ\text{C} \). With an active microchannel area of \( 2\text{cm} \times 2\text{cm} \), the heat flux removed is \( 105\text{W/cm}^2 \). Assuming a temperature rise of \( 71^\circ\text{C} \) instead, the projected heat flux removed would be \( 1165\text{W/cm}^2 \). This number compares favorably with the classic result of Tuckerman and Pease, who reported a heat flux removal of \( 790\text{W/cm}^2 \) at the same water temperature rise of \( 71^\circ\text{C} \) \(^{19}\). It should also be noted that the geometry of the present Cu MHE specimen has not been optimized. Improved MHE geometry design need to consider solid-fluid heat transfer rate together with bulk thermal conduction within the MHE body, and will be carried out in the future.

### 8.5 Conclusion

All-Cu MHE prototypes were fabricated by combining direct molding replication and solid state bonding. Heat transfer testing of the Cu MHE prototypes was conducted in a constant solid surface temperature configuration, which affords a better estimate of the solid surface temperature and consequently improved heat transfer measurements. The roughness of microchannel surfaces fabricated via molding was characterized quantitatively through optical profilometry. Optical profilometry results were used to give an estimate of heat transfer rate increase due to increased surface roughness through known macroscale correlations. Further consideration was given to thermal entrance length effects on heat transfer. Encouraging agreement between experimental heat transfer data and known macroscale correlations was obtained after the correlations were corrected for thermal entrance length and surface roughness effects. The present results demonstrate the efficacy of metal-based MHEs in accomplishing solid-fluid heat transfer, and provide fluid flow and heat transfer data upon which future MHE designs can be based.
8.6 Nomenclature

\( A \) Area (m\(^2\))
\( c_p \) Specific heat (J/kg·K)
\( D_h \) Microchannel hydraulic diameter (µm)
\( f \) Darcy friction factor
\( h \) Heat transfer coefficient (W/m\(^2\)·K)
\( H \) Channel height (µm)
\( K \) Thermal conductivity (W/m·K)
\( L \) Channel length (µm)
\( \dot{m} \) Mass flow rate of fluid (kg/s)
\( N \) Number
\( Nu \) Nusselt number
\( \Delta P \) Differential pressure (Pa)
\( p \) Microchannel perimeter (µm)
\( Pr \) Prandtl number
\( \dot{Q} \) Volumetric flow rate (m\(^3\)/s)
\( q \) Power (W)
\( Re \) Reynolds number
\( Rz \) Peak-to-valley roughness (µm)
\( T \) Temperature (°C)
\( V \) Velocity of water (m/s)
\( W \) Channel width (µm)
\( x^+ \) Dimensionless entrance length

Greek symbols
\( \alpha \) Microchannel aspect ratio
\( \beta \) Correction factor for Nusselt number correlations
\( \varepsilon \) Roughness parameter (µm)
\( \rho \) Density (kg/m\(^3\))
\( \mu \) Viscosity (N·s/m\(^2\))

Subscripts
\( App \) Apparent
\( Avg \) Average
\( c \) Channel
\( corr \) Corrected
\( dev \) Developing
\( f \) Fluid
\( fd \) Fully developed
\( i \) Inlet
\( lam \) Laminar
\( lm \) Log-mean
\( m \) Mean
\( o \) Outlet
\( s \) Surface
\( tot \) Total
\( uncorr \) Uncorrected
8.7 References


CHAPTER 9. CONCLUSION

Microsystem technologies are believed to be an important part of the contemporary technological foundation and are becoming a commercially significant specialty area in manufacturing. The design and fabrication of microscale engineering structures has the potential of generating revolutionary changes in many products over a wide range of industrial sectors. Many studies on microelectromechanical systems (MEMS) have been done for over two decades and most of them are focusing on Si-based MEMS. The choice of Si is largely due to the fact that fabrication technology for Si-based high-aspect-ratio microscale structures (HARMS) is the most mature and most widely available. Compared to Si-based devices, microdevices based on metals such as Cu and Al possess potential advantages, e.g. higher bulk thermal conductivities and increased mechanical robustness. Early examples of metal-based microdevices exist in the literature, including microchannel heat exchangers (MHEs) and magnetically driven microactuators. Overall, metal-based microdevices and microsystems have not received as much attention in comparison. One important factor responsible for the present paucity of metal-based microdevices and microsystems is the current lack of suitable fabrication techniques for metal-based HARMS. In order for metal-based microdevices to be competitive in the market place, fabrication and assembly of metal-based HARMS should be simple, fast, and reliable. The lack of such fabrication and assembly techniques for metal-based microdevices, coupled with the ready availability of technology for fabrication and assembly of Si-based MEMS, is largely responsible for the absence of metal-based microdevices and microsystems on the current MEMS market.

Metal-based MHEs promise high heat transfer coefficients together with mechanical robustness, and are of interest for a wide range of applications. Fabrication technologies capable of creating HARMS in metals such as Cu at low cost and high throughput are of particular interest. Likewise, simple and reliable bonding and assembly techniques are critical for building functional metal-based microfluidic devices. Among the different diffusional bonding techniques, the use of a chemically/structurally distinct intermediate layer at the bonding interface can lower the bonding temperature, eliminate bulk melting of the parts to be joined, promote interdiffusion, and increase the bond quality. Examples of bonding using eutectic intermediate layers exist in the literature. As one example of successful bonding of metal-based HARMS, we demonstrated bonding of Al-based microchannel structures using sputter co-deposited Al-Ge thin film intermediate layers, as reported in detail in Chapters 2 and 3. Quantitative evaluation of bond strengths was carried out as a function of various bonding parameters. Tensile bond strengths of Al pieces bonded with co-deposited Al-Ge intermediate layers were measured experimentally as a function of bonding temperature and pressure and shown to exceed 75MPa, and reaching as high as 165MPa. Other examples are the successful bonding of Cu-based HARMS using free-standing Al thin foil and Sn thin foil intermediate layers, as reported in Chapters 5 and 6. Tensile bond strengths of bonded Cu pieces were measured experimentally and to be a value of ~30MPa. By using these bonding methods, a few Al- and Cu-based MHE prototypes were successfully bonded and assembled.

Understanding bonding mechanisms and the ability of a particular bonding protocol for sealing microscale features is critical to device functionality and reliability. Scientifically, bonding of metal-based microscale features has not been studied in detail; and these studies will generate new information. Technologically, development of reliable assembly/packaging
technology for metal-based microdevices is a critical enabler for pushing metal-based microsystems from laboratory curiosity to commercial reality. Preliminary examination of the morphology and chemistry of fractured bonding interfaces suggested eutectic bonding in the case of Al and a combination of eutectic and diffusional bonding in the case of Cu. As mentioned above, mechanical testing of bonded specimens was carried out to evaluate interfacial bond strength, and revealed some counter-intuitive trends. Detailed characterizations of the micro-/nano-scale structure of buried bonding interfaces were conducted to rationalize results of mechanical testing. The mechanisms for bonding Al-based HARMS with Al-Ge thin film intermediate layers and for boning Cu-based HARMS with free-standing Al thin foil and Sn intermediate layers were investigated.

Preliminary flow and heat transfer characteristics were also studied on assembled Al- and Cu-based MHE prototypes, as reported in chapters 7 and 8. Two testing configurations, constant heat flux and constant solid surface temperature, were applied in the testing. The results show the increase of surface roughness in the replicated Al- and Cu- microchannels can cause significant improvements to microchannel heat exchanger performance. The surface roughness existing in the replicated microchannels is dependant on the surface roughness of the mold inserts, which can be controlled through the electrochemical polishing (ECP) process. It provides the option of decreasing or increasing surface roughness over the roughness intrinsic to the process used to create the mold insert. Thermal entrance length effects were also considered on heat transfer testing. Encouraging agreement between experimental heat transfer data and known macroscale correlations was obtained after the correlations were corrected for thermal entrance length and surface roughness effects. The present results demonstrate the efficacy of metal-based MHEs in accomplishing solid-fluid heat transfer, and provide fluid flow and heat transfer data upon which future MHE designs can be based.

The overall heat transfer performance of metal-based MHEs is compared to similar Si-based devices. The heat flux removal in present Cu-based MHEs compares favorably with the previous result on Si-based device at the same water temperature rise. It should be mentioned that the geometry of present Al or Cu MHE specimens has not been optimized. Improved MHE geometry design needs to consider solid-fluid heat transfer rate together with bulk thermal conduction within the MHE body, including the microchannel width, spacing between two channels, surface roughness within microchannel, and dimension of entire device. Such study should be carried out in the future.
APPENDIX. LETTERS OF COPYRIGHT PERMISSION

1. Permission of Using Published Material from Microsystem Technologies

From: Permissions Europe/NL<Permissions.Dordrecht@springer.com> Tue, Jun 2, 2009 at 9:44 AM
To: Fanghua Mei <fmei1@tigers.lsu.edu>

Dear Mr. Mei,

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 40 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.

Kind regards,
Estella Jap A Joe
Springer
Rights & Permissions
—
Van Godewijckstraat 30 | 3311 GX
P.O. Box 990 | 3300 AZ
Dordrecht | The Netherlands
fax +31 (0) 78 657 6377
estella.japajoe@springer.com

From: Fanghua Mei [mailto:fmei1@tigers.lsu.edu]
Sent: Thursday, May 28, 2009 6:20 AM
To: Permissions Europe/NL
Subject: Obtain permission

To Whom It May Concern:

I am Fanghua Mei, the first author of the following three papers in MST:
1. Eutectic bonding of Al-based high aspect ratio microscale structures, Microsyst. Technol. 13, 723-730 (2007);

I am finishing my Ph.D. Dissertation and would like to include the work described in these articles in my dissertation. I would like to confirm with you that this use is within the copyright retained for authors. Since my submission deadline is early June, I would appreciate a prompt return email. Thank you for your time and help.

Regards,

Fanghua Mei

2420 Patrick F. Taylor Hall
Department of Mechanical Engineering
Louisiana State University
Baton Rouge, LA 70803
Tel: 225-578-5947
Fax: 225-578-9195
Email: fmei1@lsu.edu

---

2. Permission of Using Published Material from Journal of Materials Research

From: Lorraine Wolf <Wolf@mrs.org>  Thu, May 28, 2009 at 7:56 AM
To: Fanghua Mei <fmei1@tigers.lsu.edu>
Cc: Lorraine Wolf <Wolf@mrs.org>

TO: Fanghua Mei
FROM: Lorraine Wolf
DATE: May 28, 2009

Because this is from your own work, MRS Journal of Materials Research policy is that as an author of this article you have the right to use this material without our permission, remembering to cite the source. Please keep this in mind for future work that is published in the JMR.

Lorraine

Lorraine Wolf
Publishing Assistant
Journal of Materials Research
Materials Research Society
506 Keystone Drive
Warrendale, PA 15086
Phone: 724-779-3004 x503
Fax: 724-779-4396
Email: lwolf@mrs.org
To Whom It May Concern:

I am Fanghua Mei, the first author of the following paper in JMR:


I am finishing my Ph.D. Dissertation and would like to include the work described in this article in my dissertation. I would like to confirm with you that this use is within the copyright retained for authors. Since my submission deadline is early June, I would appreciate a prompt return email. Thank you for your time and help.

Regards,

Fanghua Mei

2420 Patrick F. Taylor Hall
Department of Mechanical Engineering
Louisiana State University
Baton Rouge, LA 70803
Tel: 225-578-5947
Fax: 225-578-9195
Email: fmei1@lsu.edu

3. Permission of Using Published Material from Journal of Microelectromechanical Systems

From: j.hansson@ieee.org <j.hansson@ieee.org>  Thu, May 28, 2009 at 1:48 PM
To: fmei1@tigers.lsu.edu

Dear Fanghua Mei:

We are happy to grant you this permission to reprint this IEEE copyrighted paper in your thesis and have it placed on your university’s website. We have only two requirements that must be satisfied before we can consider this permission final:

1. The following copyright/credit notice must appear prominently on the first page of the reprinted paper, with the appropriate details filled in: © 2008 IEEE. Reprinted, with permission, from [IEEE publication title, paper title, and author names]

2. Additionally, if your thesis is to appear on the university’s website, the following message should be displayed at the beginning of the credits or in an appropriate and prominent place on the website: This material is posted here with permission of the IEEE. Such permission of the IEEE does
To Whom It May Concern:

I am Fanghua Mei, the first author of the following paper in JMEMS:

Fabrication, assembly, and testing of Cu- and Al- based microchannel heat exchangers, JMEMS 17(4), 869-881 (2008).

I am finishing my Ph.D. Dissertation and would like to include the work described in this article in my dissertation. I would like to confirm with you that this use is within the copyright retained for authors. Since my submission deadline is early June, I would appreciate a prompt return email. Thank you for your time and help.

Regards,

Fanghua Mei

2420 Patrick F. Taylor Hall
Department of Mechanical Engineering
Louisiana State University
Baton Rouge, LA 70803
Tel: 225-578-5947
Fax: 225-578-9195
Email: fmei1@lsu.edu
4. Permission of Using Published Material from Journal of Micromechanics and Microengineering

From: Permissions <permissions@iop.org>
To: fmei1@tigers.lsu.edu
Fri, May 29, 2009 at 9:13 AM

Dear Dr Mei,

Please find attached the permission to reproduce material from an IOPP publication.

Please acknowledge receipt of this email.

Best wishes,

Sarah Ryder
Publishing Administrator
Email: permissions@iop.org

To Whom It May Concern:

I am Fanghua Mei, the first author of the following paper in JMM:


I am finishing my Ph.D. Dissertation and would like to include the work described in this article in my dissertation. I would like to confirm with you that this use is within the copyright retained for authors. Since my submission deadline is early June, I would appreciate a prompt return email. Thank you for your time and help.

Regards,

Fanghua Mei
5. Permission of Using Published Material from Journal of Vacuum Science & Technology A

From: lucovsky@ncsu.edu <lucovsky@ncsu.edu>  Wed, Jun 3, 2009 at 7:30 AM
To: Fanghua Mei <fmei1@tigers.lsu.edu>

Yes that is OK

Professor Lucovsky, Editor-In-Chief, JVST

> Dear Dr. Lucovsky,
> I am Fanghua Mei, the first author of the following paper in JVST:
> Evaluation of bond quality and heat transfer of Cu-based microchannel heat

I am finishing my Ph.D. Dissertation and would like to include the work described in this article in my dissertation. I would like to confirm with you that this use is within the copyright retained for authors. Since my submission deadline is early June, I would appreciate a prompt return email.

Thank you for your time and help.

Regards,

Fanghua Mei

2420 Patrick F. Taylor Hall
Department of Mechanical Engineering
Louisiana State University
Baton Rouge, LA 70803
Tel: 225-578-5947
Fax: 225-578-9195
Email: fmei1@lsu.edu
Fanghua Mei was born in October, 1981, in Zhejiang Province, People's Republic of China. In 1998, he entered Shanghai Jiao Tong University, Shanghai, China, where he obtained his bachelor’s degree in materials science and engineering in 2002 and master’s degree in materials science in 2005, respectively. In August 2005, he started his graduate study in the Department of Mechanical Engineering at Louisiana State University in the United States of America. He is currently a candidate for the degree of Doctor of Philosophy in the Department of Mechanical Engineering, Louisiana State University. His research interests include thin film deposition, fabrication of metal-based MEMS, and testing of assembled MEMS devices.