INVESTIGATIONS ON THE MICRO-SCALE SURFACE INTERACTIONS AT THE TOOL AND WORKPIECE INTERFACE IN MICRO-MANUFACTURING OF BIPOLAR PLATES FOR PROTON EXCHANGE MEMBRANE FUEL CELLS

Mevlut Fatih Peker
Virginia Commonwealth University

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INVESTIGATIONS ON THE MICRO-SCALE SURFACE INTERACTIONS AT THE TOOL AND WORKPIECE INTERFACE IN MICRO-MANUFACTURING OF BIPOLAR PLATES FOR PROTON EXCHANGE MEMBRANE FUEL CELLS

A dissertation submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy at Virginia Commonwealth University.

by

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<th>Description</th>
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<tbody>
<tr>
<td>ANOVA</td>
<td>Analysis of variance</td>
</tr>
<tr>
<td>BPP</td>
<td>Bipolar plate</td>
</tr>
<tr>
<td>DOE</td>
<td>Department of energy</td>
</tr>
<tr>
<td>FEA</td>
<td>Finite element analysis</td>
</tr>
<tr>
<td>GDL</td>
<td>Gas diffusion layer</td>
</tr>
<tr>
<td>ICR</td>
<td>Interfacial electrical contact resistance</td>
</tr>
<tr>
<td>MAE</td>
<td>Membrane electrode assembly</td>
</tr>
<tr>
<td>NSF</td>
<td>National Science Foundation</td>
</tr>
<tr>
<td>PD</td>
<td>Potentiodynamic</td>
</tr>
<tr>
<td>PEMFC</td>
<td>Polymer electrolyte (or proton exchange) membrane fuel cells</td>
</tr>
<tr>
<td>PS</td>
<td>Potentiostatic</td>
</tr>
<tr>
<td>PVD</td>
<td>Physical vapor deposition</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
</tr>
<tr>
<td>SOFC</td>
<td>Solid oxide fuel cell</td>
</tr>
<tr>
<td>SS</td>
<td>Stainless steel</td>
</tr>
<tr>
<td>YS</td>
<td>Yield Strength</td>
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</table>
Abstract:

INVESTIGATIONS ON THE MICRO-SCALE SURFACE INTERACTIONS AT THE TOOL AND WORKPIECE INTERFACE IN MICRO-MANUFACTURING OF BIPOLAR PLATES FOR PROTON EXCHANGE MEMBRANE FUEL CELLS

by Mevlut Fatih Peker, Ph.D

A dissertation submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, at Virginia Commonwealth University.

Virginia Commonwealth University, 2012

Major Director: Karla M. Mossi, Associate Professor, Department of Mechanical Engineering

Micro-forming studies have been more attractive in recent years because of miniaturization trend. One of the promising metal forming processes, micro-stamping, provides durability, strength, surface finish, and low cost for metal products. Hence, it is considered a prominent method for fabricating bipolar plates (BPP) with micro-channel arrays on large metallic surfaces to be used in Proton Exchange Membrane Fuel Cells (PEMFC).

Major concerns in micro-stamping of high volume BPPs are surface interactions between micro-stamping dies and blank metal plates, and tribological changes. These concerns play a critical role in determining the surface quality, channel formation, and dimensional precision of bipolar plates. The surface quality of BPP is highly dependent on the micro-stamping die surface, and process conditions due to large ratios of surface area to volume (size effect) that cause an increased level of friction and wear issues at the contact interface.

Due to the high volume and fast production rates, BPP surface characteristics such as surface roughness, hardness, and stiffness may change because of repeated interactions between
tool (micro-forming die) and workpiece (sheet blank of interest). Since the surface characteristics of BPPs have a strong effect on corrosion and contact resistance of bipolar plates, and consequently overall fuel cell performance, evolution of surface characteristics at the tool and workpiece should be monitored, controlled, and kept in acceptable ranges throughout the long production cycles to maintain the surface quality.

Compared to macro-forming operations, tribological changes in micro-forming process are bigger challenges due to their dominance and criticality. Therefore, tribological size effect should be considered for better understanding of tribological changes in micro-scale. The integrity of process simulation to the experiments, on the other hand, is essential.

This study describes an approach that aims to investigate the surface topography changes during long-run micro-stamping of BPPs, and establish relationships between surface roughness–corrosion resistance and surface roughness–contact resistance characteristics of BPPs. Formability levels of formed BPPs and repeatability characteristics of the process were investigated. In addition, blank thickness changes, von-Mises stress, plastic strain levels and distributions of micro-stamping process were determined via finite element analysis (FEA). Test results revealed that the surface roughness change for the stamping dies and BPPs was unsteady (no trend) due to the continuous change of surface topography (i.e. asperity deformation). Sub-micron range local plastic deformations on stamping dies led to surface topography changes on BPP in long-run manufacturing case. As surface defects trigger corrosion, the correlation between surface roughness and corrosion resistance of BPPs was found to be direct. Increasing number of surface irregularities (asperities) lowered contact surface area that resulted in increased contact resistance. ZrN coated BPPs, on the other hand, did not change surface roughness, however; it improved the protection of BPPs against corrosion significantly. In
addition, ZrN coating increased the conductivity of BPPs and reduced the contact resistance between BPP and gas diffusion layer (GDL), at certain extent. As dimensional stability and repeatability was confirmed in forming of both uncoated and coated BPPs during the long run manufacturing, different formability levels were achieved for coated and uncoated samples. Lower channel height values were obtained for coated plates because of the different surface hardness of uncoated and coated plates.

In tribological size effect part of study, micro stamping experiments using three different dies with distinct channel height values at different stamping force levels were performed. It was concluded that decrease in forming die dimensions led to increase in coefficient of friction as previously reported by other researchers as one of the consequences of tribological size effect. On the other hand, coefficient of friction values were not affected by the force levels used in the experiments and simulations, whereas plastic strain, equivalent stress, and formability levels were increased with increasing stamping force, as expected.

In essence, this study proposed a methodology to investigate the long-run manufacturing effects on dimensional stability and surface characteristics of micro-stamped sheets. It also correlates these parameters to fuel cell performance measures such as interfacial contact and corrosion resistance.
CHAPTER 1:

INTRODUCTION

1.1 Research Motivation and Background

There has been an increasing demand for research efforts in micro-manufacturing/forming due to trend of miniaturization, integration, and compactness of products. Portability and lightweightness are the key desired features in electronic and medical devices, as well as energy generation and storage systems [1]. In this respect, traditional manufacturing methods were scaled down to fabricate small-sized parts with dimensions in sub-millimeter range. Micro-machining processes were performed to manufacture parts such as microscale heat exchangers, micro-chemical sensors, microscale molds. However, mass production and low cost could not be provided with micromachining processes. Thus, as an alternative, metal forming methods are preferred in microforming since these methods are appropriate for high production rates, can provide high quality final product surface, and provide high strength with a low cost [2]. Microstamping, microextrusion, microembossing, coining and microdeepdrawing were developed to manufacture microparts. On the other hand, microforming has some challenges due to elevated frictional forces at contact interface and size effects.

The challenges in microforming can be defined as [3]:

- Influence of size effect on material behavior
- Appropriate and sensitive tooling, equipment and controls requirement for microforming
- Unknowns in contact mechanics, surface interactions and microscale tribology
Reliable modeling and simulation of microforming processes

One specific field in which miniaturization has tremendous importance is fuel cell technology. Fuel cell technology is used to generate power (consuming fuel mostly hydrogen or hydrogen based fuels such as methanol, natural gas etc. and an oxidant such as oxygen) via chemical reactions. PEM fuel cells, which seem to be a future trend among other types of fuel cells, have many advantages such as high efficiency, fast response, low temperature requirement, comparatively better durability, lightweight and compactness. However, they are still not in stage of commercial competence with fossil fuel-based energy generation techniques, such as internal combustion engines (ICE), because of their high cost, and durability issues. In a recent study, fuel cell power cost is reported to be around $200/per kW [4]. Compared with the cost of power generated through internal combustion engines ($30-$50 per kW), fuel cell power cost stands as an important challenge for commercialization of fuel cell in transportation and small size portable power generation systems. Department of Energy (DOE) cost targets for PEM fuel cells has been set as $35 per kW for automotive industry [4]. PEM fuel cells having these standards are expected to be produced, and commercialized in 5-10 years in high volumes.

Bipolar plate (BPP) is one of the major components in a fuel cell stack shown in Figure 1.1. Table 1.1 shows the weight and cost percentages of bipolar plate in a fuel cell. As it can be seen from this table, although bipolar plate weight and cost reduce year by year with the development of new materials and technologies, it still constitutes a major portion of total weight and cost that need to be reduced further.
Bipolar plates (BPP) have vital functions in fuel cells. BPP has micro-channels on both sides which enable gases (H₂ and O₂) to flow through. It also provides electron conduction between cells as well as increases the strength for fuel cell. It also helps to remove the heat from inside to outside [4-8].

In order to perform these functions properly, bipolar plates must have chemical stability,

### Table 1.1 The evolution of weight and cost of BPP in PEM fuel cell [6]

<table>
<thead>
<tr>
<th>Year</th>
<th>Weight (%)</th>
<th>Cost (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2002</td>
<td>90</td>
<td>67</td>
</tr>
<tr>
<td>2004</td>
<td>78</td>
<td>37</td>
</tr>
<tr>
<td>2006</td>
<td>75</td>
<td>11-45</td>
</tr>
</tbody>
</table>

**Figure 1.1** An example of a PEM fuel cell stack [5]
electrical and thermal conductivity, low contact resistance, good mechanical strength, low gas permeability, inexpensive massive production, low cost, and uniform reactant gas distribution [6].

Metallic bipolar plates, in particular stainless steel bipolar plates, are attracting researches due to their excellent bulk conductivity, low cost, high chemical stability, high mechanical strength, negligible gas permeability, and good manufacturability. However, they have poor corrosion resistance. As a result of corrosion, contact resistance increases depending on the growth of metal oxide layers. The metal ions released from bipolar plates can contaminate the polymer electrolyte membrane. Thus, bipolar plates must be coated to increase their corrosion resistance, decrease contact resistance and extend the fuel cell life. These coating materials can be carbon-based (e.g. polyaniline, poly-pyrrole) as well as metal-based such as metal nitrides, and early transition of metal elements [9].

Beside the bipolar plate material selection, choice of manufacturing technique is also important since wrong method selection may result in breaking, bending, and squashing [10]. Considering the requirements for BPP manufacturing in terms of both precision, production rate and cost effectiveness, stamping is seen as a prominent choice for fabricating bipolar plates (BPP) in Proton Exchange Membrane Fuel Cells (PEMFC) [7]. Advantages of stamping process are their low cost and fast production cycles for bipolar plate fabrication.

In micro-stamping of BPPs, thin metal sheets are formed by precision dies as shown in Figure 1.2. In this respect, thousands of micro-channels must be formed with satisfying geometric shape and surface accuracy.
A major concern in micro-manufacturing of high volume BPPs is tribological changes and service life issues of micro-stamping dies in which BPPs are formed. These dies have micro-channels with high precision and highly smooth surface finishes on them. The surface quality of BPP produced is highly dependent on the micro-stamping die surface and process conditions due to the large ratios of surface area to volume ratios (size effects) that cause an increased level of friction and wear issues at the contact interface. Surface characteristics of BPPs such as surface integrity, surface roughness, hardness and stiffness are major features affecting the fuel cell performance. During the high volume and speed production of BPPs, mechanic characteristics of the BPPs may change because of repeated interactions between tool (micro-forming die) and workpiece (sheet blank of interest), at the contact interface. Surface mechanical characteristic values at the tool and workpiece should be monitored, controlled and kept in acceptable range throughout the long production cycles to maintain the surface quality of BPPs as illustrated in Figure 1.3. In micro-forming, highly localized stresses occur under high contact pressure, and these may easily change the surface topography of tool which leads to variations on surface
quality of bipolar plates. These topographic and tribological changes subsequently affect the corrosion and contact resistance of bipolar plates and overall fuel cell performance.

Figure 1.3 Parameters involving in fuel cell performance

1.2 Objectives of Proposed Research

The overall research objective of this study is to methodologically correlate the dimensional and surface quality requirements of bipolar plates to the interrelated parameters of micro-stamping process in mass production conditions. Specific objectives that address the research goals are as follows:

- **Objective: 1** Understand the cause, mechanism and consequences of interactions between micro-stamping process conditions (speed, force, coating, die surface roughness and hardness) and bipolar plate quality (channel formation, surface roughness) (Task 1 and 2);

- **Objective: 2** Characterize the effects of BPP coatings on the product surface quality as well as on the die wear/life issues (Task 2);
- **Objective: 3** Understand and model the influence of “tribological size effects” on micro-stamping die-BPP interface (Task 3);

- **Objective: 4** Investigate the effect of long-run manufacturing on the die and BPP surface quality, corrosion and contact resistance characteristics of BPPs (Task 4 and 5).

### 1.3 Research Approach and Plan

Based on the identified research needs and objectives as outlined in the previous sections, the research plan for this study is as follows:

**Task 1: Determination of the micro-forming parameters**

An extensive literature survey was performed to gain fundamental knowledge on microforming issues and to determine the micro-forming parameters that need to be monitored in experiments. Based on the previous researches related with my study, the factors that affect the die and bipolar plate surface quality, dimensional variation and die life has been identified as follows;

- Micro-forming technique,
- Micro-forming speed,
- Material - size - surface conditions for both tool (die) and deformed part (bipolar plate),
- Micro-channel geometry
Task 2: Investigation the effect of die surface quality on coated and uncoated blank surface quality

In task 4, uncoated and coated samples were stamped and subjected to corrosion and contact resistance tests to find out the effect of coating on bipolar plate performance.

Task 2.1: Surface inspection of both tool (die) and workpiece (bipolar plate) and the correlation between surface roughness and corrosion/contact resistance

An experimental micro-forming system with a small (750 micron feature height) insert was designed and manufactured. Large numbers of uncoated bipolar plates were fabricated, and then the surface conditions of both die insert and bipolar plate were monitored to understand the impact of repeated surface interactions. Channel heights of BPPs were measured to investigate the repeatability of tests. In addition, contact and corrosion resistance of bipolar plates were performed to understand the effect of surface roughness on corrosion and contact resistance characteristics of BPPs.

Task 2.2: Determination of the influence of bipolar plate coating on the forming characteristics and die wear/die life

All the measurements (surface roughness, channel heights) and tests (corrosion resistance and contact resistance tests) mentioned in Task 2.1 were also subjected to the coated BPPs. Finally, the results of measurements and tests for uncoated and coated BPPs were compared to understand the effect of coating on surface roughness, corrosion and contact resistance, consequently fuel cell performance.
Task 3: Development of micro-forming models for better understanding of the tribological size effect

The objective of this task is to study the “tribological size effect” in micro-forming with

- Determination of deformation characteristics (von-Mises stress, plastic strain and thickness changes),

- Determination of friction coefficient,

- Formability comparison of different geometries/dimensions of bipolar plates,

This task is divided into two parts as “physical modeling” and “numerical modeling”.

Task 3.1: Experimental modeling of micro-forming

A micro-forming test system to manufacture micro-channeled features was designed and built. Several small die inserts with different size micro-channel features have been employed in BPP manufacturing. Micro-stampings tests were performed in different force levels (100kN, 200kN and 300kN) using micro-stamping dies with different channel sizes (250µm, 750 µm and 2500 µm). The results of measurement (friction, dimensional variation) obtained from different dies and plates were compared with the numerical results to reveal the influence of “size effect”.

Task 3.2: Numerical modeling of micro-forming

Finite element (FE) based numerical models were established with commercial Ls-Dyna software. First, the geometries were built. Second, material properties and process parameters and boundary conditions were entered. Then the numerical results were analyzed. The numerical
channel height values were compared with the experimental channel height results to determine coefficient of friction for all models.

**Task 4: Experimental and numerical analysis of long run manufacturing process on the die, BPP surface quality, deformation characteristics and coefficient of friction**

The objective of this task is to investigate the effect of long run manufacturing process on the die wear, bipolar plate surface quality the relation between surface roughness and corrosion/contact resistance characteristics, the relations between increasing number of stampings and elevated levels of friction. An experimental micro-forming test system was devised to fabricate micro-channeled bipolar plates in large numbers. During the process, surface inspections (surface roughness change and, dimensional variations) were performed periodically from both die and bipolar plate surfaces. Since contact and corrosion resistance of bipolar plates have tremendous effect on fuel cell performance, their variations on the manufactured bipolar plates were observed as well. FE based numerical models were also built for investigation of deformation characteristics and determination of coefficient of friction.

**Task 5: Development of predictive models and guidelines for design and selection of bipolar plate and coating material, channel dimensions etc.**

Numerical modeling of the micro-forming operation was conducted and the results were validated by comparing experimental results.
### Table 1.2 Objectives and their corresponding tasks

<table>
<thead>
<tr>
<th>Objectives</th>
<th>Tasks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Objective 1</td>
<td>Task 1; Task 2</td>
</tr>
<tr>
<td>Objective 2</td>
<td>Task 3</td>
</tr>
<tr>
<td>Objective 3</td>
<td>Task 4</td>
</tr>
<tr>
<td>Objective 4</td>
<td>Task 4; Task 5</td>
</tr>
</tbody>
</table>

### 1.4 Dissertation Organization

This dissertation is divided into six chapters. In **Chapter 1**, Motivation, objectives, research plan, expected scientific contributions are presented. **Chapter 2** introduced a state of the art review for micro-forming technology, size effect issues and fuel cell technology and bipolar plates. **Chapter 3**, on the other hand, focuses on the effect of manufacturing on stamping tribology which is a very critical approach in micro-forming studies. This chapter also introduces the effect of coating of the bipolar plates on micro stamping tribology. **Chapter 4** was based on tribological size effect which is major challenge in micro-forming. Numerical modeling studies designed to address the tribological size effect issue was also included in this chapter. In **Chapter 5**, the effect of long run manufacturing on die wear, BPP surface quality, surface roughness and corrosion/contact resistance characteristics. The main objective of this chapter was to investigate the, friction variations in mass production-like conditions. In **Chapter 6** Summary and conclusion were discussed. Hence, proposed dissertation organization was as below:
Chapter 1: Introduction

Chapter 2: State of the art review

Chapter 3: Effect of manufacturing process and coating on micro-stamping of bipolar plate

Chapter 4: Investigations of the “tribological size effect” issue in micro-stamping

Chapter 5: Effect of long-run manufacturing on the die/BPP topography and tribological condition

Chapter 6: Summary, conclusion, recommended future work, and expected Scientific Contribution
References


[5] Future energies web site:


CHAPTER 2:
STATE OF THE ART REVIEW

2.1 Microforming Fundamentals

Electronic and electromechanic industries require continuously advancing technologies for manufacturing of micro-parts due to the increasing demand for miniaturization. Any kinds of connecting elements, fasteners, micro-screws, pins are some examples of micro-forming parts as seen in Figure 2.1.

![Figure 2.1 Parts formed with micro-manufacturing techniques [1]](image)

The miniaturization trend in formed parts results in a phenomenon called “size effect”. For example, when a material is cut into smaller sizes (i.e., scaled down), grain size of material does not change, however; the impact of each individual grain on the overall material behavior becomes more dominant [2-5]. Especially the grains in the deformed area may change the material behavior such as flow stress, hardness and interaction with contacting surfaces (i.e.,
friction, wear, heat transfer), significantly. After miniaturization of tool and workpiece, the shear stresses at contact interface as well as hardness and yield strength increase while flow stress decreases [6, 7]. High friction leads to need for higher contact forces, die wear, temperature rise and extra energy requirements. According to a previous investigation, it was shown that relative velocity on the contact area should be larger than a critical value for friction reduction [6].

A micro manufacturing system consists of five different groups as; material, tooling, process, machine/equipment and product as depicted in Figure 2.2. When a traditional forming process is scaled down to micro scale, the major effect that changes the material behavior is “size effect”. Other material characteristics, anisotropy, ductility differ depending on the size effect. The challenge with the tooling is producing very small tolerances with high surface quality. Therefore, tool material and manufacturing method selections are critical to achieve the final product dimensions with desired properties. These selections are also significant for low tool making cost and increased tool life. The problems for micro-machine and equipment grow, as the sizes are scaled down. One of the main concerns in mass production of small parts is need for high precision. Because of the adhesive forces (Van der Waals, electrostatic and surface tension), handling and holding of parts are difficult. Determination of process parameters and setting appropriate automation system are the other challenges that occur in micro-scale.
2.2 Tribology in Microforming

Tribological condition between the tool and the material is one of the most important issues in micro-metal forming. In order to understand the tribological behavior in dry friction condition, the topography of two contacting surfaces needs to be understood. The meaning of dry friction is that there is no lubricant between two solid contact surfaces. Many types of surfaces have the geometric characteristics of the surface that involve asperities and valleys as in Figure 2.3 [9]. In dry friction condition, actual contact involves only at asperities. Real contact area is much smaller than the apparent total area and increases with increased load [10]. During sliding, friction is contributed by adhesion and deformation at asperity in contact. [11]
During the long time forming process, the asperities on the dies and BPPs surfaces could be deformed and smoothened (Figure 2.4) that leads to reduce roughness. Then, some of the tips on the softer material can become plastic and cold weld on the harder material under sufficient loads. This is the description of adhesive theory of friction Figure 2.5 [12]. As a result of this phenomenon, unsteady surface roughness changes can be observed during the long run manufacturing conditions. Increase and decrease of surface roughness on workpiece were shown visually in Figure 2.6.
Figure 2.4 (a) Tool and workpiece before contact (b) Workpiece surface deformed by a rigid die

W : Load  
δ : Center deflection of the asperities  
Z : Initial summit height  
d : Current position of the top surface
2.3 Dry Friction in Microforming

Lubrication is necessary in sheet metal forming due to the contact interactions between tool and workpiece. Lubrication is commonly used in metal sheet forming to separate work piece and die surfaces, reduce interface friction, help material flow, obtain parts with required thickness, increase die life reducing wear and contact stresses [13].

However, variety of problems and cost issues related with lubrication occur. Especially in micro-scale, lubrication is not preferred because of its impacts on environment and health, difficulties
in removal of oil from the surface of tool, lubrication clogging, effects of viscous forces on
formability. Detailed issues related with lubrication were given as:

1) Cost and Environmental Issues: Subsistent toxicity of some mineral oil-based lubrication caused billions of dollars cleaning cost. Since they harm ecology and water reserves, these types of lubricants should be defused. Volatile organic solvents are commonly used to remove lubricant from formed surfaces. The problems of mineral oil-based lubrication led to an increase in usage of lubricants such as vegetable oils due to their renewability, good lubricity, low volatility, high viscosity, solvency for lubrication additives. However, use of vegetable oils was limited by their poor thermal and oxidative stability.

2) Coherence of lubricant with other process operations: Lubricant needs to be subjected to the metal sheet surface easily and removed by an inexpensive and fast method.

3) Health aspects: Recently, health risks related with the use of lubrication were appeared. Some lubricants used in manufacturing include some hazardous elements such as chlorine, sulfur, and phosphorus. Government regulations restrict more daily uses of lubrication year by year.

4) Increasing surface roughness: The thick oil film reduces the contact between the tool and workpiece surface asperities. On the other hand, plastic deformation is unrestricted under these conditions. This leads to increase the roughness on the workpiece surface. This is undesirable for metal forming processes.

5) Rust or Corrosion: Oils used in microforming trigger rust or corrosion on the tool and workpiece surface. Corrosion and rust inhibitors should be used for corrosion protection, increasing the overall cost.
6) Clinging of waste materials to the tool: Especially in mass production of micro-scale parts, waste materials cling to the dies. This interrupts production. These small materials may also affect the surface quality of workpiece.

7) Flammability: Most metal forming oils are flammable.

8) Pick-up and galling: During cold forming of tribologically difficult metals like stainless steel and aluminum, pick-up and galling may occur. Reaching a critical value of temperature causes lubrication film breakdowns that result in pick-up and galling. [13, 15-21]

Some studies showed that it is also possible to form metal sheets without lubrication. However, dry friction mechanism should be well analyzed [10]. In this study, dry friction of the micro-stamping process was found in lower levels (µ: 0.18) compared to other microforming processes such as micro-deep drawing and micro extrusion. Thus, taking the challenges of lubrication use in microforming and the relatively lower friction levels of microstamping into account, micro-stamping of BPPs was performed without lubrication.

2.4 Size Effects in Microforming Processes

2.4.1 Grain Size Effect and Feature/Specimen Size Effect

Forming processes in micro scale are affected more by the feature size of the microparts than in macro-scale. A sudden change in plastic deformation characteristics can be seen due to smaller ratio of feature size to grain size in microparts than macroparts [22]. At micro scale, a few grains in deformed area determine the material behavior and surface characteristics. Therefore, the material can not be assumed as homogeneous continuum as in the macro-scale.
This effect is so-called “size effect”, and preventing use of traditional applications for conventional forming processes in microforming operations. Therefore, systematical investigations on size effect are required. One commonly accepted theory for better understanding of size effect on the metal behavior of thin sheet metals is “surface layer model” [6]. This theory indicates that hardening of surface grains are less than other grains. After miniaturization, the ratio of surface area to volume increases. As a consequence, the surface grains determine the material behavior that leads to reduction of the strength of material.

In previous studies, two types of “size effects” were defined: “grain size effect” and “feature size effect” as shown in Figure 2.7 [5, 6]. “Grain size effect” can be explained by Hall-Petch equation [23, 24]. According to Hall-Petch equation, the material with smaller grain sizes shows higher strength than the one with larger grain size. Grain size effect plays an important role when the part is only in macro scale. As the size reduces, “feature/specimen size effect” gains importance in micro scale manufacturing. Feature/specimen size effect can be classified into two parts as feature effect and specimen effect depending on the material testing method. The smallest feature on the final part can be regarded as feature size, whereas the thickness of a sheet blank or diameter of a billet represents the specimen size. For instance, assuming micro-channels formed on initially flat sheet blank; the thickness of the blank can be considered as specimen size, as the dimensions of the channel would be feature size.
2.4.2 Tribological Size Effect

Although many studies focused on grain size effect or feature/specimen size effect, there are very limited studies on tribological size effect. In a previous study, Messner et al. studied tribological size effect for bulk material integrating experimental data to FE-based models. Numerical models of different ring tests were established. Since nomograms obtained from FE simulations are size independent, flow stress does not affect the numerical results. Thus, experimental data has to be integrated to the simulations. In the experimental part of the study, compression tests with ring specimens with different dimensions were performed. Unformed and formed cylindrical specimens for different sizes were shown in Figure 2.8. Then, size dependent experimental flow curves were used in the simulations. Friction coefficients were determined by comparison of experimental and numerical diameters. The results showed that reduction of initial specimen size led to increase of coefficient of friction [25].
Engel et al. investigated the tribological size effect with ring compression and double cup extrusion. Double cup extrusion set up was illustrated in Figure 2.9. The materials were formed moving down the upper punch to built 2 cups with cup heights of $h_u$ and $h_l$. The test results revealed friction increased with scaling down of size in the case of lubrication with oil, whereas friction was size independent [26].
De-bin et al. performed cylinder compression tests for the investigation of tribological size effect. It was concluded that size reduction led to increase friction factor in the case of lubrication. The influence of tribological size effect was not observed without lubrication [27].

Besides bulk material forming, deep drawing is also significant for sheet metal forming. Hu et al. studied on the calculations of friction between tool and workpiece using the experimental deep drawing test data as illustrated in Figure 2.10. Since tangential pressure occur in the workpiece which is very complex. Therefore, a strip was used instead of circular blank to eliminate tangential pressure. Punch force was measured from experimental tests and used in the friction calculations in deep drawing tests. A double cup extrusion process was analyzed experimentally and numerically. Friction factors for different specimen dimensions were
investigated. The results demonstrated that friction factor increased with reducing initial diameter of workpiece [28].

![Figure 2.10 Principle of strip drawing [28]](image)

In another study, Hu et al. studied the effect of miniaturization on tribological size effect in a strip-drawing test. The aim was to determine a size dependent friction function to integrate FEM simulation. Sheet thicknesses were scale down to identify the tribological size effects. The drawn strips were demonstrated in Figure 2.11. Different friction functions were obtained for different process dimensions. According the results, friction increased with miniaturization [29].
2.5 Fuel Cell Technology

Fuel cell is a device that generates power by providing combination of hydrogen or hydrogen based fuels and oxygen via chemical reaction. Power and a nontoxic byproduct-water are released as final products as well as usable heat. Among the other environmentally friendly power sources devices, fuel cell is the one with high efficiency. Due to their advantages, fuel cells are considered as part of the solution efforts to energy problems. Since there are several technical problems, fuel cells are not commercially available in the market, yet. The expensive cost, weaknesses on hydrogen production and storage methods are important challenges in their commercialization. The amount of power generated by a fuel cell depends on the type of fuel cell, operating temperature and pressures of gases. Fuel cells can be grouped in two categories: (a) Considering working temperature and type of electrolyte used, (b) power and applications area, as tabulated in Table 2.1, and Table 2.2, respectively.
Table 2.1 Classification of fuel cells considering working temperature and type of electrolyte

<table>
<thead>
<tr>
<th>Type</th>
<th>Electrolyte</th>
<th>Operation Temp. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkaline Fuel Cells (AFC)</td>
<td>KOH</td>
<td>55-90</td>
</tr>
<tr>
<td>Proton Exchange Membrane (PEM)</td>
<td>Polymer</td>
<td>50-125</td>
</tr>
<tr>
<td>Direct Methanol Fuel Cells (DMFC)</td>
<td>Sulfuric acid or polymer</td>
<td>50-120</td>
</tr>
<tr>
<td>Low Power (1-5 kW), Micro app.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phosphoric Acid Fuel Cells (PAFC)</td>
<td>Ortho phosphoric acid</td>
<td>190-210</td>
</tr>
<tr>
<td>Molten Carbonate Fuel Cells (MCFC)</td>
<td>Li – K carbonate mixture</td>
<td>630-650</td>
</tr>
<tr>
<td>Solid Oxide Fuel Cells (SOFC)</td>
<td>Stabilize zirconium</td>
<td>900-1000</td>
</tr>
</tbody>
</table>

Table 2.2 Classification of fuel cells considering power and application area

<table>
<thead>
<tr>
<th>Power Range</th>
<th>Application Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-5 kW</td>
<td>Micro-applications</td>
</tr>
<tr>
<td>5-10 kW</td>
<td>Apartment-Building</td>
</tr>
<tr>
<td>10-100 kW</td>
<td>Building</td>
</tr>
<tr>
<td>50-300 kW</td>
<td>Commercial</td>
</tr>
<tr>
<td>240 kW-10 MW</td>
<td>Power Station</td>
</tr>
</tbody>
</table>
Working principle of a PEM fuel cell is illustrated in Figure 2.12. A membrane that transmits protons throughout the membrane thickness is sandwiched between anode and cathode electrodes. At the anode side, hydrogen is decomposed into electrons and protons. As protons can pass through the membrane, electrons could not pass; since the size of electrons is much bigger than the one size of protons. In another way, membrane functions as an insulator and electrons are forced to go along on an exterior circuit. Consequently, electricity is generated. Water is produced as a byproduct via a combination of protons, electrons and oxygen ions with another catalytic process on the cathode side. In the case of using hydrocarbons, the emissions are water and carbon dioxide [30].

![Figure 2.12 Working principle of a PEM fuel cell][31]

Beside of bipolar plates discussed before, some other significant components of PEM fuel cells are polymeric membrane and electrodes. One of the reasons that PEM fuel cells are not
commercial now is the inadequacy in thermal and chemical endurance of membrane. In addition, current membranes (perfluorosulfonic acid (PFSA)) can not provide long service life (>1000 hr) due to stressess, high temperature and pressure. Performance of a membrane is also low in high temperatures. Even though no exterior forces are applied on fuel cells, the temperature difference leads to shrinking and swelling on membrane surface.

There are two electrodes used in fuel cells. As hydrogen (anode) side is negative, the positive side is called cathode. The electrodes must be electronically conductive and porous. Two chemical reactions occur in fuel PEM fuel cells. While oxidation occurs in anode side, reduction happens in cathode side. The basic reactions are given below:

$$\text{At the anode: } H_2 \rightarrow 2H^+ + 2e^-$$

$$\text{At the cathode: } \frac{1}{2} O_2 + 2H^+ + 2e^- \rightarrow H_2O$$

$$\text{Overall: } H_2 + \frac{1}{2} O_2 \rightarrow H_2O$$

The reactions are slow due to the low temperature working condition in the fuel cell. Therefore, catalysts are used to increase the reaction speed.

2.6 Bipolar Materials, Manufacturing and Research Issues:

2.6.1 Bipolar Plate Materials:

Bipolar plate materials can be classified into three groups distinctly: graphite, carbon composite and, metallic bipolar plates. Graphite material used to be used widespread for BPPs due to their excellent electrical conductivity and chemical stability. However, challenges of
graphite BPP such as high permeability, high cost and brittleness lead to seeking for alternative BPP materials. In this context, different types of composite bipolar plates (graphite-polymer, carbon/carbon composite and polymer composites) have been developed for fuel cell technology. Oak Ridge National Laboratory developed carbon/carbon composite plates in 2003 [32]. Even though carbon/carbon composites have high electrical conductivity, high strength, lightweight and low permeability, their complexity and production cost block the commercialization of carbon/carbon composites [32]. In a previous study, higher conductivity and low cost properties were reported using graphite-polymer composites [33]. On the other hand, problems regarding with working temperature and cold-starting of fuel cell issues have also been noted [34]. Polymer composite is another material considered as bipolar plate material. Since it can be manufactured by molding techniques, polymer composites are appropriate for mass production. The challenges of using polymer composites can be summarized as low electrical conductivity and low mechanical strength [35]. Comparing with other materials, metallic BPPs stand to be the most promising alternative due to their high electrical and thermal conductivity, mechanical strength, chemical stability, compatibility for mass production, cold-start features. Commonly preferred materials for metallic BPP are variety of stainless steel grades, Ni based alloys, Ti based alloys and Al based alloys. Fe based alloys are reported to be not appropriate materials for BPP since their corrosion resistance is very low [34]. Furthermore, they have low electrical conductivity. Among these metallic BPPs, stainless steel meets most of the expectations as BPP material. Thus, stainless steel BPP is commonly preferred as BPP material [31, 32, 34]. The disadvantages of stainless steel bipolar plates are reported as relatively low corrosion resistance performance and high contact resistance between bipolar plate and other components of fuel cell.
However, depending on the developments in coating material and coating technologies, it would be possible to address these challenges [32].

2.6.2. Manufacturing of Bipolar Plates

Besides the bipolar plate material, manufacturing technique of bipolar plates is also important and affects the fuel cell performance. Fabrication of bipolar plates is part of the manufacturing of the fuel cell stack. Manufacturing of fuel cell consist of four subsystems:

1) The cell stack (MEA, BPP, GDL)
2) The balance of power (BOP) (piping, cooling, valves, etc.)
3) Power conditioning
4) System control

Some fundamental requirements should be considered in manufacturing of BPPs:

- Flatness
- Parallelism of the faces
- Uniformity of flow fields

Exact control of the bipolar plate configuration and dimensions are critical. For instance, it was reported that 25µm increase in a plate’s thickness at a corner lead to 10cm tilt in an 80kW stack (approximately 400 cells) [36]. Three major manufacturing techniques used in metallic bipolar plate productions are:

1) Photo-Etching
2) Stamping
3) Machining
Machining of bipolar plates is very expensive and a low speed process. Therefore, it is not preferred in high volume bipolar plate production. Different from machining process, etching and stamping have been proved to be better choices to produce micro-channels on the bipolar plates [37]. Examples of photo-etching and stamping of bipolar plates are shown in Figure 2.13.

![Figure 2.13](image)

**Figure 2.13** Single bipolar plate of a) stamped stainless steel sheet (GenCell Corp.), b) photo etched stainless steel/titanium plate (Tech-etch, Inc.)

Photo etching is a manufacturing process where metal is selectively dissolved, and leaving a finished part. Before process begins, thin metal parts should be cleaned and covered with a photosensitive coating. Multiple channel levels can be etched onto the fuel cell plate with photo-etching process [38]. However, it is not a convenient process for mass production of bipolar plates.

Stamping of thin stainless steel sheets have been received attention due to the advantages summarized below [36]:

- Stamping method reduces the manufacturing cost and time dramatically for BPP manufacturing.
- There is almost no waste material in production of BPP.
- Bipolar plates can be produced in seconds via the fraction between die and BPP.
- Very tight tolerances can be achieved.

Even though stamping process is accepted as an appropriate choice because of the advantages mentioned above, there are still challenges need to be addressed for mass production conditions. Different from macroscale stamping, there are several unknowns and uncertainties in micro-stamping such as, contact interactions, frictional and contact forces as well as wear issues. Those aspects are more important in microscale than macroscale since the rate of surface area to volume is high in microscale. Surface roughness changes results in variations for corrosion and contact resistance of bipolar plates, which leads to altered fuel cell performance. Additionally, developments in modeling, fuel cell performance, and durability are required.

### 2.7 Time and Cost Analysis of Stamped SS316L BPPs

Comparisons of (5 in x 11in) stamped SS316L, stamped aluminum, extruded aluminum, molded graphite plates and injection molded graphite plates in terms of time and cost for were given in Table 2.3. The molded graphite bipolar plates have been commonly used because of their chemical stability and conductivity. However they are the most expensive BPP ($10.42) because of their low production rate and high amount of waste material. High cost of molded graphite BPP is one of the biggest challenges for commercialization of fuel cells. The production time, material saving and cost of graphite BPPs were decreased with injection molded technique.
The production time for graphite BPP time has reduced from 8.95 min/plate to 1.12 min/plate as the cost has dropped from $10.42/plate to $3.21/plate. However, it’s still not competitive comparing with the metallic BPPs (stamped stainless steel and aluminum) time and cost which are 0.53 min/plate for both stamped stainless steel and aluminum and $2.56/plate for stainless steel and $2.22/plate for aluminum respectively. Another disadvantage of molded graphite is their high weight (0.481 lb/plate) as the weight of stainless steel BPP and aluminum are 0.231$/plate and 0.099$/plate respectively [39].

**Table 2.3** Cost comparison for bipolar plate forming technologies [39]

<table>
<thead>
<tr>
<th>Weight and volume</th>
<th>316 SS Stamping</th>
<th>Aluminum stamping</th>
<th>Aluminum stamping</th>
<th>Molded graphite</th>
<th>Injection molded</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total weight</td>
<td>0.231</td>
<td>0.099</td>
<td>0.202</td>
<td>0.481</td>
<td>0.077</td>
<td>lb/plate</td>
</tr>
<tr>
<td>Material cost/$lb</td>
<td>$ 1.80</td>
<td>$ 0.78</td>
<td>$ 0.78</td>
<td>$ 3.50</td>
<td>$ 3.50</td>
<td>$/lb</td>
</tr>
<tr>
<td>Subtotal material</td>
<td>$ 0.42</td>
<td>$ 0.08</td>
<td>$ 0.16</td>
<td>$ 1.68</td>
<td>$ 0.27</td>
<td>$/plate</td>
</tr>
<tr>
<td>Frame and seal or “O” ring</td>
<td>$ 1.30</td>
<td>$ 1.30</td>
<td>$ 0.05</td>
<td>$ 0.50</td>
<td>$ 1.50</td>
<td>$/plate</td>
</tr>
<tr>
<td>Material cost per plate</td>
<td>$ 1.72</td>
<td>$ 1.38</td>
<td>$ 0.21</td>
<td>$ 2.18</td>
<td>$ 1.77</td>
<td>$/plate</td>
</tr>
</tbody>
</table>

**Total cost** 8.95 min/plate

| Labor rate                | 50.00           | 50.00             | 50.00             | 50.00           | 50.00           | $/hr       |
| Press capital cost        | $ 0.01          | $ 0.01            | $ 0.43            | $ 0.51          | $ 0.51          | $/plate    |
| Per plate labor           | $ 0.44          | $ 0.44            | $ 7.46            | $ 0.93          | $ 0.93          | $/plate    |
| Subtotal forming cost     | $ 0.45          | $ 0.45            | $ 4.45            | $ 7.89          | $ 1.44          | $/plate    |

**Plate finishing cost**

| Coating materials         | $ 0.50          | $ 0.90            | $ 0.90            | $ 0.90          | $ 0.90          | $/ft²      |
| Corrosion coating cost materials | $ 0.34    | $ 0.34            | $ 0.20            | $ 0.20          | $ 0.20          | $/plate    |
| Coating application cost  | $ 0.05          | $ 0.05            | $ 0.05            | $ 0.05          | $ 0.05          | $/plate    |
| Planarization cost        |                 | $ 0.35            |                 |                 |                 | $/plate    |
| Subtotal                  | $ 0.39          | $ 0.39            | $ 0.25            | $ 0.35          | $ 0.35          | $/plate    |
| Total manufactured cost   | $ 2.56          | $ 2.22            | $ 4.91            | $ 10.42         | $ 3.21          | $/plate    |

**Stack production cost**

| Plates/stack              | 136             | 136               | 136               | 136             | 136             |           |
| Cost/stack                | $ 348           | $ 301             | $ 668             | $ 1,417         | $ 440           |           |
| Weight/stack              | 31.42           | 13.46             | 27.47             | 65.45           | 10.55           |           |
2.8 Material Properties of Stamping Dies, Bipolar Plates and Coating Materials Used in the Study

2.8.1 Stamping Die

AISI Type H13 Hot Work Tool Steel, air or oil quenched from 995-1025 °C

High hardenability, excellent wear resistance, hot toughness and good thermal shock resistance are the main advantages of H13. Hardness of H13 can be improved by hardening. Applications of H13 are stamping dies, forging dies, pressure die casting tools, hot shear blades and plastic molds. H13 material is also appropriate for aluminum die casting. H13 can be welded with oxy-acetylene. Chemical composition and properties of H13 were given in Table 2.4, Table 2.5, and Table 2.6 [40].

<table>
<thead>
<tr>
<th>Component</th>
<th>Wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.32 – 0.4</td>
</tr>
<tr>
<td>Cr</td>
<td>5.13 – 5.25</td>
</tr>
<tr>
<td>Fe</td>
<td>&gt; 90.95</td>
</tr>
<tr>
<td>Mo</td>
<td>1.33 – 1.4</td>
</tr>
<tr>
<td>Si</td>
<td>1</td>
</tr>
<tr>
<td>V</td>
<td>1</td>
</tr>
</tbody>
</table>
Table 2.5 Physical and Mechanical Properties of H13 Tool Steel [40]

<table>
<thead>
<tr>
<th>Mechanical Properties</th>
<th>Value</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>7.8 g/cc</td>
<td></td>
</tr>
<tr>
<td>Hardness, Knoop</td>
<td>570</td>
<td></td>
</tr>
<tr>
<td>Hardness, Vickers</td>
<td>549</td>
<td></td>
</tr>
<tr>
<td>Tensile Strength, Ultimate</td>
<td>1990 MPa</td>
<td></td>
</tr>
<tr>
<td>Tensile Strength, Yield</td>
<td>1650 MPa</td>
<td></td>
</tr>
<tr>
<td>Elongation at Break</td>
<td>9 %</td>
<td></td>
</tr>
<tr>
<td>Modulus of Elasticity</td>
<td>210 GPa</td>
<td>at 20 °C</td>
</tr>
<tr>
<td>Bulk Modulus</td>
<td>140 GPa</td>
<td>Typical for steel</td>
</tr>
<tr>
<td>Poisson’s Ratio</td>
<td>0.3</td>
<td>Calculated</td>
</tr>
<tr>
<td>Machinability</td>
<td>50 %</td>
<td>Based on 1 % carbon steel.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>As 100 % machinability</td>
</tr>
<tr>
<td>Shear Modulus</td>
<td>81 GPa</td>
<td>Estimated from elastic modulus</td>
</tr>
</tbody>
</table>

Table 2.6 Descriptive properties of H13 Tool Steel [40]

<table>
<thead>
<tr>
<th>Descriptive Properties</th>
<th>Value</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Annealing Temperature</td>
<td>850 – 870 °C for 4 hours</td>
<td>Furnace cool 20 °C per hour max.</td>
</tr>
<tr>
<td>Stress Relieving Temperature</td>
<td>600 – 650 °C for 2 hours</td>
<td>Cool in still air; always stress relief before hardening.</td>
</tr>
</tbody>
</table>
2.8.2 Bipolar Plate

Stainless Steel (SS316L)

SS316L has more corrosion resistivity than most of the stainless steels such as SS 302-304. Other advantages of type 316L are higher creep resistance, excellent formability, rupture and tensile strength at high temperatures. It is commonly used in industrial applications such as chemical, pharmaceutical industry, surgical and medical tools, surgical implants, evaporator & handling equipment, petroleum refining equipment, textile industry equipment, heat exchanger tubes.

Maximum carbon rate for most standard grades of stainless steel is 0.08%. Standard grades of stainless steels are appropriate to use for non-welded parts. On the other hand, L-grades stainless steel include 0.03% carbon maximum. Strengths of the L-grades of stainless steel are lower than standard stainless steels. L-grades of stainless steel are resistant to heat treatments. Chemical composition and properties of SS316L were given in Table 2.7, Table 2.8 [41].
### Table 2.7 Chemical composition of SS316L [41]

<table>
<thead>
<tr>
<th>Component</th>
<th>Wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>62.045 – 72</td>
</tr>
<tr>
<td>Cr</td>
<td>16 – 18</td>
</tr>
<tr>
<td>Ni</td>
<td>10 – 14</td>
</tr>
<tr>
<td>Mo</td>
<td>2 – 3</td>
</tr>
<tr>
<td>Mn</td>
<td>2</td>
</tr>
<tr>
<td>N</td>
<td>0.1</td>
</tr>
<tr>
<td>S</td>
<td>0.03</td>
</tr>
<tr>
<td>C</td>
<td>0.03</td>
</tr>
<tr>
<td>Si</td>
<td>0.75</td>
</tr>
<tr>
<td>P</td>
<td>0.045</td>
</tr>
</tbody>
</table>

### Table 2.8 Physical and mechanical properties of SS316L [41]

<table>
<thead>
<tr>
<th>Physical Properties</th>
<th>Value</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>7.99 g/cc</td>
<td></td>
</tr>
<tr>
<td>Mechanical Properties</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hardness, Rockwell B</td>
<td>79</td>
<td></td>
</tr>
<tr>
<td>Tensile Strength, Ultimate</td>
<td>558 MPa</td>
<td></td>
</tr>
<tr>
<td>Tensile Strength, Yield</td>
<td>290 MPa</td>
<td>0.2 % YS</td>
</tr>
<tr>
<td>Elongation at Break</td>
<td>50 %</td>
<td>In 2 inches</td>
</tr>
<tr>
<td>Modulus of Elasticity</td>
<td>193 GPa</td>
<td>Tension</td>
</tr>
<tr>
<td>Modulus of Elasticity</td>
<td>77 GPa</td>
<td>Torsion</td>
</tr>
</tbody>
</table>
2.8.3 Coating Material

Zirconium Nitride (ZrN) with 1 µm thickness

ZrN is a hard ceramic material. Applications of ZrN are coating medical devices, industrial parts, automotive and aerospace. It is also commonly used for coating of some parts which is in high wear and corrosive environments. ZrN coating is subjected to different types of materials with PVD (Physical Vapor Deposition) method. It has light gold color. Properties of ZrN were given in Table 2.9 [42, 43].

<table>
<thead>
<tr>
<th>Properties of Zirconium Nitride</th>
<th>ZrN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular formula</td>
<td>ZrN</td>
</tr>
<tr>
<td>Molar mass</td>
<td>105.23 g/mol</td>
</tr>
<tr>
<td>Appearance</td>
<td>Yellow-brown crystals</td>
</tr>
<tr>
<td>Density</td>
<td>7.09 g/cm$^3$ (20°C)</td>
</tr>
<tr>
<td>Melting point</td>
<td>2980°C</td>
</tr>
<tr>
<td>Solubility in water</td>
<td>Insoluble</td>
</tr>
</tbody>
</table>

2.9 Properties of Profilometers Used in the Study

2.9.1 3D Profiling System

Wyko NT1100 was used for wear and failure analysis. 3D surface roughness measurements were provided by Wyko NT1100 (Veeco, 2650 E. Elvira Rd., Tucson, AZ 85706 USA). Table 2.10 shows the specifications of Wyko NT1100 [44].
Table 2.10 Specifications of Wyko NT1100 [44]

| SYSTEM |
|------------------|--------------------------------------------------------------------------------------------------|
| Measurement Technique | Optical phase-shifting and white light vertical scanning interferometry |
| Measurement Capability | Three-dimensional, non-contact, surface profile measurements |
| Measurement Array | Max. array 736 x 480 |
| Software | Wyko Vision32 software running under Microsoft Window XP |

| PERFORMANCE |
|------------------|---------------------------------------------------------------------------------|
| Vertical Measurement Range | 0.1 nm to 1 mm |
| Vertical Resolution | < 1Å Ra |
| Field of View | 8.24 mm to 0.05 mm |
| Reflectivity | 1% to 100% |

| DIMENSIONS AND WEIGHT |
|-----------------------|------------------------------------------------------------------|
| Dimensions | 399 mm W x 508 mm D x 737 mm H(15.5 in. W x 20 in. D x 29 in. H) |
| Weight | does not exceed 56.7 kg (125 lbs) |

2.9.2 2D Profiling System

Dektak 150 was used for 2-D surface roughness measurements and channel height measurements in the study (Veeco, 2650 E. Elvira Rd., Tucson, AZ 85706 USA). Specifications of Dektak 150 were demonstrated in Table 2.11 [45].
**Table 2.11** Specifications of Dektak 150 [45]

<table>
<thead>
<tr>
<th>SYSTEM</th>
<th>Contact stylus profilometry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measurement Technique</td>
<td>Two-dimensional surface measurements</td>
</tr>
<tr>
<td>Measurement Capability</td>
<td>0.03 to 15mg</td>
</tr>
<tr>
<td>Stylus Force</td>
<td>Dektak software running under Windows XP</td>
</tr>
<tr>
<td>Software</td>
<td>120,000</td>
</tr>
<tr>
<td>PERFORMANCE</td>
<td></td>
</tr>
<tr>
<td>Scan Length Range</td>
<td>55mm standart</td>
</tr>
<tr>
<td>Vertical Range</td>
<td>1mm</td>
</tr>
<tr>
<td>Vertical Resolution</td>
<td>1Å max. (at 6.55μm range)</td>
</tr>
<tr>
<td>Lateral Accuracy</td>
<td>&lt;0.1% (on 55mm scan)</td>
</tr>
<tr>
<td>DIMENSIONS AND WEIGHT</td>
<td></td>
</tr>
<tr>
<td>Dimensions</td>
<td>292mm W x 508mm D x 527mm H</td>
</tr>
<tr>
<td>Weight</td>
<td>34kg (75lbs.)</td>
</tr>
</tbody>
</table>
References


[41] Lenntech Inc. website: www.lenntech.com/stainless-steel-316l.htm#ixzz1c2DJep1B.


CHAPTER 3:

INVESTIGATIONS ON THE VARIATION OF CORROSION AND CONTACT RESISTANCE CHARACTERISTICS OF METALLIC BIPOLAR PLATES MANUFACTURED UNDER LONG-RUN CONDITIONS

Tribological variations, surface conditions (roughness, hardness, coating) and surface interactions between micro-stamping dies and bipolar plate blanks play a critical role in determining the surface quality, channel formation and precision of bipolar plates. This study is aimed to understand the cause, mechanism and consequences of interactions between micro-stamping process conditions (speed, force, coating, die surface roughness and hardness) and bipolar plate quality (channel formation, surface roughness). The ultimate goal is to interrelate such interactions, if exist, to the bipolar plate performance (corrosion and contact resistance, and durability). A total of 2000 repeated micro-stamping of 51μm-thick uncoated and coated SS316L sheet blanks into an array of 750μm micro-channels were performed using 175-220kN force levels with constant stamping speed of 1mm/s. Microscopic examinations (surface roughness and channel height) were conducted periodically on both die and coated & uncoated plate surfaces to observe topographic variations. In addition, corrosion and contact resistance tests were carried out in the same intervals. Analysis of variance (ANOVA) technique was used to determine the significance of the process parameters on channel height, roughness, corrosion and contact resistance differences. The results revealed similar roughness trends for die and plate surfaces during 2000 micro-stampings. ZrN coating with 1μm thickness dramatically improved corrosion and contact resistance behavior of plates.
3.1 Introduction

Fuel cell technology is used to generate power via chemical reactions consuming hydrogen or hydrogen based fuels such as methanol, natural gas etc. and an oxidant such as oxygen. Proton exchange membrane fuel cells (PEMF), as shown in Figure 3.1, are considered to be an alternative to the internal combustion engines (ICE) for transportation applications. PEMFCs offer high efficiency, fast response, low operating temperature, comparatively long durability, low noise, lightweightness and compactness.

A PEM fuel cell mainly consists of ion conducting membrane, anode and cathode electrodes, bipolar plates and catalyst. Bipolar plate (BPP) is one of the primary components having important functions in a fuel cell stack. Tsuchiya and Kobayashi estimated the BPPs’ portion in total fuel cell stack weight and cost as 79%, and 45% in 2004, respectively [1]. In another study by Li and Sabir in 2005, these values were updated as 60%, and 30% [2]. With the continuous improvements in manufacturing technologies and optimizing the overall cost, a recent study noted that the cost of bipolar plates has fallen to about 25% of the stack [3].

BPPs have micro-channel arrays on both sides, which enable gases (H\textsubscript{2} and O\textsubscript{2}) flow through and distribute it equally for fast and efficient reactions. BPPs also provide electron conduction between cells as well as increase the strength and flexibility of fuel cell stack, which is quite vital in transportation vehicles. It also helps to dissipation of heat from the reaction sites, hence increasing the life of membranes [4, 5]. In order to perform these functions properly, bipolar plates must have chemical stability, electrical and thermal conductivity, low contact resistance, good mechanical strength, low gas permeability, and micro-channels for uniform reactant gas distribution [4].
Compared to other materials, metallic BPPs stand to be the most promising alternative due to their high electrical and thermal conductivity, mechanical strength, chemical stability, compatibility for mass production, and low cost. Commonly preferred metallic materials for BPPs are different grades of stainless steel alloys, Ni based alloys, Ti based alloys and Al based alloys. Fe based alloys are reported to be not appropriate materials for BPP since their corrosion resistance is very low that results in lower output in fuel cells [5]. Quite a few researches noted the suitability of stainless steel as BPP material [5-8]. The disadvantages of stainless steel bipolar plates were reported as relatively low corrosion resistance performance and high contact resistance between bipolar plate and other components of fuel cell. However, depending on the developments in coating material and coating technologies, it would be possible to address these challenges [9-11].

**Figure 3.1** Schematic of a PEM fuel cell and component thicknesses [Alternative Energy News]
Besides proper selection of the bipolar plate material, choice of manufacturing technique is also important factor for robust and cost-effective manufacturing. High-volume commercial manufacturing has been identified as one of the potential roadblocks to a future hydrogen economy [12]. Several methods have been proposed including machining, molding [13,14] stamping, hydroforming [15], flexible forming [16], electromagnetic forming, etc. [17]. Considering the requirements for BPP manufacturing in terms of both precision, production rate and cost effectiveness, stamping is considered as a prominent choice for fabricating bipolar plates [13]. In this process, precision dies with micro-machined channels are used for deforming thin metal sheets as shown in Figure 3.2. Hundreds of micro-channels, with total lengths of the order of 100-1000 m, must be embossed on each side of the bipolar plates. Advantages of stamping process are their low cost and fast production cycles for bipolar plate fabrication. In a single proton exchange membrane fuel cell (PEMFC) used for a typical passenger car, there are about 150 to 300 bipolar plates. Also a large set of micro-channels are incorporated inside each bipolar plate to flow liquid coolant. Therefore, each bipolar plate must satisfy stringent geometric accuracy (thickness and flatness), shape accuracy (channel dimensions, shape and distribution) and surface-quality requirements, while simultaneously fulfilling cost (< $3/kW² or ~$1/plate based on DOE cost targets for 2015) and production-rate (> 2 plates/sec) constraints [18].
Satisfying the stringent geometric, shape, and surface-accuracy requirements mentioned above necessitates developing a thorough understanding and modeling tools for controlling the accuracy and surface integrity of the micro-machined dies, as well as determining the process conditions and die wear during the micro-embossing process. A major concern in micro-manufacturing of high volume BPPs is the tribological changes and service life issues of micro-stamping dies in which BPPs are formed. These dies have micro-channels with high precision and highly smooth surface finishes. The surface quality of BPP produced is highly dependent on the micro-stamping die surface and process conditions due to the large ratios of surface area to volume ratios (size effects) that cause an increased level of friction and wear issues at the contact interface. Surface characteristics of BPPs such as surface roughness, hardness and stiffness are major features affecting the fuel cell performance. During the high volume and fast production rates of BPPs, mechanical characteristics of the BPPs may change because of repeated interactions between tool (micro-forming die) and workpiece (sheet blank of interest), at the contact interface. Evolution of surface mechanical characteristics at the tool and workpiece should be monitored, controlled and kept in acceptable range throughout the long production cycles to maintain the surface quality of BPPs. In micro-forming, highly localized stresses occur
under high contact pressure, and these may easily change the surface topography of tool which leads to variations on surface quality of bipolar plates. These topographic and tribological changes subsequently affect the corrosion and contact resistance of bipolar plates and overall fuel cell performance.

Although there are quite a few studies focused on tribological behavior in micro-forming based on the short-run experimentation, surface interactions between die and blank, changes in contact conditions and mechanics and the consequent effects on the product (in this case BPP) surface quality need to be studied in detailed, particularly considering the effects of long-run production conditions.

This study describes an experimental approach that aims to investigate the surface topography changes during long-run micro-stamping of BPPs, and establish relationships between surface topography and contact-corrosion resistance changes. Since the fuel cell performance is affected by the changes in corrosion-contact resistance of BPPs, surface topography changes are directly related with fuel cell efficiency that can be altered with the variations of surface topography. The methodology followed in this study was to monitor surface topography changes on both die and formed plate surfaces, and relate them to the corrosion-contact resistance of BPPs at certain production intervals. In the next section, experimental conditions and procedures for micro-stamping, corrosion and contact resistance tests are explained. In the third section, experimental findings and their analyses were presented. Conclusions are summarized in the fourth section.
3.2 Experimental conditions and procedures

3.2.1. Material

SS316L metallic sheet alloy with a thickness of 51µm (0.051mm) was chosen as material of interest for forming of uncoated and coated blanks. Stainless steel or Fe-Based alloys have been deemed to be good candidate as BPP material in early fuel cell studies due to their low cost, electrical and thermal conductivity and easy formability. Nonetheless, studies reported its poor corrosion resistance and unsuitability of its use without surface modification [3,7,19]. Mechanical and chemical properties of SS316 used in this study are given in Table 3.1 and Table 3.2, respectively.

2000 pieces of SS316L sheet blanks were prepared from 70mm x 70mm square sheets. 1000 of these sheet blanks were micro-stamped to fabricate 1000 uncoated BPPs, whereas the other 1000 were, first, coated with a ZrN using PVD coating technology, and then micro-stamped to produce 1000 coated BPPs. Coating properties are given in Table 3.3.

Table 3.1 Mechanical Properties of SS316L (as provided, Brown Metals, Rancho Cucamonga, CA)

<table>
<thead>
<tr>
<th>Condition/Tempering</th>
<th>Annealed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus of Elasticity (Tension)</td>
<td>193 GPa (28 x 10^6 psi)</td>
</tr>
<tr>
<td>Ultimate Tensile Strength</td>
<td>711 MPa (103,100 psi)</td>
</tr>
<tr>
<td>Yield Strength at 0.2% offset</td>
<td>341 MPa (49,400 psi)</td>
</tr>
<tr>
<td>Percent Elongation in 2 inches (*)</td>
<td>56.00%</td>
</tr>
<tr>
<td>ASTM Grain Size</td>
<td>10</td>
</tr>
</tbody>
</table>

- *The measured elongation will be less as thickness decreases to 0.002” and less.*
Table 3.2 Chemical composition of SS316L (as provided Brown Metals Company, Rancho Cucamonga, CA)

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>N</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.021</td>
<td>1.48</td>
<td>0.033</td>
<td>0.001</td>
<td>0.43</td>
<td>16.2</td>
<td>10.03</td>
<td>2.06</td>
<td>0.43</td>
<td>0.04</td>
<td>Bal</td>
</tr>
</tbody>
</table>

Table: 3.3 Coating Conditions (as provided by Tanury Industries, Lincoln, RI)

<table>
<thead>
<tr>
<th>Coating</th>
<th>ZrN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>1µm</td>
</tr>
<tr>
<td>Temperature</td>
<td>60 °C</td>
</tr>
<tr>
<td>Deposition Time</td>
<td>16.Min</td>
</tr>
<tr>
<td>Deposition Current</td>
<td>450A</td>
</tr>
<tr>
<td>Bias Voltage</td>
<td>75V</td>
</tr>
<tr>
<td>Sccm* Nitrogen</td>
<td>500</td>
</tr>
<tr>
<td>Sccm Argon</td>
<td>800</td>
</tr>
<tr>
<td>Pressure (m Torr)</td>
<td>5</td>
</tr>
</tbody>
</table>

* Sccm: Standard cubic centimeter per minute

3.2.2 Micro-stamping Experimental Set up and Conditions

Dies and inserts shown in Figure 3.3 were used in micro-stamping experiments. The die set mainly consists of male and a female inserts. Each insert was machined from H13 tool steel and had 26 arrays of micro channels with 750µm in depth as depicted in Figure 3.4. The micro-stamping set up was assembled into an Instron Satec 400 HVL (Instron Corp., Norwood, MA, USA), and was used in compression mode to provide a necessary stamping force. Figure 3.5 shows the formed plates using the stamping test setup.
Figure 3.3 Micro-stamping setup inserts & dies

Figure 3.4 (a) Insert, (b) Cross-sectional shape of female insert

Figure 3.5 (a) Example of stamped plates, (b) Digital microscope image of formed bipolar plate
3.2.3. Experimental Procedure

In the first set of experiments, 1000 coated blanks were micro-stamped with 175-220kN stamping force range, and stamping speed of 1mm/s. During the experiments, microscopic surface examinations (surface roughness and channel heights) were performed periodically (at every 200 stampings) for both die inserts and BPP surfaces to investigate topographic variations as depicted in Figure 3.6. In order to investigate the effect of surface topography changes on the corrosion resistance and contact resistance performances of bipolar plates, potentiodynamic corrosion resistance tests, and contact resistance tests were also carried out at every 200 stampings for at least two BPP samples. Followed by the micro-stamping of coated blanks, 1000 micro-stampings were performed using uncoated blanks. Same microscopic examinations and performance tests were applied in this test phase, as well.

![Figure 3.6 Experimental and measurement sequence of 1000 micro-stampings for both coated and uncoated blanks. (Surface inspections, roughness measurements, corrosion and contact resistance tests were performed at the beginning and after every 200 stampings.]

3.2.4 Measurements

Two different measurements were acquired from formed plates as surface roughness and dimensional measurements, which provides useful information on the topography and form
changes caused by manufacturing. Microscopic examinations were also performed to detect the possible visual change on the surfaces.

3.2.4.1 Surface Roughness Measurements

Surface roughness of BPP is important since BPP is in contact with other components of fuel cell such as gas diffusion layer (GDL), which in turn, is in contact with the membrane electrode assembly (MEA). Electrons are transferred from one cell to another via these contact surfaces and, any minute irregularities or undesired features on BPP surface will directly affect the contact conditions with GDL [20, 21]. Surface topography also affects the contact pressure distribution on the MEA, thus changing the electron transfer conditions directly. Similarly, any scratches or other surface irregularities may trigger the corrosion on BPPs in humid environment of fuel cell stacks. Long run manufacturing may elevate these surface roughness variations leading to drastic performance changes for fuel cells. Therefore, corrosion and contact resistance tests were performed on the BPPs at every 200 micro-stampings to correlate the surface topography changes with corrosion and contact resistance (CR and ICR) characteristics. Methodology followed for corrosion and contact resistance tests are given elsewhere by authors in an earlier study [22].

In this study, surface roughness values of both bipolar plates and stamping dies were measured with 2D and 3D measurement systems. At least three different types of roughness parameters (Ra, Rq, Rt for 2D and Sa, Sq, St for 3D) were obtained shown in Figure 3.7. Definition of these roughness parameters are given as follows:
**Ra, Sa**: Ra and Sa are the most commonly used roughness parameters. Ra is the absolute value of profile heights over a given length (area).

\[
Ra = \frac{1}{L} \int_{0}^{L} Z(x) \, dx \quad \text{(for 2D)}
\]

\[
5a = \frac{1}{A} \int_{0}^{L} \int_{0}^{Ay} Z(x, y) \, dx \, dy \quad \text{(for 3D)}
\]

Despite of widespread use, Ra has weaknesses as it does not include structure information and does not show the differences between peaks and valleys.

**Rq, Sq**: Determines the root-mean square value of 2D and roughness corresponding to Ra. Rq is statistically more meaningful parameters.

\[
Rq = \sqrt{\frac{1}{L} \int_{0}^{L} y^2(x) \, dx}
\]

**Rt or St**: The vertical height from the lowest valley to the highest asperity. It’s also called “peak to valley roughness”.

\[Rt = Rp + Rv\]

Rp: Maximum peak roughness

Rv: Maximum valley roughness [7,25]
Surface roughness inspections from various locations on both die and formed plate, as shown in Figure 3.8, were performed. Measurement for a certain point was repeated three times to ensure the repeatability of results. Microscopic pictures for the die and plate were given in Figure 3.9.
3.2.4.1.1: 2-d Roughness Measurements

A contact type profilometer (Dektak 150, Veeco Instruments Inc., Tucson, AZ, USA) was used in 2D roughness measurements. Roughness was measured both at the channel peaks and valleys. Instead of one whole line measurement, partial line measurements from five different locations, as shown in Figure 3.8, were acquired for comprehensive and accurate representation of surfaces. To address the repeatability of measurements, surface roughness
measurements were performed at least three times for each point. Table 3.4 shows the scanning parameters used in the roughness measurements.

2.4.1.2: 3-d Roughness Measurements

Wyko NT1100 optical profiler (Veeco Instruments Inc., Tucson, AZ, USA) was used for 3D roughness measurements. Surface roughness was measured at the channel peaks and valleys. As in 2D, in 3D roughness measurements were taken from five different locations as shown in Figure 3.8, and surface roughness inspections were performed three times. Scan parameters are given in Table 3.5.

**Table 3.4 2-d roughness scan parameters**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Scan Length</strong></td>
<td>5 mm</td>
</tr>
<tr>
<td><strong>Duration</strong></td>
<td>30 sec.</td>
</tr>
<tr>
<td><strong>Force</strong></td>
<td>3.00 mg.f</td>
</tr>
<tr>
<td><strong>Measurement location</strong></td>
<td>Channel peaks &amp; valleys</td>
</tr>
</tbody>
</table>

**Table 3.5 3-d roughness scan parameters**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Scanned Area</strong></td>
<td>450.6 x 600.8µm</td>
</tr>
<tr>
<td><strong>Magnification</strong></td>
<td>10.55</td>
</tr>
<tr>
<td><strong>Sampling</strong></td>
<td>938 x 67nm</td>
</tr>
<tr>
<td><strong>Array size</strong></td>
<td>640 x 480</td>
</tr>
</tbody>
</table>
3.2.4.2 Dimensional Measurements of Micro-Channels

Monitoring of the channel dimension variations is also important since it will directly affect the repeatability of micro-stamping process and dimensional stability of formed bipolar plates. Channel height values were measured using an optical microscope (Nikon Eclipse LV100). Alternatively, channel height measurements on the micro-stamped plates were conducted using Dektak 150 surface profiler. In addition, die surface was inspected for probable visual changes and surface defects using optical microscope.

3.2.4.3 Corrosion Resistance Tests

The surface of metallic BPPs can deteriorate in corrosive working environment as in real fuel cell conditions. This may result in reduced efficiencies and power losses which consequently lead to additional operation and maintenance costs. It is reported that surface topography variations influence corrosion resistance behavior of metallic BPPs [23]. In this study, potentiodynamic corrosion tests were performed to observe the corrosion resistance behavior of BPPs. Micro-stamped BPPs were exposed to corrosive environment by applying O₂ gas bubbling, which represents the cathodic conditions in a real fuel cell. The tests were carried out in 80°C to imitate the PEM fuel cell working conditions.

3.2.4.4 Contact Resistance Tests

Four-probe method was used for the investigation of interfacial contact resistance (ICR) between GDL (gas diffusion layer) and BPP sample. Before subjected to ICR tests, BPPs were degreased in ultrasonic acetone bath. Then, a stack was constituted sandwiching the GDL and BPP between two gold coated copper current collector. This stack was then compressed by means of an electromechanical testing system (MTS Insight 30, MTS System Corp., Eden Prairie,
under the incrementally increasing load levels. ICR values were measured under increasing pressure from 20 N/cm² to 284 N/cm² with 24N/cm² increments. When measuring the ICR, the bulk resistance of BPPs and GDLs were considered negligible since bulk resistance values of parts were too small compared to the ICR (interfacial contact resistance) values. Two different plates produced for same process conditions were tested under same pressure value to confirm repeatability of test system.

3.3 Results and Discussion

3.3.1 Repeatability of stamping process and effect of coating on channel formability

The channel height values measured using a surface profilometer was plotted in Figure 3.10 for after every 200 stampings. The results indicate that maximum channel height variations are %10.2 and %10.8 for the uncoated and coated stamped plates, correspondingly. Obvious channel height differences were observed between coated and uncoated samples (Average height is 278 µm for uncoated plates whereas coated plates has 214 of µm average channel height value). This can explained by the difference in surface hardness values of the coated and uncoated plates. Micro-hardness values of uncoated and coated blanks were measured as 157 and 170 (HV₀.２), respectively. The die channel height measurements, on the other hand, showed insignificant (sub-micron) changes only.

ANOVA analysis was performed to understand if the channel heights of coated and uncoated plates differ significantly during the 1000 stampings. A p of 0 (zero), which is significant for α of 0.05 (confidence interval of 95%), was obtained from comparison of uncoated and coated plate channel heights. However, channel height variations for both coated
and uncoated samples during each 1000 production cycle were not found to be statistically significant (p=0.166 and p=0.131 for uncoated and coated plates respectively), suggesting that dimensional stability of the micro-stamped plates, so the process were robust. No significant visual surface pattern change was observed on the die surfaces when the die surface before and after 1000 stampings as shown in Figure 3.11.

![Channel Height Measurements](image)

**Figure 3.10** Channel Height measurements of Uncoated and Coated BPPs
Surface inspections were performed on the uncoated and coated, unformed and formed plate, presented in Figure 3.12, using optical microscope (Nikon Eclipse LV100). Significant visual pattern change was observed before and after stamping of the blank for both uncoated and coated BPPs. On the other hand, the visual surface pattern change between 1\textsuperscript{st} BPP stamped and 1000\textsuperscript{th} BPP was not clear. Surface pictures were also taken with Scanning Electron Microscope (SEM) on uncoated and coated samples before and after forming shown in Figure 3.13. Some irregularities were observed on formed uncoated and coated samples.
Figure 3.12 Surface inspection on the surfaces of a) uncoated-unformed blank, b) 1st uncoated BPP, c) 1000th uncoated BPP, d) coated-unformed blank, e) 1st coated BPP, f) 1000th coated BPP
3.3.2 Elemental Microanalysis of ZrN Coated BPPs

Cross section of coated BBP and a plot of x-ray counts vs. energy were shown in Figure 3.14 and Figure 3.15, respectively. The cross sectional SEM image confirmed the average thickness of ZrN coating ($t \approx 1 \mu m$). Various elements in the BPP were corresponded by energy peaks. Most elements built multiple peaks. For instance, strong $K_\alpha$ and $K_\beta$ peaks were demonstrated by iron.
EDX microanalyses were performed for quantitative determination of the elemental composition of the coated BPPs. These microanalyses reveal the predominance of iron, with peaks at 6.400 keV and 7.100 keV, Zr with a peak at 2.050 keV, Cr with a peak at 5.400 keV. Minor amounts of Ni with a peak at 7.400 keV have also been detected. Elemental quantitative analyses yield the weight of the elements present in Table 3.6. As a result, SEM and EDX analysis revealed a higher amount of Zr material among some other elements on coated BPP surfaces.

![Cross section of SS316L BPP coated with 1µm-thick ZrN](image)

**Figure 3.14** Cross section of SS316L BPP coated with 1µm-thick ZrN
Figure 3.15 Elemental EDX microanalyses of the coated BPP (ZrN-1µm), (a typical spectrum)

Table 3.6 Typical elemental EDX microanalysis (Data are expressed as both weight and atomic percentages.)

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZrL</td>
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<tr>
<td>CrK</td>
<td>13.34</td>
</tr>
<tr>
<td>FeK</td>
<td>52.25</td>
</tr>
<tr>
<td>NiK</td>
<td>7.46</td>
</tr>
</tbody>
</table>
3.3.3 Effect of micro-stamping process on the surface topography

Surface roughness measurements were taken on a rectangle surface (450.6µm x 600.8µm) on the BPPs and stamping dies shown in Figure 3.16. The measurements results revealed that roughness on the plate surface increased significantly (e.g. from 0.17 µm to 1.75µm). 3-d surface roughness measurements for coated & uncoated SS 316L plates as well as for forming die were illustrated in Figure 3.17 for one of the measurement locations (3A) as the measurements. Measurements from other locations exhibited similar patterns. Surface area roughness ($S_a$) values were presented for the micro-stamped uncoated and coated plates. Variations (%) in surface roughness of the female die peak and plate valley were tabulated in Table 3.6 roughness variations for female die peak during micro-stamping of 1000 uncoated were between ~2.4 - 5%, and were ~0.5 - 6% for 1000 coated plates. Variations for uncoated and coated BPP valley locations, on the other hand, were found to be in the range of ~0.6 - 8% and ~1 - 12%, respectively. When die and micro-stamped bipolar plate roughness values were compared, it was observed that the variations for the die surface roughness values are lower than that of micro-stamped blanks, as expected. However, the variations were observed to be in a very narrow range (less than 1µm) for die and plate roughness values. Increased number of micro-stampings is necessary to reveal even longer-term manufacturing effects and accurate relations between the die-blank surface interactions. It was also observed that the surface roughness change for the die and plates was unsteady. It is assumed that, as discussed in Section 2.2, the continuous change of surface topography (i.e., asperity) due to smoothened asperities and cold welded tips led to varying surface interactions between stamping dies and BPPs during 1000 micro-stampings. A trend might be observed in a longer run manufacturing case. However, the results thus far revealed mostly similar roughness changes for die and uncoated and coated plates.
Coating material did not demonstrate any clear difference in terms of surface roughness after fabrication of BPPs.

ANOVA analysis was also performed to reveal the significance of the roughness variations. The roughness changes on the uncoated and coated BPPs and on the die during micro-stamping were examined separately. During the analysis of roughness variation on both die and BPPs, three different measurements at five different locations at the same interval of micro-stamping (i.e., 1st, 200th, 400th, 600th, 800th, 1000th) were taken into account. According to ANOVA results, roughness variations on die surface was negligible (p=0.288) during the micro-stamping of coated plates whereas it was found to be significant (p=0.035) for micro-stamping of uncoated plates suggesting that die surface conditions would change significantly in long-run micro-stamping of uncoated BPPs, potentially affecting the BPP surface condition, and hence the fuel cell performance, consequently. However, changes in surface roughness values obtained between uncoated and coated BPPs at the corresponding stages of micro-stamping were found to be insignificant.

Figure 3.16 Surface roughness change after micro-stamping
3.3.4 Effect of surface roughness on the corrosion resistance of the micro-stamped uncoated and coated BPPs

Potentiodynamic corrosion test results for uncoated and coated samples were presented in Figure 3.18. In the figure, high current density values stand for low corrosion resistance. When
current density values of uncoated and coated BPPs were compared, it was noted that uncoated BPPs have higher current density (low corrosion resistance) than the coated BPPs. Average current density for uncoated BPPs was measured as 7.04 µA.cm⁻² while coated BPPs yielded an average current density of 2.05 µAcm⁻². Hence, it can be concluded that the coating of SS316L blanks with ZrN improved the protection of BPPs against corrosion significantly due to the corrosion protection characteristics of ZrN, as discussed in Section 2.8.3. It is also noted that the recorded corrosion current density values are close to satisfy the target level (1 µA.cm⁻²) set by U.S. Department of Energy [24]. Figure 3.19 shows the trends for corrosion current density and the surface roughness values at corresponding steps. Similar to surface roughness variations, corrosion resistance of BPPs showed unsteady variations during 1000 micro-stamping process both for coated and uncoated cases. Coated and uncoated BPPs showed very similar trends when their roughness vs. corrosion resistance performances were compared. Current density values are increased when the surface roughness values are increased. This means that surface roughness increase resulted in increased corrosion resistance. In addition, it was observed that the corrosion resistance variation for coated BPPs was lower then that of uncoated BPPs, suggesting that coating of BPPs not only increases the level of corrosion resistance but also offers less variation in corrosion behavior. However, additional and longer-run micro-stamping experiments might be needed to establish a direct relation with a high certainty between surface roughness variation and the corrosion resistance performance of bipolar plates.

ANOVA analyses showed that there was no significant difference in the current density variation for both uncoated and coated BPPs taken at different stages of micro-stamping runs (i.e., 1ˢᵗ, 200ʰ, 400ʰ, 600ʰ, 800ʰ, 1000ʰ) as p=0.123 and p=0.333 was obtained for uncoated and coated BPPs, respectively.
**Figure 3.18** Potentiodynamic corrosion test results for uncoated and coated stamped samples w/O₂ (200kN/s-1mm/s)
Figure 3.19 Average roughness-corrosion resistance relations for a) coated b) uncoated plates

3.3.5 Effect of surface roughness on the contact resistance of uncoated and coated BPPs

Average contact resistance values of uncoated and coated BPPs were measured as 293mΩ.cm² and, 132mΩ.cm² respectively as seen in Figure 3.20. It is deduced that coating material (ZrN) increased the conductivity of BPPs, and reduced the contact resistance between
BPP and GDL, at certain extent. Nevertheless, the contact resistance values were still much higher than the DOE target levels [24]. Figure 3.21 depicts the relation between surface roughness and contact resistance variation for coated and uncoated BPPs. Although there is not a strong correlation due to the low roughness changes on the BPPs, the relation between roughness and contact resistance in uncoated and coated BPPs was found to follow the same trend. The reason might be the increasing area of asperities led to lower contact surface area. Similar to surface roughness changes on the BPPs, contact resistance of micro-stamped BPPs did not follow a certain pattern. Further investigations are necessary to obtain a stronger correlation between high contact resistance and high surface roughness variations as obvious from the ANOVA. It resulted in insignificant differences for contact resistance tests results (for uncoated and coated BPPs, p=0.134 and p=0.368, respectively).

Figure 3.20 Contact resistance test results for uncoated and coated stamped plates
Figure 3.21 Average roughness-contact resistance relations for (a) coated plates (b) uncoated plates
3.4 Conclusions

1000 coated + 1000 uncoated blank were formed into fuel cell bipolar plates via stamping process, consecutively to observe the surface topography changes and its effect on corrosion and contact resistance behavior.

Surface topography changes were encountered at insignificant levels for both die and formed bipolar plate surfaces. It was concluded that although there were unsteady changes in roughness values during the stamping of the BPPs, the surface roughness trends for mating surfaces (female die peak-plate valley) were similar.

The results also showed different formability levels for coated and uncoated samples. Higher channel height values were obtained for uncoated BPPs that are believed to be caused by different surface finishes as well as hardness properties of uncoated and coated plates. Dimensional stability and repeatability was confirmed in forming of both uncoated and coated BPPs during the long-run manufacturing. The die channel height measurements, on the other hand, showed insignificant (sub-micron) changes.

Corrosion resistance test results demonstrated that corrosion resistance of coated plates is much higher than uncoated plates, as forecasted initially. Boosting effect of ZrN coating was observed against corrosion, though corrosion resistance was not at desired level.

Contact resistance test results indicated that uncoated plates exhibited higher level of contact resistance compared to coated plates. It can be deduced that ZrN is favourable but not sufficient to satisfy the DOE target levels for both corrosion and contact resistance values. The relation between surface roughness and contact resistance was deemed to be direct.
As a general conclusion, longer runs of micro-stamping experiments are proposed to establish a stronger correlation between topography changes and corrosion, contact resistances, based on the evidence of findings.

3.5 Contributions

Although there are quite a few studies focused on tribological behavior in micro-forming based on the short run experimentation, surface interactions between die and blank, changes in contact conditions and mechanics and the consequent effects on the product (in this case BPP) surface particularly considering the effects of long-run production conditions were not studied enough. This study is unique since it includes all these information.

Investigations on tribological behavior in micro-forming mostly focused on micro-deep drawing and micro-extrusion. Since very limited number of studies based on stamping process which is appropriate for mass production in micro scale, this study is significant.

Surface treatment techniques were commonly used in surface topography studies. In this study, coating was applied on sheet blanks. As a coating material, ZrN was selected taking into account the corrosion resistance and contact resistance behavior of some other coating materials such as CrN and TiN. These tests were made in other studies of our group. After forming the ZrN coated blanks, corrosion and contact resistance tests were performed. So, this study is significant, since it gives the comparison between coated and uncoated BPPs in terms of surface topography changes during long run manufacturing conditions and corrosion and contact resistance behavior.
This study attracts attention since formability levels and repeatability characteristics of uncoated and coated BPPs were assessed. It revealed that coated BPPs showed lower formability than uncoated BPPs due to the hardness difference between uncoated and coated BPPs.

Significance levels of surface topography changes of stamping dies and BPPs were analyzed and demonstrated in this part of the study via analyses of variance (ANOVA) technique.
References


CHAPTER 4:

INVESTIGATIONS OF MICRO-SCALE SURFACE INTERACTIONS AND TRIBOLOGICAL SIZE EFFECT IN MICRO-STAMPING OF SS316L SHEETS

Micro-stamping is a very convenient sheet-metal forming technology providing small tolerances, surface accuracy, low cost and fast production of thin metallic sheets. Compared to macro-forming operations, tribological changes in micro-forming processes are more dominant and critical. Therefore, tribological size effect should be considered for better understanding of tribological changes in micro-scale. The integrity of process simulation to the experiments is essential. The objective of this study is to establish numerical and experimental models for micro-stamping of stainless steel blanks regarding the tribological size effect and determine the effects of feature size of the dies and stamping force onto friction, von-Mises stress, plastic strain, part thickness changes. Numerical models of micro-stamping process for the dies with different channel sizes, 250 µm, 750 µm and 2500 µm were established in commercially available FEA package Ls-Dyna. Several friction conditions were applied and runs performed and then, von-Mises stress, plastic strain distributions, and thickness changes for the micro-stamping process were obtained. Experimental micro-stamping tests were conducted to verify the established FE models. The channel heights of formed parts obtained from experimental and numerical simulations for 100kN, 200kN and 300kN stamping force conditions were compared to investigate friction coefficient occurred at the die-stamped part interface. The results indicated that the von-Mises stress, and plastic strain increased with the increasing stamping force whereas part thickness was reduced, as expected. The dies with 250 µm, 750 µm and 2500 µm channel
sizes showed different magnitudes and distributions of von-Mises stress and plastic strain. In addition, different friction coefficients were encountered for the micro-stamping dies with 250 µm, 750 µm and 2500 µm channel sizes. The lower channel size the higher coefficient of friction was observed as a result of tribological size effect.

4.1 Introduction

The demand for miniaturization is incessantly growing with the technological developments in micro-electromechanics, electronic health care and automobile industries. Overall worldwide market value of micro- and nanomanufacturing sector was forecasted as $25 billion by 2009 [1]. Specifically, the market size for micro medical manufacturing in which micro components and mechanisms are involved is expected to reach $ 23 billion, by 2014 [2]. As a result of this demand, the research and industrial activities on mass manufacturing of micro-scale parts such as micro-gears, pumps, connector pins, miniature screws, contact springs has been a trend. In this respect, metal forming methods are preferred and adapted to microforming since these methods are appropriate for high production rates, and can provide high quality final product surface, and high strength with a low cost [3-6]. On the other hand, it is challenging to produce form micro-scale parts in big quantities and very tight tolerances compared to macro-scale process. Some of these challenges that need to be properly addressed are as follows; (1) Influence of size effect on material behavior, (2) Unknowns in contact mechanics, surface interactions and macro scale tribology, (3) Lack of reliable modeling and simulation of micro-forming processes, (4) Machine and equipment problems [6,7]. Different from macro forming, in micro forming the ratio of surface area to volume of workpiece is high that causes size effect
In addition, due to the higher ratio of surface area to volume, an increased level of friction and wear issues at the contact interface are experienced [10,11]. The friction at the tooling/workpiece interface in sheet metal forming operations determined the formability level of the sheets [12-14]. Thus, the energy and force required for forming sheets can be minimized and longer tool life and better surface quality of tool-workpiece can be provided via reduction of friction [12, 15, 16]. Therefore, determination of coefficient of friction is critical particularly in micro-scale [17]. FE based models needs to be integrated to the experiments for more reliable friction analysis. Quite a few studies are available in literature on the Hall-Petch equation based grain size effect, however; limited number studies investigated the tribological size effect and determination of coefficient of friction. Hu and Vollertsen carried out strip drawing tests and FEA to identify the tribological size effect [18]. They concluded that friction increased with miniaturization. Bin et al. investigated tribological size effect through cylinder compression tests and found that as tribological size effect encountered in lubricated condition whereas it is not observed in dry condition [19]. Engel studied the frictional size effect performing double-cup-extrusion tests and revealed that scaling down the process dimensions increased the friction seriously [20]. In a similar study by Liu et al., size effect and effect of the friction coefficient on the micro extrusion process with FEA was studied and the results showed that the increasing ratio of the surface to the volume increased the friction coefficient that leads to increase in forming load [21]. In an another attempt by Peng et al, micro-scale soft punch stamping process, a rigid die and rubber as a flexible medium involves in only, was investigated with experiments and simulations. They noted that sheet blanks with smaller grain size is prone to attain higher formability. They noted that friction forces increase with miniaturization that leads to reduce formability [22].
Compared to micro strip/deep drawing and micro extrusion, micro-stamping is more prevalent technique in producing micro-parts and has not been investigated in detail, thus far. Current paper is therefore, dedicated for investigating tribological size effect via performing micro-stamping experiments with thin metallic sheet blanks and establishing FE models and their validation. It aims also establishing FE models and their validation.

4.2 Experimental conditions

SS316L metallic sheet alloy with a thickness of 51µm (0.051mm) was chosen as material of interest for forming of blanks, since SS316L offers low cost, electrical and thermal conductivity and easy formability [23, 24]. Total number of 36 SS316L sheet blanks were prepared in 30mm x 70mm dimensions. Rectangular strip blanks were preferred in the current study based on the previous research noting that the die corners cause tangential pressure in the blank and causes problems in numerical calculations [18, 25]. 3 different die pairs with different channel heights, 250, 750 and 2500 µm (representing the scale factors of X, 3X, and 10X) shown in Figure 4.1 were used to stamp the sheet strips. Each die pair was made from H13 tool steel with the dimensions of 40mm X 40 mm X 20 mm and had 67, 26, 9 arrays of micro channels, respectively. Blank holders used in the test setup were made from rubber material. Rubber blank holders protected the strips against the possible micro-cracks during the forming process. The optimum dimensions of blank holders were determined regarding the highest channel height that can be attained while avoiding the micro-cracks on the blank. The variables in micro-stampings was the blank holder dimensions and channel sizes of the dies used while same sheet strip and blank holders materials were used in all experimentation. Dies and blanks with almost same roughness values were employed in experiments to minimize the effect of surface quality on results [7, 26].
Figure 4.2 illustrates the micro-stamping procedure employed. The test setup mainly consists of a male and a female dies. The micro-stamping setup was assembled into an Instron Satec 400 HVL (Instron Corp., Norwood, MA, USA), and this machine was used in compression mode to provide a required stamping force. Then, rectangular sheet blanks were micro-stamped with three different stamping force levels 100, 200 and 300 kN stamping force and under constant stamping speed (1 mm/s). Figure 4.3 shows the strips stamped using the dies with 250 µm, 750 µm and 2500 µm channel heights while Table 4.1 summarizes the experimental plan conditions.

Figure 4.1 Stamping dies with (a) 250 µm, (b) 750 µm and (c) 2500 µm channel heights
**Figure 4.2** Description of micro-stamping process

**Figure 4.3** Stamped SS316L sheet strips with dies (a) 250 µm, (b) 750 µm and (c) 2500 µm channel heights
Table 4.1 Experimental Cases

<table>
<thead>
<tr>
<th>Forming Die Channel Height (µm)</th>
<th>Stamping Force (kN)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>100</td>
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<tr>
<td>250</td>
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<tr>
<td></td>
<td>300</td>
</tr>
</tbody>
</table>

4.3 FE model for micro-stamping experiments

Finite element based simulations are appropriate for the prediction of the performance of the stamping process. However, FE modeling softwares don’t consider the size effect. Thus, experimental data that determines size effect should be integrated to the numerical model [13].

The numerical simulations were performed using commercial explicit/implicit finite element code Ls-Dyna for the micro-stamping processes employed three different channel sized dies (250, 750 and 2500 µm). In order to simplify the analysis of stamping process and reduce the simulation time, 2-dimensional half models were built in Ls-Dyna. At first, die, blank, and blankholder geometries were prepared using SolidWorks CAD tool. Then, the models were prepared, as demonstrated in Figure 4.4, for finite element analyses in Ls-Dyna. The FE models consisted of 37172, 28840 and 28613 4-node quadrilateral elements for the processes with 250, 750 and 2500 µm channel height dies, respectively. Male and female dies were defined as rigid materials, whereas the blanks and blank holders were specified as deformable bodies. Elastic
mechanical properties for blanks, male and female dies such as elasticity modulus, density and poisson ratio were acquired from literature as $E = 193$ GPa, $\rho = 7.99$ g/m$^3$, $\nu = 0.31$, respectively [27]. Power law material model with strength coefficient value (K) of 1058 and strain hardening exponent (n) of 0.28 were utilized for blanks. Blatz-Ko Rubber material model with mass density of 0.95 t/mm$^3$ and shear modulus of 28 N/mm$^2$ was used for blankholder. Coulomb friction model was defined for all contact surfaces. All contact conditions between upper (male) die and blank, lower (female) die and blank, blank and blankholder, blankholder and lower die (female) were selected as “2D Automatic Node to Surface” contact algorithm [28]. A very wide range of coefficients of friction values ($\mu = 0.001, 0.01, 0.02, 0.03, 0.04, 0.05, 0.1, 0.2$ and $0.3$) were employed in the simulations. The upper die was moved down with a constant stamping speed of 1 mm/s, and a blank holder force of 3000 N was applied on blank holder. Finally, implicit solver was imposed as solution algorithm in the simulation due to allow for much larger time steps [29].

Figure 4.4 2D FE models of stamping process for the dies with the channel dimensions of (a) 250 µm, (b) 750 µm and (c) 2500 µm.
4.4 Results and Discussion

The experimental conditions and corresponding numerical results for micro-stamping simulations are presented in this section, and effect of process parameters like stamping force and surface features on results such as sheet formability and friction conditions are discussed.

4.4.1 Effect of stamping force and die channel size on sheet formability

As it is well known in literature, friction plays an important role in sheet metal forming operations as it affects the formability [11, 20, 28]. In this study, the formability characteristics of SS316L sheet blanks were investigated experimentally and numerically. Micro-stamping experimental conditions were simulated with FEA; and resulting highest strain, von-Mises stresses as well as maximum thickness changes for different force levels and die channel heights as listed in Table 4.2. As expected, von-Mises stress, part thickness changes and plastic strain values increased with increasing stamping force. After performing 100 kN stamping force, maximum thickness change was occurred as 25.3 µm in the die with 750 µm channel height (from 51 µm to 25.7 µm) whereas minimum thickness change was observed as 1.4 µm in the 2500 µm channel height (from 51 µm to 49.6 µm). However, increasing stamping force from 100 kN to 200 kN and 200 kN to 300 kN lead to increase the thickness change at most in the die with 2500 kN channel height as 2.3 µm (from 1.4 µm to 3.7 µm) and 1.9 µm (from 3.7 µm to 5.6 µm) respectively.

Die channel size was also effective on formability [30]. As it is presented in Table 4.2, the lowest von-Mises stress, strain levels and thickness changes were recorded for the sheet strips formed with 2500 µm channel height die. As larger channels let the workpiece displace into the channel easier, the formability of the sheets is limited. When the parts formed with 250
μm and 750 μm are compared in terms of deformation characteristics, the higher von-Mises stress, plastic strain and thickness changes values were obtained for the strips formed with 750 μm die. Because small features of the die with 250 μm channel height resisted to reach higher stress-strain levels and thickness changes. Experimental data (load-time curve) shown in Figure 4.5 confirmed these results. It was seen during the same time increment that the energy transferred to the sheet strips was higher for 750 μm case than 250, and 2500 μm cases. The reason for higher plastic strain in experiments with 750 μm die case might be due to the use of thicker rubber parts (0.1 mm thick in 250 μm, 0.2 mm in 750 μm case).

It was seen that the strain and von-Mises stress distributions for formed strips followed similar trends, as such only strain distributions were presented in Figure 4.6. Higher stress values were encountered for the curved parts of the strips due to increasing stretching effect in these locations.
### Table 4.2 Experimental conditions and corresponding FEA results for the micro-stamped strips

<table>
<thead>
<tr>
<th>Forming Die Channel Size (µm)</th>
<th>Stamping Force (kN)</th>
<th>Thickness Change (µm)</th>
<th>Plastic Strain</th>
<th>Max. von-Mises Stress (MPa)</th>
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</thead>
<tbody>
<tr>
<td>250</td>
<td>100</td>
<td>10.5</td>
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<td>5.6</td>
<td>0.13</td>
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</tbody>
</table>

**Figure 4.5** Load-time curves obtained from experiments for micro-stamping with different dies
4.4.2 Effect of die channel height on the friction coefficient

Friction coefficient can vary depending on material, surface quality of tool/workpiece, lubrication and also the normal pressure on the contact surface [25]. Surface features on the dies also dictate the friction levels in microforming [30]. In this part of the study, friction coefficients between the sheet strips and dies at certain force levels were estimated numerically and measured from real samples. To this goal, different Coulomb coefficient of friction values were imposed to FE models (See Table 4.3) and analyses performed. Then, the channel heights of the sheet
strips micro-stamped were acquired from FEA results and compared with corresponding experimentally obtained ones. Table 4.3 shows the FEA estimations and experimentally obtained channel height values for each tested case. Based on this channel height comparison the closest channel height values are used to estimate corresponding coefficient of friction value ($\mu_{\text{exp}}$). The experimental channel height measurements ($h_{\text{exp}}$) given in Table 4.3 were obtained by using a contact type profilometer (Veeco Inc., Tucson, AZ). For example, sheet strip stamped with 250 $\mu$m die yielded channel height values of 162, 183, and 190 $\mu$m for 100, 200, and 300kN stamping force levels, experimentally. Considering these values listed in Table 4.3, experimentally obtained channel height values fall into the range where coefficient of friction is in between 0.04 and 0.05. Hence, it is concluded that the coefficient of friction during the micro-stamping of strips at 100 kN and 250 $\mu$m die channel height case is in the range of 0.04-0.05. Similarly, as seen in Figure 4.7, coefficient of friction value was estimated in the range of 0.01-0.02 for stamping of strips at 100 kN and in 2500 $\mu$m channel height die. For the part stamped with 750 $\mu$m of die channels, the channel height values for 100kN, 200kN and 300kN stamping force levels were acquired as 630 $\mu$m, 654 $\mu$m and 652 $\mu$m, respectively. These channel height values were found to be in between the channel height values attained from FEA for coefficient of friction values of 0.02 and 0.03. Coefficient of friction values were estimated with an accuracy of 0.01 for all tested cases. Another important finding was that the coefficient of friction value did not change significantly for the same forming die with increasing stamping force level. However, some changes might be seen in higher or lower force levels. It can be concluded that geometry of the forming die is more effective than the stamping force ranges experimented in this study.
Table 4.3 Experimentally and numerically obtained channel height values for different force and forming die conditions.

<table>
<thead>
<tr>
<th>Die Channel Size (µm)</th>
<th>Coefficient of Friction Imposed in FEA (µ)</th>
<th>Channel Height Values Obtained for Different Forces in FEA (µm)</th>
<th>100kN</th>
<th>200kN</th>
<th>300kN</th>
</tr>
</thead>
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<tr>
<td></td>
<td>0.1</td>
<td>712</td>
<td>804</td>
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<td></td>
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<td></td>
<td>0.03</td>
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<td></td>
<td>0.04</td>
<td>681</td>
<td>713</td>
<td>752</td>
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<tr>
<td></td>
<td>0.05</td>
<td>692</td>
<td>785</td>
<td>777</td>
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<td>0.1</td>
<td>660</td>
<td>749</td>
<td>728</td>
<td></td>
</tr>
</tbody>
</table>

$\mu_{exp}$: Estimation for coefficient of friction in the experiment

$h_{exp}$: Estimation for channel height in the experiments
Figure 4.7 Channel height measurements in 100kN stamping force for friction coefficient of (a) 250 µm, (b) 750 µm, and (c) 2500 µm
4.5 Conclusion

Formability and surface interactions for between micro-stamped SS 316L sheet strips and micro-stamping dies were studied both experimentally and numerically. Channel height formation was taken as measure for formability. Micro-stamping experiments were performed using three different dies with distinct channel height values (x, 3x, 10x) at different stamping force levels to investigate the tribological size effect. It was found that when the forming die dimensions get smaller the frictional conditions get harsher. Hence, tribological size effect was confirmed with the results. Interestingly, increasing force level used in the experiments and simulations did not affect the coefficient of friction value in micro-forming of sheet strips with a certain die. On the other hand, plastic strain, equivalent stress so the formability levels were increased with increasing stamping force, as forecasted initially.
References


[27] AK Steel Corporation, West Chester, Ohio, USA. Available also at: www.aksteel.com. Last access date: 09/24/2011.


CHAPTER 5:

SURFACE TOPOGRAPHY EVOLUTION DURING LONG-RUN MICRO-STAMPING OF BPPs AND ITS EFFECT ON CORROSION AND CONTACT RESISTANCE CHARACTERISTICS

In long-run micro-stamping process, surface topography and tribological conditions can vary because of the repeated surface interactions between micro-stamping die and bipolar plates (BPPs). Long run stamping process does not only impact die wear/life but also the bipolar plate geometric accuracy, formability and surface quality characteristics. These characteristics, in turn, would affect the corrosion and contact resistance of BPPs, consequently the fuel cell performance. Therefore, understanding of the topography and tribological changes caused by micro-stamping process conditions is critical. The aim of this study was to investigate the reasons and consequences of the interactions during the mass manufacturing of micro-stamped sheets, and establish correlations between surface interactions vs. corrosion and contact resistance of BPPs. In experimental part of this study, 10,000 SS316L sheet blanks with 51µm in thickness were micro-stamped using a set of stamping dies with 750µm micro-channels under 175-225kN stamping force levels with constant stamping speed of 1mm/s. Surface inspections (surface roughness and channel height) were monitored periodically. Corrosion and contact resistance tests were also carried out on the BPPs. Analysis of variance (ANOVA) technique was performed to investigate the significance of the variations of the surface roughness, channel heights, corrosion and contact resistance of BPPs. On the other hand, Three-dimensional (3D)
finite element models of micro-stamping process were established. von-Mises equivalent stress, plastic strain and final sheet thickness values and distributions were determined and compared for different process conditions. The channel height values obtained from experimental and numerical modeling were compared to determine the coefficient of friction at the contact interface of micro-stamping dies and BPPs. The variation of the coefficient of friction was investigated at every 1000-stamping interval as well. The results revealed that the roughness values for the female, male dies and BPPs followed similar trends during 10,000 micro-stampings. The corrosion resistance variation as a consequence of long-run manufacturing was found to be statistically insignificant, and the relation between surface roughness and corrosion resistance was found to be indirect. On the other hand, direct correlation was observed between roughness and contact resistance. Finally, experimental and numerical channel height analyses results showed that the coefficient of friction did not change considerably during the mass production of BPPs, at least within the 10000 stamping cycle.

5.1 Introduction

Proton exchange membrane (PEM) fuel cells have attracted interest from researchers due to their high efficiency and cleanliness. PEM Fuel cells are appropriate for the portable power and transportation applications mainly due to their low operating temperature levels and relatively quick-start features. However, cost and durability issues prevent PEM fuel cells from widespread use [1]. Since each fuel cell produces 0.6-0.9 V only, hundreds of fuel cells should be stacked together to be used in applications where much higher voltages are needed. If the mass manufacturing of PEM is considered, the cost issue becomes more critical. When the cost
of fuel cell power is compared with the internal combustion engines, fuel cell power is 4-10 times more expensive ($30-$50/kW vs. $200-$300/kW) [1-4].

Bipolar plate stands for high volume and high cost component of PEM fuel cells (60-80% of stack weight, 30-45% of stack cost) [5]. Molded graphite material is widespread BPP material for PEM fuel cells due to their excellent electrical conductivity and chemical stability. However, graphite BPP is not competitive in the energy market because of high manufacturing cost and time which is $10.42 and 45 seconds per plate, respectively [6]. Formed thin metallic sheets are promising candidates for a PEM fuel cell stack with high efficiency and low cost [7]. In addition of these advantages, metallic BPPs offer high electrical and thermal conductivity, chemical stability, compatibility for mass production, mechanical strength, cold-start features which are crucial in long-term stability of fuel cell applications [8]. Considering the corrosion resistance requirement of BPPs, stainless steel stands forward as the most appropriate material among the other metallic BPP materials such as Ni based alloys, Ti based alloys and Al based alloys [8].

Fabrication is also critical for the low cost and high quality BPPs. There are several forming methods proposed for different materials such as machining for graphite BPPs, stamping process for metal BPPs and molding for polymer-carbon composites [9-11]. Among these BPP fabrication methods, stamping, as illustrated in Figure 5.1, is the most competitive process since high production rates and cost effectiveness are possible with this technique [9]. Geometric accuracy (thickness and flatness), shape accuracy (channel dimensions, shape and distribution) and surface quality requirements are satisfied by stamping process. Since the surface quality of BPPs is highly affected by tribological conditions and die wear, understanding of material behavior and die/BPP surface interactions in micro-scale is very critical. Previous studies also showed that friction conditions in micro-scale is more aggressive than that for macro-scale
because of the higher surface to volume ratio so called “size effect” [12-13]. During the high volume production of BPPs, high contact pressures cause highly localized stresses which result in topographic and tribological changes. These changes, in turn, may influence the corrosion and contact resistance behavior of BPPs, consequently the fuel cell performance. Therefore, surface topography alterations should be monitored and kept in acceptable range throughout the long-run manufacturing conditions.

**Figure 5.1** Micro-stamping process and the resulting bipolar plate with micro-channel array on a large surface

Previous investigations on the topography and tribological behavior in micro-forming were performed mostly with short-run processes. This study, on the other hand, aimed for monitoring the topographic and tribological changes, contact conditions and their effects on the formed product (in this case BPP) in long run manufacturing conditions to obtain more reliable and realistic results since in real production conditions micro-stamping of BPPs is supposed to take place for ten thousands, if not hundred thousands, of cycles.
This research study consists of experimental and numerical analyses. In the experimental part of the study, investigations of the surface topography and tribological changes on the die and BPP interface during long-run micro stamping process were performed. Furthermore, correlations between surface topography and corrosion/contact resistance characteristics of BPPs were examined. In the numerical part, finite element (FE) based models were established to observe stress-strain conditions and thickness change distributions on the formed BPPs. The determination of coefficient of friction during the long run micro-stamping process was based on the finite element analyses in comparison with experimental findings.

5.2 Experiments and Numerical Analyses

5.2.1 Materials

SS316L metallic sheet alloy with a thickness of 51 µm was chosen as BPP material. Mechanical and chemical properties of SS316L were presented in Table 5.1 and 5.2. 10,000 of SS316L sheet blank samples were prepared in 70mm x 70mm dimensions. Blank-holder force mechanism was employed using a rubber pad as illustrated in Figure 3.

5.2.2 Micro-stamping Experimental Setup and Conditions

The forming dies (lower and upper dies) used in micro-stamping experiments are shown in Figure 5.2. Each die had 26 arrays of micro channels with 750µm height. The dies were machined from H13 hot-work tool steel. High hardenability and excellent toughness are the main properties of H13 tool steel. In order to apply the force required to form the blanks, die set
up was assembled into an Instron Satec 400 HVL (Instron Corp., Norwood, MA, USA), and used in compression mode.

**Table 5.1** Mechanical Properties of SS316L (as provided, Brown Metals, Rancho Cucamonga, CA)

<table>
<thead>
<tr>
<th>Condition/Tempering</th>
<th>Annealed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus of Elasticity (Tension)</td>
<td>193 GPa (28 x 10^6 psi)</td>
</tr>
<tr>
<td>Ultimate Tensile Strength</td>
<td>711 MPa (103,100 psi)</td>
</tr>
<tr>
<td>Yield Strength at 0.2% offset</td>
<td>341 MPa (49,400 psi)</td>
</tr>
<tr>
<td>Percent Elongation in 2 inches</td>
<td>56.00%</td>
</tr>
<tr>
<td>ASTM Grain Size</td>
<td>10</td>
</tr>
</tbody>
</table>

**Table 5.2** Chemical composition of SS316L (as provided Brown Metals Company, Rancho Cucamonga, CA)

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>N</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.021</td>
<td>1.48</td>
<td>0.033</td>
<td>0.001</td>
<td>0.43</td>
<td>16.2</td>
<td>10.03</td>
<td>2.06</td>
<td>0.43</td>
<td>0.04</td>
<td>Bal</td>
</tr>
</tbody>
</table>

**Figure 5.2**: Micro-stamping dies used to form SS 316L sheet blanks
5.2.3. Numerical Simulations

Finite element is quite useful to model very complex metal forming processes where analytical solutions are not available in most cases. In this study, commercially available explicit/implicit finite element code Ls-Dyna was employed to simulate the micro-stamping process. Deformation characteristics and coefficient of friction variations were aimed to be calculated through finite element analyses. Due to the symmetry nature of the problem, a quarter 3-d model was established to reduce the simulation time shown in Figure 5.3. The blanks and blankholder were defined as deformable bodies and modeled with 4-node quadrilateral elements while female and male dies were assigned as rigid parts. The total number of elements used in the models was 52,338. In analysis, at least 11 different FEA were performed with different coefficient of friction values using Coulomb’s friction model. In order to determine the range of coefficient of friction experienced in micro-stamping tests, first, a few FEA with the coefficient of frictions of 0, 0.1, 0.2, 0.3, 0.4 and 0.5 were applied and their corresponding channel height results were compared with the experimental results. Then, iterative finite element analyses were performed until the change in coefficient of friction value with respect to previous step is reduced under a level of 0.01. All contacting surfaces were specified as “contact forming one way surface to surface”.available in Ls-Dyna [14]. Blankholder force of 3 kN was applied on sandwiched sheet blank between upper and lower die flat surfaces. Simulations were performed on a personal computer equipped with 2.8 GHz hyper-threading featured processor and 8 GB of RAM. Average computational time of 20 min. was recorded for analyses.

As it is known in size effect studies, experimental data should be integrated into finite element model as input parameters since size effect is not taken into account intrinsically in the FE models [15-17]. Therefore, experimental data stamping speed (1 mm/s) and load-
displacement were used in finite element simulations. Material properties used in the simulations were presented in Table 5.3.

**Table 5.3:** Material properties for SS 316L (AK Steel, www.aksteel.com)

<table>
<thead>
<tr>
<th>Material model</th>
<th>Power law, $\sigma = 1058. \varepsilon^{0.28}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus of elasticity ($E$, GPa)</td>
<td>193</td>
</tr>
<tr>
<td>Poisson’s ratio ($\nu$)</td>
<td>0.31</td>
</tr>
<tr>
<td>Density ($\rho$, g/cc)</td>
<td>7.99</td>
</tr>
</tbody>
</table>

**Figure 5.3** 3D, quarter FE model of stamping process used in fabrication of BPPs

### 5.2.4 Methodology and Measurements

In this study, experimental and numerical analysis were performed. A flowchart showing the steps for experimental and numerical procedures is given in Figure 5.4. In experimental part of the study, process parameters such as stamping force, and stamping speed were set 200kN, 1 mm/s, respectively. Then, 10,000 blanks were stamped with the same force and speed levels. In
Figure 5.5, an example of stamped BPPs is presented. Frequency of experimental measurements and tests along 10,000 stamping process is depicted in Figure 5.6. After micro-stamping every 1000 blanks, surface roughness and channel height measurements from at least three BPP, lower and upper dies (i.e., male and female dies, respectively) were taken. After every 2000 stampings, three BPPs were subjected to corrosion tests. Contact resistance tests were applied onto another three BPPs. Multiple measurements at certain intervals aimed to address repeatability of measurements and to detect possible variations.

In numerical analyses FE models were constructed for the micro-stamping of BPPs. First, the geometrical model was built. Material properties were derived from the existing information from the manufacturers and literature. Then, boundary and loading conditions such as blank holding and stamping speed were applied on the model. Upon performing numerical analyses, thickness distribution, effective plastic strain and von Mises stress values were recorded from each analysis. Channel height values of the BPPs obtained with using different coefficients of friction were compared with the experimentally obtained channel height findings to estimate the coefficient of friction values experienced at the contact interface. After every 2000 of micro-stamping, variation of coefficient of friction was estimated with above given procedure.
Figure 5.4 Methodology of micro-stamping process experiments, measurements and finite element analyses

Figure 5.5 Example of micro-stamped plates
5.2.5 Measurements and Tests

Surface roughness and dimensional measurements were conducted on BPP surfaces and channels, respectively. These measurements are important to understand the topography changes caused by manufacturing. In order to monitor the visual changes on the surfaces, microscopic examinations were also performed. Details of roughness and dimensional measurements as well as corrosion and contact resistance tests are given in subsequent sections.

5.2.5.1 Surface Roughness Measurements

Surface roughness of BPPs plays very important role since BPPs are in contact with the other components of fuel cell such as GDL. Any undesired features and irregularities on the contact interface between BPP and GDL can directly affect the electron transfer from cell to cell [18, 19]. Contact pressure distribution on the MEA can also be influenced by the surface topography of BPP. In addition, the factors that increase surface roughness such as scratches and surface disorders can also increase the corrosion risk for BPPs. When mass production of BPPs is considered, the surface roughness of BPPs might change dramatically. Therefore, corrosion and contact resistance tests were applied on three BPPs at every 2000 micro-stampings to
investigate the variations and possible relations between surface roughness, and corrosion, and contact resistance characteristics of BPPs. Procedures followed for corrosion and contact resistance tests were presented in an earlier study [20].

In this study, surface roughness values of both bipolar plates and stamping dies were measured with 3D measurement systems. Wyko NT1100 optical profiler (Veeco Instruments Inc., Tucson, AZ, USA) was used for 3D roughness measurements. Surface roughness values of dies and plates were measured at the channel peaks and valleys from three different locations on both die and formed plate, as shown in Figure 5.7. Measurement for a certain point was repeated three times to ensure the repeatability of results. Scan parameters are given in Table 5.4.

![Figure 5.7 Roughness measurement locations](image)

**Table 5.4 3-d roughness scan parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Scanned Area</strong></td>
<td>450.6 x 600.8μm</td>
</tr>
<tr>
<td><strong>Magnification</strong></td>
<td>10.55</td>
</tr>
<tr>
<td><strong>Sampling</strong></td>
<td>938 x 67nm</td>
</tr>
<tr>
<td><strong>Array size</strong></td>
<td>640 x 480</td>
</tr>
</tbody>
</table>
5.2.5.2 Dimensional Measurements of Micro-Channels of BPPs

Channel height values of BPPs were measured for the determination of friction coefficients. Details about determination of friction coefficients were presented in “Methodology” section of this chapter. Monitoring of channel height is also significant to investigate dimensional stability of BPPs and repeatability of the process. A contact type profilometer (Dektak 150- Veeco Instruments Inc., Tucson, AZ, USA) was used in channel height measurements.

5.2.5.3 Corrosion Resistance Tests

Humid working environment can trigger corrosion issues for BPPs. This leads to efficiency drops and power loses in fuel cells. It was reported that corrosion resistance behavior was affected by the surface topography variations [21]. In this study, the influence of surface topography on corrosion resistance was investigated by performing potentiodynamic corrosion tests. A working electrode (BPP sample), a graphite counter electrode, an Ag/AgCl reference electrode, acid tank and a gas bubbler at the bottom of the tank were the components of corrosion test setup. Corrosion cell was placed into a furnace and heated up to 80°C to simulate the working temperature of PEMFC. To represent the cathodic condition in a real fuel cell, O₂ gas purged into the corrosion cell.

5.2.5.4 Contact Resistance Tests

Four-probe method was used to determine interfacial contact resistance (ICR) between GDL (gas diffusion layer) and BPP. A stack was built placing GDL and BPP between two gold coated copper current collector. Then, an electromechanical testing system (MTS Insight 30, MTS System Corp., Eden Prairie, MN, USA) was used to compress the stack under the
incrementally increasing load levels. ICR values were obtained under increasing pressure from 20 N/cm² to 284 N/cm² with 24N/cm² increments. Compared to the ICR (interfacial contact resistance) values, bulk resistance values of BPP and GDLs were too small. Thus, the bulk resistance of BPPs and GDLs were not considered in the ICR measurements. Three different plates were subjected to ICR tests to verify the repeatability of measurements.

5.3 Results and Discussion

5.3.1 Effect of micro-stamping process on the surface topography

Figure 5.8 demonstrates the 3d surface roughness measurements for lower (female) die, upper (male) die and SS316L BPPs obtained at the location 2B (shown in Figure 5.7). Other two roughness measurement locations illustrated similar patterns. Average roughness variations (%) for the locations on BPPs during micro-stamping of 10,000 BPPs were 5.5% whereas the variations for female and male dies roughness values were found to be 2.9% and 9.3% respectively. When roughness values for female and male die, and BPP were compared, the highest and lowest roughness change (%) were observed on male and female dies, respectively. The roughness changes for the dies and plates were observed to be unsteady, and narrow-ranged. It is believed that the sub-micron range local plastic deformations on the dies led to continuous topography changes on the surfaces during the stamping of 10,000 BPPs. Results also indicated similar roughness changes for dies and BPPs. As the surface roughness of the dies reduced, the surface roughness of BPPs also decreased.

ANOVA statistical analysis was performed to exhibit the significance of roughness variations. The roughness changes for female, male dies and BPPs were analyzed separately.
Three different measurements form three different locations at the same interval of micro-stamping (i.e., 1\textsuperscript{st}, 1000\textsuperscript{th}, 2000\textsuperscript{th}, 3000\textsuperscript{th}, 4000\textsuperscript{th}, 5000\textsuperscript{th}, 6000\textsuperscript{th}, 7000\textsuperscript{th}, 8000\textsuperscript{th}, 9000\textsuperscript{th}, and 10000\textsuperscript{th}) were considered during the ANOVA analysis of dies and BPPs. ANOVA results indicated that the roughness variations on female, male dies and BPPs were all significant (p=0.000, p=0.000 and p=0.002). Considering these results, it can be reached to the conclusion that surface roughness on BPPs was directly affected by the roughness on female and male die surfaces.

No significant visual surface pattern change was observed on the die surfaces when the die surface before and after 10,000 stampings as shown in Figure 5.9.

![Graph showing surface roughness variations along 10,000 micro-stamping for die and micro-stamped BPPs](image)

**Figure 5.8** Surface roughness variations along 10,000 micro-stamping for die and micro-stamped BPPs
5.3.2 Effect of surface roughness on the corrosion resistance of the micro-stamped BPPs

Average roughness value for BPPs was measured as 1.65 μm while corrosion tests for BPPs yielded an average current density of 11.48 μA.cm\(^{-2}\). This current density value is higher than target level (1 μA.cm\(^{-2}\)) set by U.S. Department of Energy [22]. Trends for surface roughness and potentiodynamic corrosion test results of SS316L BPPs were presented in Figure 5.10. As in the case of roughness variations for BPPs, corrosion changes of BPPs demonstrated unsteady changes as well. When the roughness and corrosion performances are compared, similar trends were observed. As roughness values are increased, current density values observed to be increased. In other words, increase in BPP roughness led to drop on corrosion resistance.
ANOVA analyses, on the other hand, revealed that the differences in both surface roughness and current density measurements for BPPs were insignificant (p=0.087 and p=0.403 respectively) at different stages of micro-stamping runs (i.e., 1st, 2000th, 4000th, 6000th, 8000th, 10000th). A well-established correlation could not be established since Thus, a direct correlation between surface roughness and corrosion resistance of BPPs can be addressed after longer-run experiments.

![Figure 5.10](image-url)  
**Figure 5.10** Average roughness-corrosion resistance variations along 10,000 micro-stamping for micro-stamped BPPs

### 5.3.3 Effect of surface roughness on the contact resistance of BPPs

Average contact resistance values of BPPs were measured as 234 mΩ.cm² whereas the average roughness values of BPP were obtained as 1.65 µm. Comparing with the DOE contact
resistance target level (10 mΩ.cm²) [11], measured contact resistance values of BPPs were found to be much higher. The trend between surface roughness and contact resistance is illustrated in Figure 5.11. Similar to surface roughness changes on the BPPs, contact resistance of micro-stamped BPPs was unsteady. Nonetheless, the relation between surface roughness and contact resistance of BPPs was found to follow the same trend. It was assumed that the increasing surface area due to flattening of asperities can lead to lowers the contact surface area. According to ANOVA analysis results, the roughness variations for the BPPs were insignificant (p=0.096), while the contact resistance changes were significant (p=0.027) at different stages of micro-stamping runs (i.e., 1st, 2000th, 4000th, 6000th, 8000th, 10000th). In order to observe significant changes on surface roughness on BPPs, further investigations are necessary. Thus, a direct relation between surface roughness and contact resistance of BPPs can be stated.

Figure 5.11 Average roughness-contact resistance variations along 10,000 micro-stamping for micro-stamped BPPs
5.3.4 Effect of micro-stamping process on the variation of coefficient of friction

Since aggravated frictional conditions are experienced in microforming processes, determination of friction coefficient is more critical in micro-scale than macro scale. Thus, once can get valuable information on the severity of tool/workpiece interaction, and deformation characteristics of the tool and workpiece can be predicted with the determination of friction coefficient [23-25].

In this part of the study, friction coefficient variations at different stages of micro-stamping runs (i.e., 1st, 2000th, 4000th, 6000th, 8000th, 10000th) were investigated. Several FE simulations were conducted with different coefficients of friction. Corresponding channel height values were obtained from each simulation. These values, then were compared with the measurement obtained from real samples by using a 2D (contact type) profilometer (Dektak 150-Veeco Inc., Tucson, AZ). Figure 5.12 shows the numerically and experimentally obtained channel height values for BPPs. The results revealed that all of the experimental channel height values obtained at different stages of micro-stamping runs (i.e., 1st, 2000th, 4000th, 6000th, 8000th, 10000th) were between 232.67µm and 239 µm which corresponds the friction coefficient value between 0.18 and 0.19 as observed from FEA results. This implied that, the overall coefficient of friction experienced at the real micro-stamping operation is in between 0.18 and 0.19, and it did not show significant variation during the 10,000 stamping tests.
5.3.5 Effect of micro-stamping process on deformation characteristics

Process parameters affect the deformation characteristics. Therefore, analyzing the equivalent stress, plastic strain, and thickness change distributions for the micro-stamped parts should carefully be examined to prevent from excessive stresses or strains on both tooling and microformed part. This information is also essential for better die/process design as well as longer tool life. Figure 5.13 presents von-Mises, plastic strain and thickness change distributions on the stamped BPPs under 200kN stamping force and 1 mm/s stamping speed. The distributions were obtained for the coefficient of friction value of 0.19 which was determined from corresponding FEA and experimental measurements. As it can be seen from Figure 5.13, the highest equivalent stress, plastic strain, and thickness changes for microstamped BPP were recorded approximately as 750 MPa, 0.16 and 8μm, respectively at the corner of ribs. Failures on the BPP surfaces were experienced above these values at corners as shown as failure locations in Figure 5.14.
Figure 5.13 The distributions of (a) von-Mises stress, (b) plastic strain and (c) final material thickness over the micro-stamped surface
5.4 Conclusions

This study investigated the effect of long-run manufacturing conditions on the surface topography, corrosion and contact resistance of micro-stamped BPPs. Several FEA analyses were conducted to estimate the coefficient of friction values occurred at contact interface.

Surface roughness analyses for forming die and BPPs demonstrated significant changes during the whole range of experiments. Although without patterned roughness changes was observed, the roughness trend between the microforming die parts (male, female dies) and micro-stamped BPPs was observed to be similar.

Corrosion resistance test results showed that surface treatment is required to improve the corrosion resistance of plates to reach the DOE corrosion resistance targets. Corrosion
resistance change during the long-run manufacturing was insignificant, and the relation between the surface roughness and corrosion resistance was found to be indirect.

According to contact resistance tests, SS316L plates exhibited much higher contact resistance than DOE target levels. Contact resistance test results exhibited significant changes over 10,000 micro-stamping process. The correlation between surface roughness and contact resistance of BPPs was deemed to be direct.

The dimensional measurements of channel heights showed insignificant channel height changes at different stages of micro-stamping runs (i.e., 1st, 2000th, 4000th, 6000th, 8000th, 10000th). This implied to dimensional stability of formed products and repeatability of the process. Affected by these findings, coefficient of friction variation was negligibly small and was found to be between 0.18 and 0.19. Finite element analyses provided also useful information on the experienced stress, strain, thickness change magnitudes and valuable data for better die design and longer tool life.
References


6.1 Summary

In this research, micro-scale surface interactions at the tool and workpiece interface are investigated. As a case study, manufacturing of stainless steel bipolar plates were chosen. Comprehensive understanding of tribological effect and size effect between tooling and deforming materials is aimed to be gained to address the die wear, bipolar plate surface quality, and process durability issues in mass production of micro-channels on bipolar plates.

Extensive literature survey has been made to determine the micro-forming parameters that to be monitored in experiments. Also, background search on fuel cells and bipolar plate manufacturing is performed to relate the micro-forming study to the manufacturing of BPPs. Based on the preliminary literature survey conducted so far, the factors that affect the die and bipolar plate surface quality, dimensional variation and die life were identified as control parameters.

Problems associating with die wear/die life issues which result in deformation of metallic bipolar plates and attempt to find out the effects of process parameters and the effect of coating conditions on die life and bipolar plate surface quality were described in chapter 3. Specifically, microstampings experiments at 175-220 kN force levels with constant stamping speeds were
performed using 0.002” (51µm) thick uncoated and coated SS316L sheet blanks. Microscopic examinations (surface roughness and channel heights) were applied periodically from both die and coated & uncoated plate surfaces to investigate topographic variations. In addition, corrosion and contact resistance tests were carried out in the same periods.

In chapter 4, experimental and numerical models were performed to investigate the formability characteristics of SS316L sheet blank. In numerical part, 2-dimensional FE based models were established. The distributions of von-Mises stress and plastic strain were monitored. Channel heights of the sheet plates for different coefficient of frictions were obtained. In experimental part of the study, SS316L sheets were stamped under three different force levels (100kN, 200kN, 300kN) using the dies with different channel sizes (250 µm, 750 µm and 2500 µm). Then experimental channel height values were taken. These channel height values were compared with the numerical channel heights to determine the coefficient of frictions with 0.01 accuracy.

In chapter five, 10,000 of SS316L blanks were stamped under 200kN stamping force and 1 mm/s stamping speed. Surface topography variation for female, male dies and BPP were monitored periodically during the 10,000 stampings. Corrosion and contact resistance tests were performed at different stages of micro-stamping runs (i.e., 1st, 2000th, 4000th, 6000th, 8000th, 10000th) to investigate the corrosion/ contact resistance variations during the long-run manufacturing condition. A 3-dimensional FE model was conducted to the experiments to find the deformation characteristics of the stamping tests. As practiced in chapter 4, experimental channel height values of BPPs were compared with the numerical channel heights for determination and variation of coefficient of friction during the 10,000 stamping tests.
6.2 Conclusions

-Surface topography variations and dimensional measurements of channel heights during 1000 uncoated and 1000 coated tests

From the studies conducted so far, 1000 coated + 1000 uncoated blank were formed into fuel cell bipolar plates via stamping process, consecutively to observe the surface topography changes and its effect on corrosion and contact resistance behavior. Roughness variations at different extents were observed on the die and plate surfaces that is believed to be caused by different surface finishes as well as hardness properties of die and plate. It was concluded that although there was some unsteady changes in roughness values during the stamping of the BPPs, the surface roughness trends for mating surfaces (female die peak-plate valley) were similar. However, more stamping tests are required to correlate surface topography changes. The results also showed different formability levels experienced for coated and uncoated samples. Higher channel height values were obtained on uncoated samples. Dimensional stability and repeatability was observed in forming of both uncoated and coated samples.

-Corrosion resistance variations during the stamping of 1000 uncoated and 1000 coated BPPs and the roughness-corrosion resistance correlations

Corrosion resistance test results demonstrated that corrosion resistance of coated plates is much higher than uncoated plates. From the roughness point of view, as roughness on coated plates surfaces increased, current density reduced, and this lead to an increase in corrosion resistance. On the other hand, increasing surface roughness on uncoated plates yielded increased corrosion resistance.
-Contact resistance variations during the stamping of 1000 uncoated and 1000 coated BPPs and the roughness-contact resistance relations

Contact resistance test results indicated that contact resistance of uncoated plates was found to be higher compared to coated plates. Although there is no clear pattern between roughness and contact resistance, the relation between those was found to be indirect.

-Correlation between surface feature size and formability level

The effect of die channel size on the formability level was important. The die with largest channel size (2500µm) design demonstrated lower level of deformation since displacement was dominant and the strip displaced into channels rather than stretched. The smallest size die feature (250µm) prevented the plastic deformation of strip. When three feature die design were compared in terms of plastic deformation, the highest value was obtained at the die with 750 µm. Thinning behavior also showed similar trend with the plastic deformation.

-Correlation between surface feature size and friction coefficient

Friction levels were found to be increasing with decreasing feature size as known “tribological size effect” in the literature. The coefficients of friction were obtained from the comparison of experimental and numerical channel heights. After the channel heights were determined in the numerical model, the corresponding coefficient values were selected as the coefficients of friction of the process. Although the friction coefficients may change depending on the location on the die/BPP interface, the coefficients of friction can be taken as general friction coefficient.
-Surface topography variations and dimensional measurements of channel heights during 10,000 stamping of SS316L sheets

Statistically significant surface topography changes were observed, when the surface roughness values of female die, male die and BPPs were compared. The roughness results indicated that the roughness variation on a die directly affected the other die and BPP. For instance, when the roughness of female die increased, roughness values of male die and BPP mostly increased. As a conclusion, the die with high quality surface is required for high surface quality of BPP.

-Corrosion resistance variations during 10,000 stamping of SS316L sheets and the roughness-corrosion resistance correlations

The corrosion resistance test results indicated insignificant corrosion resistance variation during 10,000 stampings of SS316L sheets. However, indirect relations were observed between surface roughness and corrosion resistance of BPPs. This might be caused by the reasons that increasing surface area of asperity or defects on rough surfaces triggered corrosion.

-Contact resistance variations during 10,000 stamping of SS316L sheets and the roughness-contact resistance correlations

According to the statitical analysis (ANOVA) results, the contact resistance variations of BPPs were significant. Similar to the roughness variations, unsteady changes were observed in terms of contact resistance of BPPs. The results showed that as surface roughness of BPPs increased, the contact resistance of BPPs also increased.
-The coefficients of friction variations during 10,000 stamping of SS316L sheets

Since dimensional measurements of experimental channel height variations were insignificant, the repeatability of stamping tests was provided. All channel heights at different stages of micro-stamping runs (i.e., 1\textsuperscript{st}, 2000\textsuperscript{th}, 4000\textsuperscript{th}, 6000\textsuperscript{th}, 8000\textsuperscript{th}, 10000\textsuperscript{th}) corresponded the same friction of coefficient during 10,000 stamping of SS316L sheets.

6.3 Recommended Future Work

-Improvement of coating material on SS316L sheets considering tribological size effect

Surface tribology and tribological size effect are important issues for the determination of BPP surface quality which influences the corrosion and contact resistance, consequently fuel cell performance. As a surface treatment method, coating can affect the surface quality of BPPs. Coating on SS316L BPPs can improve the surface tribology, so that higher surface quality on BPP can be obtained using appropriate coating material (e.i. ZrN, TiN and CrN). Tribological conditions on the interface of stamping die and BPP can change different than uncoated BPPs. Thus, coated strip sheets with different coating materials and different coating thickness values should be stamped using the dies with different channel sizes. Numerical analysis should be conducted to experimental analysis. The contribution expected will be the understanding of tribological size effect on coated SS316 sheets.
In this study the effect of coating of BPPs on micro-stamping tribology and corrosion/contact resistance characteristics were studied. However, coating of micro-stamping dies can also improve the surface topography and tribological conditions on the BPPs. Therefore, large number of stamping process should be performed on SS316L using coated dies. During the process, surface inspection measurements (surface roughness and dimensional measurements of channel heights) should be performed periodically from both die and bipolar plate surfaces in certain periods. Since contact and corrosion resistance of bipolar plates have tremendous effect on fuel cell performance, their variations on the manufactured bipolar plates should be observed as well.

In this study, effect of die surface quality on BPP surface quality during mass manufacturing of BPPs was investigated. However, the effect of die feature size on BPPs should be studied. Surface topography and tribological variations of BPPs can be affected by the die feature size as well as die surface quality due to surface to volume ratio so called “size effect”. In chapter 4, it’s concluded that the tribological conditions (i.e. coefficient of friction) are different for different size die features. Long-run stamping process using the dies with different channel sizes should be performed to monitor the effect of die feature size on BPP surface quality.
6.4 Scientific Contributions

The following scientific contributions are expected.

• Characterization of the effects of BPP coating on surface topography of the dies and BPP, corrosion and contact resistance characteristics of BPP, consequently PEM fuel cell performance

• Comprehensive understanding and modeling of tribological size effect in micro-forming applications and particularly micro-stamping of SS316L bipolar plates.

• Determination of the effects of process parameters on tribological behavior of the die and BPP

• Comprehension of the effect of long-run manufacturing on the tool (stamping dies) and workpiece (SS316L sheet blanks) surface quality, corrosion and contact resistance characteristics of BPP, consequently PEM fuel cell performance during the micro-stamping of SS316L

• Numerical model establishment for a particular micro-forming operation and its validation

• Understanding of the deformation characteristics (von-Mises stress, plastic strain and thickness variations) and distributions in micro-stamping process
APPENDIX 1

Potential-Current Density Relations for 1000 Uncoated + 1000 Coated Stamping Tests

Potentiodynamic Results for uncoated-coated Stamped (200kN.s^{-1} -1mm.s^{-1}) Samples w/ O2
APPENDIX 2

Male Die Roughness Changes For Five Different Locations During Long-run Manufacturing

![Male Die Roughness Changes](image)

- **Average Roughness (µm)**
- **Number of Samples Formed**
APPENDIX 3

2D (Contact Type) Surface Roughness Measurements For Five Different Locations

Female Die Surface Roughness (Contact Type)

![Graph showing female die surface roughness measurements for five different locations.](image)

- **0**
- **0.5**
- **1**
- **1.5**
- **2**
- **2.5**
- **3**

**Number of Samples Formed**

**Average Roughness (µm)**

- **1A**
- **1C**
- **3B**
- **6A**
- **6C**