Effect of manufacturing conditions on the corrosion resistance behavior of metallic bipolar plates in proton exchange membrane fuel cells

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A B S T R A C T

Metallic bipolar plates are one of the promising alternatives to the graphite bipolar plates in proton exchange membrane fuel cell (PEMFC) systems. In this study, stainless steel (SS304, SS316L, and SS430), nickel (Ni 270), and titanium (Grade 2 Ti) plates with an initial thickness of 51 μm were experimented as bipolar plate substrate materials in corrosion resistance tests. In addition to unformed blanks, SS316L plates were formed with stamping and hydroforming processes to obtain bipolar plates under different process conditions (stamping force, hydroforming pressure, stamping speed, hydroforming pressure rate). These bipolar plates, then, were subjected to corrosion tests, and the results were presented and discussed in detail. Potentiodynamic polarizations were performed to observe corrosion resistance of metallic bipolar plates by simulating the anodic and cathodic environments in the PEMFC. In order to determine the statistical significance of the corrosion resistance differences between different manufacturing conditions, analysis of variance (ANOVA) technique was used on the corrosion current density (I_{corr}, μA cm^{-2}) values obtained from experiments. ANOVA for the unformed substrate materials indicated that SS430 and Ni have less corrosion resistance than the other substrate materials tested. There was a significant difference between blank (unformed) and stamped SS316L plates only in the anodic environment. Although there was no noteworthy difference between unformed and hydroformed specimens for SS316L material, neither of these materials meet the Department of Energy's (DOE) target corrosion rate of ≤ 1 μA cm^{-2} by 2015 without coating. Finally, stamping parameters (i.e. speed and force levels) and hydroforming parameters (i.e. the pressure and pressure rate) significantly affected the corrosion behavior of bipolar plates.

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1. Introduction

Hydrogen fueled proton exchange membrane fuel cell (PEMFC) systems which produce electrical energy from reduction oxidation reactions is one of the promising alternative energy generation methods due to high efficiency, minimal emissions and low noise. PEM fuel cell is environmentally friendly and allow for easy start-up at low temperatures. Consequently, PEMFCs have found prominent potential applications in the automobile industry. However, PEMFC technology has some disadvantages such as high manufacturing cost and durability issues.

PEMFC mainly consists of the membrane, the catalyst layer, and the bipolar plates (BP). The bipolar plate, as one of the critical components of PEMFC, constitutes 80% of the weight and 45% of

the cost of the hydrogen fuel cell stack [1,2]. A recent study estimated the bipolar plate cost as \$1.41 plate^{-1} and energy generated through bipolar plates as \$3.53 kW^{-1} (based on 1000 mW cm^{-2}). Even though these values satisfy DOE 2010 target of \$5 kW^{-1}, it further needs to be improved to meet DOE 2015 target of \$3 kW^{-1} [3]. Bipolar plates have several functions in the fuel cell system: providing mechanical strength and support to the stack, carrying hydrogen and oxygen to the electrodes, separating the individual cells in the stack, conducting electrons between cells, and facilitating water and thermal management within the cell [4]. Hence, bipolar plates must possess high conductivity, low permeability, manufacturability without major problem, and low material and manufacturing costs [5,6]. Metallic bipolar plates are seen as formidable candidates since they meet such major requirements. On the other hand, metallic bipolar plates can be corroded due to the acidic environment of the PEMFC. In some cases, the plates form passive layers as metals corrode on the surface. The formation of passive layers results in high contact resistance which deteriorates the high conductivity for the bipolar plates. In addition, ions

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are released from the metal and enter the membrane, reducing its efficiency due to membrane poisoning. These conditions adversely impact the performance of the fuel cell [7].

Substrate material and manufacturing techniques are considered as important factors impacting the corrosion resistance of bipolar plates. Each metal exhibits different characteristic under corrosive conditions. In addition, the manufacturing method affects the surface topography of the bipolar plates. Consequently, the corrosion and conductivity behaviors are affected. Several studies in the literature aimed to find out the optimum substrate material that will provide high corrosion resistance and lower interfacial contact resistance [1,4,8–10]. Two electrochemical tests namely potentiodynamic and potentiostatic polarization tests are the most commonly conducted tests to observe corrosion behavior of metallic blank (unformed) plates by simulating the anodic and cathodic environments in the PEMFC [11,12]. However, bipolar plates used in the actual fuel cell environment are manufactured via deformation processes such as stamping. Hence, their surface characteristics and dimensions are entirely different from those of blank substrates as mostly reported in the literature.

The purpose of this study is to investigate the effect of manufacturing process and substrate material on the corrosion resistance of metallic bipolar plate in PEM fuel cell using statistical analysis tools. Therefore, several substrate materials and two different manufacturing methods were chosen in order to determine proper substrate materials and suitable manufacturing conditions for future PEMFC bipolar plate corrosion resistance research.

2. Experimental procedure

In this study, stainless steels (SS304, SS316L, and SS430), nickel (Ni 270), and titanium (Grade 2 Ti) sheet blanks with a thickness of 51 μm were chosen as unformed substrate materials for corrosion testing. Moreover, hydroformed and stamped SS316L plates with a thickness of 51 μm were compared with SS316L blanks in terms of corrosion resistance. Potentiodynamic polarizations were conducted to determine corrosion resistance of metallic bipolar plates by simulating the anodic and cathodic environments in the PEMFC. In addition, Tafel analysis was applied to measure corrosion current density (I_{corr}) of the specimen (blank or formed bipolar plate). In order to determine the statistical significance of the corrosion test results, ANOVA (analysis of variance) tests were conducted on the I_{corr} values obtained for each specimen.

Stainless steels are preferred for fuel cell applications due to their relatively high corrosion resistance among metallic bipolar plates as an alternative to graphite plates [13,14]. In a previous research by authors, corrosion resistance of SS304 substrate material was studied in detail [15]. It was found that SS430 and Ni had low corrosion resistance. Ti and Ni plates were included in this study to compare the behavior of pure metals in corrosion tests even though it is known that Ni and Ti are more expensive than the stainless steels. However, this study mainly focused on SS316L, which has high corrosion resistance and low cost as manufactured bipolar plate material [11]. SS316L with surface treatment was also suggested for potential bipolar plate in PEMFC by Lee et al. [16].

2.1. Manufacturing of bipolar plates

SS316 plates were formed to bipolar plates with flow channels using hydroforming and stamping processes. Hydroforming involves a blank that is shaped between an upper die (female) and high pressure fluid from bottom. For stamping, a blank sample is compressed between upper and lower dies to produce the channels. Detailed information and description of these processes and equipment were reported in a recent study [17]. For hydroforming,

20 and 40 MPa pressure levels and 1 MPa s^{-1} pressure rate were used in the experiments whereas force levels of 100 and 200 kN and 1 mm s^{-1} punch speed were used in the stamping process.

2.2. Corrosion resistance experiments

Manufactured bipolar plates and unformed specimens were tested using potentiodynamic polarization method to assess the corrosion behavior of the samples according to the procedure mentioned in [11]. A Solartron 1287 Electrochemical Interface potentiostat and CorrWare software (Scribner Associates Inc., Southern Pines, NC, USA) were used to produce Tafel Plots which will be explained under "potentiodynamic polarization test" section – potential (E) vs. the logarithm of the current ($\log I$).

2.2.1. Sample preparation procedure

After acetone application, the samples were put in an ultrasonic acetone bath for 30 min in order to remove oil and stain residues from the surface. Followed by cleaning process, the specimens were taped (Fig. 1b) with the Teflon tape except the active area of the bipolar plate. Samples were put into the sample holder set (given in Fig. 1b) in a tight form that the passive (taped) area is protected from the acid exposure (Fig. 1b). After the tests, it was assured that there was not acid diffusion and residue into Teflon taped passive area. In order to simulate the PEMFC environment, 0.5 M sulfuric acid (H_2SO_4) was used as the working electrolyte. Prepared specimens (working electrodes) were placed into the acid tank. Then, the corrosion test setup (Fig. 1a) was placed into the electrical furnace which was heated to 80 °C prior to tests.

2.2.2. Potentiodynamic polarization test

A polarization method such as potentiodynamic polarization is one of the commonly used laboratory-type corrosion tests. This technique can provide significantly useful information regarding the corrosion mechanisms, corrosion rate and susceptibility of specific materials (stainless steels and alloys) to corrosion in designated environments. One of its numerous advantages is allowing to non-destructive and on-site examination [18,19]. The standard test method for conducting potentiodynamic polarization resistance measurement was set by ASTM G59-97 standard [20].

The potentiodynamic experiments were performed in the potential range of −1.2 to 0.8 V at a rate of 1 mV s^{-1}. All of the potentials are given with respect to the reference electrode which is equivalent to standard hydrogen electrode (SHE) in the potential range of −1 to 1 V. Two different potentiodynamic conditions (H_2 purge for anode side and O_2 purge for cathode side) were tested. A corrosion cell consisting of the bipolar plate as a working electrode, a graphite counter electrode, and an Ag/AgCl (silver/silver chloride) reference electrode was used for the potentiodynamic experiments and were connected to the Solartron potentiostat. Detailed experimental setup and procedure is given in [15].

In order to quantify the corrosion current density of the samples in all the graphs, Tafel analysis [21] was conducted on the Tafel Plots using CorrView software. This method involves extrapolating the anodic and cathodic reactions by finding tangent lines to the anodic and cathodic sections of the graph. The intersection of the two tangent lines gives the corrosion current density, I_{corr}. The CorrView software found I_{corr} values automatically using two points above and below E_{corr}, the corrosion potential.

Corrosion current density value was determined by two different methods, namely Tafel extrapolation and Fit AutoTafel. Due to small differences between the results of the two methods; only "Fit AutoTafel between cursors" method was applied to obtain I_{corr} value for all graphs given in the rest of the study.
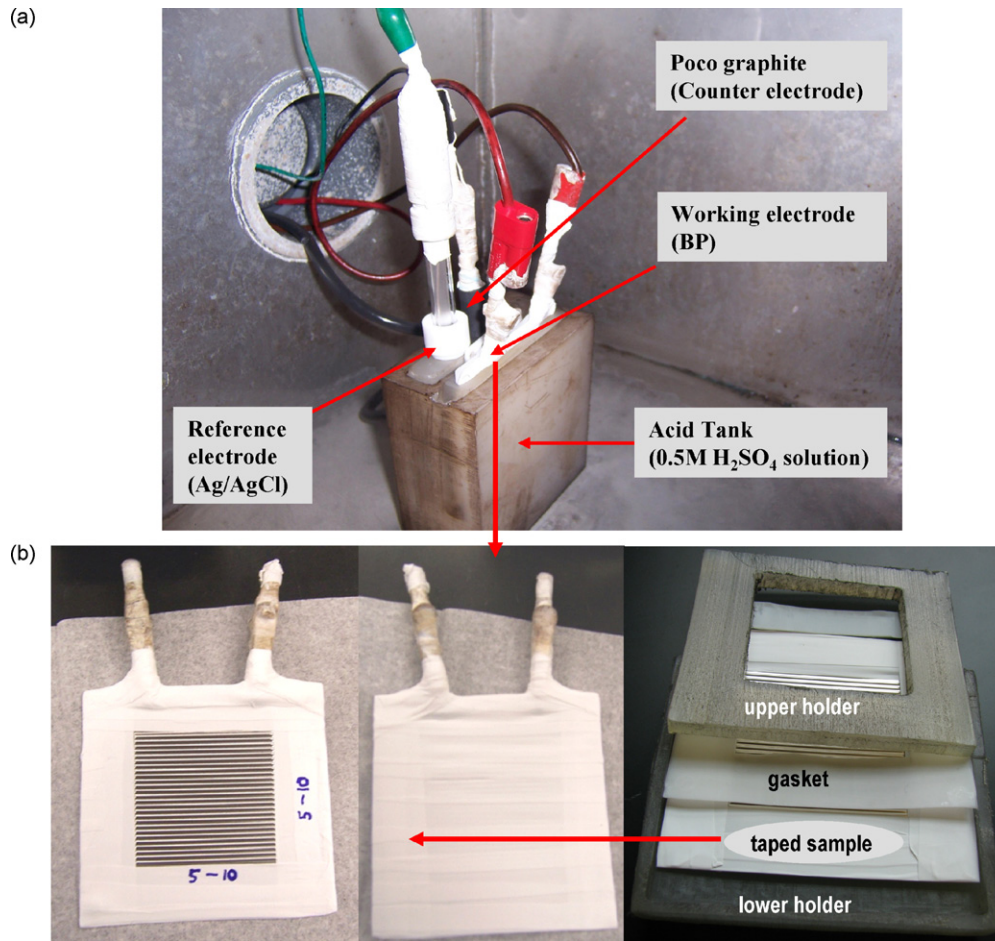
Fig. 1. Corrosion test setup in the furnace (a) front (tested), back side (covered) and the holder set of bipolar plate (b).

3. Results and discussion

3.1. Potentiodynamic test results

The anodic Tafel Plots (H_2) showed more peaks and noise than their cathodic (O_2) counterparts. Additionally, corrosion current densities were higher in anodic side than cathodic environments. This observation suggests that the purge gas (H_2 or O_2) did impact the corrosion behavior of those substrates at different potentials. The following sections are dedicated to the potentiodynamic test results of unformed and formed plates from different materials.

3.1.1. Unformed SS304, SS316L, SS430, Ti and Ni Plates

In this section, the corrosion current densities of unformed samples are analyzed. Table 1 shows the mean corrosion current densities for experimental groups (SS316L, SS304, SS430, Ti, and Ni), which is the average of three test results. Fig. 2 shows the anodic (H_2) results while Fig. 3 shows the cathodic (O_2) condition test results.

From Figs. 2 and 3, it can be observed that the Ti plate shows the highest corrosion resistance in the cathodic side, followed by
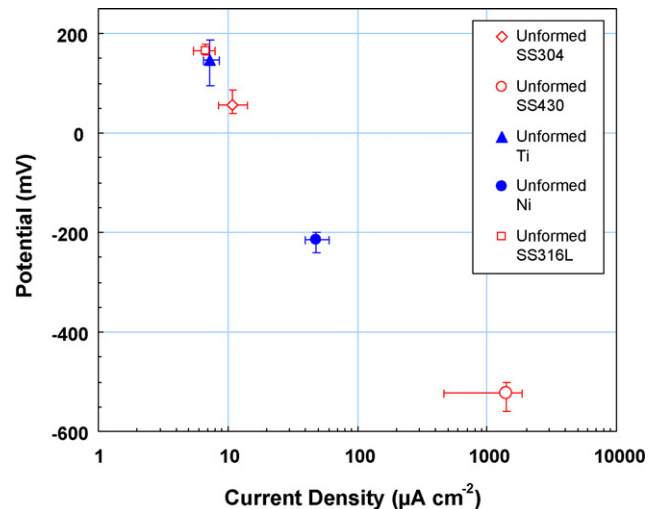

Fig. 2. Current density data with H_2 for unformed SS304, SS316L, SS430, Ti, Ni.

Table 1
Corrosion current density (mean) data for unformed SS304, SS316L, SS430, Ti, Ni plates.

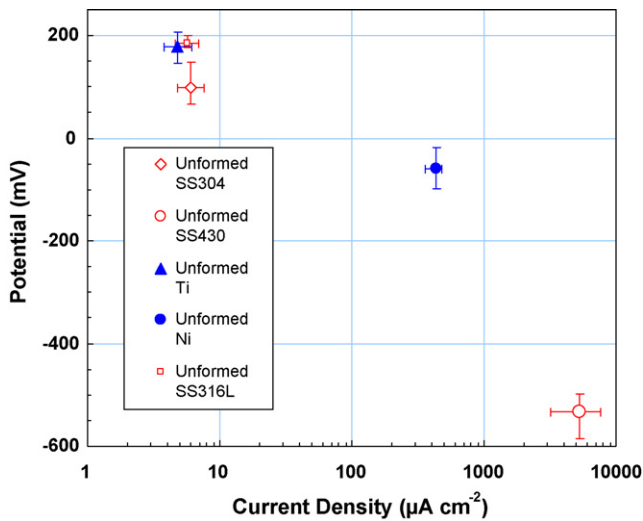|	Mean I_{corr} ($\mu A\,cm^{-2}$) for SS304	Mean I_{corr} ($\mu A\,cm^{-2}$) for SS316L	Mean I_{corr} ($\mu A\,cm^{-2}$) for SS430	Mean I_{corr} ($\mu A\,cm^{-2}$) for Ti	Mean I_{corr} ($\mu A\,cm^{-2}$) for Ni
H_2 purge (anodic tests)	10.92	6.68	1400	7.25	48.15
O_2 purge (cathodic tests)	5.99	5.68	5238	4.83	431.48

Fig. 3. Current density data with O_2 for unformed SS304, SS316L, SS430, Ti, Ni.

SS316L, SS304, Ni, SS430 samples, respectively, while SS316L plate yields the lowest corrosion current density in the anodic side. The same results can also be inferred from the Table 1 which indicates that Ti with O_2 purge and SS316L with H_2 purge have the lowest current densities. Having a lower corrosion current density represents a higher corrosion resistance [12]. Although SS430 demonstrated the worst corrosion behavior in current study, Pozio et al. found in their study that Niobium (Nb) coated SS430 could be an appropriate substrate material in terms of the corrosion and contact resistances for bipolar plates in PEMFC [22]. Similarly, Weil et al. revealed that boronization of nickel could help increasing the corrosion resistance of Ni bipolar plates [23].

3.1.2. Unformed and formed SS316L plates

Corrosion resistances of formed and unformed SS316L bipolar plates are presented and discussed in this section. Table 2 tabulates the mean I_{corr} data for unformed, stamped and hydroformed groups consisting of three specimens for each case.

The corrosion resistance of the SS316L samples was evaluated by their corrosion current densities and the corrosion potentials. Figs. 4 and 5 show these results in anodic and cathodic environments, respectively. According to these graphs, the plates corrode in the anodic side more than they do in the cathodic side. Wang and Northwood obtained similar results for SS316L in their research emphasizing that cathodic (O_2) side was four times more corrosion resistant compared to anodic side (H_2) [11]. Moreover, hydroforming process did not demonstrate as much of a corrosion augmenter impact on the bipolar plate as the stamped plates. Hydroformed plates produced with 20 MPa pressure and 1 MPa s^{-1} rate showed very similar corrosion characteristics to the unformed plates. Hydroformed plates performed better than unformed samples in the cathodic condition, as well. This can be illuminated with the elimination of residual stresses and having smooth surface after deformation process [15]. In contrast, hydroforming under 20 MPa and 1 MPa s^{-1} condition did not produce the expected channel depth due to the low manufacturing pressure [17].
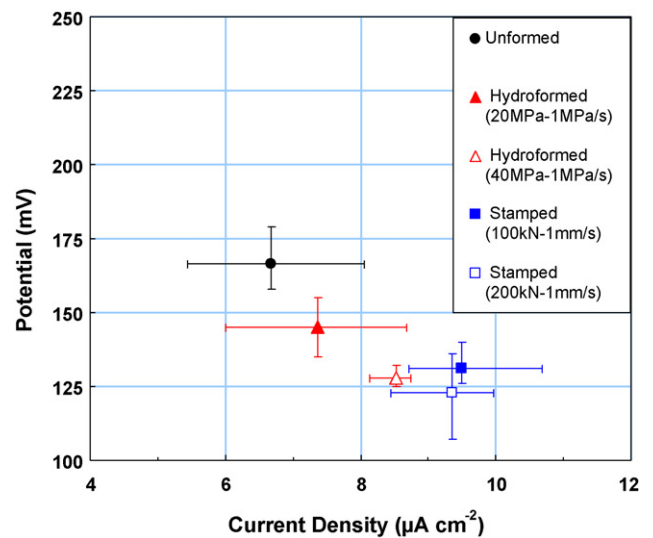

Fig. 4. Current density data with H_2 for unformed and formed SS316L from potentiodynamic test results.
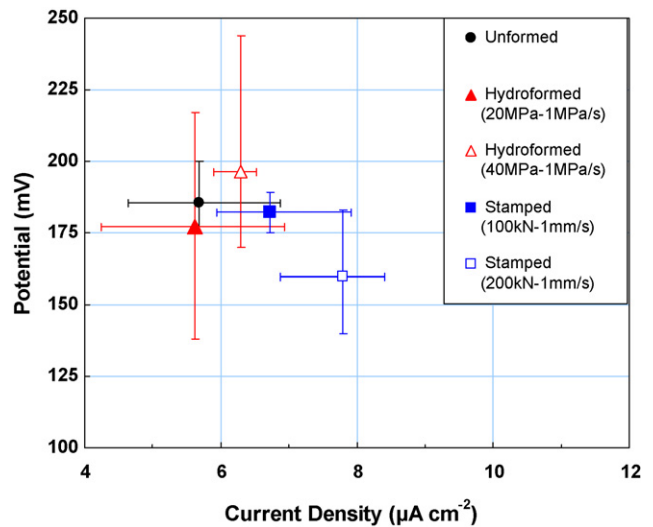

Fig. 5. Current density data with O_2 for unformed and formed SS316L from potentiodynamic test results.

4. Analysis of variation (ANOVA)

This part of the study aimed to investigate the impact of substrate material, purge gas, and manufacturing method and their variation on the corrosion resistance of a PEM fuel cell bipolar plate. To this aim, several analysis of variance (ANOVA) analyses were conducted on the I_{corr} data for each data set in order to determine the statistical significance of the corrosion test results. 95% confidence interval (CI) plots for the mean were obtained to compare unformed and formed plates in terms of their corrosion resistance. For reliable statistical analysis, all experimental groups had at least three repeated trials.
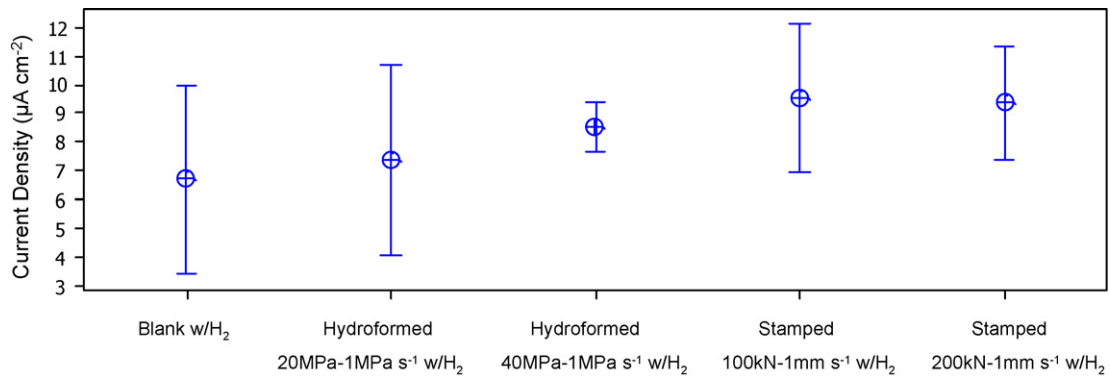
Table 2
Corrosion current density (mean) data for unformed, hydroformed and stamped SS316L plates.

SS316L	Mean I_{corr} ($\mu A\,cm^{-2}$) for blank	Mean I_{corr} ($\mu A\,cm^{-2}$) for hydroforming (20 MPa–1 MPa s^{-1})	Mean I_{corr} ($\mu A\,cm^{-2}$) for hydroforming (40 MPa–1 MPa s^{-1})	Mean Icorr ($\mu A\,cm^{-2}$) for Stamping (100 kN–1 mm s^{-1})	Mean Icorr ($\mu A\,cm^{-2}$) for Stamping (200 kN–1 mm s^{-1})
H_2 purge (anodic tests)	6.68	7.37	8.52	9.5	9.36
O_2 purge (cathodic tests)	5.68	5.62	6.29	6.73	7.79

Fig. 6. 95% confidence intervals of blank, hydroformed and stamped SS316L plates in H_2 environment.

Before statistical analyses, it was assumed that SS316L and SS304 stainless steel would be more corrosion resistant than the other materials tested (SS430, Ni, and Ti). Unformed SS316L would also perform better than its stamped and hydroformed counterparts. In addition, the anodic (H_2) environment would increase the corrosion current density more than the cathodic condition.

According to the ANOVA analysis, results for unformed substrate materials indicated that there was an important difference among the unformed group samples that SS430 and Ni had significantly lower corrosion resistance than the others. Moreover, there was a significant difference in the anodic (H_2) condition test results for the unformed, hydroformed and stamped SS316L plates at an α of 0.05 ($p = 0.029$). Confidence intervals of the SS316L plates are shown in Fig. 6. The stamping speed-force levels and the hydroforming pressure and pressure rate were found to be significantly affecting the corrosion resistance of the bipolar plates. Further ANOVA tests were carried out to determine which process affects the corrosion behavior of the SS316L samples more by comparing the test results of formed samples, and unformed samples. Differences between blank (unformed) SS316L plates and stamped SS316L specimens in the anodic environment were found to be significant. Comparison of interval plots for both anodic and cathodic sides for unformed SS304, SS430, Ti, Ni and formed with unformed SS316L plates are given in the next sections.

4.1. Comparison of unformed SS304, SS316L, SS430, Ti and Ni

First, an ANOVA test was performed on the five unformed substrate groups (SS304, SS316L, SS430, Ti, and Ni) to determine

whether the substrate material that has a significantly high or low corrosion current density than the others in both anodic and cathodic conditions. Confidence intervals of these groups are given in Fig. 7. The test showed a significant difference in terms of the corrosion behavior among those groups. The ANOVA test attained a p-value of 0.000, which is significant for α of 0.05 (confidence interval of 95%). In addition, further analysis indicated that SS430 and Ni (Table 1) showed greater corrosion current densities than the other substrates tested in both anodic and cathodic environments.

Subsequently, SS304, SS316L and Ti, which displayed lower current density data than the other materials, were analyzed in terms of anodic and cathodic conditions. Analysis indicated that there was no significant difference between the unformed SS304, SS316L and Ti at an α of 0.05.

4.2. Comparison between the SS316L unformed and hydroformed plates

Another series of significance tests were carried out to determine whether the hydroforming process conditions (20 and 40 MPa) significantly affected the corrosion current density values (Fig. 8). These ANOVA tests showed that there was no significant difference at an α of 0.05 between the blank and hydroformed SS316L at 20–40 MPa pressure and 1 MPa s^{-1} pressure rate for both anodic and cathodic environments. Therefore, it can be inferred that the hydroforming process is a more promising method in terms of corrosion resistance variation and consequently manufacturing purposes. Similar conclusions for robustness of hydroforming process were noted in another publication in the literature [17].
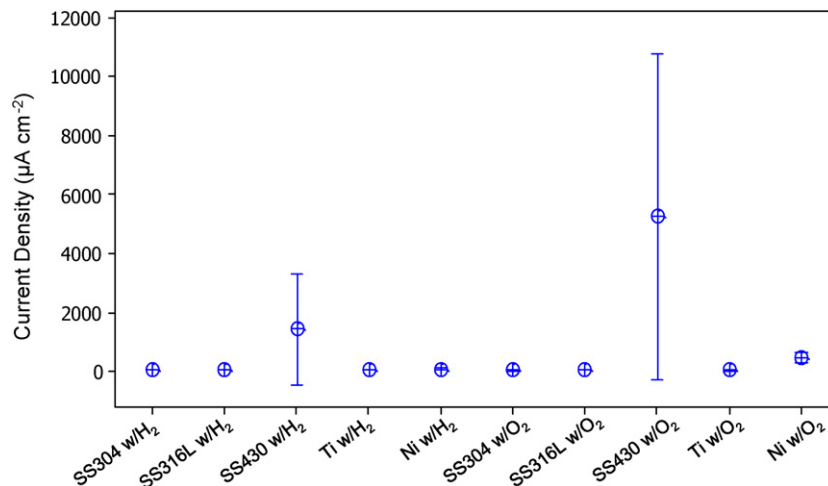

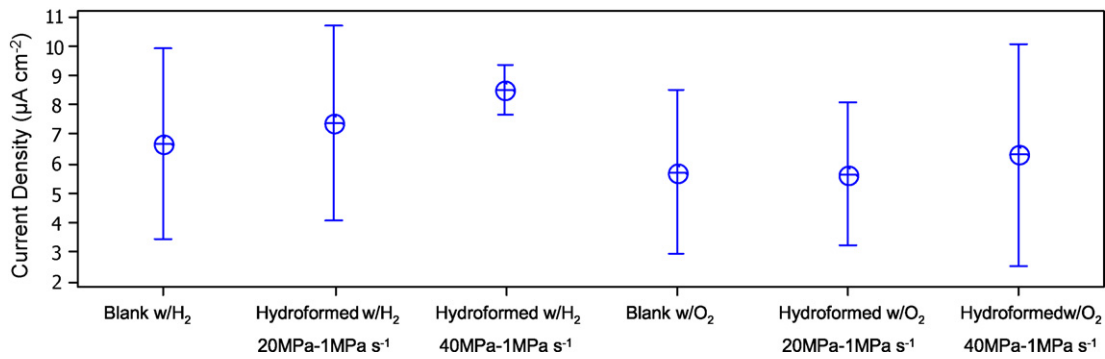**Fig. 7.** Interval plot for unformed SS304, SS316L, SS430, Ti and Ni in anodic and cathodic conditions.

Fig. 8. Comparison between blank and hydroformed SS316L in anodic and cathodic conditions.
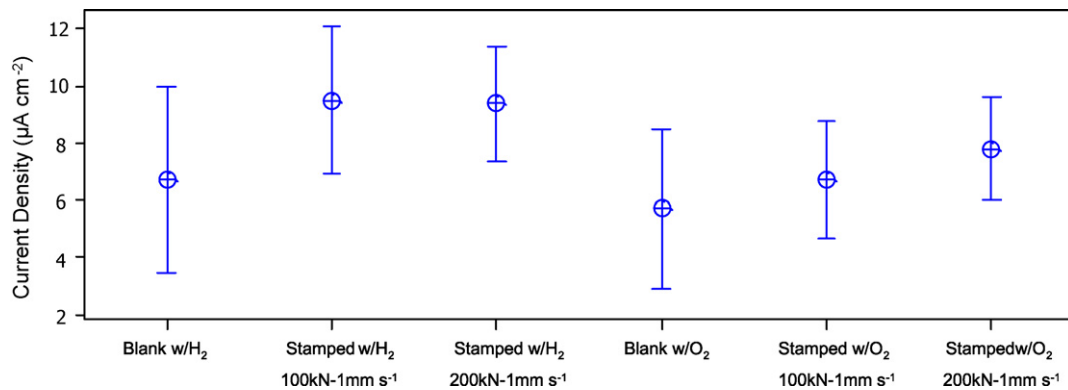

Fig. 9. Comparison between blank and stamped SS316L samples in anodic and cathodic conditions.

4.3. Comparison between the SS316L unformed and stamped plates

The effect of the stamping on the corrosion resistance of bipolar plates was evaluated by comparing their corrosion current densities with that of the unformed plates. According to the ANOVA test that yielded a p-value of 0.002 which is lower than an α of 0.05, there was a significant difference in the corrosion current density of unformed and stamped SS316L samples at 100–200 kN force and 1 mm s^{-1} force speed regardless of force level or environment (H$_2$ or O$_2$) (Fig. 9). Hence, the stamping process is found to be negatively affecting the corrosion resistance significantly due to its increasing corrosion current on bipolar plates. However, stamping process is an important alternative method due to its ability to produce metallic bipolar plates inexpensively and rapidly. Therefore, coated blanks or coating of stamped plate approaches should be considered in order to satisfy DOE targets for durability, corrosion and contact resistance, weight per kW, and cost requirements.

5. Conclusions

According to the corrosion test results and ANOVA statistical analyses, the effect of the speed–force levels in stamping and the pressure and pressure rate in hydroforming were found to be significant on the corrosion resistance of bipolar plates. Effect of manufacturing conditions on the formability of micro-channels, on the other hand, was reported elsewhere [17,24].

In addition, the corrosion test results for unformed substrate materials indicated that SS430 and Ni have significantly less corrosion resistance than all other substrates tested. Moreover, the SS304, SS316L, and Ti groups showed similar corrosion resistance, in general. Consequently, SS430 and Ni were found to be not suitable as bipolar plate substrate materials for the PEMFC use without surface treatment. 304, and 316L stainless steel alloys as well as Ti, on the other hand, can be considered in the future as potential PEMFC bipolar plate materials. It should be noted that Ti has high cost and oxide formation problems. When comparing the corrosion resistance values for SS316L and SS304, it was revealed that SS316L performed better in the simulated PEMFC environment than SS304. Polarization test results showed that SS316L produced lower current density in both anodic and cathodic sides. A similar conclusion was pointed out in an earlier study [25].

Confidence intervals of the mean corrosion current densities for metal substrates were higher than 1 μA cm^{-2} (target value by DOE by 2015), and therefore, the metal substrates are considered to be corroded by themselves too much for use in the PEM fuel cell. According to the corrosion test graphs, the lowest current density of the samples was determined as 4.83 μA cm^{-2} (unformed Ti). This value is still above the target of 1 μA cm^{-2}. Moreover, the passivation of the substrate materials would stimulate the interfacial contact resistance (ICR) of the bipolar plate, reducing the conductivity, and efficiency. It was concluded that none of the substrates tested in this study would meet the 2015 DOE goals. However, surface modifications could increase the corrosion resistance of these substrates dramatically.

Future studies regarding the corrosion behavior of the metallic bipolar plates should include coated substrate materials. In literature, many studies aimed to determine an inexpensive, pinhole-free coating that defends the substrate effectively [4,26–28]. From these studies it is clearly seen that, there are many coating alternatives and surface modifications techniques that could increase the corrosion resistance of bipolar plates to satisfy the DOE requirements. These coatings could be applied to SS304 and SS316L to decrease corrosion current densities to less than 1 μA cm^{-2} in order to achieve bipolar plates which could be used in

PEMFC devices with compatibility and high performance. In next phase of this study, several coating types such as TiN, ZrN and CrN with different thicknesses will be applied onto SS316L substrate materials, and the effect of coating materials on the improvement of corrosion and contact resistances will be investigated. The effect of coating process sequence (coating applied prior to manufacturing and after manufacturing to bipolar plates) will be studied, as well.

Acknowledgments

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