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Technical Evaluation of Proton Exchange Membrane (PEM) fuel cell performance – A review Tabbi Wilberforce^{1*}, O. Ijaodola¹, Emmanuel Ogungbemi¹, F.N. Khatib¹, T. Leslie¹, Zaki El

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Abstract

This investigation considered recent advancement made in material selection for bipolar plate (BP) for PEM fuel cell. The mechanical characteristics as well as the chemical characteristics of each material currently used for mass production of bipolar plate were also captured. The report delves more into the various techniques used in the coating of materials for BP. Several coating materials were also discussed. The impact of these coating materials on the efficiency of the fuel cell is also captured. Techniques adopted by researchers in coating applications with experimental justification are also captured in this report. The advantages and disadvantages of different surface treatment used in the development of bipolar plates is critically evaluated in this report. A conclusion was therefore deduced that for the cost of fuel cell to reduce to compete with other energy generation mediums, it is recommended that BPs are coated in order to curtail the effect of corrosion on the efficiency of the cell. Coating of BP improves corrosion resistance, interfacial contact resistance and corrosion current density. This report hence will serve as a manual for the fuel cell research community in their quest of improving the overall performance characteristics of fuel cells.

Key words: Bipolar plate, Interfacial contact resistance (ICR), Stainless steel, Polarization curve, Coating

1.0 Introduction

Energy is considered as the building block of life. The high reliance on fossil product for energy generation as well its environmental impact is the main contributing factor for the need to consider alternative forms of energy generation [1]. According to Yifei et al, 2016 [2] most fossil reserves are depleting gradually hence their sustainability cannot be guaranteed. Bizon and Phatiphat, 2018 [3] further argue that the prices of fossil commodities in recent times has been fluctuating making the cost of energy generation unstable. Places where some of these fossil products are extracted in their natural state have seen several unrests leading to the loss of properties and lives over the last few decades with Nigeria being a practical example. Scientists are considering other medium of energy generation due to the issues raised earlier. It must be noted that fossil products continue to dominate the energy industry till date [4-6]. Priya et al, 2018 [7] in their research concluded that renewable energy therefore remains the only alternative to fossil products as they are environmentally friendly. PEM fuel cells according to researchers are one of the types of renewable energy generation mediums that will give a better competition with fossil fuel [8-12]. This is mainly because they are environmentally friendly with no amount of carbon dioxide being released into the atmosphere during the energy generation process [13]. PEMFCs are energy converting device that transforms hydrogen and oxygen into electricity using membrane electrode assembly (MEA) as the platform where electrochemical reaction occurs. Platinum is often used as the catalyst to speed up the chemical reaction process [14]. The byproduct for the electrochemical reaction is hydrogen and heat. Fuel cells in general are highly efficient means of energy generation but PEM fuel cells have fast start up time and they are very robust. They also require less maintenance as there are no moving parts in their build up. The absence of no moving parts implies that the issue of wear and tear and excessive heat leading to huge losses is not a challenge in the usage of PEMFCs. All these are some merits in generating energy using PEMFCs. The main disadvantage in the usage of PEMFC is the cost of the device [15]. They are currently expensive compared to other type of energy generation. The cost of PEMFCs is often due to the weight of the device, the optimization processes on input parameters (flow rate, pressure, temperature etc.) as well as the modification of the components in the fuel cell. An important component in the cell is the BP. They operate as the medium where reactive substances enter the fuel cell and serves as the exit point f or the byproduct of the cell [16-20].

The bipolar plate (flow plate) also functions as the collection point for all produced current from cell [21 – 25]. This means that a poorly designed bipolar plate will reduce the efficiency of the cell. Since most BPs in recent times are made up of metals, there is the need to consider coating the flow plate with suitable material to prevent corrosion but increase the electrical and thermal conductivity [26 – 30]. The work considers some specific research conducted on stainless steel and titanium. Due to their easy availability and cost, stainless steel is sometimes used in the production of BP. The ICR as well as I_{corr} of ferritic stainless steel was compared with SS316L. The order of the ICR was from 100 to 200m Ω cm² (SS444 > SS436 > SS441 > SS434 > SS316L > SS446. According to another investigation conducted by another researcher, the I_{corr} of SS316L, SS321 and SS347 are 0.26 x 10⁻⁴, 0.8 x 10⁻⁵ and 1.58 x 10⁻⁵ mAcm⁻² respectively. ICR of uncoated austenite stainless steel is normally greater than 100m Ω cm⁻² in anodic and cathodic region of PEMFC [18]. A conclusion can therefore be made that the bare stainless steel does not meet the US Department of Energy (DOE) criteria shown in Table 1.

Property	Unit	2017	2020
Weight	KgKW ⁻¹	< 0.4	0.4
H ₂ permeation rate	$cm^{3}(cm^{2}s)^{-1}$	<1.3x10 ⁻¹⁴	1.3x10 ⁻¹⁴
Cost	\$KW-1	3	3
Corrosion at anode &	µAcm ⁻²	<1	<1
cathode			
Electrical conductivity	Scm ⁻¹	>100	>100
Flexural strength (ASTM	MPa	>25	>25
D790-10)			
Area specific resistance	Ω -cm ²	0.02	0.01

 Table 1: Specifications for material used for bipolar plates

The flow plates Corrosion characteristics for SS316L was also investigated by a group of researchers using varying Sulfuric acid (H₂SO₄) concentrations via electrochemical techniques. Increasing the concentrations of the H₂SO₄ from 0 to 1 M according to their investigations made the I_{corr} increase appreciably from 0.84 to 640 mAcm⁻² and that of the interfacial contact resistance from 10 to $50m\Omega cm^{-2}$. Their work further stated that the ICR between SS316L and carbon paper decreased as H₂SO₄ concentrations increased. This condition changed when the passive film thickness increased. It also showed the need for the coating of SS316L using a thin conductive and protective layer.

This paper focuses on coated bipolar plates for proton exchange membrane (PEM) fuel cells applications. It involves investigation into the different materials used for coating bipolar plates, the process of stamping bipolar plates and various surface treatment adopted for the development of bipolar plates. Materials investigated include carbon, chromium, nitrogen, nickel, noble metals, non-ferrous alloys and aluminium. All these materials have potentials in have been highly considered by researchers in this field. Furthermore the process of stamping the bipolar plates for both bare and coated metals – hydroforming, is discussed. Surface treatments considered in this work include chemical approach, treatment via heat, ion implantation, spray coating and vapour deposition (VD).

1.1 Carbon coatings

The best coating suitable for stainless steel according to Feng et al [31] is carbon films. This conclusion was after performing potentiodynamic experiment and potentiostat experiment to determine rate of resistance to corrosion and the rate of corrosion for uncoated and coated material using carbon.



Fig. 1: Carbon film on SS316 (Authorization to reproduce obtained from ref. [32])

Feng et al [31] kept samples in a resin (epoxy) having an area of 10 x 10 mm². By means of soldering, wire made of copper was fixed to the back of each material. Electrode system adopted

by the group was also made up of sheet of platinum. The platinum sheet functioned as counter electrode. The reference electrode was made of calomel electrode. The research group further performed electrochemical test in 0.5 M sulfuric acid to excite the hash PEM fuel cell conditions. Prior to electrochemical experiment, they recorded the open circuit potential against time for an hour to make sure the sample was electrically stable. The characteristics and how stable the uncoated and carbon coated materials behaved was also carried out using the potentiostatic test. The potentiostatic experiment was performed within 8.9 – 9hours at a potential of 0.6 V against the SCE but purged with air. The solution was then collected and inspected to ascertain the quantity of Iron, Chromium, Nickel and Molybdenum ions in the solution. The sample was then separated from the epoxy resin and all impurities eliminated using ethanol ultrasonically. The samples then went through scanning electron microscopy (SEM) prior and after the electrochemical experiment. This was to determine the processes as well as the impact of the corrosion on the bare and carbon coated Stainless Steel 316L. Using hydrophobic materials is one approach of curbing flooding in the cell stack as the H_2O prevents easy movement of air or oxygen by blocking the flow channel or the pores in the GDL. Finally, data physics OCA20 contact angle system was utilized to determine the water contact angle on the carbon coated Stainless Steel 316L [31]. The ICR was also determined using the approach suggested by Davies and Brady according to Taherian, 2012 [32] as shown in Fig. 2 below.



Fig. 2: Diagram for the set up used to determine the ICR (Authorization to reproduce obtained from ref. [34])



Fig. 3: Topology obtained from SEM a) cross sectional view b) thin film of carbon stainless steel 316L34 (Authorization to reproduce from [34])

Fig. 3 indicates the topology for thin film of carbon on stainless steel 316L material. From Fig. 3, it can be seen that the carbon film is made granules of carbon that are spherical, having diameters between $0.1 - 1\mu m$. The carbon utilized also had a thickness of $3.3 \mu m$ as shown in Fig. 3b. The entire duration for the coating was 4hrs. Accumulation of the carbon spheres is what creates the carbon film which is very compact and dense as well. The carbon film serves as a layer that reduces the rate of corrosion on the substrate. Fig. 4 shows the Raman spectrum of carbon coated stainless steel 316L.



Fig. 4. Carbon on stainless steel 316L [authorization to reproduce obtained from ref. 34] The G – band from Fig. 4 is at almost 1568cm⁻¹ as well as the D – band at 1390 cm⁻¹. A suggestion has been made that the D peak intensity to the G - peak must have a relation that is linear to the graphite crystallite size. The magnitude of the D – band is bigger compared to the G – band. This means the crystallites for the graphite is small but disordered band is large. Another suggestion has also been made that the G and D peak are as a result of sp² only. Feng et al [31] further determined the potentiodynamic polarization characteristics for uncoated as well as the carbon coated stainless steel 316L in 0.5 M sulfuric acid using 2ppm HF at 80°C and the results obtained is captured in Fig. 5.



Fig. 5: The uncoated and carbon coated SS316L potentiodynamic curves in 0.5M in Sulfuric acid solution using 2ppm HF at a temperature of 80°C purged with a) air b) hydrogen [authorization to reproduce obtained from ref. 31]

The potentials for the corrosion of the stainless steel 316L at cathodic as well as anodic region of the fuel cell according to their findings was -0.286V against saturated calomel electrode as well as -0.307 against saturated calomel electrode respectively. Half – cell potential for e (O₂/H₂O) $[O_2 + 4H^+ + 4e^- 2H_2O]$ is always higher than that of $e(H^+/H_2)$ $[2H^+ + 2e^- H_2]$, the rate of corrosion potential of SS316L is always higher at the cathodic region and in the anodic region of the fuel cell. A passive thin film is also created on the surface. Corrosion potential for the stainless steel 316L which was carbon coated was observed to be nobler compared to that of bare stainless steel 316L (247mV against saturated calomel electrode and 192mV against saturated calomel electrode in the cathodic and anodic conditions respectively. When the corrosion potential is high, the chemical inertness also goes high and this implies good corrosion resistance. The polarization curve obtained for the bare SS316L was similar to austenitic SS. This can further be divided into 3 main regions that is, active area, passive area and trans passive area. Their investigation showed a high passivating current of 1mAcm⁻² as captured in Fig. 5 because of active dissolution and oxidation of the metal. In a situation where the potential is increased to 0.9 V against SCE, there was a boost in the current density due to the oxidation of $Cr^{3+}(Cr^{3+}Cr^{6+})$. Main difference in carbon coated Stainless steel 316L is that there is no passive

region for carbon coated SS316L. The potentiodynamic curves using air to excite the PEM fuel cell cathodic region is also captured in Fig.10. Red dotted line shows the cathode operational potential at 0.6V against saturated calomel electrode whiles the current density for passivation of the stainless steel 316L at the cathodic region is 11.26µAcm⁻² but the current density of stainless steel 316L coated using carbon decreases to 1.85µAcm⁻². When the current density for the anode is small, the rate of corrosion is low signifying the durability of the material. The potentiodynamic polarization curve based on their investigation is showed in Fig. 5 b. Flow plate for PEMFC corrodes when a potential is applied to it (cathode 0.6V against SCE and -0.1 V against Saturated calomel electrode). Feng et al [31] further did a potentiostatic test to ascertain the resistance to corrosion for PEMFC conditions. At the cathodic region, a 0.6V against saturated calomel electrode was used during the potentiostatic experiment and purged using air whiles at the anodic region, a -0.1V against saturated calomel electrode was applied while purging with hydrogen. The potentiostatic curve they obtained at 0.6V against SCE for bare as well as coated SS316L sample is also presented in Fig. 5. The current density of bare/uncoated stainless steel 316L reduces appreciably at the initial stages but stabilizes at 1.4µAcm⁻² from Fig. 5. The reduction in the current density was due to the formation of a passive film on the material being investigated. Comparing current densities for the potentiostatic and potentiodynamic test exhibited some differences attributed to the passive film. Accumulated charge for the uncoated SS316L as well as carbon coated SS316L is also shown in Fig. 6 insert. It can also be observed that there is correlation between charge as well as time and this is linear. The current stabilization (total passivation) can therefore be deduced to occur after nearly an hour. The current density stabilizes faster for the carbon coated stainless steel 316L and kept constant at 2.4μ Acm⁻² [32]. There is a linear correlation observed during the testing time from the charge time curve showing that passivation is halted when the coated stainless steel 316L was investigated. This caused stability in the PEM fuel cell cathodic region. Current density of SS316L according to the investigation is less compared to the carbon coated in the stainless steel 316L in the potentiostatic experiment. The passive film created on stainless steel decreases corrosion rate.



a) b)
Fig. 6: Bare and coated stainless steel 316L potentiodynamic graph a) Simulated cathodic region
b) anodic condition (authorization to reproduce obtained from ref. [31]).

Figure. 6(b) explains the potentiostatic experiment for -0.1V against saturated calomel electrode generated from uncoated and coated stainless steel 316L using carbon around the anodic region. Decay of the current density for the SS316L was very rapid, underwent a positive negative switch as well as slowly. It shows initial passive film on the uncoated Stainless steel 316L was unstable within the PEM fuel cell. Fig. 7 shows the surface topology micrographs for the coated and uncoated carbon.



Fig. 7: Surface topology micrographs for the uncoated and coated Stainless steel 316L (authorization to reproduce obtained from ref. [33])

Feng et al [31, 34] further observed the uncoated and coated stainless steel 316L after the potentiodynamic investigation by adopting the SEM techniques. Rate of corrosion for the bare SS316L was high at both electrodes of the fuel cell (Anode and Cathode). Fig. 7 (b) shows the various corroded cells on stainless steel 316L after the polarization at the cathodic region of the fuel cell but the corrosion layer formed on the surface. Topology of the surface for coated SS316L did not exhibit any disparity but whitish substances were noticed on the samples shown

in Fig. 7(d) to (f). It implies that steel made of carbon is stable and is capable of serving as a layer to withstand corrosion. Fig. 8: shows the ICR results obtained.



Fig. 8: Interfacial contact resistance against compaction force for a) coated and uncoated SS316L b) carbon coated stainless steel 316L after the potentiostatic test (authorization to reproduce obtained from ref. [31]).

Li et al [35] al also conducted several experiments on aluminum bipolar plate to increase their ability to resist corrosion as well as improve the conductivity of the flow plate electrically. It must be noted that aluminum flow plates are 65% lighter compared to stainless steel. They deposited different kinds of coatings on the aluminum bipolar plate (Titanium Nitride, Chromium Nitride, Carbon, Carbon/Titanium Nitride and Chromium/Chromium Nitride). Aluminum alloy 5052 (AA – 5052) was used. Results obtained from the SEM showed that the coat made of carbon layer is denser compared to Titanium Nitride and Chromium Nitride. Potentiodynamic test showed that there was some improvement with the corrosion resistance for all the coatings. A detailed comparison for all the SEM images they obtained after the potentiostatic test, C/CrN and concluded that the multilayer coating shows the best stability. The chemical composition of the AA – 5052 they used is as shown in Table 2. Samples were split into 12mm x 12mm with thickness of 4mm and polished up using water proof abrasive paper.

Magnesium	Silicon	Copper	Zinc	Manganese	Chromium	Iron	Aluminum
2.2-2.8	0.25	0.10	0.10	0.10	0.15-0.35	0.40	obtained

Table 2. Aluminum alloy 5052 chemical composition

Fig. 9 shows the morphology of Titanium Nitride, Chromium Nitride, Carbon, Carbon/Titanium Nitride and Carbon/Chromium Nitride from a field emission scanning electron microscopy. To determine corrosion on the sample, the surface morphology technique was done after the potentiostatic tests. Fig. 10 shows the scanning electron microscopy (a) Titanium Nitride, (b) Chromium Nitride, (c) Carbon film, (d) Chromium/Titanium Nitride film, (e) Chromium/Chromium Nitride film. The characteristics of the uncoated and coated Aluminum alloy-5052 were determined using an acidic medium [35]. The electrochemical verification was done by purging the solution using air before and during the test [32]. Again, to ascertain the electrochemical stability, the potential against time was registered for an hour [34].



Fig. 9: Scanning Electron Microscopy image for (a) Titanium Nitride, (b) Chromium Nitride, (c)Carbon film, (d) Carbon/Titanium Nitride film, (e) Chromium/Chromium Nitride film (authorization to reproduce obtained from ref. [32]).



Fig. 10: Sectional Scanning Electron images for (a) Titanium Nitride, (b) Chromium Nitride, (c)Carbon film, (d) Carbon/Titanium Nitride film, (e) Chromium/Chromium Nitride film(authorization to reproduce obtained from ref. [32]).

Li et al [35] also measured the interfacial contact resistance for coated Aluminum alloy – 5052. The structure was made of Toray TGP – H – 090) which is a conducting carbon paper placed between the sample and 2 Cu plates. The drop in voltage is summing all the ICRs each sample found in the set up. In coating, the usual expectations are to have a dense, smooth as well as homogenous coating layer on the sample especially for physical vapour deposition coatings. This helps the ability for the material to resist corrosion to increase. Fig. 9 shows the image of

Titanium Nitride, Chromium Nitride, Carbon, Carbon/Titanium Nitride and Carbon/Chromium Nitride obtained from SEM. It is observed that there are some defects and pinholes on the Titanium Nitride and Chromium Nitride layers captured in Fig. 9 (a) and Fig. 10(b). Observable defects reduced the corrosion resistance of the Aluminum Alloy – 5052. Titanium Nitride as well as Chromium Nitride coatings were less dense than that of carbon coatings with spherical carbon granules. Li et al [35] further argued that even though the surface of the multi-layer is usually rough because of the Titanium Nitride and Chromium Nitride coatings being uneven, multilayer can create pinholes. The Carbon/Chromium Nickle multilayer coating was seen to be homogenous compared to Carbon/Titanium Nickle multilayer shown in Fig. 9 (d) and (e). The sectional images for the various coatings are shown in Fig. 10. The thickness for Titanium Nitride, Chromium Nitride, Carbon, Carbon/Titanium Nitride and Carbon/Chromium Nitride were 1.5mm, 1.3mm, 0.8mm, 2.5mm and 2.2mm respectively. The potentiodynamic curves for the bare and coated Aluminum Alloy – 5052 material for PEM fuel cell conditions is captured in Fig. 11. Most negative corrosion potential was observed for the uncoated AA-5052 which was nearly 1.4V against MSE. Corrosion potential for the coated AA-5052 was noble compared to the uncoated one implying that the AA – 5052 can resist corrosion better than uncoated AA – 5052 and the same was also observed for the chemical inertness.



Fig. 11: The potentiodynamic curves for the coated and uncoated AA-5052 in proton exchange membrane fuel cell a) purged with H_2 (anodic conditions) b) purged with air (cathode conditions) (authorization to reproduce obtained from ref. [31])

1.2 Chromium coatings:

As explained earlier chromium nitride (CrN) is one of the types of coatings applicable to stainless steel as well because it reduces the I_{Corr} and ICR [31, 34]. Park et al [30] carried out experiment to examine the strength for Stainless steel 430 metal BP after applying a coat using Chromium Nitride/Chromium for Direct Methanol Fuel cells. It was observed according to the investigation that the Chromium Nitride/Chromium coated sample reduced the Interfacial Contact Resistance from 200m Ω cm⁻² for uncoated Stainless Steel 430 to 4m Ω cm⁻² for the Chromium Nitride/Chromium coated SS430). I_{Corr} was nearly 10⁻⁵mAcm⁻² in uncoated stainless steel 430 as well as $10^{-7} - 10^{-6}$ mAcm⁻² for the coated Stainless Steel 430. Using the process of nitridation of alloys made of Chromium such as Nickle Chromium alloys and ferritic Chromium stainless steel, Brady et al [42] tried by means of experiment to reduce the ICR. Well-known method of creating coats on stainless steel is by Chemical vapour deposition and Physical Vapour deposition [36]. The process of modifying the surface using heat nitridation is an approach of reducing the ICorr and ICR of BP forming a mixture of nitride/Chromiumnitride/oxide. When temperatures are high (900°C), thermal nitridation generates Chromium Nitrides that are discrete. This results in chromium depleted areas, reducing the opposition to corrosion. Precipitations such as Chromium Nitrides and Titanium Nitride as well as Chromium - depleted regions are more conductive compared to Chromium oxide. This is created when the nitridation occurs at high temperature. This explains that at very high temperature nitridation reduces the ICR. There is absence of Chromium from the matrix at high temperature conditions and this reduces the corrosion resistance. Atomic diffusion reduces whenever the nitridation temperature reduces which in effect reduces the thickness of the coat. This in effect causes the corrosion current density to increase as well as the ICR [37]. They therefore explained that the nitridation temperature must be optimized. The investigations conducted by M.J. Kelly, et al [38] considered the thermal nitridation for two separate temperatures (700°C and 900°C) stainless steel. Their report suggested that the ICR for stainless steel reduced after nitridation. This reduces the contact electrical resistance. At 700°C the coated steel according to their investigation had the best corrosion properties in PEM fuel cell conditions but coated steel at 900°C had relatively low properties. The explanation they gave was that at low temperatures, stainless steel nitridation created a layer and this protected the metal beneath the coat from the possibility of corroding. An explained was also given that the interfacial contact resistance value

and I_{corr} simulated anode conditions and I_{Corr} in simulated cathode conditions for SS446M that has undergone nitridation but at low temperature was $6m\Omega \text{cm}^{-2}$, 1 x 10^{-6}Acm^{-2} and 1 x 10^{-7}Acm^{-2} respectively and these are less compared to the bare SS446M. Another researcher also reported that that the ICR can also be reduced further to 10 m Ω cm⁻² using plasma nitridation of SS316L at 370°C for over two hours and that of SS304L as well but the I_{Corr} was higher than 10mAcm⁻². Tina et al also projected that performing coating at higher temperature of 370°C will lead to precipitation hence an increase in depletion and reduced resistance to corrosion. Optimization of the temperature of nitridation of stainless steel 316L was performed by Hong et al. They varied the temperatures at which the coating was done between 257, 317 and 377 °C. Smallest value for the Interfacial contact resistance obtained at a temperature of 317° C was 13 m Ω cm⁻² whiles the smallest value for the current density was obtained at 257°C (3.43 x 10⁻⁶ Acm⁻² at 0.6V). The outcome of their investigations confirms the fact that optimizing the temperature of coating process reduces the Interfacial Contact Resistance. The process temperature can also be reduced using high – density plasma nitiriding and this in effect nullify the Cr depletion [37]. Other authors investigated the merits of treating stainless steel 316L at lower temperatures for 180 minutes at 900oC using chromium. Using shot peening to active surface method helped the substrate to be pretreated and reduced the chromizing temperature. The I_{Corr} for the chromized SS316L was 3 x 10^{-7} Acm⁻² and the ICR values was 23 m Ω cm⁻² and these values were 3 times less than that of bare SS316L [38 – 45]. A homogenous and heavy coat made of chromium on 1045 steel also investigated by Bai et al. Outcome of their investigation revealed that the phases of the material were made of carbides as well as chromium ferritic oxides [46]. Power density produced using the metal BPs was greater than that of graphite flow plates. They argued that carbon steel can be compared to that of graphite for the manufacturing of bipolar plates in PEM fuel cell. The price tag of carbon steel bipolar plates is less compared to metal bipolar plates. Other researchers also conducted investigations using chromium carbide on aluminum as well as stainless steel 316 samples. Their report exposed the fact that using chromium carbide gave a relatively reduced ICR as well as good resistance to corrosion. Testing the longevity of fuel cell using aluminum that is coated as bipolar plate at cell temperature of 70°C gave a minimum power degradation because of the corrosion of the metal. The behavior of some coatings electrochemically like Chromium Nitride/Titanium Nitride on Stainless steel 316L was also investigated. The work was aimed at examining Chromium/Nitride/Titanium Nitride coat on

stainless steel 316L. Protective efficiency (0.76 mAcm²) according to the electrochemical investigations was high when the coating of Chromium Nitride/ Titanium Nitride thickness was kept at 1:9. Another investigator examined Nickle – Chromium on Stainless steel316L and concluded that the interfacial contact resistance reduced from 380 to 200 m Ω cm⁻² for the Nickel Chromium – coated Stainless Steel 316L after polarization [45].

1.3 Coatings containing Nitrogen

Coatings containing Nitrogen like Titanium Nitrides on stainless steel is very common [51]. Using Titanium Nitride coated SS304 as bipolar plate has also been investigated. Titanium Nitrides and Titanium (II) Nitrides (Ti₂N) coatings gave a lower interfacial contact resistance, 25 and 26 m Ω cm⁻² respectively. A reduced I_{Corr} of 0.0131 and 0.0145 μ Acm⁻². To effectively validate the results, the test must be conducted for longer period of time. Several protective coatings were deposited on stainless steel samples (stainless steel 304, stainless steel 310 and stainless steel 316) using PVD approach [52]. The outcome of the investigation met the target set by the DOE for bipolar. Interfacial contact resistance for the stainless steel 316L (300 m Ω cm⁻²), Zirconium – coated stainless steel (1000 m Ω cm⁻²) and Zirconium Nitride -coated stainless steel (160 m Ω cm⁻²) showed that even though Zirconium is good as coating material to combat corrosion, the interfacial contact resistance increases. Again, coatings made of Au reduces I_{Corr} and ICR less than the criteria set by the DOE as long as the thickness of gold coating is greater than 10nm especially on the cathode region. Some results also showed that Chromium Nitride is the best types of coat for SS BP of PEM fuel cells [53-55].

1.4 Ni – P electroless plating

Electroless plating technique is one strategy for coating BP. The industry is one of the sectors where Ni – P coating on stainless steel can be found. Using expensive organic material is one of the reasons why this method according to the research community is not the very best approach of coating even though it is simple and requires uniform solution bath at specific temperature and power of hydrogen. Homogeneity of the thickness of the coat via electroless approach is more compared to electroplating. Electroless plating approach was used to coat Ni – P on stainless steel 316L. Potentiostatic test using Ni – P made under favorable conditions were performed [56]. A negative current was generated for all times showing the cathode being protected. There

were no metal ions identified in the solution even after hours of potentiostatic treatment. Bulk resistance is reduced whenever the bipolar plate made of stainless steel was coated using Ni-P layer and the performance of the cell increased compared to existing cells on the market. The Ni-P coat on stainless steel 316L by Copper - interlayer showed maximum output current when compared to the proton exchange membrane fuel cell. A researcher conducted an intensive investigation coating AA 5251 using Ni – P and Ni – Co – P by means of electroless and electroplating approach. The corrosion current of Ni-Co-P coated increase by nearly 4 times compared to the bare AA5251 material. Corrosion current using electroless and electroplating approach were 3.21×10^{-5} Acm⁻² and Ni – P was 1.13×10^{-7} Acm⁻². The ICR values was very high when the electroless method was used (114 m Ω cm⁻²) and this is higher compared to the values obtained using electroplating (m Ω cm⁻²). Their investigation showed that using Ni-P coating was not the best for bipolar plates [57- 60].

1.5 Coating using Noble metals

Gold, silver and platinum are noble metals that are suitable as coating materials on stainless steel due to the protection being provided by them as well as improving the conductivity of the stainless steel. Silver has good electrical conductivity, excellent corrosion resistance and very cheap. Through the process of ion implantation, a group of researchers coated a thin layer of silver on SS316L. According to the results they obtained from the potentiostatic tests, there was a reduction in the I_{Corr} from 10 to 0.7μ Acm⁻² after the implantation using Ag. Their work also showed an improvement of the ICR values from 312 to 78 m Ω cm⁻². It must be noted that the value for the ICR can reduce immensely due to the method of coating, environmental conditions during the coating, as well as thickness of the coating [61- 64].

1.6 Different coating materials

ICR characteristics of Niobium – implanted SS316L was investigated [31]. Results obtained showed that the current density of Niobium implanted Stainless steel 316L reduced in simulation in PEM fuel cell environment far below 1μ Acm⁻². The dissolution rate from the induced coupled plasma results showed that the Niobium – implantation reduced. Depth profiles for XPS techniques showed that a passive film is created made of NbO. The outcome of their investigation showed that using Niobium improved the corrosion resistance as well as the flow of

electricity of Stainless Steel 316L in PEM fuel environment under simulated conditions. Their report did not consider the ICR test [65]. Another method that can reduce the corrosion resistance and interfacial contact resistant is doping using another element. Cerium element was used by Lavigne et al on the surface of stainless steel 316L. The steel surface was initially modified by keeping it in a steel solution made of CeO₈S₂ and Na₂SO₄ for two hours. The final data they collected from their investigation also showed that the Ce-modified stainless steel was less than the DOE criteria. There was also reduction in the ICR values they obtained from 152 m Ω cm⁻² for SS316L to 33 m Ω cm⁻² for the modified stainless steel [66].

1.7 Coatings made of Nonferrous alloy.

Except for the price, Ni-based alloys are good coating material for metal bipolar plates. Monel, Hastelloy and Incoloy are examples of these materials. Ferrous alloys have low corrosion resistance as well as low ICR [67]. Cost of Ni-based alloys are higher than that of ferrous alloys. The variation in prices for Ni – based and ferrous alloys makes manufacturers of BPs prefer the latter for commercial applications. The cost, weight, accessibility and stampability of aluminum alloy makes them preferable by the research community as the best coating material but they are weak in strength compared to SS. Titanium is expensive but possess low corrosion resistance as well as low stampability when compared to stainless steel.

1.8 Alloys made using Aluminum

Nitride coatings like Chromium Nitride and Zirconium Nitride on Al-5083 bipolar plates using Physical Vapour Deposition appraoch was examined thoroughly. The results obtained from the potentiodynamic curve depicted that aluminum – Chromium Nitride sample gave good corrosion resistance compared to Zirconium Nitride/Chromium Nitride coat under simulated conditions.

Multi layered bipolar plate is more fragile compared to the monolayer. A method of reducing the rate of corrosion for metal BPs is using composite layer. An Aluminum - based bipolar palte coated using polypropylene composite was also investigated by M.J. Kelly, et al [38]. The inter layer was made of carbon black and carbon paper. This was to lower the ICR between the aluminum and composite layer. Their work exposed the fact that the interfacial contact resistance was less than 21 m Ω cm⁻² as well as the I_{Corr} was less than 1µAcm⁻² [68]. Polymer based composite on Al was also researched using wet spraying [69]. Their investigation could not meet

the US department of Energy criteria because applying the coat by means of spray produced defects on the sample [70].

1.9 Alloys made of Nickel

Three commercial alloys were studied by Brady et al. These were Hastelloy G -30, Hastelloy G - 35 and AL 29 - 4C. The interfacial contact resistance of Al 29 - 4C, Hastealloy G-30 and Hastealloy G-35 reduced after nitridation to 10 m Ω cm⁻² (at 150 Ncm⁻²). Their surfaces became resistant to corrosion due to nitridation at the anodic current density falling below 1μ Acm⁻². Forming semi – continuous Chromium Nitride surface layers on (Hastealloy) and AL 29 – 4C via nitridation was also performed. The application of these metal bipolar plates is small because of their high price [69]. The resistance to corrosion on uncoated metals was equally investigated [70]. Their work showed that the current density of stainless steel 316L, stainless steel 321, stainless steel 347, Inconel 625, Incoloy 825, Hastelloy C-276, Tantalum and Titanium were 26, 8, 15.8, 4, 6.4, 4.8, 1.26 x 10⁻² and 1260µAcm⁻² respectively. stainless steel 316L, stainless steel 321, stainless steel 347, Inconel 625, Incoloy 825 and HastellovC-276 at 80°C was 12.6, 20, 50, 0.1, 4 as well as 0.8µAcm⁻² respectively. At 120°C only Tantalum and at 80°C Inconel 625 and HastelloyC-276 meet the standard set by the DOE (1µAcm⁻²). Tantalum has very high corrosion resistance but it is not a cheap metal (very expensive). The most suitable material for bipolar plates are HastelloyC-276 and Inconel625. From commercial perspective, stainless steel especially SS316L are conducive for the manufacturing of BP because their prices are lower, and they are also abundant [71]. The formability of Stainless Steel 316L is higher compared to other metals such as Titanium and this characteristics of stainless steel 316 aid in stampability.

1.9.1 Titanium alloys

Titanium a type of metal which is very light (density 4.51 gcm⁻³). It has hexagonal close packed configuration. Titanium is normally coated using an oxide layer that is nonconductive. It must be noted that when Titanium is compared to some metals with Face Centered Cube structure for example, Nickel and stainless steel 316L, the formability of Titanium is low hence weaker stampability. The oxide layer coated on Titanium surface causes the interfacial contact resistance to increase considerably. The corrosion and conductive layers of Ti has been coated in several investigations conducted by several researchers in order to improve their performance. Using

pulsed bias arc ion plating approach, Titanium – Gold film was coated on Titanium samples which led to interfacial contact resistance as low as 4.3 m Ω cm⁻². The current density of Titanium / Titanium – Gold was nearly 10µAcm⁻² and this is greater than the US, Department of Energy standard. Nitrogen plasma ion implantation was conducted at low temperature of 100°C and high temperature of 370°C in order to enhance the corrosion resistivity as well as ICR of titanium samples by Feng et al. [34].

2.0 Hydroforming – stamping of bipolar plates.

As explained earlier, graphite and polymer based composite material used for bipolar plates are fading away [35] and being replace by metallic bipolar plates because the metallic sheets can be stamped up to low thickness below 0.051nm. Laser welding process can be used in metallic bipolar plates making put them at a better advantage compared to the other materials. Two methods for creating channels on large materials to build flow plates for PEMFC applications is hydroforming and stamping. Fig. 12 shows the process of performing stamping whiles Fig. 13 shows hydroforming approach but there are two main observable defects using these two processes. These are, the breaking of the sample during the forming stage and unsteady distribution of flow. Channel rib depth is optimized to reduce the unsteady distribution of the flow [32]. A researcher concluded that the distribution of the flow is dependent on channel rib depth. Optimization of the depth therefore affects the distribution of the flow [66].



Fig. 12: Schematic diagram for the process of stamping (authorization to reproduce obtained from ref. [32])



Fig. 16: Schematic diagram of a machine for the hydroforming process (authorization to reproduce obtained from ref. [33])

At the channels peak, the hydroformed bipolar plate yielded a low surface roughness value compared to the stamped bipolar plate. Again, hydroforming shown in Fig. 13 reduces dimensional variations compared to stamping process. There are two major concerns that must be addressed during the micro forming process and high production rate of bipolar plates. The dimensional error because of manufacturing technology controlled is the first issue. The next issue is related to shape error because of spring back in the stamping technique, stress in wielding process, unequal and clamping pressure. To improve the efficiency of the cell, it is always recommended, that pressure dissemination via the cell is uniform. The dimensional error as well as the shape error all lead to the unsteady stress distribution hence the unbalanced distribution of flow also affects performance between the flow plate and Gas Diffusion Layers [33]. The variation of channels and ribs as well as the surface roughness are all classified under

dimensional error. The formability and contour of the surface as well as the quality of metal bipolar plate using stamping and hydroforming have all be researched. According to their investigations, hydroforming makes the bipolar plate have lower dimensional variations.

2.1 Bare Metals

A material classified under bare material is Stainless steels. This is because they possess good strength, stable chemically, flow of gas through them is low, many types of alloy choice as well as can be applied for mass production of the fuel cells and also relatively cheap. The major challenge using this material is corrosion. Different authors have considered using stainless steel as bipolar plate [31 - 33] and their investigation showed that the rate of corrosion is low and the output of the cell is very reliable for hours. Wang et al [73] conducted a research and also concluded that stainless steel is good for making of bipolar plates [74 – 75]. Their work showed that austenitic (349TM) and Ferritic (AISI446) stainless steel with high chromium content are good for bipolar plate material even though ferritic needs improvement.

2.2 Coated Metals

Nickle, titanium, stainless steel and aluminum are considered as suitable for BPs but to prevent corrosion, they must be coated using good coating covering. Coats applied on materials are supposed to be very conductive as well as stick to the metal substrate without showing the base metal out and they must also be very close to avert formation of micro – pores on the coated metal to reduce coating expanding [36]. Today two types of coatings are being investigated. These are coatings made of metals and carbons. Coatings made of carbon include usage of graphite, polymer, as well as mono polymers. The coatings made of metals involves the usage of metals that are noble. Today there are more research being conducted on coated stainless steel (316L) as bipolar plates [37,38]. An investigation conducted by wind et al shows that using BP that is gold coated stainless steel also improved the efficiency of the fuel cell just like graphite. Coating of stainless steel 316L was proposed by M.J. Kelly, et al [38]. Coating of metallic bipolar plates still have some challenges due to pinhole defects [40,41]. Brady et al [41, 42] as a way of curbing the impact of pinhole defect due to coating manufactured bipolar plate using thermal nitridation process. This approach is yet to be verified. Researchers around the world are

working to enhance the performance of the fuel cell via coating the BPs mostly for the automotive industry but they are yet to be commercialized.

3.0 Summary of surface treatment adopted for development of BPs.

Metallic BPs as explained are susceptible to corrosion and rust when their surface comes into contact with an environment that is acidic and moist especially as can be seen in fuel cells. the various research work that concerned coating of the metals by some individuals and research groups have be presented earlier but this part of the paper will give a summary of the current methods used in coating of bipolar plates. The methods considered are chemical, treatment using heat, spraying, vapor deposition, and Ion implantation approach.

3.1 Chemical approach

The compositions from substrate to the final coated material metallurgically was studied electrochemically using passive film by a group of researchers and their work saw a high improvement of the bipolar plates corrosion resistance. Their approach made the surface morphology to become very smooth as well as glossy appearance and this enhanced the gas and H_2O movement in the flow channels on the flow plate and decreases the resistance as a result of surface morphology. Electrochemical characteristics of the cell stack was also evaluated by another group of researchers and their work was directly linked to the working temperature and lesser impact on pressure [34]. There is the need for human intervention whenever coating process like cyclic voltammetry and electroplating process is being used. A solvent for the anode and cathode is also required. Using electrochemical approach as the method of coating gives varying I_{corr} or doping percentage. According to the DOE, chemical experiments must be conducted beyond 24hrs in solvent. The materials must be checked for the possibility of it becoming corroded over a period in harsh conditions. This process in effect will help reduce the manufacturing cost but the results obtained via this process are generally inconsistent.

3.2 Treatment using heat

Traditional nitriding process are plasma, gaseous and salt bath nitriding [76]. Its a commonly used thermochemical treatment method of applying a coat on metallic flow plates using nitrogen

and carbon (nitrocarburizing) [77, 78]. Diffusion on substrate of the metallic surfaces at temperatures between 480 and 580°C also occurs on the surface. The development of materials as well as expectations of the various components of the materials gave rise to several technological approaches being implemented. Some challenges of using plasma nitriding (NP) are arcing, hollow cathode effect, sputtering and corner. The corner effect is a phenomenal treatment process which involves plasma nitriding (PN), gas nitriding (GN) as well as carburizing. Another approach used in active screen plasma nitriding (ASPN) gave good results but to uniformly complete nitriding, enough bias activation is required. This implies that nitriding increases the corrosion resiustance, surface hardness and fatigue strength. The merits of traditional PN without their demerits related to metals are all used in ASPN [79].

3.3 Ion implantation

This is a finishing approach utilized on the surfaces of metals using ionized atoms and some specific elements such as Nickel, Molybdenum, Chromium etc., extracted via electrical means, accelerated and geared towards the area being treated using vacuum chamber. The ions then perforate and become attached but there is loss of energy due to elastic collision with target atoms according to a research performed by Dearnaley, 2013 [76]. The performance of the implanted layer is higher because treatment depth very thin This method is described as the line of sight approach. There are other methods like metal dielectric deposition, plasma deposition, annealing and photolithography. The method results in smaller doses (1011/cm²) compared to chemical deposition. Doses for various levels are regulated precisely using this approach and the doping uniformity is very constant. There is low possibility of contamination because the process is often carried out in a vacuum, but the approach involves the usage of expensive equipment amounting to millions of pounds. The passive layer composition modification using ion implantation method for 316L stainless steel flow plates has been researched thoroughly. Interfacial contact resistance also reduced appreciably form $312m\Omega cm^{-2}$ to 36 m Ωcm^{-2} . Co – implantation using Ni – Cr reduced the ICR from 255 m Ω cm⁻² to 22 m Ω cm⁻² as the resistance of the corrosion increased appreciably. The work done by another researchers, Zhang et al [90] also saw the I_{Corr} of the sample with a coating layer being kept at $10^{-6.5}$ Acm⁻² and the ICR for the sample being coated for the cathode conditions in a simulation process surged up appreciably from 35 m Ω cm⁻² to 60 m Ω cm⁻². The work they conducted buttressed the fact that ion

implantation led to long term stability in proton exchange membrane conditions. The values obtained for ICR and I_{corr} are still greater than that of the target set by the US, DOE, which is 10 m Ω cm⁻² and 1µAcm⁻² though this approach improved the performance of the fuel cell successfully. There was good uniformity obtained from one wafer to the other giving a good flow channel purposely for equal distribution of reactant gases in PEM fuel cell.

3.4 Applying coat using spray

Spray gun is used during thermal spraying and air brushing to apply a coat on the surface of a metal. The material used for the coating sometimes comes as powder, ceramic rod as well as wire. Some investigators like Husby [77] employed the air brushing methods and the results generated for the current density values were between 0.11µAcm⁻² to 0.54cm⁻² for stainless steel flow plates. Results obtained had the best current density after the surface was coated and this met the target set by the DOE which is less than 1µAcm⁻². Fabricated multilayer coated onto substrate can be done using this conventional air brush approach. The thickness of the coating, spraying time, gaps in between sample and the air brush as well as the temperature for the substrate have all been examined via air brushing approach. According to a research conducted by Susanna et al [78], the coated sample by means of spray had 270nm of thickness and 80nm as the roughness of the surface. The target distance was kept between 100 and 180nm, whiles the pressure was also maintained between 10 - 18 pound per square inch at ambient temperature to nearly 70°C and the spraying was performed for a period of 30 seconds. The combustion flames/electric arc are the main disparity between thermal spraying and other spraying processes. The I_{Corr} results obtained according to an investigation carried out by Gago et al [79] is 1.10 x 10 Acm⁻² and although this value was low, it still could not meet the set target. The maximum thickness of the thermal spray on the metallic plate was maintained between 0.6 - 0.8 nm. with the surface roughness between 4 - 15mm. A researcher also argued in his report that using arc process in thermal spraying resulted in some deposits on the metal surface and this enhanced the coating percentage which in effect gave good protection for the material especially in an aquatic environment. This also indicates excellent uniformity absorbance within the plates, which will reduce the possibility of the part coated from damaging [78].

3.5 Vapor deposition (VD)

The methodology used for coatings are same when using vapour deposition approach, but the technology utilized as well as the processes involved physically differ slightly. Heating or sputtering is employed to deposit layers on the substrate of a metal in physical vapour depositions (PVD). In terms of corrosion resistance, PVD coatings are better compared to electroplating process due to the hardness of coating when PVD approached is used. The I_{Corr} obtained using PVD coating was 0.00029µAcm⁻² which indicates good corrosion resistance compared to electroplating method which was 1.9 µAcm⁻². PVD method is less harmful to the environment compared to electroplating approach. Durability that covers topcoats are not needed in PVDS because abrasion resistance, temperature and strength are all excellent [80]. The corrosion resistance for the single layered coating of TiN and Graphite - iCTM improved when the multilayer coated TiN + C was used. An investigation conducted also showed that 10nm Au coatings would be the best coating material for further future investigation. Chemical process is employed in the chemical vapour deposition (CVD) approach where high quality as well as good characteristics of a solid material is used as the material used for the coating process. The ICR and I_{corr} obtained falls within the US Department of Energy targets of $<10m\Omega cm^{-2}$ and $<1\mu A cm^{-2}$ whenever CVD approach is used. The inductive coupled plasma (ICP) combined with physical vapor deposition method of gas mixture (TiCl₄, BCl₃, H₂ and Ar) was also investigated. TiB₂ films were also seen as being very hard. It is cost saving and increases production. Again, the plasma surface alloying (PSA) approach is used in VD group [80 - 85]. The PSA using nitrides as well as carbon was also performed by Dong et al. The approach utilized enhanced resistance to corrosion on austenitic stainless steel. The usage of the PSA was also employed by Wang et al where they used Niobium and Carbon diffusion samples to treat materials to generate reduced current density (0.051-0.058 μ Acm⁻²) and interfacial contact resistance of 8.47m Ω cm⁻² [86 - 89]. A new type of approach called hybrid plasma surfacing co - alloying approach was also investigated. Interfacial contact resistance values of the samples coated were $9m\Omega cm^{-2}$ and long duration experiments were carried out to determine impact of corrosion on the cell stack. Another research conducted is using arc ion plating (AIP) [90]. The two experiments conducted by Zhang et al [91] using titanium and SS by the AIP approach concluded that nanocomposite Ti – Ag – N improved the resistance to corrosion as well as conductivity of the material. In extremely high oxidized atmosphere, titanium plate showed good resistance to corrosion, low

interfacial contact resistance at 2 m Ω cm⁻² under 140 Ncm⁻² and this increased the electrical conductivity. The next investigation conducted was to enhance the performance by using CrN_{0.86} film coated stainless steel 316L flow plates that limited the interfacial contact resistance to 8.8 m Ω cm⁻² under 100 Ncm⁻² and the I_{Corr} reached 0.1µAcm⁻² [92, 93]. From the results obtained for the two-research conducted, it can be deduced that the high film density obtained using the arc ion plating was the main contributing factor to the high performance of the coated bipolar plates. Cooling water system is needed to discharge heat for VD technique in some special cases at extremely high temperature [94- 101].

4.0 Conclusion

The investigation presented coating techniques and materials for PEM BPs. Several materials currently used for the manufacturing of fuel cell bipolar plate were discussed. Materials like composites are being encouraged by researchers in the manufacturing of bipolar plate because they reduce the weight of the cell. Even though metals and graphite continue to dominate the fuel cell market in terms of manufacturing of bipolar plates, researchers recommended that metals are coated to increase the longevity of the fuel cell. This will further reduce the prices of fuel cell in general. There is still more research work being conducted on metal and nonmetallic material to ascertain their overall effect on fuel cell performance. Optimization of all the surface treatment techniques captured in this report is still being researched to determine the best conditions that would yield the maximum performance of the fuel cell. A conclusion can therefore be deduced that for fuel cell to become commercially viable and to enhance their competition with other energy generation mediums for the automobile industry and portable applications, they must meet the target set by the US, Department of Energy. For the BPs to meet these criteria, coating becomes very necessary especially when they are made of metals. This improves the interfacial contact resistance, current density and the corrosion resistance.

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