

EXPERIMENTAL INVESTIGATION ON THE INFLUENCE OF PRE-CARBURIZATION ON THE SINGLE FUEL CELL PERFORMANCE OF CHROMIZED STAINLESS STEEL BIPOLAR PLATES

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ABSTRACT

This paper reports preliminary attempts at improving the in-situ performance of chromized 304 stainless steel bipolar plates via the introduction of a pre-carburization step prior to chromizing. 304 stainless steel bipolar plates with parallel flow field design were pre-carburized at 900°C for 3 hours and subsequently chromized at 1040°C for 3 hours. The surface modified plates were thereafter assembled into a 5cm² active area single Proton exchange membrane (PEM) fuel cell. The performance and durability of the single fuel cell with pre-carburized chromized bipolar plates (PCCBP) at room temperature was evaluated and compared to that of a non-carburized chromized stainless steel bipolar plate (CBP) tested under identical conditions. The results showed that the PCCBP attained a peak power density of 18.20 mW/cm², which was about a double-fold higher than that of CBP. The ten-hour durability test, however, indicated the need to further improve the stability of PCCBP.

KEYWORDS: *Chromizing, Current density, PEM fuel cell, Pre-carburization, Stainless steel bipolar plates.*

INTRODUCTION

Proton exchange membrane fuel cells (PEMFC) are electrochemical devices which have low operating temperatures, high power density, quick start-up capacity and rapid response to varying load advantages over conventional power sources. Therefore, these devices have been considered as potential power source for portable, stationary and transportation devices (Pollet *et.al.*,2012; Moreno *et al.*,2015). There are, however, many challenges to the widespread use of PEMFC for such application.

One of the major challenges to the successful commercialization of PEMFC is the high cost and low durability of the bipolar plate, a key component of PEMFC stacks. The bipolar plates perform critical functions that influence the overall performance and lifetime of PEMFC. These plates electrically connect adjacent cells in PEMFC stacks, facilitate uniform distribution of reactant gases over the entire active electrode area and provide pathway for removal and management of

by-products (Moreno *et al.*, 2015; Antunes *et al.*, 2010, Taherian 2014). As the most repeated component in PEMFC stacks, the bipolar plates account for a significant proportion of the total cost of PEMFC stack (Karimi *et al.*, 2012; Asri *et al.*, 2017, Oladoye *et al.*, 2014). Conventionally, these plates are fabricated from non-porous graphite due to its high corrosion resistance and surface conductivity. On the other hand, graphite is brittle, permeable to gas and expensive to mass produce. Hence, alternative materials such as stainless steel alloys, which have lower cost, easy machinability and availability of a wide range of alloys advantages over graphite, have been considered for bipolar plate application. On the other hand, the low corrosion resistance of stainless steels in the acidic and humid PEMFC environment coupled with its low surface conductivity due to the presence of a semi-conductive surface oxide layer are major concerns with the use of this ferrous based metal as bipolar plate material. Metallic ions generated during corrosion could contaminate the membrane and reduce its efficiency while the semi-conductive nature of the passive oxide layer, which increases in thickness during the corrosion process, increases the ohmic losses generated during operation (Antunes *et al.*, 2010; Karimi *et al.*, 2012) These challenges, which promote power output degradation, have necessitated the need for surface modification of stainless steels for enhanced performance in PEMFC environments.

Surface modification of stainless steels for bipolar plate applications have been extensively studied. The literature has shown that different types of coatings ranging from metal-based coatings such as chromium nitrides and chromium carbides and carbon-based coatings such as graphite and diamond like carbon (DLC) coatings have been investigated [Antunes *et al.*, 2010; Karimi *et al.* 2012; Taherian, 2014, Brady *et al.*, 2006). Chromium-based coatings deposited by pack cementation, in particular, have been shown to enhance the performance of various stainless steels in simulated and real PEMFC environments (Lee *et al.*, 2009; Cho *et al.*, 2008, Oladoye *et al.*, 2016; Wen *et al.*, 2010; Bai *et al.*, 2010; Yang *et al.*, 2010). According to the literature, pack chromized coatings on stainless steel for bipolar plate application can either be deposited at high temperature ($\geq 1000^{\circ}\text{C}$) (Lee *et al.*, 2009; Cho *et al.*, 2008, Oladoye *et al.*, 2016) or at lower temperatures such as 700°C combined with pre-rolling (Wen *et al.*, 2010; Bai *et al.*, 2010) or 900°C with shot peened substrates (Yang *et al.*, 2010). These coatings irrespective of the processing routes have generally been reported to improve the performance of bare stainless steel alloys in simulated PEMFC environments. Bai *et al.* (2010) had reported the single fuel cell performance of chromized 430, 420 and 316 stainless steel bipolar plates produced by a combination of rolling pre-treatments and low temperature chromising at 700°C for 2 hours. Oladoye *et al.* (2016) have also recently demonstrated that the peak power density of the single fuel cell

assembled with chromized 304 stainless steel bipolar plates produced by conventional chromising at 1040°C for 3 hours was two-fold higher than that of the uncoated bipolar plate. Although, these studies have generally indicated that chromized stainless steel could be viable for bipolar plate application; there is, however, the need for further improvement on the in-situ performance of chromized stainless bipolar plates in order to make these materials viable for commercialization of PEMFC.

A possible route to enhance performance of chromized stainless steel bipolar plates is to reduce its interfacial contact resistance (ICR) with the gas diffusion layer (GDL). It is well known that ohmic resistance arising from ICR between the bipolar plate and the GDL is a major contributory factor that promote output power degradation in PEMFCs. Several studies have also demonstrated that the lower the ICR between these components the better the performance of PEMFCs (Cho *et al.*, 2005; Peng *et al.*, 2014; Toops *et al.*, 2010, Brady *et al.*, 2006). Hence in this study, the possibility of enhancing the single fuel cell performance of chromized 304 stainless steel bipolar plate via the introduction of a pre-carburization step prior to chromizing process was investigated. The proposed method involves the deposition of a carbon rich layer on the substrate prior to chromising. The essence of the carbon rich layer is to promote the formation of chromium carbide and nitride coatings in lieu of conventional chromized coatings which consist of chromium in solid solution of iron, complex chromium iron carbide and chromium iron nitride (Hosmani *et al.*, 2014; Iorga *et al.*, 2014). The formation of these electrically conducting phases in the absence of iron solid solution could enhance the surface conductivity of chromized stainless steel alloy and ultimately its single fuel cell performance.

MATERIAL AND METHODS

Two pairs of 35 mm x 35 mm x 2 mm bipolar plates with serpentine flow field design as shown in Fig. 1 were fabricated in-house from commercially available 304 stainless steel sheet. A pair of the bipolar plates was chromized at 1040°C for 3 hours in a pack of chromium, ammonium chloride and alumina powders as described in a previous study (Oladoye *et al.*, 2016). Another pair of the bipolar plates was pack-carburized in a pack of charcoal and sodium carbonate powders at 925°C for 3 hours and quenched in water prior to chromising at 1040°C for 3 hours.

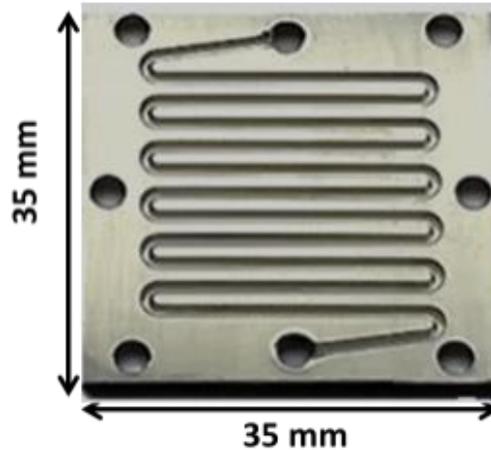


Fig. 1: Typical bipolar plate used in this study

2.2 Single fuel cell testing

The coated bipolar plates were assembled in pairs with a catalyst coated Nafion MEA (Fuel cell store, USA), Toray carbon paper gas diffusion layer (TGP-H-120, Fuel cell store, USA) and a silicon gasket (Fuel cell store, USA) into two single fuel cells shown in Fig. 2. Each single fuel cell had an active area of 5 cm^2 and was assembled at a compaction pressure of 125 N/cm^2 . Prior to the initial polarization of the cells, they were purged with nitrogen gas, after which, humidified hydrogen gas and compressed air was supplied to the anode and cathode side of the fuel cell at a flow rate of 50 ml/min respectively. All experiments were performed at room temperature. Open circuit potential was first measured after which current variation with cell voltage was measured, and power density was evaluated from the current-voltage data. The durability of the single fuel cells was investigated using a Gamry Interface 1000™ potentiostat/galvanostat. The test was conducted for 10 hours at constant voltage of 0.6 V using chronoamperometry technique. The cells were thereafter dismantled and EDX analysis of the GDLs was examined using with an INCA Energy dispersive X-ray (EDX) spectrometer (Oxford instruments).

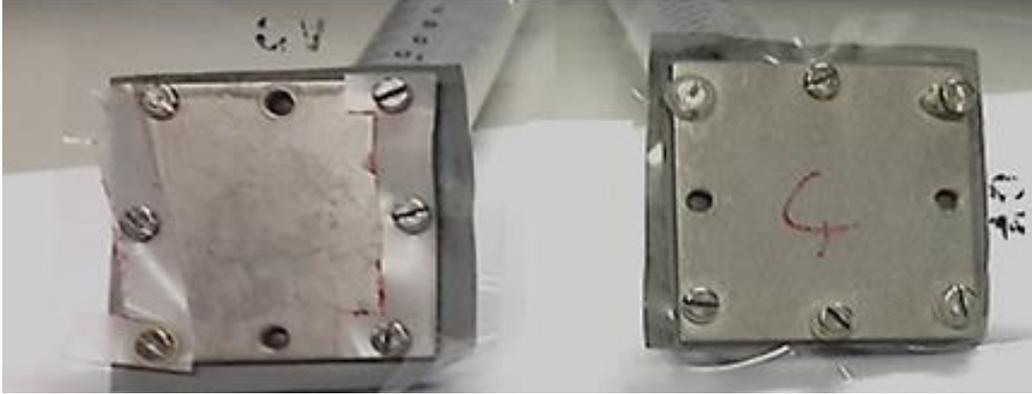


Fig. 2: Single fuel cells used in the study

In order to evaluate the effect of ICR on the performance of the cells, ex-situ ICR of both pre-carburized and non-carburized chromized 304 stainless steel coupons was measured using Wang's method (Wang *et al.*, 2003). Details of the experimental procedures can be found in previous studies (Oladoye *et al.*, 2016; Baroutaji *et al.*, 2017) The X-ray photoelectron spectroscopy (XPS) analysis of the outermost layer of the pre-carburized and non-carburized chromized 304 stainless steel coupons were accomplished with a commercial Kratos Axis Ultra XPS model with Al X-ray source. Core level spectra of selected elements were obtained at constant pass energy of 20 eV and 0.05 stepwise using adventitious C1s (285.8 eV) as reference.

RESULTS AND DISCUSSION

Fig. 3 a & b presents the current-voltage (I-V) and power density curves of the single fuel cell with non-carburized chromized bipolar plates (CBP) and pre-carburized chromized bipolar plates (PCCBP) respectively. At zero current, both CBP and PCCBP cells exhibited a similar voltage of approximately 0.92 V. However, as current was increasingly drawn from the cell, a rapid decrease in the voltage of CBP compared to that of PCCBP cell was observed. At the typical operating voltage of PEMFC (0.6V), PCCBP attained a 50% performance improvement

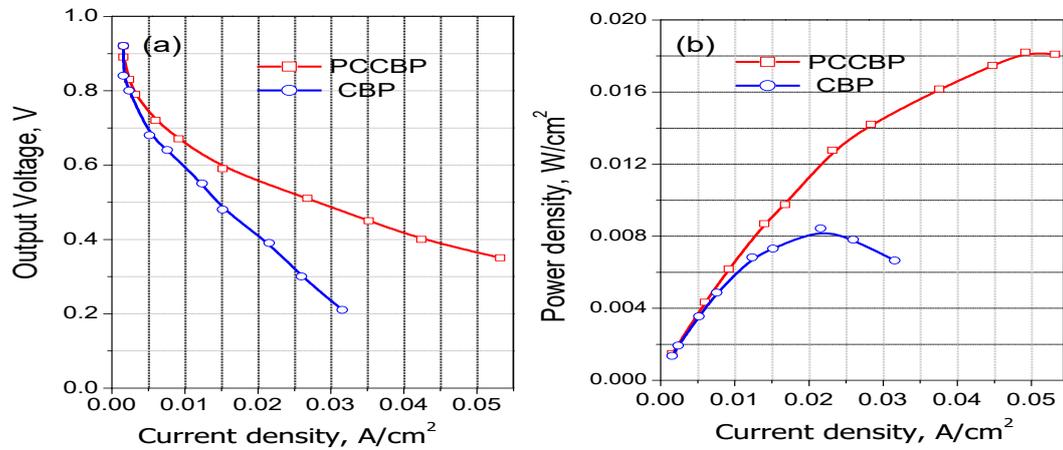


Fig. 3: Initial polarization of the single fuel cells

on the current density of CBP attaining a current density of 0.010 A/cm². Similarly, Fig. 3b indicated that the latter attained a higher maximum power density than the former. PCCBP attained a maximum power density of 18.20 mW/cm² at a current density of 53.2 mA/cm² while CBP cell attained a maximum power density of 8.47 mW/cm² at a current density of 21.60 mA/cm².

Researchers such as Bai *et al.* (2010), Cho *et al.* (2005) and Toop *et al.* (2010) had reported that ICR between PEMFC components play a significant role in determining the performance of PEMFCs. In this case, the single fuel cells had identical components (except the bipolar plate) and were operated under similar conditions. Hence, the improvement in performance of PCCBP over that CBP could be attributed to a lower contact resistance between the bipolar plate and the

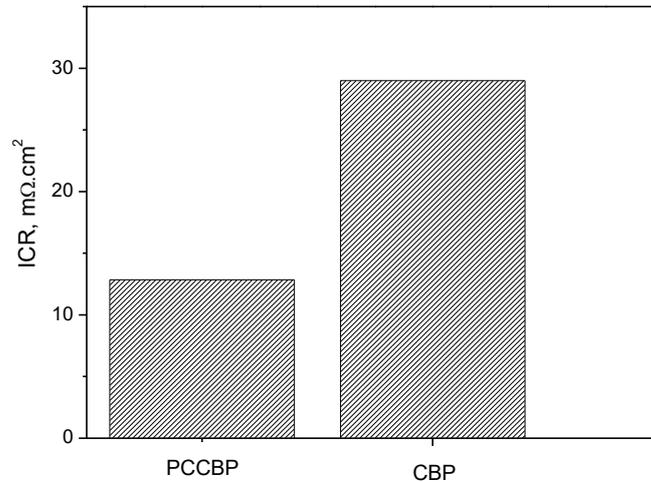


Fig. 4: ICR of PCCBP and CBP at 125N/cm²

GDL in PCCBP compared to that between the same components in the CBP. Nevertheless, in order to confirm this, ex-situ ICR was measured for both PCCBP and CBP coupons, at 125 N/cm², the assembling pressure of the single fuel cells. The ex-situ ICR of the coupons shown in Fig. 4 indicated that PCCBP coupon exhibited about 56 % decrease in the ICR of the CBP coupon, which was found to be 29 mΩ.cm². Surface conductivity of bipolar plate is known to be related to surface composition and roughness (Brady *et al.*, 2006; Karimi *et al.*, 2012). In this case, the effect of roughness could be considered negligible as both bipolar plates were subjected to similar surface preparation. Hence, the decrease in ICR observed in PCCBP coupon over that of the CBP coupon could be attributed to the difference in surface composition of the outermost layer of the metal. This fact was further confirmed using XPS. Table 1 shows the XPS analysis for of the two materials. The Cr2p, N1s and C1s narrow spectrum of both materials in Table 1 indicated the presence of chromium carbide (C₇C₃), Chromium nitrides (CrN and Cr₂N) and CrOxNy (chromium oxy nitrides) on the outermost layer of PCCBP coupon. These phases are well known to be electrically conductive except CrOxNy, whose electrical conductivity range between that of CrN and Cr₂O₃ depending on its oxygen content (Castaldi *et al.*, 2011). However,

Table 1: Energy of the photoelectron peak in the Cr2p, C1s and N1s of PCCBP and CBP.

Material	Cr2p _{3/2} (eV)	Cr2p _{1/2} (eV)	C1s (eV)	N1s	O1s	Phases detected	Reference
PCCBP	574.6	584.0	283.3	-	-	Cr ₇ C ₃	(Detroye <i>et al.</i> , 1999; Jiang <i>et al.</i> 2012)
	-	-	283.6	-	-	-	
	576.7	586.3	-	398.3	532	CrOxNy	(Agouram <i>et al.</i> , 2004;
	-	-	-	396.5	-	CrN	Subramnian <i>et al.</i> , 2011;
				397.4	-	Cr ₂ N	Lippitz <i>et al.</i> 2005)
CBP	574	583	282.2	397.3	530	(Cr,Fe) ₇ C ₃	(Detroye <i>et al.</i> , 1999; Jiang <i>et al.</i> , 2012, Lippitz <i>et al.</i> , 2005)
	-	-	-	-	-	Cr(Fe) ₂ N	
	-	-	-	-	-	Cr ₂ O ₃	

comparison of the XPS analysis of the coupons in this study to that of that chromium oxy nitride films in previous studies (Agouran *et al.*, 2004; Subramanian *et al.*, 2011) suggest that the CrOxNy in PCCBP could be CrN with traces of Cr₂O₃. On the other hand, XPS analysis of the outermost layer of CBP as shown in Table 1 revealed that complex chromium carbide (Cr (Fe)₇C₃), complex chromium iron nitrides (Cr (Fe)₂N and chromium oxide (Cr₂O₃) were formed on the outermost layer of CBP. Comparing the composition of the outermost layer of both PCCBP and CBP coupons, it is proposed that the presence of Fe solid solution in chromium carbide could have accounted for the increase in ICR of CBP compared to that of PCCBP. However, further studies would be needed to fully understand the role of Fe on the surface conductivity of chromized stainless steel. Furthermore, unreacted carbon in the carburized layer, which would be buried under the chromium carbide and nitride layer, could also have contributed to the enhancement of the surface conductivity of PCCBP as carbon is known to be electrically conductive.

Fig. 5 shows the short-term durability result for PCCBP and CBP single fuel cells. PCCBP exhibited higher current density than CBP throughout the duration of the test. However, its current density continued to decrease steadily compared to that of CBP, which stabilized after about 1.5 hours of operation. At the end of the ten-hour test, rate of current density degradation in PCCBP was estimated to be about 0.14 A/hr while that of CBP was about 0.07 A/hr. It is noted that current density degradation in both cells was quite high, suggesting that current density degradation may not only be due to corrosion of bipolar plates but also to factors such as possible drying out of the MEA evident by the initial sharp decrease in current density for both cells. Nevertheless, in order to evaluate the influence of corrosion on the performance, the single fuel cells were dismantled in order to characterize the metallic ions in the MEA. On dismantling the cell, a brownish corrosion product was found on the cathode side GDL of PCCBP while none was observed on the cathode side GDL of CBP while none was observed on that of GDL of CBP.

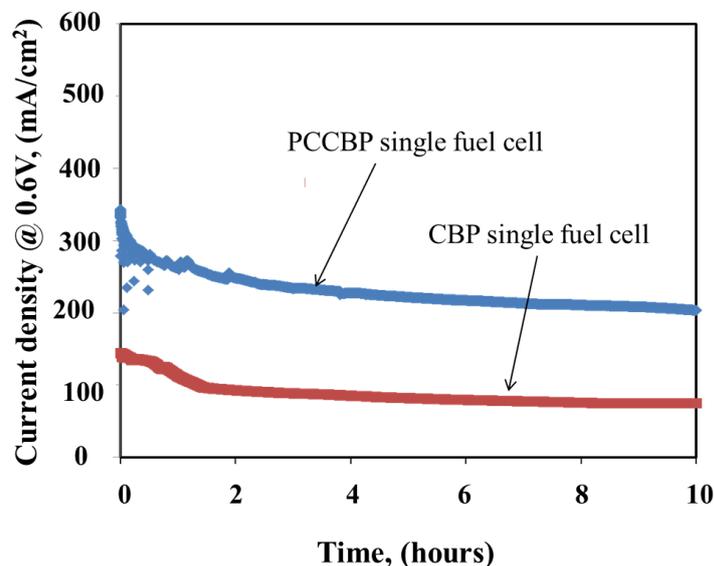


Fig. 5: Short-term durability test for PCCBP and CBP

Fig. 6 a & b presents the EDX spectrum of the cathode side GDL of CBP and PCCBP respectively. From the EDX spectra it can be deduced that corrosion was more severe in PCCBP than CBP with the EDX spectrum of the former showing more intense peaks for Fe, Mn, Ni and Si, which are constituent elements of the substrate. The presence of chromium, which was not in the EDX spectrum of CBP,

is also evident in Fig 6b. The poor corrosion resistance of PCCBP in the cathode PEMFC environment could be attributed to sensitization i.e. formation of chromium carbide at the grain boundaries, which leave the matrix devoid of chromium, an essential element for corrosion resistance in austenitic stainless steels. This behaviour of PCCBP also shows that a chromizing process after pack-carburizing may not adequately restore the chromium content of pack-carburized stainless steel in contrast to the work of Iorga *et al.* (2014). It could also be deduced from the durability test result (Fig. 5) that pack carburizing degraded the corrosion resistance of chromized 304 stainless steel, which is inconsistent with the work of Sulamain *et al.* (2016), who reported that pack carburizing, enhanced the corrosion resistance of 304 stainless steels in sodium chloride solution, hydrochloric acid and distilled water.

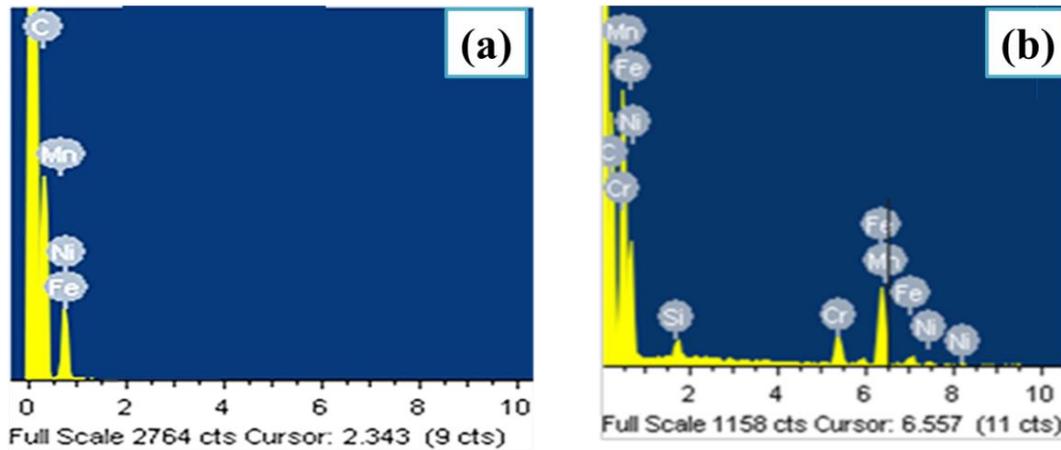


Fig. 6: EDX spectrum of (a) CBP (b) PCCBP after the durability test

The result presented in Fig 5 further suggest that employing low temperature carburizing techniques such as Swagelok gas carburizing could significantly improve the durability of PCCBP. Vaibhav *et al.* (2008) had reported that the proprietary Swagelok process facilitated the formation of a colossal supersaturated carbon layer on 316 stainless steel instead of iron carbide layer formed by pack-carburizing. Hence, corrosion resistance of 316 stainless steel was not degraded in simulated PEMFC, however, its ex-situ ICR, which was estimated to be about 75 $m\Omega/cm^2$ between typical compaction pressure of PEMFC (100-150N/cm²) was too high for PEMFC application. It is proposed that chromizing such gas carburized stainless steel could make a combination of pre-carburization and chromizing a viable method for enhancing the in-situ performance of chromized stainless steel.

CONCLUSION

In this study, it was demonstrated that the introduction of a pre-carburization step prior to chromizing of 304 stainless steel resulted in about 56% reduction in the ex-situ ICR of chromized stainless steel at 125 N/cm². Consequently, the single fuel cell performance of CBP was significantly enhanced. However, the short-term durability test, indicated that current density degradation in PCCBP was higher despite the double fold increase in its current density over that of CBP. Post-test analysis of the cathode side GDL of both single fuel cells indicated that the higher degradation rate observed in PCCBP could be attributed to its susceptibility to corrosion. The poor corrosion performance of this material could possibly be due to sensitization during pack-carburization. Hence, it could be concluded that adopting a carburizing technique which does not degrade the corrosion resistance of stainless steel is crucial for successful adoption of pre-carburizing and chromizing for enhancing the in-situ performance of chromized stainless steel bipolar plate.

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