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Active screen plasma surface co-alloying of 316 austenitic stainless steel with both nitrogen and niobium for the application of bipolar plates in proton exchange membrane fuel cells

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ABSTRACT

Austenitic stainless steel has been researched as a promising candidate material for bipolar plates in proton exchange membrane fuel cells. However, its interfacial contact resistance (ICR) is about 16 times higher than that of the Department of Energy (DOE) target ($10 \text{ m}\Omega \text{ cm}^2$), which leads to undesirable fuel cell performance. In this work, a new hybrid plasma surface engineering process, based on active screen plasma co-alloying, has been developed to simultaneously alloy 316 austenitic stainless steel (316 SS) surfaces with both nitrogen and niobium. The results demonstrated that the layer structure of the modified surfaces can be tailored by adjusting the treatment conditions. All the plasma treated 316 SS samples exhibited significantly reduced ICR below the DOE target of $10 \text{ m}\Omega \text{ cm}^2$. The corrosion resistance of the N/Nb co-alloyed 316 SS was much better than active screen plasma nitrided and marginally better than the untreated material.

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Introduction

As an efficient, clean and quite power source, proton exchange membrane fuel cells (PEMFCs) have received extensive interest in the last decade mainly due to the concerns over severe air pollution caused by conventional power sources and the depletion of fossil energies. Significant improvement has been made recently in the efficiency and performance of PEMFCs. However, the wide commercial application of

PEMFCs has been retarded, to a large extent, by the low mechanical strength and the high fabrication cost of graphite bipolar plates [1].

To this end, great efforts have been made to explore metallic bipolar plates, such as aluminium alloys [2,3], titanium alloys [4,5] and amorphous alloys [6,7]. Among them, austenitic stainless steels (SS) have attracted more and more attention as promising candidates for bipolar plate application mainly because of their good mechanical performance, relatively low cost and ease of manufacture

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[8–10]. However, there are still some limitations of austenitic stainless steels and hence technical challenges to be addressed. For example, their insufficient corrosion resistance [11,12] and poor conductivity due to the formation of passive oxide layer [13,14] can lead to undesired degradation of the power output of PEMFCs. It is known that surface modification has been successfully used to improve the surface properties of materials and components. Hence, it could be a promising method to improve the surface conductivity and/or corrosion resistance of stainless steel bipolar plates [15–18].

In our recent paper, a low-temperature active screen plasma nitriding (ASPN) technique is reported to modify the surface properties of 316 SS [19]. The surface conductivity of 316 SS has been successfully improved by the low-temperature ASPN induced nitrogen-supersaturated S-phase case. For example, the interfacial contact resistance (ICR) can be reduced from 158 m Ω cm² for the as-received 316 SS to 33 m Ω cm² for the ASPN treated surfaces. However, the ICR of the ASPN treated 316 SS surface is still larger than the target (10 m Ω cm²) set by Department of Energy (DOE) [20]. Clearly, alloying with interstitial nitrogen alone cannot meet the DOE target and new plasma surface co-alloying techniques with both interstitial and substitutional alloying elements could be a desirable approach.

Niobium and its compounds are well-known for their excellent corrosion resistance and surface conductivity. Many researchers have applied niobium coating onto the surface of stainless steel bipolar plates to improve their performance in PEMFC environment. Different methods have been explored such as cladding [21,22], physical vapour deposition (PVD) [23,24] and electro-deposition [25] to improve the corrosion resistance and surface conductivity of stainless steel. However, the bonding strength between Nb layer and the substrate is low after annealing treatment [26]. Electro-deposition of Nb improves the corrosion resistance of 304 SS but leads to the degradation of surface conductivity due to the formation of NbO and Nb₂O₅.

In view of the problems associated with Nb coatings, Feng et al. [27] utilizes ion implantation method to introduce Nb into 316 SS. The corrosion current density of the Nb implanted 316 SS is reduced effectively but due to the formation of Nb oxide, the surface conductivity of the Nb implanted stainless steel is still much higher than the DOE target of 10 m Ω cm².

Clearly, new surface engineering technologies need to be developed to further improve the surface electrical conductivity of 316 SS. This paper reports a new hybrid plasma surface technology which combines low-temperature active screen plasma co-alloying with both nitrogen and niobium with deposition of a thin surface niobium layer on the top. The microstructure and composition of the plasma treated surfaces were fully studied by X-ray diffraction (XRD), energy dispersive spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM). The surface conductivity and corrosion behaviour were also evaluated. Experimental results have demonstrated that the ICR of the plasma co-alloyed 316 SS surfaces is well below the DOE target (10 m Ω cm²).

Materials and methods

Materials and sample preparation: Commercial 316 austenitic stainless steel (316 SS) was selected as the substrate, and its chemical composition is (wt%): 0.06% C, 17.20% Cr, 1.30% Mn, 2.20% Mo, 11.70% Ni, 0.014% S and Fe balanced. Coupon samples of 6 mm in thickness were cut off from 1 inch (25.4 mm) hot rolled bars using a SiC cutting wheel. The front side of the coupon samples was firstly wet ground using silicon carbide abrasive paper up to #1200 grit and subsequently polished using 6 μ m and 1 μ m diamond pastes. The backside was wet ground up to #1200 grit. Prior to treatment, samples were washed with soapy water, cleaned with acetone in ultrasonic bath for 5 min, and finally dried under hot flowing air.

Cross sections were cut from treated coupon samples and mounted in conductive bakelite. The mounted samples were wet ground and then polished using the same procedures as described above. In order to reveal the micro-structure of the cross sections, an etching agent containing 25 ml H₂O, 25 ml HNO₃ and 50 ml HCl was used. After etching for about 20 s, samples were rinsed immediately with water then acetone, and dried in hot flowing air.

Active screen plasma treatments: Active screen plasma surface co-alloying treatments with both N and Nb (ASPA(N + Nb)) were conducted in an AS Plasma Metal 75 kVA industrial scale furnace and the experimental setting for the ASPA(N + Nb) treatment is shown in Fig. 1, schematically.

The small metal active screen was placed on the worktable of the large furnace, where bias was applied. The lid of the active screen was specially designed to carry niobium. The sample was insulated by ceramic and hence stayed at a floating potential. The effect of bias on the small metal active screen was studied using three different biases: 5%, 10% and 15% of the main power supply of 15 kVA, and the treatment parameters as well as the sample codes are listed in Table 1.

Characterization methods: The phase constituent of the plasma treated samples was investigated using XRD (Bruker D8 Advance) with Cu K α radiation ($\lambda = 0.154$ nm). The surface morphology and the cross-sectional microstructure of the samples before and after treatment were observed using SEM (Jeol 7000). The chemical compositions of the treated surfaces and cross-sections were examined by EDS (Oxford Instrument Inca). The composition and chemical state of the elements were analysed by XPS (Theta Probe). Elemental concentration depth profiles were determined by glow discharge optical emission spectroscopy (GDOES) (Leco GDS-750). The hardness depth profiles were probed using nano-indentation (Nano Test 600).

Corrosion tests: The corrosion behaviour of untreated and plasma treated 316 SS samples under different conditions was evaluated by electrochemical tests consisting of potentiodynamic polarization and potentiostatic polarization. A standard three electrodes system was employed in the polarization tests. A platinum rod, a saturated calomel electrode (SCE) and the to-be-measured sample were acted as the counter electrode, the reference electrode and the working electrode, respectively. The Gamry electrochemical workstation was used to measure and record the corrosion data. To simulate the working environment of PEMFC, a sulphuric acid

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