



Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Characterization and corrosion resistance of TiZr coating on SS304 stainless steel using cathodic arc evaporation techniques

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ARTICLE INFO

Article history:

Received 3 September 2016

Revised 26 December 2016

Accepted in revised form 12 January 2017

Available online xxxx

Keywords:

Physical vapor deposition (PVD)

Cathodic arc evaporation (CAE)

Polymer electrolyte fuel cell (PEFC)

Metal bipolar plates

ABSTRACT

In this study, an alternating titanium and zirconium (TiZr) nano-composite layer is deposited on a SS304 stainless steel surface to improve the corrosion resistance of bare SS304 in an acid environment with and without F ions as well as in a NaCl salt solution using cathodic arc evaporation techniques. The microstructures of this TiZr coating are studied and correlated with corrosion resistance. The corrosion resistance of a TiN/ZrN coating, which has an alternating titanium nitride/zirconium nitride (TiN/ZrN) multilayer, is compared with the corrosion resistance of the TiZr coating. The results show that the TiZr coating (layer thickness of 3.66 μm) has a nano-composite structure consisting of alternating Ti and Zr layers with a hexagonal closest-packed (hcp) arrangement. The corrosion rates of SS304 coated with a TiZr layer are much lower than those of uncoated SS304 in H_2SO_4 with and without F ions as well as in the NaCl salt solution. Additionally, the corrosion resistance of our TiZr coating is better than that of TiN/ZrN, CrN, TiN, TiN/CrN and CrN/Ti coatings and a bulk $\text{Ti}_{25}\text{Zr}_{75}$ alloy. The excellent corrosion resistance of the TiZr coating is attributed to the alternating Ti and Zr nano-composite structure with an hcp arrangement, which provides an effective impediment to active corrosive ions during anodic dissolution. Furthermore, the TiZr coating has lower interfacial contact resistance than the SS304, even after corrosion in H_2SO_4 with F ions, and it offers less surface resistance than the TiN/ZrN coating. This makes the TiZr coating a better protective coating for SS304 bipolar plates.

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1. Introduction

Titanium (Ti) has been extensively investigated as a biomedical material due to its good mechanical strength, excellent biocompatibility, high acidic corrosion resistance, and humid condition, in which no corrosion occurred at a high positive over-potential [1–4]. Some of the characteristics of zirconium (Zr) are similar to those of Ti, such as low density, good mechanical properties, high melting point, high corrosion resistance, and excellent biocompatibility [5]. Adding up to 50 at.% of Zr into pure Ti could increase the hardness of the alloy to about 2.5 times that of pure Ti [5]. This occurred because Ti and Zr formed TiZr alloys that strengthened the solid solution due to the difference between the atomic radii of Ti and Zr (1.47 and 1.62 Å, respectively). The alloys' enhanced mechanical and corrosion resistance make them excellent candidates for different applications, especially in the biomedical field, such as for dental implants, dental prostheses, bone plates, and artificial joints [6–7]. Most TiZr alloys are prepared by melting the two bulk metals together to produce certain compositions [1–2,8–9].

There have been only a few reports concerning the coating of TiZr alloys prepared by the magnetron sputtering technique. M.G. Rodriguez-Hernandez and colleagues [5] studied the mechanical properties of deposited TiZr and TiZr–C coatings fabricated using pulsed-DC magnetron sputtering at room temperature. The results demonstrated that the TiZr coating had a columnar dense packed morphology without pores, voids, or visible defects. This coating was partly crystalline with a mainly martensitic hcp α phase, although a ω phase was also detected. These phases caused the hardness and elastic modulus of the TiZr coating to be better than those of the bulk TiZr alloy.

Due to the enhanced mechanical, corrosion resistance, and electron conductivity properties of the TiZr coating, it is worthwhile to examine the possibility of using it as a protective coating for metal bipolar plates to improve the corrosion resistance of metal substrates such as stainless steel. The anti-corrosive properties of metal substrates, especially stainless steel, are not good in polymer electrolyte fuel cell (PEFC) operating environments containing H_2SO_4 with and without F ions. Metal-based bipolar plate materials used in PEFC environments, such as titanium, stainless steel, and copper alloys, have been widely investigated due to their ability to be easily mass produced, high mechanical strength, low interfacial contact resistance (ICR), and good corrosion resistance [3,10–12]. In order to improve the corrosion resistance of

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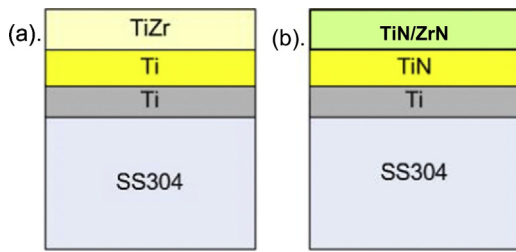


Fig. 1. Diagrams of the proposed layer structure of (a) TiZr and (b) TiN/ZrN samples.

metal bipolar plates, coatings with good corrosion resistance, such as Au, TiN/ZrN, CrN, TiN, TiN/CrN, and CrN/Ti coatings prepared using the physical vapor deposition (PVD) technique, have been extensively studied in the last few decades [4,13–18]. Among those coatings, the TiN/ZrN coating on SS304 stainless steel [14] showed an excellent corrosion resistance in H_2SO_4 and acid with F ions. However, the surface resistance of SS304 coated with a TiN/ZrN layer was also increased. Unlike the TiN/ZrN coating, the TiZr coating contains only metal elements but has enhanced corrosion resistance and mechanical properties. Therefore, it is worthwhile to investigate the electrochemical behaviors of TiZr alloy coatings comprised of only metal elements in a simulated PEFC environment containing H_2SO_4 acid with and without F ions. Furthermore, to the best of our knowledge, no studies have reported on TiZr alloy coatings prepared using cathodic arc evaporation physical vapor deposition (CAE-PVD).

This study aims to deposit alternating Ti and Zr layers on SS304 stainless steel using the CAE-PVD technique in order to improve the anti-corrosion properties of SS304 in H_2SO_4 acid with and without F ions as well as in NaCl salt corrosive media. The structural design of the thin film is shown in Fig. 1(a). The Ti layer between the TiZr coating and SS304 substrate acts as an interlayer to improve the adhesion of the TiZr coating to the SS304. This study investigates the microstructure, surface resistance, ICR and anodic potentiodynamic polarization of the TiZr coating.

2. Experiment

2.1. Surface pre-treatment of SS304 substrate

Table 1 presents a chemical analysis of the SS304 stainless steel used in this study. The oval-shaped SS304 specimens were cut with dimensions of 20 mm × 30 mm × 1 mm using the pressure cutting technique. Before coating, the specimens were first sanded using 240, 320, 400, 600, 800, 1200, 1500, 2000, and 2400 grit silicon carbide papers with a grinder and then polished with 0.05 μm alumina powder on rubbing fur. The specimens were thoroughly degreased, ultrasonic cleaned with detergent, rinsed with deionized water, and finally dried in a hot oven at 80 °C for 4 h.

2.2. Preparation of TiZr coating

The TiZr coating on SS304 was prepared using a CAE-PVD system like that reported in our previous publication [14]. Briefly, this system consists of a stainless steel chamber, vacuum system, arc generation system, DC source system, and control system. In order to deposit the TiZr layer on the SS304, we used high-purity Ti and Zr targets as the cathodes and an SS304 or Si wafer substrate as the anode.

Table 1
Chemical compositions of SS304 stainless steel used in this study.

Metal	C	Mn	Si	Ni	Cr	N	S	P	Fe
SS 304	Max:0.08	2.0	0.75	8.0–12.0	18.0–20.0	0.1	0.03	0.045	Balance

Table 2
Surface and sheet resistances of TiZr, TiN/ZrN samples and uncoated SS304.

Specimens	Surface resistance (m Ω)	Sheet resistance (m Ω /sq.)
SS304	0.45	2.04
TiZr	0.64	2.90
TiN/ZrN	0.66	2.99

The substrate surface was cleaned before beginning the coating process by argon-plasma for 20 min and Ti ion bombardment for 1 min with chamber pressure below 5×10^{-2} Pa and substrate biases of -1000 V and -500 V, respectively. We firstly deposited the Ti interlayer with a chamber pressure and substrate bias of 1–2 Pa and -150 V, respectively, for 3 min. The second Ti layer was obtained by setting the substrate bias at -120 V for 5 min. Finally, the TiZr layer was prepared with the substrate bias set at -80 V and a chamber pressure of 3–4 Pa for at least 20 min. The resultant sample was denoted “TiZr.”

We also prepared a SS304 substrate coated with a TiN/ZrN layer to understand the differences between the properties of the nitrile compound and pure TiZr alloy. The structure of the TiN/ZrN coating is shown in Fig. 1(b). The Ti and TiN layers between the SS304 substrate and the TiN/ZrN layer act as interlayers. The coating parameters for the Ti interlayer were the same as those used to create the TiZr sample. The second TiN interlayer was created by setting the substrate bias at -120 V with N_2 gas feeding. Finally, the chamber pressure and substrate bias were set at 2–4 Pa and -80 V to obtain the TiN/ZrN layer. The resultant sample was denoted “TiN/ZrN.” All the process temperatures and the rotation speed of the specimens on the vehicle for preparing these two samples were maintained at 180–200 °C and 2–4 rpm, respectively. The power-on time, power of Ti and Zr targets,

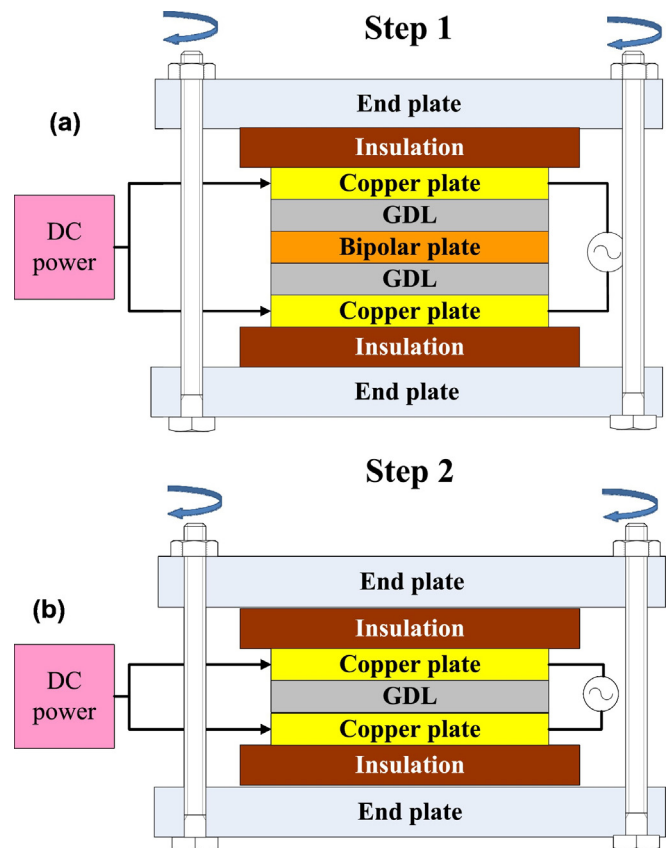


Fig. 2. Diagrams of the homemade resistance cell for the interfacial contact resistance measurement. (a). measurement of R_{total1} ; (b). measurement of R_{total2} .

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