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Short Communication

Effect of phosphoric acid and perchloric acid on Electropolishing of additive manufactured 17-4 PH stainless steel and its characterization

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Additive manufactured 17-4 PH stainless steel is widely used and is the material for this study. The processing of the work piece for additive manufacturing is difficult and the surface is very rough. The surface roughness is reduced by electropolishing using phosphoric acid and perchloric acid solutions, in order to compare their efficiency. Surface morphology is determined using SEM. A 3D white light interferometer measures the surface roughness and electrochemical testing is used to determine corrosion resistance after electropolishing.

Keywords: Electropolishing; Additive Manufacturing; 17-4 PH stainless steel; Surface roughness

1. INTRODUCTION

Additive manufactured 17-4 PH stainless steel is characterized by high wear resistance, good corrosion resistance, good mechanical properties and high yield strength. It is used for aerospace applications, including aircraft fittings, nuclear reactor components, gears, jet engine parts and aircraft components [1-4]. Due to the difficulty in processing 17-4PH stainless steel, meeting the requirements for surface quality is a demanding task. Ultra-precision machining allows the dimensional accuracy of the workpiece to be very precise and the flatness and roughness of the surface is controlled well. Current precision grinding and polishing processes include grinding, lapping, mechano-chemical polishing and chemo-mechanical polishing to form shapes precisely but there is also processing deterioration and residual stress so electrochemical processing is necessary [5-8].

As-fabricated objects that are produced by additive manufacturing suffer from surface roughness, which is caused by powder particles sticking to the contour of the molten surface during manufacturing. Roughness causes stress concentration and crack initiation so it decreases the fatigue performance [9,10]. Roughness must be decreased using a post process. This study determines the effect of the electropolishing process, which increases the surface quality without direct physical contact.

Electropolishing began at the beginning of 20th century [11-13]. The first studies involving practical applications were by Jacquet, who gained a patent for the process in 1930 [14]. Methanol sometimes replaces water as a solvent [15-16]. Electropolishing is an electrochemical method that is accelerated by the external voltage to level and smooth the sample surface. The components of the electrolytic cell include a cathode, an anode, a stir bar and an electrolyte to allow the reaction [17].

On the anode side, metal ions diffuse into the electrolyte and form metal salts during the electropolishing process. When the concentration of the metal salts reaches saturation, the sample surface forms a metastable film, which has high resistance and viscosity. This surface film inhibits crystallographic etching and causes brightening [18-19]. The mechanism by which the surface film forms is a main advantage of electropolishing process in relation to other post processing techniques because it allows micro surface improvements. However, if the surface film becomes too thick, the diffusion of metal ions is inhibited, which affects the polishing. The surface film must achieve a proper thickness [19-21]. To achieve uniform polishing, the surface film is controlled by the agitation speed, the temperature and the external voltage.

This study uses phosphoric acid and perchloric acid as an electrolyte for the electropolishing system and determines the effect of electropolishing for additive manufactured 17-4 PH stainless steel. The metal surface is smooth after electropolishing. Samples that are fabricated by an additive manufacturing process are characterized before and after the electropolishing process, in order to determine the change in surface morphology. The Electrochemical properties of different electropolishing system are also determined.

2. EXPERIMENTAL

2.1. Materials and electropolish parameters

This study uses additive manufactured 17-4 PH stainless steel. Its elemental content is detailed in a previous study by the authors [22].

Table 1. The electropolishing composition and parameters

Electropolishing solution	Operating voltage (V)	Operation time (min)
10 wt.% H_3PO_4 solution	10	5, 7, 10
10 wt.% $HClO_4$ solution	10	5, 7, 10

This study uses phosphoric acid and perchloric acid for electropolishing and the previous study used sulfuric acid. The electropolishing parameters are shown in Table 1.

2.2. Microstructural characterization

A scanning electron microscope (SEM) of JEOL SCANNING MICROSCOPE (model JSM-IT100)) was used to observe the surface morphology of the specimens. Digital 3D white light interferometry (Chroma 7503) was used to measure the surface roughness of the samples. The micro-hardness of each sample was measured using a Vickers micro-hardness device (HVS-1000), using a load of 100 g for 10 s.

2.3. Electrochemical properties measurements

Electrochemical impedance experiments were performed on a computer-controlled potentiostat (VERSASTAT4, Princeton Applied Research) in 3.5wt. % sodium chloride (NaCl) aqueous solution at 25 °C. A Hg/KCl electrode was used as a reference and a platinum plate served as the auxiliary electrode. The exposed surface area of the specimen was 1.77 cm^2. All measurements were performed at the open circuit potential of the system. The amplitude of the sine wave for this study is 5 mV and the stimulation frequency ranges from 100k Hz to 5M Hz [23-26]. The impedance spectra are fitted by the software using a suitable equivalent electrical circuit (ZSimpWin 3.21).

3. RESULTS AND DISCUSSION

Figure 1 shows the SEM surface morphology of an untreated specimen and various plates that are treated by electropolishing for a short period of time (5 min). The surface of the untreated additive manufactured 17-4 PH stainless steel is covered with spherical particles, as shown in Figs. 1(a) and 1(b), because the additive manufacturing powder cools and is deposited on the surface in the molten state, so the surface is very rough. After electropolishing additive manufactured 17-4 PH stainless steel using phosphoric acid for 5 min, the surface morphology of the spherical particles becomes less and smaller but the surface of the sample is not yet flat and smooth, as shown in Figs. 1(c) and 1(d). When additive manufactured 17-4 PH stainless steel is electropolished using perchloric acid for 5 min, the surface morphology of the spherical particles is also reduced and smaller. The surface of the sample becomes flatter and smoother, as shown in Figs. 1(e) and 1(f).

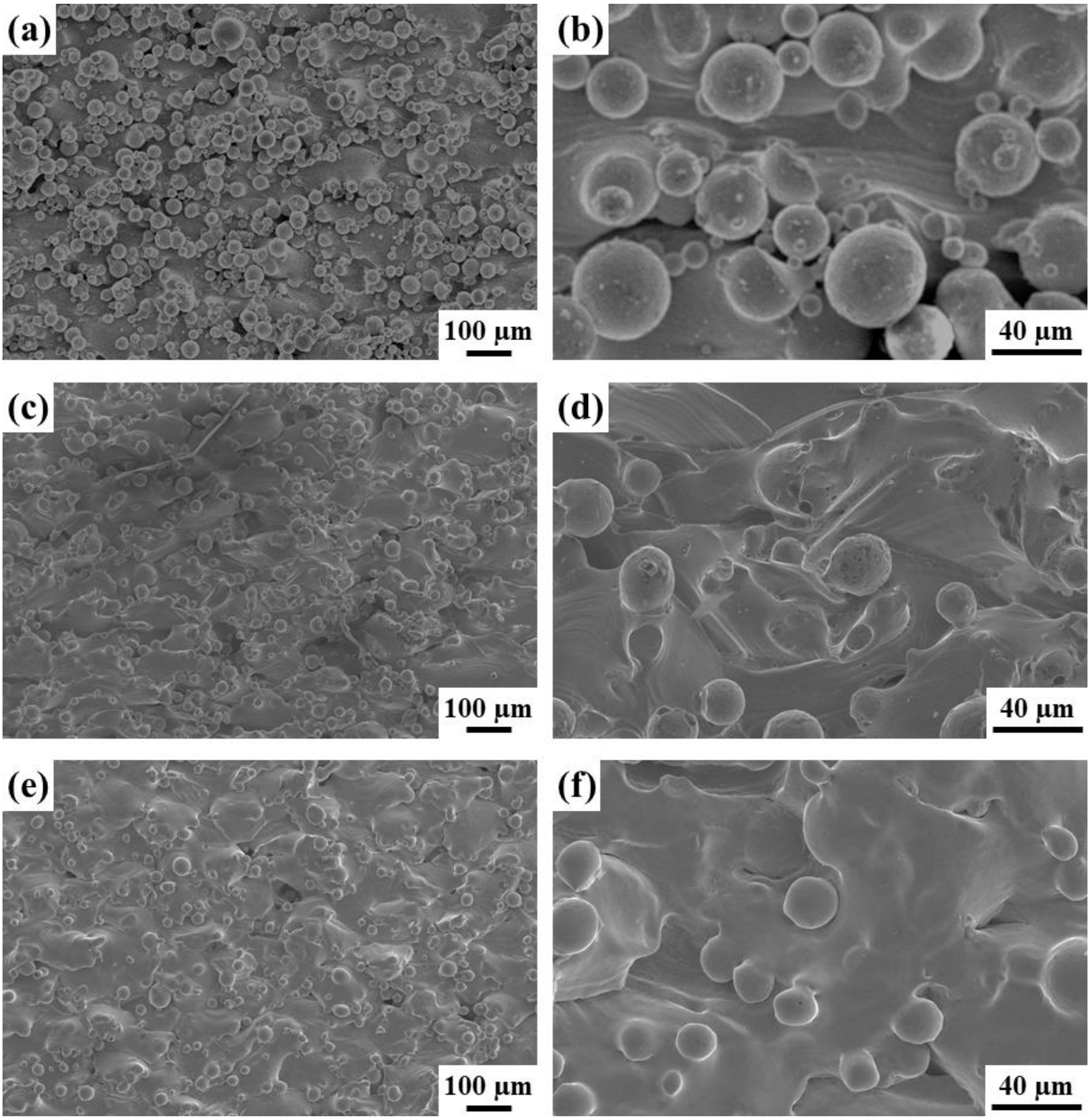

Figure 1. The SEM surface morphology results for (a) untreated and (b) the enlarged part in (a); (c) electropolishing using phosphoric acid for 5 min and (d) the enlarged part in (c); (e) electropolishing using perchloric acid for 5 min and (f) the enlarged part in (e) for additive manufactured 17-4 PH stainless steel

After electropolishing for 7 min and 10 min, the SEM surface morphology is shown in Figure 2. Electropolishing additive manufactured 17-4 PH stainless steel using phosphoric acid for 7 min reduces the number of spherical particles and there is fluctuation in the surface, as shown in Fig. 2(a). If the processing time is increased to 10 min, the surface morphology exhibits no protrusions but there are some pits, as shown in Fig. 2(b). Electropolishing additive manufactured 17-4 PH stainless steel using perchloric acid for 7 min gives a surface morphology that is similar to that for phosphoric acid, as shown in Fig. 2(c). After electropolishing for 10 min, the surface morphology is relatively smooth, as shown in Fig. 2(d).

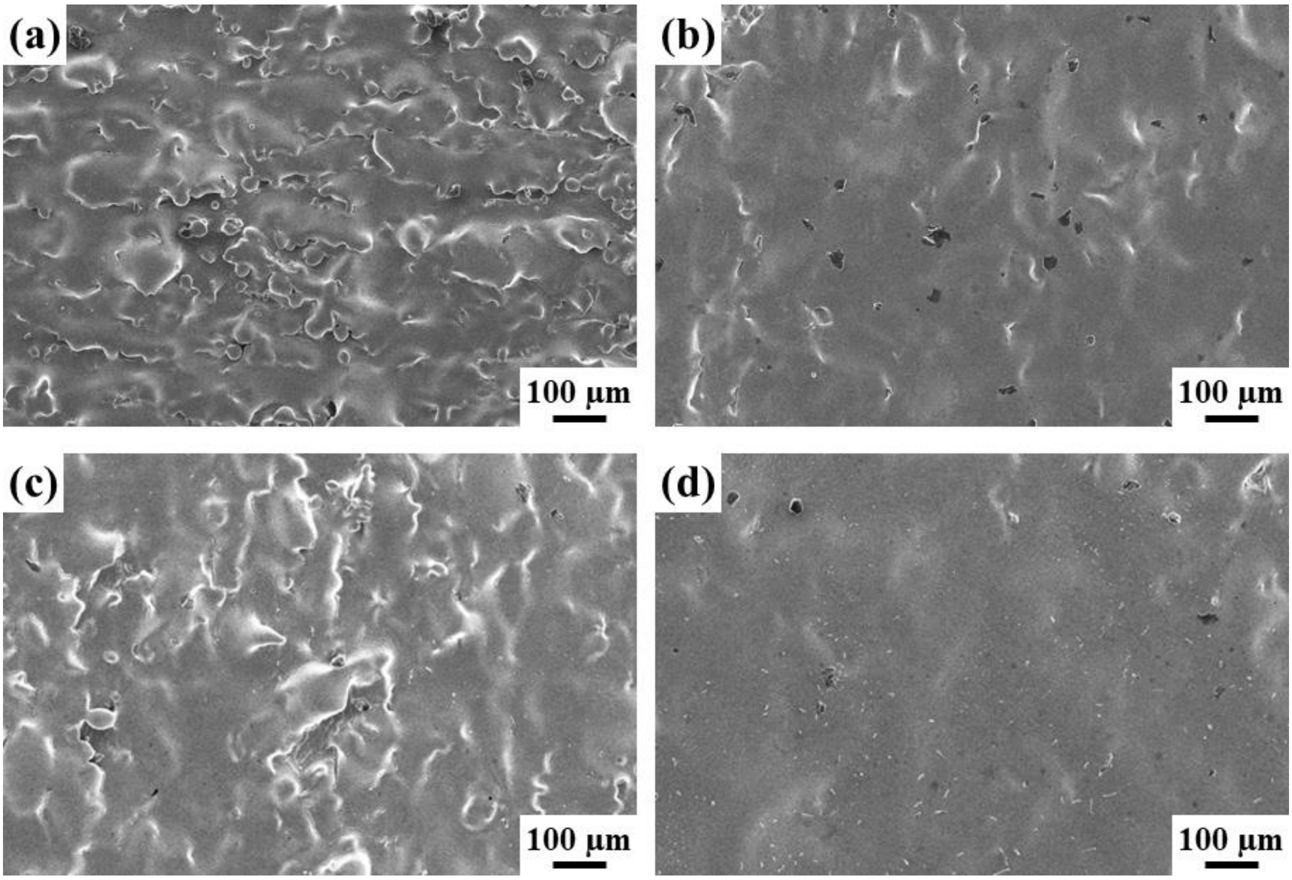

Figure 2. SEM surface morphology results for additive manufactured 17-4 PH stainless steel that is electropolished using phosphoric acid for (a) 7 and (b) 10 min and using perchloric acid for (c) 7 and (d) 10 min

To verify the surface morphology that is determined using SEM, a 3D white light interferometry was used to measure the surface roughness. The surface roughness mapping results for electropolishing of additive manufactured 17-4 PH stainless steel using phosphoric acid for 5, 7 and 10 min are shown in Fig. 3. A previous study [22] showed that the surface roughness for the untreated sample is 11.69 µm. As the duration of electropolishing using phosphoric acid increases, the surface roughness decreases from 7.96 µm (5 min) to 2.91 µm (10 min). As the duration of electropolishing using perchloric acid increases, the surface roughness decreases from 5.96 µm (5 min) to 1.76 µm (10 min). This also shows that the use of perchloric acid as electrolyte gives a more significant reduction in surface roughness than phosphoric acid. This result is consistent with the SEM surface morphology results (Fig. 1 and Fig. 2).

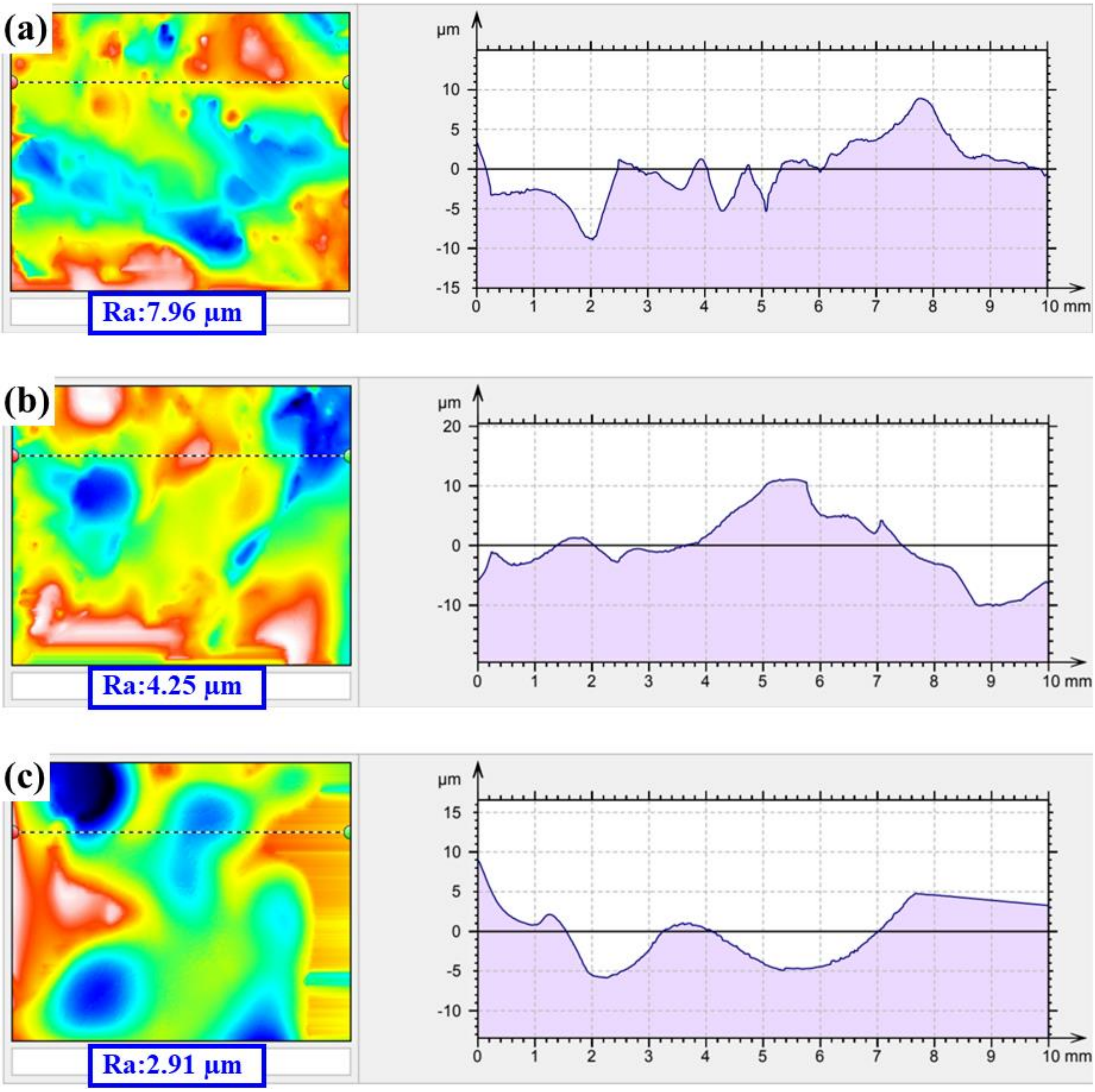

Figure 3. The surface roughness mapping results for additive manufactured 17-4 PH stainless steel that is electropolished using phosphoric acid for (a) 5, (b) 7 and (c) 10 min

Figure 5 shows the hardness of additive manufactured 17-4 PH stainless steel that is mechanically ground with emery paper of 400 to 1200 grit and for different electropolishing treatments. After electropolishing, the hardness of the sample is greater than that of a mechanically grounded sample, as shown in Fig. 5. Improving the surface roughness prevents stress concentration due to material surface defects, reduces mechanical vibration and wear and increases the service life of the workpiece material [5-8].

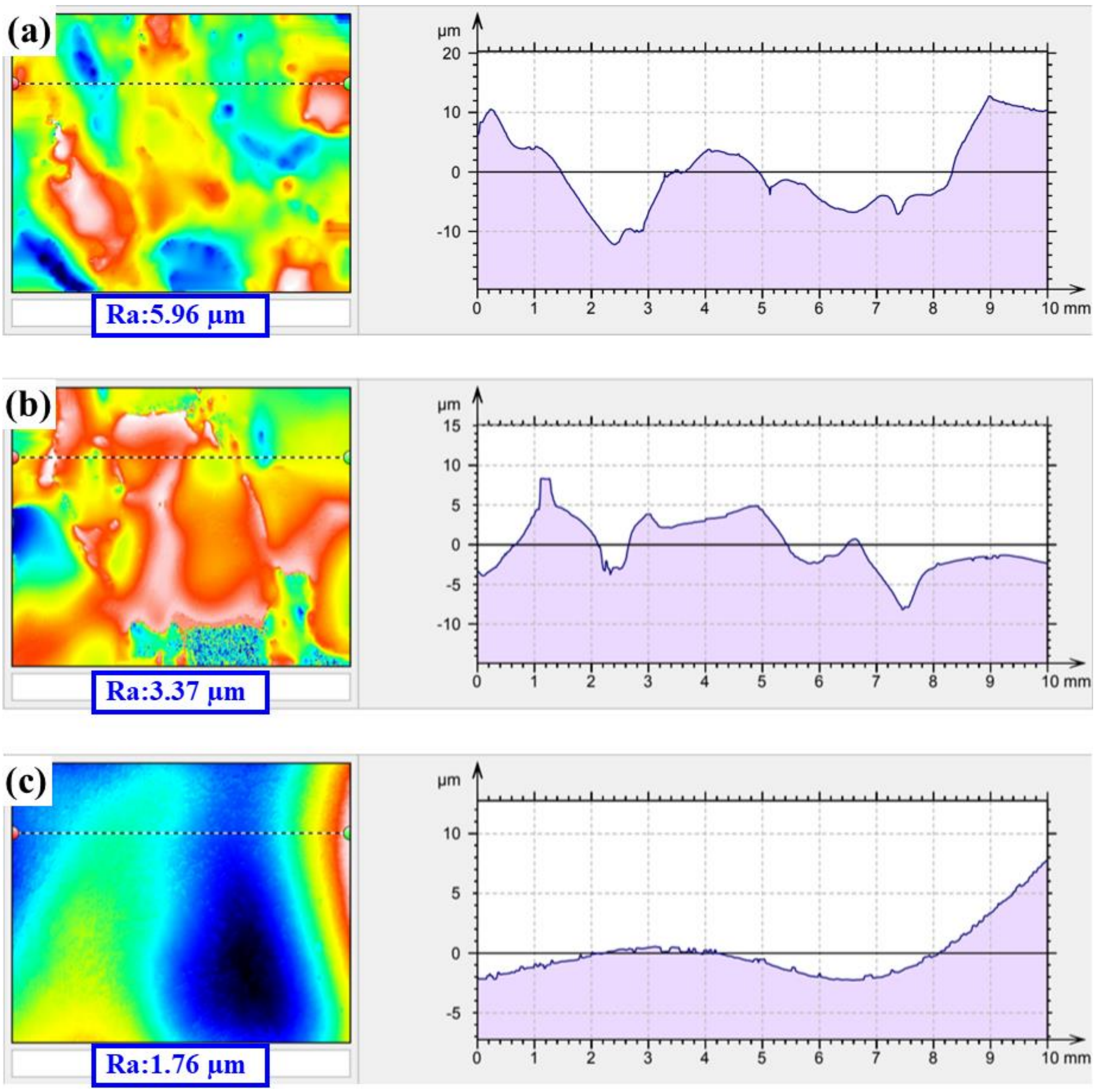

Figure 4. The surface roughness mapping results for electropolishing of additive manufactured 17-4 PH stainless steel using perchloric acid for (a) 5, (b) 7 and (c) 10 min

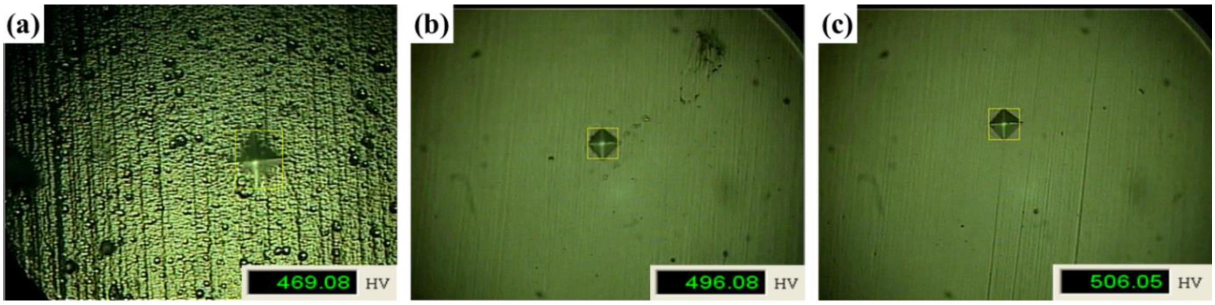

Figure 5. The hardness of additive manufactured 17-4 PH stainless steel that is (a) mechanically ground using emery paper of 400 to 1200 grit and which is electropolished using (b) phosphoric acid and (c) perchloric acid for 10 min

Figure 6 shows the Niquest and Bode result for additive manufactured 17-4 PH stainless steel for an untreated sample and for a sample that is electropolished using phosphoric acid for 5, 7, 10 min in 3.5 wt.% NaCl solution. The impedance of the sample after electropolishing is greater than that of the untreated sample, as shown in Fig. 6As the duration of electropolishing increases, corrosion resistance increases. The absolute impedance ($|Z|_{f=0.01\ Hz}$) at low frequency (0.01 Hz) for electropolishing using phosphoric acid for 10 min is 8 times that of the untreated sample, as shown in Fig. 6(b).

Figure 7 shows the Niquest and Bode results for additive manufactured 17-4 PH stainless steel for an untreated sample and one that is electropolished using perchloric acid for 5, 7, 10 min in 3.5 wt.% NaCl solution. The impedance of the sample after electropolishing is greater than that of the untreated sample, as shown in Fig. 7. As the duration of electropolishing increases, the corrosion resistance increases. The absolute impedance ($|Z|_{f=0.01\ Hz}$) at low frequency (0.01 Hz) for electropolishing using perchloric acid for 10 min is 10 times that of the untreated sample, as shown in Fig. 7(b). Fig. 6 and Fig. 7 show that the results for corrosion resistance for electropolishing using perchloric acid are better than those for phosphoric acid.

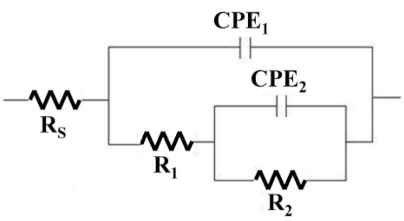

Figure 8. The equivalent circuit models for the simulation of the EIS plots for various additive manufactured 17-4 PH stainless steel samples.

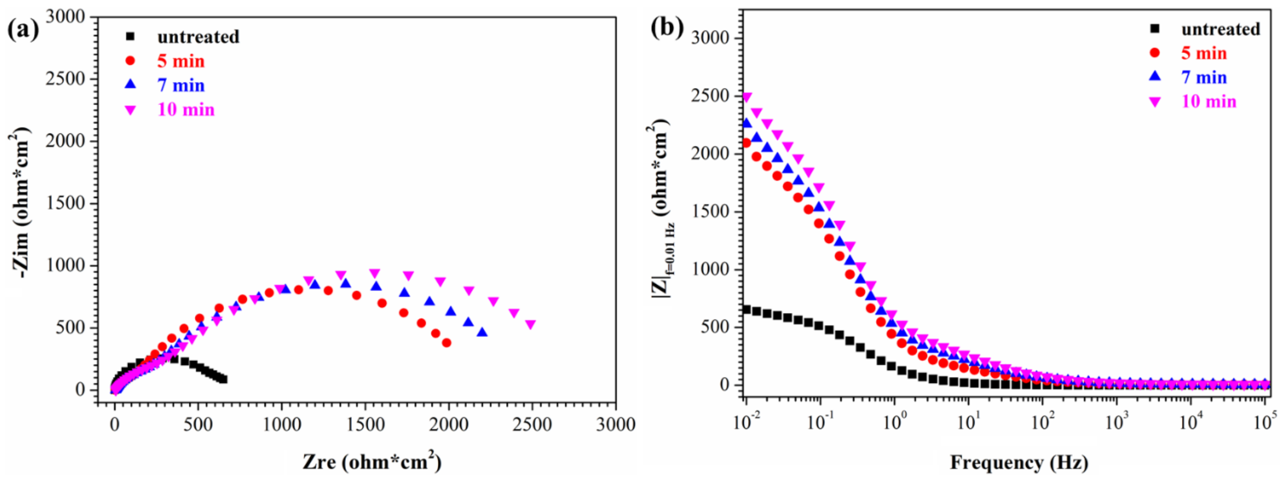

Figure 6. The (a) Niquest and (b) Bode results for additive manufactured 17-4 PH stainless steel that is untreated and which is electropolished using phosphoric acid for 5, 7, 10 min in 3.5 wt.% NaCl solution

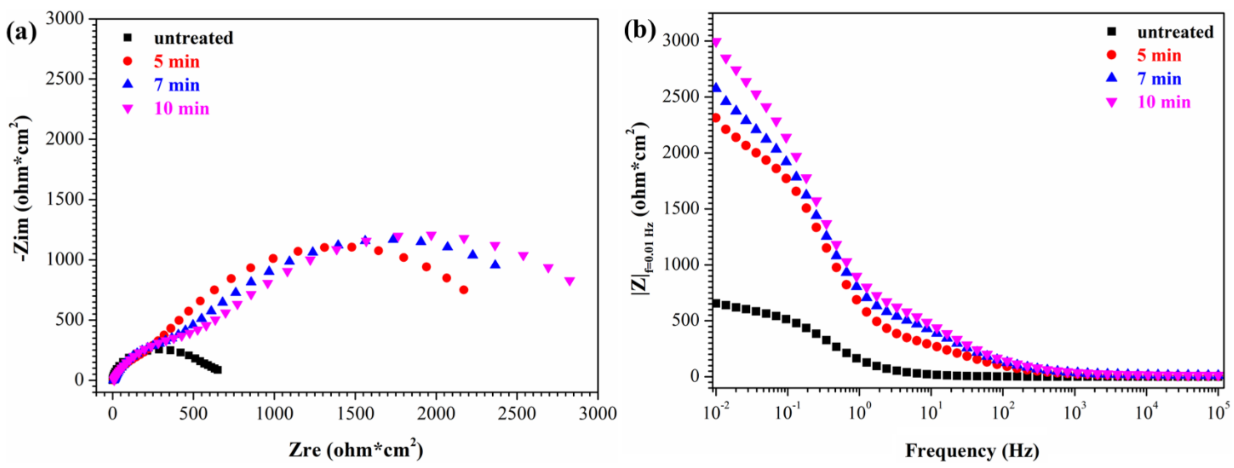

Figure 7. The (a) Niquest and (b) Bode results for of additive manufactured 17-4 PH stainless steel that is untreated and which is electropolished using perchloric acid for 5, 7, 10 min in 3.5 wt.% NaCl solution

The Niquest and Bode data is simulated using the equivalent circuit in Fig. 8. R_s is the solution resistance; R_1 is the passive layer resistance, in parallel with a constant-phase element (Q_1); R_2 is the charge transfer resistance (electron transfer) of the Faradaic process on the metal surface, in parallel with a constant-phase element (Q_2) [23-26]. The simulated values for the electric elements using ZSimpWin 3.21 software are listed in Table 2.

Table 2. The fitted value for the equivalent electric circuit extrapolated from EIS simulations for various additive manufactured 17-4 PH stainless steel plates

	Untreated sample	Phosphoric acid bath			Perchloric acid bath		
		5 min	7 min	10 min	5 min	7 min	10 min
R_s (ohm.cm^2)	13.2	14.4	15.9	16.8	16.3	19.1	15.7
Q_1 (s^n.μohm^{-1}.cm^{-2})	911	238	168	141	102	76	71
Q_1 - n	0.93	0.89	0.89	0.89	0.88	0.89	0.88
R_1 (ohm.cm^2)	563	259	374	446	338	480	650
Q_2 (s^n. μohm^{-1}.cm^{-2})	3,851	432	523	489	429	546	514
Q_2 - n	0.79	0.76	0.73	0.71	0.74	0.71	0.75
R_2 (ohm.cm^2)	150	1,939	2,107	2,391	2,312	2,507	2,675

4. CONCLUSIONS

Electropolishing is shown to decrease roughness on the surface of additive manufactured 17-4 PH stainless steel. The following lists the conclusions for this study:

1. In terms of surface morphology, the surface of the untreated sample is covered with spherical particles, and the height is large, so the surface roughness is poor.
2. The roughness of the additive manufactured 17-4 PH stainless steel sample improves from an initial value of 11.69 to 1.76 μm after electropolishing using perchloric acid for 10 min.
3. Electropolishing using phosphoric acid and perchloric acid system decreases roughness and increases hardness and corrosion resistance.

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