

# ROUGHNESS REDUCTION OF ADDITIVELY MANUFACTURED STEEL BY ELECTROPOLISHING

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## ABSTRACT:

Improving surface finishing is the critical step in the application of an additively manufactured (AM) component in high fatigue and corrosion sensitive fields. This paper provides insights into surface properties of electropolished AM component made up of 316 stainless steel with >6% carbon. We performed surface analysis with profilometer, scanning electron microscope, and AFM to investigate the electropolished and unpolished AM components. The first major discovery of the present work is that our optical profilometry study provided an estimate of the amount of material to be removed to achieve a shiny and smooth surface on 316 stainless steel AM part. One needs to remove more than 200  $\mu\text{m}$  thick layer from the surface to make a mirror-smooth surface. Such estimation can enable researchers to incorporate requisite tolerance during the design stage itself. The second important finding of our work is that ultra-smooth microstructure on the AM surface appeared when preferential material removal occurred along the boundary of the hexagonal microscopic features. Additionally, our optical profilometry studies provided an analysis of several roughness parameters on the electropolished surface. Electropolishing was effective in reducing the surface roughness below  $\sim 0.1 \mu\text{m}$  RMS over microscopic regions. Observation of sub- $\mu\text{m}$  RMS roughness was consistently observed with the optical profilometer, SEM, and AFM. SEM study revealed a significant change in the microstructure of the electropolished samples in a medium and highly smooth state. We also conducted a water contact angle study and spectroscopic reflectance study on electropolished and unpolished AM component surfaces. Our study revealed that electropolishing is a highly promising route for improving the surface finishing of AM components.

**KEY WORDS:** Additive manufacturing; roughness; electropolishing; 3D printing; SEM.

## INTRODUCTION:

Additive Manufacturing (AM) can produce nearly ready to use highly complex engineering components. However, to make such AM parts functional, it is critical to improving the surface finish of the AM components. An AM part with a rough surface will be vulnerable to premature failure during fatigue loading. It has been observed that increasing surface roughness dramatically reduces the fatigue strength of an engineering component [1, 2]. Similarly, higher surface roughness significantly reduces the high-temperature strength or creep strength [3]. Higher surface roughness is also associated with increased susceptibility to corrosion [4]. Also, the integration of the AM part with high surface roughness in a complex engineering system with multiple components can create reliability issues. High surface roughness may prevent intimate contact between the component surfaces and hence lead to loose connections [3] and vibration

generation. In addition to the surface roughness issue, an AM component may have a significantly different surface composition as compared to bulk. Surface finishing is essential to remove the scale from the surface of AM parts to restore the properties of the bulk material on the surface as well.

However, improving surface finish for an AM component can be very challenging based on its intricate design [5, 6]. Popular surface finishing approaches like machining, extrude honing, and sandblasting, may not be applicable for complex AM components [5, 6]. Here we focus on the utilization of electropolishing techniques to improve the surface roughness of metal AM components. Our rationale for choosing electropolishing is based on its flexibility in dealing with complex shapes and geometries. Electropolishing has a unique advantage because it can reduce surface roughness wherever the counter electrode can be placed in the proximity of target surfaces, and electrolyte solution touches the target surface of a metal AM component [5, 6]. Advantageously, an electropolishing solution or electrolyte can easily reach intrusions and hidden surfaces, which may be inaccessible by other surface finishing approaches [5]. During electropolishing, the AM component becomes an anode, and a counter electrode is utilized as a cathode[7]. The anode and cathode are submerged in an acidic electrolyte, and a current is applied. Electropolishing improves surface finish by removing metal from the surface of a piece, ion by ion [7, 8]. During electropolishing, several improvements commence on AM surfaces: (i) Electropolishing eliminates surface burrs from delicate and intricate AM components. (ii) Electropolishing can effectively improve the surface finishing of the AM surface by removing scale, oxides, chemicals, and surface irregularities. Hence, this process can make a steel surface corrosion resistance by eradicating the surface imperfections that serve as corrosion initiation sites and significantly improve fatigue life improvement by eliminating micro-cracks and other surface defects on AM parts. Several recent studies have applied electropolishing on AM components [9-11]. However, there are limited studies on 316 steel with > 6% carbon. Such steel tends to form cementite like the intermetallic phase. This iron carbide phase exhibits different physicochemical properties as compared to other phases present in 316 steel. This 316 steel may be utilized in many critical applications and are likely to benefit from the AM technology. In this study, we have applied electropolishing based surface finishing on 316 steel with high carbon content. The efficacy of the electropolishing is dependent on several factors: temperature, agitation, electrolyte composition, and time. [7, 12]. We previously summarized our process optimization efforts [13].

This paper reports insight we learned after the extensive surface characterizations after the electropolishing process of 316 high carbon steel AM components. We conducted optical profilometry and scanning electron microscopy (SEM) and AFM to quantify the changes in surface roughness and microstructure due to electropolishing.

## **EXPERIMENTAL DETAILS**

The electropolishing experiments were conducted on the 316 steel AM samples using previously reported optimized process [13]. These samples were prepared with EOSINT additive manufacturing machine. The 316 stainless steel metal powder was utilized for this study. The powder particle size was > 50  $\mu\text{m}$ . The typical composition of the finished AM components and the powder was 17-19% chromium, 13-15% nickel, 6-8% carbon, 2-3% molybdenum, trace elements, and balance iron. The AM components were produced by direct laser sintering of ~20  $\mu\text{m}$  thick layers. To investigate the utility of the electropolishing approach for reducing surface roughness and improving surface texture, we focused on four factors: electropolishing time,

temperature, agitation, and electrolyte composition [7, 12]. To limit the number of factors and make our optimization study manageable, we fixed the current density around  $80 \text{ A/dm}^2$ . We also referred to numerous prior studies to determine suitable current density for electropolishing on steel AM samples [6, 7, 12, 14, 15]. We conducted Taguchi design of experiments for finding the optimum levels of electropolishing time, temperature, agitation, and electrolyte composition [13]. We produced 100 mL electrolyte solution in water by adding 85% phosphoric acid and 98% sulfuric acid. Other than water, solution X contained 41 g phosphoric acid and 45 g sulfuric acid, solution Y contained 49 g phosphoric acid and 41 g sulfuric acid, and solution Z contained 15 g phosphoric acid and 63 g sulfuric acid.

Table 1: L9 Taguchi design of experiment for electropolishing [13].

| Tr<br>ial<br># | Time<br>(s) | Tempe<br>rature<br>(°C) | Acid-<br>compo<br>sition | Agitatio<br>n (rpm) |
|----------------|-------------|-------------------------|--------------------------|---------------------|
| 1              | 180         | 82                      | X                        | 0                   |
| 2              | 180         | 93                      | Y                        | 200                 |
| 3              | 180         | 104                     | Z                        | 400                 |
| 4              | 240         | 82                      | X                        | 400                 |
| 5              | 240         | 93                      | Y                        | 0                   |
| 6              | 240         | 104                     | Z                        | 200                 |
| 7              | 300         | 82                      | Z                        | 200                 |
| 8              | 300         | 93                      | X                        | 400                 |
| 9              | 300         | 104                     | Y                        | 0                   |

After conducting the nine trials, we conducted roughness measurement and determined the difference with respect to the roughness of the unpolished sample[13]. Taguchi analysis suggested that temperature was the most influential parameter for electropolishing. Out of 100% influence scale, the temperature parameter accounted for ~40%. The contribution of agitation and electrochemical bath composition were ~32% and ~24%, respectively. The electropolishing time was found to be the least influential [13]. Finally, we utilized Taguchi analysis to yield the optimum combination of levels for four parameters to produce the optimum electropolishing. According to the Taguchi analysis, electropolishing duration 300 seconds, temperature 104 °C, composition X, and agitation at 200 rpm was to yield the greatest improvement in surface smoothing after electropolishing.

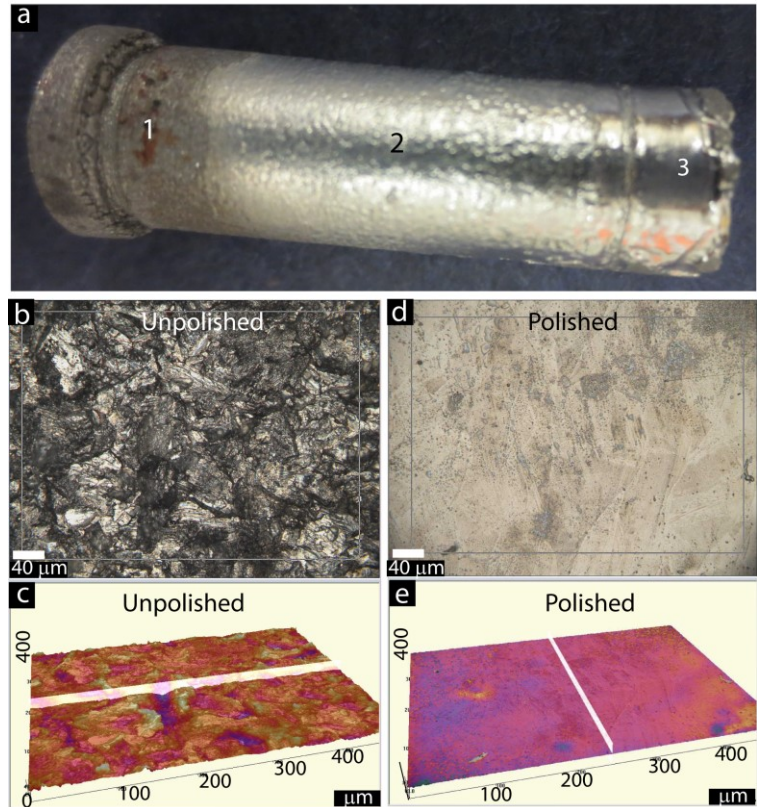
To conduct electropolishing experiments, we utilized a glass beaker with an acidic electrolyte. Heating and agitation were performed by using a hot plate with a magnetic stirrer capability. We utilized Scilogex 86143101 Model MS-H280-Pro hot plate with magnetic stirrer. In the electrolyte bath, an AM sample and counter electrode were submerged. To expose the identical amount of surface area in all the experiments, we conducted lithography on AM

samples. Shipley 1813 photoresist was dip-coated on AM samples in a class 1000 cleanroom environment. Subsequently, the photoresist coated samples were heated on the hot plate at 100 °C for 20 minutes. Prolonged baking at high temperature was accomplished to make photoresist sufficiently hard so that it can withstand acid solution and other experimental conditions. Successively, we opened a  $\sim 0.5 \text{ cm}^2$  window in the photoresist shield by selective chemical dissolution of photoresist. All the prepared samples were subjected to electropolishing. We produced a 100 mL electrolyte solution in water by adding 85% phosphoric acid and 98% sulfuric acid [7, 16]. After each experiment, the photoresist was removed from each AM sample, and roughness measurements were performed on the electropolished and unpolished areas to determine the efficacy of each experiment. The optical profilometry was performed with Zeta 20 and Filmetrics optical profilometers. We studied surface with Phenom XL SEM and NaioFlex AFM.

## RESULTS AND DISCUSSION:

After determining the suitable combination of electropolishing parameters, we attempted surface roughness reduction on cylindrically shaped samples (Fig. 1). We hypothesized that longer duration electropolishing would gradually flatten out high and low sections on the surface of as produced AM samples. Hence, prolonged electropolishing is expected to yield highly smooth morphology.

We observed that a stainless steel cylindrical sample could be electropolished to yield very smooth surface roughness. In this study, we are specifically discussing a sample with a gradual change in surface roughness after electropolishing treatment. The unpolished section is designated as zone 1, the area with medium electropolishing is designated as zone 2, and the lowermost section with maximum electropolishing is designated as zone 3 (Fig. 1a). The prolonged electropolishing with the optimized parameters produced a very smooth surface (Fig. 1a). Optical images of the zone 3 (smoothest electropolished area) and Zone 1 (unpolished rough area) are shown in Fig. 1b, and Fig. 1c. The roughness measurement was performed in the  $\sim 495 \text{ } \mu\text{m} \times 372 \text{ } \mu\text{m}$  area.



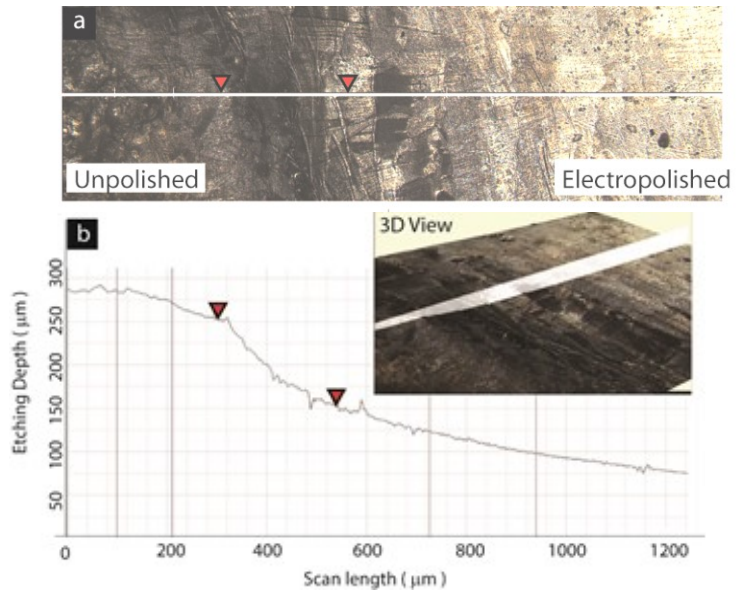
**Figure 1:** Electropolishing of a cylindrical AM component: (a) Cylindrical AM component showing unpolished (1), and electropolished (2, 3) sections. (b) Optical microscope image of the top side and the (c) 3D perspective image in the unpolished area. Optical microscope image of the (d) top side and the (e) 3D perspective image from the electropolished area.

The smoothest electropolished area showed  $\sim 0.091 \mu\text{m Ra}$  (Fig. 1d-e) and unelectropolished area exhibited  $\sim 1.88 \mu\text{m Ra}$ . However, it must be noted that roughness measurement over a broad area of the electropolished section includes many porosities. The  $495 \mu\text{m}$  long line scan on the electropolished surface in the smoothest region yielded a roughness of  $0.07 \mu\text{m Ra}$ . This result signifies that electropolishing can achieve an even more smoother surface finish.

We also investigated the amount of material removal necessary to achieve the highly smooth surface finish. To estimate the required material removal, we determined the difference in height for the electropolished and unpolished area. The depth profiling was accomplished by measuring the sample height along with a scan traversing from unpolished to the electropolished area (Fig. 2a). The two red color arrows mark the area where the sharp change in height was determined. The typical difference was around  $\sim 200 \mu\text{m}$  (Fig 2b). This result is critically important in determining the tolerance required to accommodate electropolishing as the post-processing step during the design phase of AM components. The inset figure shows a cross-section along the scan direction. The left side of the 3D view corresponds to the unpolished surface height. The white area in the 3D view shows that as one moved from unpolished to the polished area, material removal occurred (Fig. 2b).

We have also electropolished the samples with internal volumes. The internal surface was not affected by the electropolishing on the AM samples, where a counter electrode could not be placed in the proximity of the internal surfaces. We also found that electropolishing can be performed on the internal volume of the AM steel components by considering the counter electrode access during the design stage. We prepared cubic AM samples with  $\sim 1 \text{ cm}$  diameter holes to allow the counter electrode to enter into the internal volume easily. Electropolishing effectively reduced the surface roughness of the internal volume. Hence, one can design an AM component by considering the needs of electropolishing in mind. We have discussed the topic of internal surface finishing elsewhere [17, 18].

We measured a wide range of surface roughness parameters after electropolishing. For the quantitative analysis, we determined the maximum peak height ( $S_p$ ), maximum valley depth ( $S_v$ ), the maximum height difference between peak and valley ( $S_z$ ), arithmetic mean height ( $S_a$ ), and root means square (RMS) of height ( $S_q$ ). The surface skewness factor ( $S_{sk}$ ) and surface kurtosis ( $S_{ku}$ ) were also determined. We recorded these roughness parameters in  $0.2 \times 0.17 \text{ mm}^2$  area on eight different locations. The magnitude ( $S_p$ ) was  $166.35 \pm 18.65 \mu\text{m}$  for the unpolished AM surface, which was reduced to

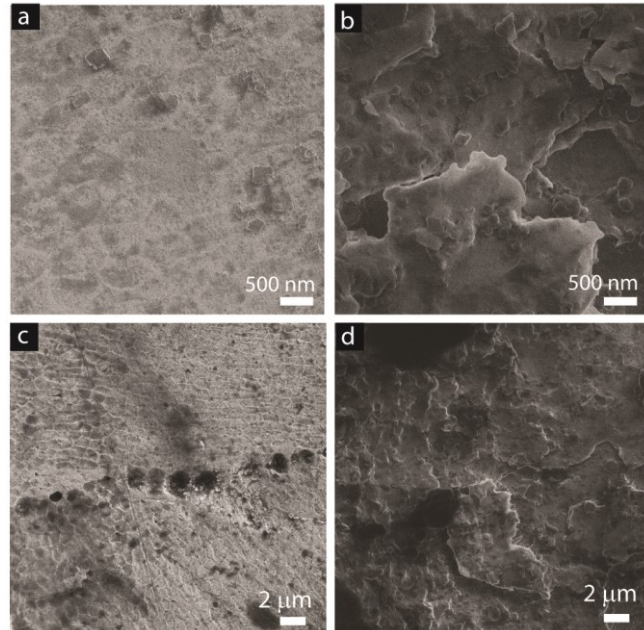


**Figure. 2.** Thickness difference between unpolished and electropolished area. (a) Optical microscope image showing the white color line along which thickness measurement was performed. (b) Etching depth vs. distance profile along the white color scan line on image (a). The inset of (b) shows the difference in height with respect to unpolished section plane.

16.38±6.65  $\mu\text{m}$  for the electropolished AM samples. Similarly, the depth of the valley ( $S_v$ ) was 60.12±20.10  $\mu\text{m}$  for the AM surface before electropolishing. After electropolishing,  $S_v$  reduced to 28.12±8.31  $\mu\text{m}$ . The difference between the height of the tallest peak and deepest valley ( $S_z$ ) for the AM sample before electropolishing was 226.44±17.67  $\mu\text{m}$ . After electropolishing,  $S_z$  reduced to 44.50±13.45  $\mu\text{m}$ . The surface roughness parameter ( $S_a$ ) for the unpolished surface was 13.88±2.65  $\mu\text{m}$ . After electropolishing  $S_a$  reduced to 3.0±0.75  $\mu\text{m}$ . The RMS roughness ( $S_q$ ) for the unpolished AM surface was determined to be 17.37±3.02  $\mu\text{m}$ . After electropolishing  $S_q$  decreased to 3.77±0.85  $\mu\text{m}$ . The surface skewness factor ( $S_{sk}$ ) magnitude was 0.10±0.98 for the unpolished surface, which indicates that the number of hills and valleys are almost in the same proportion to one another. However,  $S_{sk}$  parameter became negative for the electropolished samples indicating the dominance of cavities.  $S_{sk}$  was -0.29±0.85 for the electropolished sample. The surface kurtosis ( $S_{ku}$ ) describes the peakedness of the surface topography. If  $S_{ku}=3$ , then the distribution is ideal Gaussian-like.  $S_{ku}$  was calculated for the whole area and determined to be 2.2 for the unpolished AM sample and 3.4 for the electropolished. This data indicates that an electropolished sample possessed a higher proportional number of valleys.

We also performed scanning electron microscopy (SEM) to investigate the difference in composition and microstructure of the electropolished and unpolished area. The Electron Dispersive Spectra (EDS) study showed that surface composition was entirely different and contained high oxygen content. Interestingly, after the removal of ~ 200  $\mu\text{m}$  thick material from the surface, AM component surface exhibited typical stainless steel composition. The composition of the electropolished area was akin to the composition of stainless steel powder utilized for the AM. It is noteworthy the unpolished surface (Fig. 3a) was gray in color and signified the change in composition. The typical steel surface is shiny and has silver color as shown in the electropolished area (Fig. 1). This result is of critical importance when welding and coating like processes are considered for the AM component. Also, physio-chemical properties of the unpolished surface are entirely different as compared to the electropolished stainless steel surface.

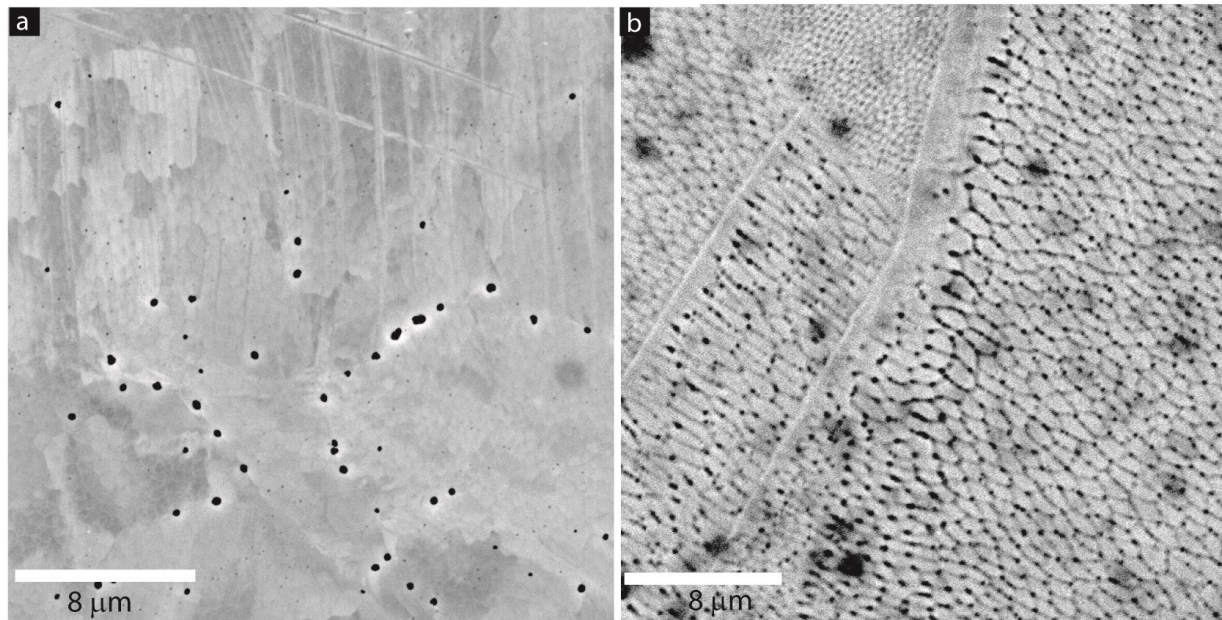
The microstructure of the electropolished and unpolished surface was expected to be quite different. We performed a SEM study and found that at high magnification, the electropolished surface was predominantly featureless (Fig. 3a), as compared to the unpolished area (Fig. 3b). The unpolished section on the additively manufactured cylinder contained the flaky pattern. However, setting the SEM parameters to yield higher contrast revealed various microstructural features (Fig. 3c). On the other hand, the unpolished section at the same magnification still exhibited a flaky pattern (Fig. 3d). Such a flaky pattern is expected to serve as the site of defect



**Figure 3.** SEM images from the zone 1 and zone 3. (a, c) Images from electropolished area at various magnification. (b and d). Images from the unpolished area at various magnifications.

or crack generation during fatigue loading. The wear property of AM components with flakes will also be entirely different as compared to the polished surface.

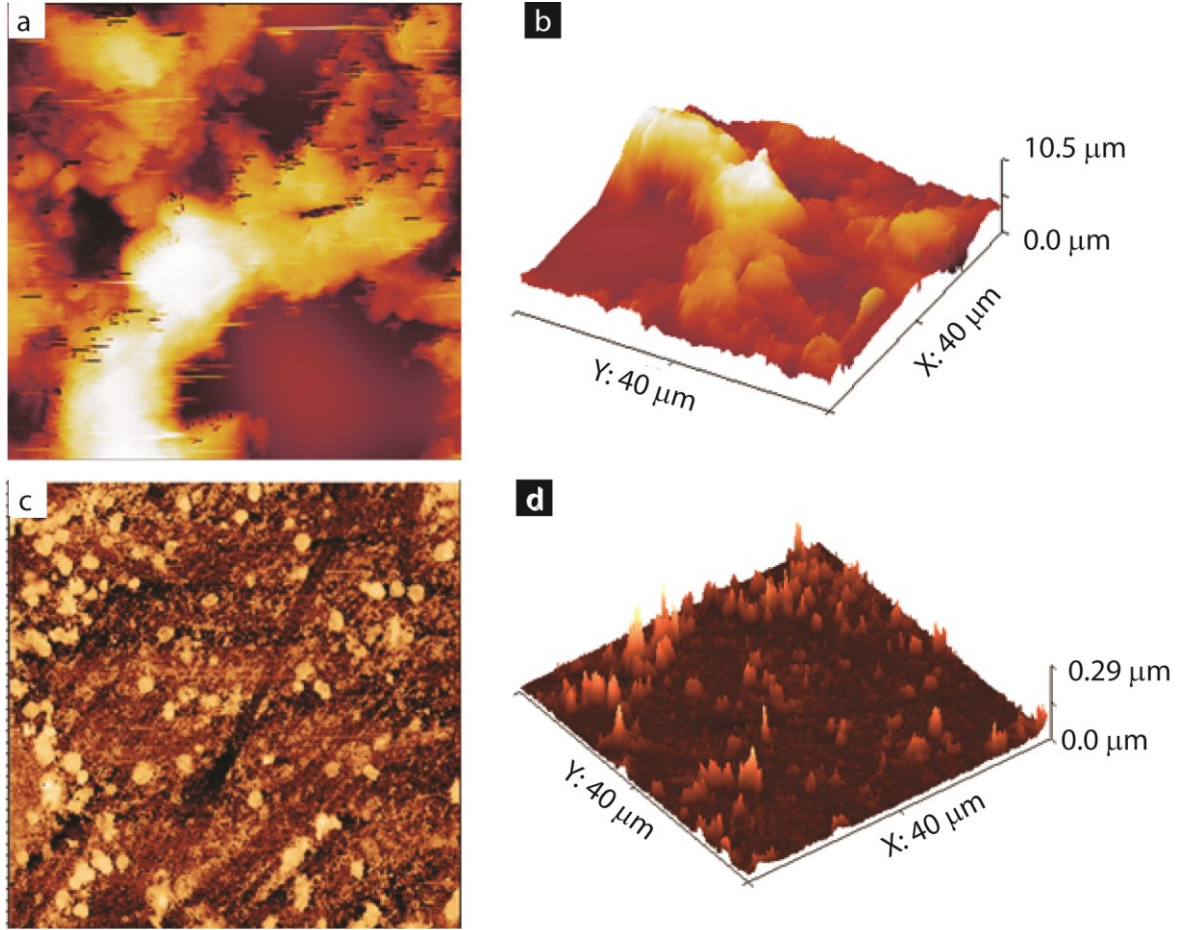
We compared the microstructure of the AM sample after optimal and excessive electropolishing. For this analysis, we utilized SEM's backscattering detector. We found that an optimally electropolished 316 AM sample yielded quite regular microstructure with numerous pits or cavities (Fig. 4a). The overall surface is quite smooth and exhibited  $\sim 0.5 \mu\text{m}$  roughness (Fig. 4a). It is noteworthy that Fig. 4a also contains sub-micron microstructural features, which are faintly observable in Fig. 4a. We also investigated the possibility of etching away the pits or cavities seen in Fig. 4a. For this objective, we conducted electropolishing for an extended period and studied the 316 AM steel sample. The microstructure of the excessively electropolished AM area turned out to be significantly different (Fig. 4b). As seen in the SEM image, the several pits area started to disappear, but many continued to persist (Fig. 4b). This study suggests that pits or cavities observed after electropolishing possessed different depth.



**Figure. 4.** SEM images obtained using backscattering detector from (a) optimally electropolished 316 AM sample and (b) from excessively electropolished AM sample.

Interestingly, the excessively electropolished section showed the presence of near hexagonal microstructural regions (Fig. 4b). The small hexagonal regions contained rather smooth or flat interior regions, but the boundary region of the hexagonal microscopic feature was preferably etched (Fig. 4b). It appears that the boundary region is more susceptible to etching as compared to the interior region. We surmise that at the microscopic region's grain boundary, possessed a cementite phase that is typically found in high carbon 316 steel grade as used in this study. Presumably, the cementite along the grain boundaries etched away faster than the low carbon phase present within the interior of the grain.

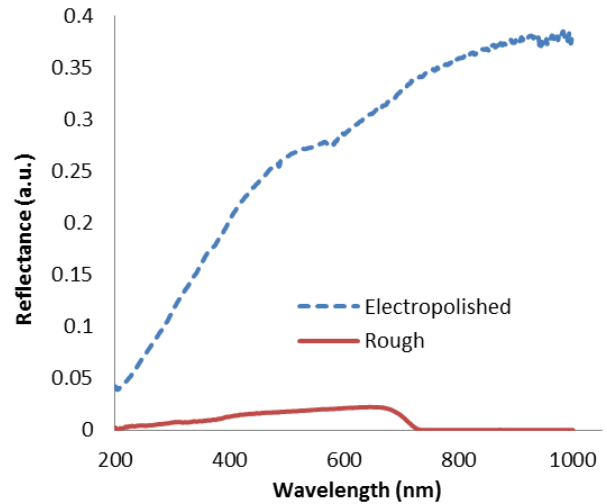
We have also employed AFM to study the impact of electropolishing on AM surface. The top view of the unpolished AM surface was highly uneven (Fig. 5a). The features observed in the AFM micrograph (Fig. 5a) were akin to the pattern observed in the SEM study on the unpolished surface (Fig. 3a). The 3D perspective image (Fig. 5b) shows that before the application of surface finishing treatment unpolished surface contained several  $\mu\text{m}$  hills and valleys. The AFM image of the electropolished sample (Fig. 5c) appeared much different as compared to the



**Figure 5:** AFM study showing abrasive basted AM sample's (a) topography and (b) 3D perspective images. (c) topography and (f) 3D perspective of the electropolished sample. The dimensions of panels in (a), (c) and (e) is  $40 \times 40 \mu\text{m}^2$ .

unpolished sample. However, the cavities of the electropolished samples appeared like small mounts in the AFM image (Fig. 5c). Presumably, it is due to the artifacts produced by the bumping of the AFM cantilever at the cavities. This AFM study suggests that for better estimation, one can perform very slow AFM scans in very small pockets to get zoomed-in views. The 3D perspective view of the electropolished sample shows the feature heights are only varying over a few nm ranges (Fig. 5d). AFM images show that electropolishing was able to bring the roughness level down to the sub- $\mu\text{m}$  range. This information could not be reliably obtained from SEM images.

We performed spectroscopic reflectance to further determine the properties of the top surface of an AM component before and



**Figure 6:** Reflectance characteristics of the electropolished and unpolished (rough) surface of an AM steel component.

after electropolishing. We utilized the Semiconsoft M probe reflectivity meter for this study. The reflectance from the as-produced AM component surface was significantly less than that from the electropolished areas. Moreover, the bare AM sample also was unable to absorb radiation beyond 730 nm wavelength (Fig. 6). This result indicates that the AM surface may absorb radiation. However, the electropolished AM component reflects the radiation back. This property is of critical importance where the AM component is expected to be radiation sensitive. It is noteworthy that the amount of radiation absorbed can vary the temperature of the AM component. Hence design engineers should be cognizant about the characteristics of various surface finishing approaches.

To understand the implication of surface quality on the adhesion properties of different coatings, we studied the difference in the surface chemistry of the unpolished and electropolished AM samples. For the study of surface chemical properties, we utilized water contact angle measurement. In this experiment, ~100  $\mu\text{L}$  water drop was introduced on cleaned and dried AM sample surfaces. The unpolished 316 AM steel sample formed a  $\sim 90^\circ$  angle at the junction of water, AM surface, and air. However, the electropolished AM sample yielded a  $\sim 45^\circ$  contact angle. The smaller contact angle indicates that the electropolished sample became significantly hydrophilic, as compared to the unpolished AM sample. Further study by XPS like sensitive surface measurement may provide more profound insights about the surface chemistry of electropolished samples.

## CONCLUSIONS

In this paper, we demonstrated the utilization of the electropolishing approach to improving the surface finish of as produced 316 high carbon stainless steel metal additive manufacturing components. The results of the Taguchi design of experiments were implemented on cylindrical shaped AM samples. Electropolishing with optimized parameters could drastically improve the surface finish of the AM components. The typical surface roughness decreased below  $\sim 0.1 \mu\text{m Ra}$ . Electropolishing also removed the scale from the AM component surface and brought out the surface with typical stainless-steel composition and bright luster. We performed roughness measurements over microscopic regions with optical profilometry, atomic force microscopy (AFM), and scanning electron microscopy (SEM). Results from three techniques corroborated with each other. We also performed large area roughness with an optical profilometer to provide additional insights. The electropolished surface possessed a significantly smaller defect population on the AM surface. We also found that nearly  $\sim 200 \mu\text{m}$  thick material should be removed during electropolishing to attain a highly smooth surface finish. Scanning electron microscope imaging revealed that as produced AM steel component had flaky morphology, along with pits like features. The microstructure of the electropolished surface was dramatically better than that of as produced AM surface. Electropolishing was effective in improving the surface finishing of the AM component if a counter electrode could be placed in the proximity of the AM component. In future studies, we also plan to study the effect of electropolishing based surface roughness on the fatigue properties of additively manufactured components.

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