Rapid surface preparation for three-dimensional characterization of defect and microstructure of metal additive manufacturing using electrochemical jet

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Highlights
- EJSP for versatile characterization of additively manufactured metal is proposed.
- EJSP can achieve both bulk removal and surface generation with the varying current.
- EJSP provides both microstructure-defined and polished surface topography.
- EJSP spatially reveals defects and microstructures of SLM parts at multiscale.
- EJSP enables three-dimensional reconstruction of the SLM microstructure by slicing.

Abstract
Process-induced volume defects and unsuitable microstructure have inhibited the effective quality assurance of parts manufactured by selective laser melting (SLM). Elucidating the complex interaction between the process, defect/microstructure, and performance is of critical importance. In this work, we have developed and demonstrated the capabilities of an electrochemical jet surface processing (EJSP) method to easily and effectively uncover defects and unveil the crystal microstructure and fusion mechanism in SLM parts of SUS316L and AlSi10Mg respectively. Experiments show that the EJSP method is highly effective in localized three-dimensional microstructural unveiling which eliminates the need for conventional sample preparation by polishing, and internal defects and retained fusion signatures at multiple layers are readily identified within seconds. Furthermore, EJSP unveils three-dimensional structural information at both micro- and nano-scale to facilitate crystallography and phase analysis. This unique approach has high potential to significantly improve qualification methods of SLM parts, which benefits in-depth research of microstructure characteristics and their formation mechanism in the SLM process with high efficiency and low cost.

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1. Introduction

Additive manufacturing (AM), also referred to as 3D printing, is revolutionizing the traditional industrial production concept and...
method by providing an unprecedented new manufacturing solution. With its strong support for design innovation with large geometric freedom and large-scale personalized customization, AM has been developing rapidly and continues to expand its application fields [1]. Laser powder bed fusion (L-PBF) is currently the most widely used technology for metal additive manufacturing. As a typical L-PBF process, selective laser melting (SLM) locally melts a thin metal powder layer and fuses it to the previous layer by scanning high-power-density laser with the guidance of computer-aided design and control. As compared with traditional metal manufacturing technologies such as forging, welding, and machining, it features advantages of no need for tools and molds, high material utilization, and a short production cycle. Further, as an AM process, SLM can manufacture metal parts with complex geometries without the design constraints of traditional manufacturing technologies.

On the other hand, the metal additive manufacturing process involves rapid melting and solidification of powder materials under a high laser energy input. The local temperature of the materials changes drastically due to the quick movement of the laser beam during the AM process, which causes very complex hydrodynamic behavior of material flow and fusion. In addition, while repeated laser scanning the periodic and rapid change of temperature is resulted at previously built materials, leading to complex phase transformation and large internal residual stress. This usually leads to the initiation and propagation of cracks [2]. Therefore, the SLM process inevitably causes the formation of various defects and unwished microstructures, which have become the most critical drawback of this process. Generally, the SLM process-induced internal defects of parts include cracks, voids, porosity, lack of fusion, inclusion defects, inhomogeneity and anisotropy of metal microstructure, and so forth, which significantly reduces the mechanical properties of the product, especially the fatigue resistance and fracture strength [3–5]. The plasticity and toughness of the AM materials are usually lower than that of the wrought ones, and thereby the AM products are more likely to undergo fractures [6]. Further, they also cause negative effects on the corrosion resistance of the built part [7]. Limited quality control of the AM process has been recognized as the critical factor to the deficiency [8]. Therefore, a better understanding of the microscopic behavior of material fusion/solidification and the formation mechanism of microstructures/defects becomes of importance to improve the part quality and process effectiveness.

So far massive research has been undertaken dealing with the characterization and identification of the defect and microstructure of the AM parts. As shown in Fig. 1, these characterizations can be used to establish the accurate correlation between process conditions and final part quality, which is of great significance to establish a simulation and control model of the process. Several non-destructive detection techniques such as ultrasonic detection [9], Archimedes method [10], X-ray micro-computed tomography [11], and laser ultrasonic detection [12] have been under development for identification of the internal defects of the AM parts. On the other hand, destructive characterization methods of microstructure analysis by optical and electron microscopy, etc., still play a key role in providing direct, reliable, detailed, and high-resolution microstructural information for the analysis, evaluation, and control of the AM process. Cutting and mechanical polishing followed by chemical or electrochemical etching of the AM parts has been the general routine to prepare the sample for observation and characterization. However, the tedious sample preparation procedures are considerably complicated and inefficient. While for the difficult-to-cut as-built materials this conventional sample preparation method suffers from further increased technological and time cost. Furthermore, the prepared sample surface is commonly two-dimensional (i.e., planar surface), which only provides limited information of the microstructure of single surface orientation. To show three-dimensional spatial microstructure information, multiple samples with different surface orientations (e.g., along the built, scanning, and tangential directions) should be prepared, which additionally causes low efficiency [13]. Therefore, an advanced sample preparation technique for AM parts characterization which provides comprehensive information of internal structure, and is amenable to the large-area industrial application is in urgent need.

Electrochemical jet machining (EJM) has been demonstrated previously as both a micromachining and surface modification (i.e., texturing and polishing) method featuring no contact, no heat, no alteration of material, and high process flexibility and versatility [14–17]. Especially, based on the surface texturing ability of EJM, Speidel et al. showed the potential of EJM to prepare textured surfaces for rapid characterization and determination of planar crys-
Electrochemical jet surface processing (EJSP) method is proposed as a novel approach to unveil the defects and microstructures of as-built AM parts and to efficiently prepare the polished surface for EBSD characterization. Through applying electrochemical jet on SLM SUS316L and AlSi10Mg, and by using multiscale and selected characterization methods, their internal structure information for the understanding of the material growth mechanism was obtained. Meanwhile, process-induced defects and microstructure were verified by the proposed method to demonstrate its capacity for establishing linkage between process parameters and the internal structure of built parts. Further, since the performance of built parts closely depends on the internal structure, the proposed method in this work is considered a potential way to enable the integrated process-structure-performance design of SLM with high efficiency, as shown in Fig. 1.

2. Experimental procedure

2.1. Sample preparation

The selective laser melting method was utilized to prepare the specimens. Two different types of commercial AM materials, SUS316L and AlSi10Mg, were used in this study. Table 1 shows the SLM conditions used. The laser scanning strategies in the SLM process were illustrated in Fig. 2.

2.2. Electrochemical jet surface processing and procedure

The proposed electrochemical jet surface processing method for AM-built parts is schematically shown in Fig. 3. The EJSP method employs a fine stream of eco-friendly neutral electrolyte as the tool to expose the material inside and generate analysis surface by anodic dissolution realized by the electrical bias between the jet nozzle and the specimen [14]. EJSP features high locality at a micro-

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**Table 1**

Printing conditions of SLM for different materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>SUS316L</th>
<th>AlSi10Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser power [W]</td>
<td>140</td>
<td>350</td>
</tr>
<tr>
<td>Scanning interval [mm]</td>
<td>70</td>
<td>130</td>
</tr>
<tr>
<td>Powder size [μm]</td>
<td>15–53</td>
<td>20–63</td>
</tr>
<tr>
<td>Laser spot diameter [μm]</td>
<td>50</td>
<td>80</td>
</tr>
<tr>
<td>Scanning rate [mm/s]</td>
<td>800</td>
<td>1650</td>
</tr>
<tr>
<td>Layer thickness [μm]</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>SLM equipment</td>
<td>Hans-M-100</td>
<td>SLM Solution 125</td>
</tr>
<tr>
<td>Scan strategy</td>
<td>Bi-directional scanning with a rotation of 67° each layer</td>
<td></td>
</tr>
<tr>
<td>Laser source</td>
<td>CW Yb-YAG Laser, Wavelength: 1064 nm</td>
<td></td>
</tr>
</tbody>
</table>

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Fig. 2. Schematic of laser scanning strategies.

Fig. 3. Schematic of the principle of EJSP for unveiling SLM parts.
scale level owing to the small unveiling area resulting from the micro-scale jet. The processing area is determined by the jet diameter, $d$, and the current density, $J$, which enables selective appraise of discrete areas. Current density ($J$) is concentrated inside the jet and generally exhibits a Gaussian-type distribution with a cylindrical nozzle [19], thus leading to a varied dissolution rate at the unveiling area according to Faraday's law. As a result, a dimple of a hemispherical surface that exposes the internal material three-dimensionally is obtained, which gives a volumetric display of the specimen inside. Thus, built defects can be readily detected by EJSP. Meanwhile, the microscopic etch-rate can be selective, anisotropic, and crystallography-dependent, thus enabling EJSP to generate complex but characteristic nanoscale topography of the built material. Therefore, both macro-scale material removal and nano-scale crystalline topography can be simultaneously generated by EJSP. Owing to the bulk removal characteristics, EJSP can be directly applied to rough surfaces. As shown in Fig. 4, the generated surface can be directly characterized by microscopies, and traditional pre-treatment of the specimen such as cutting, mechanical polishing, and etching, as mentioned previously, becomes unnecessary. Furthermore, as the jet diameter is restricted to less than several hundred $\mu$m, a high current density can be readily generated, leading to ultra-high efficiency of surface generation for analysis, which makes it attractive from an industrial perspective. Large area and selective surface unveiling can also be realized by translating the jet over the specimen arbitrarily, which is particularly amenable to industrial application.

The EJSP experiments were performed using a lab-developed set-up, as schematically shown in Fig. 5. An electrochemical jet with a diameter of 0.31 mm is generated from a nozzle (Stainless steel 304) with a pressured pump and fluid passage. NaNO$_3$ aqueous solution of 20 wt% is used as the electrolyte. Constant current is supplied between the nozzle and workpiece (i.e., specimen) to enable the electrolytic reaction, and the current density is varied to change the materials removal rate and surface characteristics. For large-area unveiling, the electrochemical jet is translated along a preset path by a positioning stage. Unless specified, all the experiments were carried out under conditions shown in Table 2. The
Fig. 7. Material-dependent characteristic dimple topographies and defects of SLM AlSi10Mg unveiled by EJSP. (current density \( J \): 200 A/cm\(^2\), processing time \( t \): 4.5 s).

Table 3
EDX analysis results of element composition of different locations in Fig. 7(f).

<table>
<thead>
<tr>
<th>Location</th>
<th>Al (wt.%)</th>
<th>O (wt.%)</th>
<th>Si (wt.%)</th>
<th>Mg (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>48.83</td>
<td>42.86</td>
<td>6.97</td>
<td>1.35</td>
</tr>
<tr>
<td>2</td>
<td>50.27</td>
<td>43.87</td>
<td>3.46</td>
<td>2.40</td>
</tr>
<tr>
<td>3</td>
<td>54.27</td>
<td>41.14</td>
<td>1.55</td>
<td>3.05</td>
</tr>
<tr>
<td>4</td>
<td>53.10</td>
<td>38.85</td>
<td>4.79</td>
<td>3.26</td>
</tr>
<tr>
<td>5</td>
<td>86.00</td>
<td>5.01</td>
<td>7.86</td>
<td>1.13</td>
</tr>
</tbody>
</table>
obtained surface of the specimen is subsequently ultrasonically cleaned in acetone, ethanol, and deionized water and characterized without further treatment.

2.3. Characterization methods

The surface topography of the specimen was analyzed by scanning electron microscopy (SEM, Zeiss Merlin) and atomic force microscopy (AFM, Bruker Dimension Edge). An electron backscatter diffraction (EBSD) detector (EDAX, Digiview 4) equipped in the SEM was used to unveil the crystallographic orientations of the specimen. Energy Dispersive X-ray Spectroscopy (EDX, Oxford INCA X-stream-2) was used to identify the elemental composition of the specimen. The surface profile obtained by EJSP was measured using a surface profiler (SURFCOM NEX 031 DX-12) and a laser scanning confocal Microscope (KEYENCE VK-X1000). The sur-

![Fig. 8. Characteristic dimple topographies and defects of SLM SUS316L unveiled by EJSP. Both micro- and nano-scale microstructures, such as micro defects, melting tracks, grains, and substructures inside grain can be observed at different orientations under high magnifications. (current density $J$: 200 A/cm$^2$, processing time $t$: 4.5 s).](image)
face profiler and a white light interferometer (Taylor Hobson CCI HD) were employed to measure the surface roughness of the resulted surface by electrochemical jet.

3. Results and discussion

3.1. EJSP unveils defects and characteristic microstructural features three-dimensionally

According to Faraday’s law, the material dissolution rate is proportional to the current density. Therefore, for a stationary jet, a hemispherical dimple is resulted on the specimen due to the bulk materials removal that corresponds to the Gassuan-type current density distribution. As shown in Fig. 6, it is demonstrated that a strong dependence of the generated surface topography on the material. Additionally, owing to the three-dimensional hemispherical surface of the generated dimple, cross-sections perpendicular to x, y, and z axes (as shown in Fig. 3) of the SLM parts are exposed. Further, different topography can be confirmed at the dimple base and dimple sidewall for the same material due to the differed etching behavior under different current densities, which will be discussed in detail in Section 3.2. This is considered as an advantage of EJSP to unveil the material microstructures more comprehensively at the multi-scale. In both cases, especially in AlSi10Mg, the SLM process signature of melting track, and the rotation angle of laser scanning between each layer can be vividly confirmed. Moreover, as described later, the grain-dependent topography such as grains, cells, grain boundaries in different melting tracks can be observed at high magnifications, indicating the anisotropy and etch-rate selectivity of EJSP of the AM parts. This can be attributed to several mechanisms, including element-selective etching considering different standard oxidation potentials of the elements, and preferential grain-boundary corrosion, etc. The following shows detailed discussions on the two specimens, respectively, to better understand the characteristics of the EJSP-generated surface.

In AlSi10Mg alloy, both the top view and cross-section of the melting track can be observed on the base and sidewall surface of the EJSP-generated dimple, respectively (Fig. 7(a) and (b)). Further, the observed melting tracks either align parallelly or cross each other with an intersection angle of 67°, corresponding to the rotation angle of laser scanning. At higher magnification, the etched surface shows a characteristic nanoporous microstructure with numerous nanoscale holes in it as a result of selective etching (Fig. 7(c) and (d)). Pitting corrosion occurs at the boundary area where the current density is minimum (Fig. 7(e)). The inside of the pitting spot is also composed of a large number of nanoholes, implying the characteristic etching behavior of AlSi10Mg. According to previous research, the walls of nanoholes are considered as the etching-resistant eutectic Al-Si network [20]. On the other hand, various built defects of the material, including oxides formed by the combination of Al with residual oxygen (evidenced by the corresponding EDX data presented in Table 3), porosity defect, and lack of fusion, are readily uncovered at the EJSP-generated surface, as evidenced in Fig. 7(f)-(h). This demonstrates the effectiveness of EJSP for detecting defects in AM parts.

Fig. 8(a) shows the surface topography of the dimple generated in SLM SUS316L sample by EJSP. Similarly, a large difference in the surface features between the jet center and marginal area can be told due to the gradient of current density. On the other hand, the material removal behavior shows a significant difference in comparison to AlSi10Mg. Specifically, the resultant surface topography of SUS316L at the bottom of dimple is much smoother and...
the unveiled fusion boundary and the molten pool is less obvious, indicating an electrochemical polishing effect at the jet center under high current densities (Fig. 8(c), (e) and (g)). Meanwhile, the varied etching behavior at the dimple sidewall caused by the low current density is more obvious in SUS316L. For example, fusion boundaries, melting tracks, grains, and numerous cellular columnar structures with different growth orientations, which are primarily determined by the heat gradient during solidification, are easily confirmed at the dimple sidewall (Fig. 8(b), (d) and (f)). Especially at the dimple boundary, distinct fine columnar cells with a diameter of 300 nm and a length of several microns can be vividly observed, (see Fig. 8(h)), showing the crystal growth orientation during the printing process [13]. However, this grain information can hardly be obtained in the case of AlSi10Mg, indicating their material-dependent etching behaviors in the NaNO3 solution. According to the AFM analysis shown in Fig. 9, the surface at the dimple base of SUS316L is composed of numerous parallel-aligned porous nanostructures with a surface roughness Ra = 16–20 nm. The shapes of nanostructures are either lamellar or near round with submicron dimensions, as in the figures, which is considered the residual of the columnar cell structures with different growth orientations after the electrochemical polishing. On the
Fig. 11. Surface-generation mechanism and characteristics of EJSP. (a) The mechanism of microstructure-dominated dissolution and (b) electrochemical polishing effect during the electrochemical jet processing at different current densities. Change of current density cause variation of diffusion layer thickness with different ion concentrations, which determines the etching kinetics. (c) Schematic illustration of the different dissolution behaviors of EJSP at different regions in the unveiling. (d) Different surface responses resulting from the EJSP method for different characterization purposes.

Fig. 12. EBSD analysis based on the obtained surface by EJSP. (a-b), (d-e) and (g-h) show the SEM images of the EJSP-generated dimple in SLM SUS316L, rolled SUS316L, and SLM AlSi10Mg respectively. The rolled SUS316L is included here for comparison purposes. The insets in (a), (d), and (g) show the surface topography of the dimple base obtained by a white light interferometer. (c), (f) and (i) show the obtained EBSD results corresponding to (b), (e), and (h). (Jet diameter: 1.43 mm, current density $J$: 200 A/cm$^2$, processing time $t$: 1.5 s).
other hand, similar to AlSi10Mg, the built defects in the material can also be confirmed (Fig. 8(b)), demonstrating the versatility of the EJSP method for defect uncovered.

3.2. Modulation of surface topography by current density for different characterization purposes

As an electrochemical method, the dissolution profile and surface topography of EJSP are determined by the current density. Thus, the unveiling depth can be readily adjusted by controlling the current density. In Fig. 10, the dissolution depth shows a linear increase with rising the current density. Meanwhile, as can be seen in the figures, the current density also exerts a significant influence on the resultant surface topography. At lower current densities between 20 and 50 A/cm² (Fig. 10(a) and (b)), the material microstructures such as melting track and fusion boundary are of high amplitudes and can be vividly identified, indicating that the material microstructures dominate the EJSP-generated surface topography. This is rather beneficial for the characterization of the microscopic building process of AM. On the contrary, the surface is smoothened and the amplitude of surface microstructures is significantly reduced at excessively high current densities (≥100 A/cm², Fig. 10(c) and (d)), especially at the jet center, indicating an electrochemical polishing effect. This can be attributed to a thick diffusion layer with high ion concentrations formed at high current densities, which makes etching kinetics dominated by the mass transfer and thus eliminates the effect of different microstructures with distinct electrochemical properties, as shown in Fig. 11(a) and (b) [18,21,22]. As a result, the melting track becomes less obvious due to even dissolution on the generated surface. Nevertheless, grain-dependent textures of small amplitudes resulted from crystallographic etching mechanism can still be observed at higher magnifications, as shown in Fig. 10(g) and (h). Further, this kind of surface can be directly used for EBSD analysis due to its relatively low roughness. As shown in Fig. 12(a) and (b), the base of the resulted dimple in SLM SUS316L obtained by EJSP

Fig. 13. Selective unveiling of large area by translating jet over the (a) SLM AlSi10Mg and (b) SUS316L specimen. (current density J: 200 A/cm², translation speed v: 0.1 mm/s).

Fig. 14. Surfaces at different depths can be readily unveiled by varying the jet translation speed. These micrographs at each depth can be used to reconstruct the three-dimensional microstructure of the AM metal. (material: SLM AlSi10Mg, current density J: 200 A/cm²).
exhibits rather low roughness of $S_a = 5.85$ nm, and an EBSD image of the as-machined surface of dimple base is readily obtained where the grain size, shape, and crystal orientation can be observed. For comparison, the EJSP result of the rolled SUS316L plate is also included in the figure, from which the influence of the manufacturing process on the resulted metallographic structure can be easily told. While regular polygonal grains and linear grain boundaries are observed in rolled SUS316L specimen (Fig. 12(f)), the SLM SUS316L specimen exhibits various elongated grains with irregular grain boundaries due to the large temperature gradient during the build process (Fig. 12(c)). Further, Fig. 12(g) and (h) show the resulted dimple surface in SLM AlSi10Mg obtained by EJSP which is directly subjected to EBSD analysis. The resulted dimple of SLM AlSi10Mg exhibits a rougher surface ($S_a = 1.12 \mu m$) than SLM SUS316L because of the material-dependent etching behaviors. Nevertheless, the EBSD result can still be acquired, as shown in Fig. 12(i). This is an advantage of EJSP because the traditional EBSD sample preparation procedures, such as mechanical and/or electropolishing, can be eliminated. Based on the above discussion, the surface-generation mechanism and characteristics of EJSP of SLM metal are schematically shown in Fig. 11(c) and (d). According to the results, the applied current density should be properly selected during EJSP, and an optimum current density exists when attempting to unveil the material microstructures from different aspects and scales.

3.3. Selective unveiling of large area by scanning

Large-area unveiling was carried out with jet translation. By controlling the trajectory of the jet translation, selective analysis of specified surface areas can be achieved. Fig. 13 shows the generated characteristic topography on two different materials by single-path unidirectional linear scanning. Despite large differences in the surface topography defined by material microstructures, the characteristic features resulted from SLM including molten pool boundary, fusion boundary, porosity defect, inclusions, etc., are readily unveiled. Taking AlSi10Mg for example, porosity defect with a size of several microns and the inclusions which contain unmelted powers or other inclusions can be observed in Fig. 13(a).

The unveiling depth of EJSP can be easily controlled by controlling the total charge supplied via changes in scanning speed. As shown in Fig. 14, different jet translation speeds were used to machine grooves of different depths, leading to the disclosure of internal material at different build depths. The material microstructures can be unveiled even at a very small dissolution depth of 7 \mu m (Fig. 14(d)). We must note that the dissolution degrees of the melting tracks at the same location (the dotted line of Fig. 14(a)) differ considerably although the current density is the same, indicating their inhomogeneous etching resistance. Further, owing to the Gaussian-type distribution of current density inside the jet, multiple fusion layers of the SLM specimen, as shown in Fig. 14(a), can be uncovered by increasing the dissolution depth. The stepped profile observed in the inset of Fig. 14(a) indicates the different layers of laser track unveiled by EJSP, which makes EJSP a unique and advantageous method for unveiling multilayer volumetric information in the bulk.

Fig. 15 shows another example of the obtained surface of scanning EJSP of SLM SUS316L sample. The cross-stacking of melting tracks with an intersection angle of 67° can be evidently seen (Fig. 15(c)). The microstructure of each track shows that the molten pool is composed of cellular columnar structures of different lengths. A comparison of scanning and stationary EJSP indicates the former can provide further information on the growth orientations and corrosion behavior of cellular columnar structures. This is because, as illustrated in Fig. 11(c), the low current density region will finally pass the process area during the scanning process. Thus the polished surface at the jet center is roughened again by selective etching under the low current density, leading to a unveil of microstructure-defined surface topography. Furthermore, as shown in Fig. 15(d), the cellular columnar structures show a nano-column shape and are arranged in an orderly manner in a local area. The surface of
Fig. 16. (a)-(f) Observation of the dissolution process of SLM AlSi10Mg sample by varying the jet scanning times, showing the evolution process of surface topography. (g) Schematic illustration of the reconstruction of the three-dimensional microstructure of SLM AlSi10Mg unveiled by three scans of EJSP. (current density $J$: 200 A/cm$^2$, translation speed $v$: 2 mm/s).
the nano-pillars, with an average diameter of about 300 nm, shows a porous structure. The obtained information can be used to understand the microscopic characteristics of the build material and provide guidance for adjusting the SLM process parameters to achieve desired microstructures.

3.4. Synchronous slicing and crystallographic texturing for three-dimensional reconstruction of microstructure by multiple scans of EJSP

To design and develop a high-performance SLM part, a thorough understanding of the microstructure, especially its profile that is practically three-dimensional, is necessary. As revealed in our study, the crystallographic surface with different orientations (i.e., three-dimensional hemispherical surface) unveiled by EJSP offers the hint for the three-dimensional profile of molten pool and grain. However, the observed surface crystallography with a certain depth in the as-built parts still has somehow limits in precisely reflecting the real three-dimensional profile of them. Herein, to further grasp the precise profile information of the microstructure in as-built parts, EJSP with multiple scans was applied to reveal the microstructure information at different depths. The evolution process of the resulted surface topography during multiple EJSP scans was investigated via in-situ observations. To realize this, we firstly selected a site of interest and marked it on the specimen, and then the marked area is subjected to EJSP scans. The change in the surface topography of the observation spot was observed by SEM after each scan. As shown in Fig. 16(a) and (b), even a one-time scan can unveil the appearance of intermittent melting tracks and grains. With the increase of scan time (Fig. 16(c)-(f)), the crystallography with different depths can be unveiled due to the combined ability of bulk materials removal and surface texturing of EJSP. Further, with equidistantly increasing the unveiling depths, the observed two-dimensional profiles of molten pool and grains change accordingly, which indicates the in-depth information of these microstructures like their profile changes along the built direction and spatial distribution. More importantly, theoretically, with these observed two-dimensional micrographs with equal depth interval, the real three-dimensional profile of these microstructures can be reconstructed, which will benefit further microscopic investigation of the as-built parts, such as microstructure-based simulation. Fig. 16(g) shows a preliminary example of reconstruction of microstructure with the unveiled crystallography by EJSP. Because of its rapid and simultaneous slicing and crystallographic texturing ability, EJSP exhibits high potential to be applied as an effective crystallography preparation method for the three-dimensional reconstruction of microstructure in AM parts.

4. Conclusions

An electrochemical jet surface processing method that allows localized rapid surface preparation for characterization of the built defects and microstructural and crystallographic features of SLM parts with the minimum procedure was presented and demonstrated. The microstructural information of SLM parts can be readily elucidated by EJSP which creates complex but characteristic topographies on the unveiling specimen surface. Our key findings are summarized as follows:

I. The internal microstructures of as-built SLM AlSi10Mg and SUS316L specimens can be unveiled by EJSP with no need for surface pretreatment such as complicated polishing procedures. The important features for SLM process, such as fusion interface, molten pool boundary, and so forth, can be directly revealed. Further, defects, such as porosity defect, lack of fusion, etc., and solidification structure, such as cellular columnar crystal structure, and columnar dendrites are readily uncovered by the EJSP method.

II. EJSP is capable of simultaneously creating macro- and micro-structures on the specimen surface by bulk material removal and nano-scale crystallographic corrosion phenomena, respectively, taking advantage of the varying current density distribution inside the jet. Thus, it enables multi-scale detection of the micro-sized built defects and characterization of the nano-sized crystallographical microstructure of SLM parts at the same time, which are commonly difficult to realize with traditional methods.

III. Featuring a gradient current density in the unveiling area, EJSP exhibits gradient etching to show the difference in corrosion behavior of the material under different current densities, enabling comprehensive unveil of the material inside at the multi-scale level. For example, the substructure formed in solidification, i.e., the cellular structure, can be identified by the crystallography-defined etching anisotropy at low current densities.

IV. Owing to volumetric material removal, EJSP is capable of showing three-dimensional information of material inside. For example, the microstructures of the SLM part both in the deposition plane and perpendicular to the plane can be shown at the same time during the unveiling.

V. Combining its abilities of volumetric material removal and surface texturing, EJSP can precisely remove the materials layer-by-layer and synchronously unveil the crystallographic texture of exposed surface of SLM parts by multiple scans. With this unique merit of EJSP, three-dimensional reconstruction of the microstructure of SLM parts can be easily realized with multiple two-dimensional crystallography with the determined depth interval.

To summarize, EJSP provides an efficient and effective approach through which process-dependent microstructural defects and crystalline topographies can be rapidly generated using an electrolyte jet. Without altering the material, the EJSP method unveils three-dimensional multi-scale material information for in-depth analyzing the formation behavior and mechanism of defects and microstructures in SLM process. Meanwhile, EJSP features low cost, high efficiency, and high locality thus enables large-area analysis of AM parts at high efficiency, which makes it amenable to large area industrial applications. It is expected the inspection technique can help to establish a linkage model between process parameters and microstructural features in SLM production, which is of great significance to build high-quality metal parts.

CRediT authorship contribution statement

Jiajun Lu: Conceptualization, Methodology, Validation, Investigation, Data curation, Visualization, Writing – original draft. Weidong Liu: Methodology, Writing – original draft. Xiaogang Hu: Resources. Shuai Wang: Validation, Writing – original draft. Guoping Tang: Resources. Yonghua Zhao: Conceptualization, Methodology, Resources, Formal analysis, Funding acquisition, Supervision, Project administration, Writing – original draft, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
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Data availability

The data used to support the findings of this study are available from the corresponding author upon request.

References