Experimental and numerical investigation of 17–4PH stainless steel fabricated by laser powder bed fusion and hot isostatic pressing

Jaehoon Bae1,2,4, Min-kyeom Kim1,4, Eunyoung Oh1, Kyung-Tae Yang3,1 and Jonghwan Suhr1

1 Department of Mechanical Engineering, Sungkyunkwan University, 2066, Seobu-ro, Jangan-gu, Gyeonggi-do, Republic of Korea
2 Korea Military Academy, 574, Hwarang-ro, Nowon-gu, Seoul, Republic of Korea
3 IT Development Division, LG Electronics, 10, Magokjangang 10-ro, Gangseo-gu, Seoul 07796, Republic of Korea
4 These authors contributed equally to this work.

E-mail: suhr@skku.edu

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Abstract
Meticulous design and optimization of additive manufacturing (AM) are essential for obtaining high-quality metallic products, particularly using laser powder bed fusion (L-PBF). However, its potential in applications is limited because of the lack of understanding of AM. This makes the process parameter optimization time and cost-consuming. Here, the L-PBF process is employed to minimize defects and enhance the mechanical properties of 17–4PH stainless steel specimens, coupled with modeling. The optimal manufacturing parameters were determined by evaluating the relative densities of the as-built parts and thermal deformation. Either high or low energy densities resulted in high porosity and a higher energy density results in greater thermal deformation, attributed to the high mismatch in thermal expansion, while the surface roughness of as-built products is not as good as commercially available products. The hot isostatic pressing process improved the mechanical properties of the printed product by reducing the porosity and recrystallizing microstructures.

1. Introduction
Laser powder bed fusion (L-PBF) is a metal 3D printing technique where metal powders are melted and stacked layer by layer (figures 1(b), (c)). It is suitable for manufacturing complex structural parts for aerospace, medical, and military applications. Though L-PBF is promising, the quality of the printed products depends on the trial-error-based determination of the manufacturing process parameters [1]. Because the L-PBF process experiences several heating/cooling cycles, material phase transformations (solid–liquid), and localized high-temperature gradients, it results in heterogeneous microstructures, high surface roughness, high porosity, spatter, delamination, crack, residual thermal stress, and deformation [1–5]. It is essential to determine the optimal energy density (E_s) by systematically controlling the process parameters in order to manufacture the desired products cost-effectively; Note that E_s is shown in table 1, where P, V_s, D_s, and D_l are the laser power (W), printing speed (mm s⁻¹), hatch spacing (mm), and layer thickness (mm), respectively.

Predicting manufacturing processing conditions can prove useful. However, individual experimental or numerical studies on L-PBF processing optimization have been available [6–8]. Herein, by combining experimental and numerical approaches, the L-PBF manufacturing process is investigated to control the porosity, surface qualities, microstructures, and mechanical properties of as-built parts. The effect of post heat treatment on the parts fabricated via L-PBF was studied by examining their microstructures and mechanical properties.

2. Materials and methods
Both a microscope and laser particle analyzer confirmed that D10, D50, and D90 of commercially available 17–4PH powder are 10.82, 17.53, 34.06 μm, respectively, which was used in L-PBF (figure 1(a)). The process is
depicted schematically in figure 1(b). After then, the powder layer thickness\((D_t)\) was carefully designed based on the particle movement and spreading by a recoater blade. Since the layer thickness should be more than the D90 of powder, layer thickness was chosen as 40 \(\mu\)m, which is the allowable minimum thickness of powder layer (table 1). This is because the thin thickness, which is thinner than powder diameter, inhibits the particle from being spread well, whereas the thick thickness deteriorates the wettability of bead [9]. The melt pool morphology and size determined the hatch spacing\((D_h)\) with which the overlap rate of melt pools is recommended to range from 20 to 50\%. The hat spacing, therefore, was designed as 0.08 mm considering the melt pool sizes (99.57–132.07 \(\mu\)m), which determined the energy densities of 70.31–97.66 J mm\(^{-3}\).

All specimens were built using selective laser melting equipment (SITI-SLM250, Shanghai Industrial Technology Institute-SITI) (figure 1(c)). Figure 1(d) shows the cubic specimens (25 \(\times\) 25 \(\times\) 25 mm\(^3\)) fabricated by varying the process parameters (table 1) to measure the porosity (ASTM B962–08). All specimens were vertically cut in half for examining the pores or other defects at the cross-section center using an optical microscope (OM). The surface roughness and morphologies of as-built products were characterized by 3D measurement system (VR-3000, Keyence).

To predict the thermal deformation, cantilevered beams (figure 1(e)) were fabricated [10]. The inherent strain and volumetric heat energy were calibrated considering 17–4PH SS properties, including temperature-dependent mechanical properties like the coefficient of thermal expansion, Young’s modulus, etc. After calibration, process analysis was performed under various conditions (table 1) using commercially available AM software (Simufact Additive 4.1) to predict the residual stress distribution and corresponding deformation. Figure 3(a) compares the maximum deformation obtained via simulation and 3D scanning measurements at
three energy densities ($E_v = 70.3, 84.9, 97.7 \text{ J mm}^{-3}$). The maximum deformation prediction agrees with the measurements (100 μm resolution), with error < 2% at 70.3 and 84.9 J mm$^{-3}$.

Mechanical testing, such as hardness and tensile test, and microstructural analysis using OM and electron backscatter diffraction (EBSD) were conducted on the specimens (figure 1(f)) fabricated under optimum printing conditions (figure 3(e)) at a strain rate of 2 mm min$^{-1}$ according to ASTM E8. To investigate the post-processing effects on the microstructures and properties, hot isostatic pressing (HIP)-treated specimens heated for 240 min at 1140 °C and 1100 bar and cooled to 400 °C for 120 min were prepared according to ASTM F3184–16.

3. Results and discussion

To design the process parameters, surface roughness and defects such as thermal deformation and porosity after manufacturing were investigated using simulations and experiments. The surface roughness of as-built product should attain the same level of commercially available product, if it will be used for engineering applications without post processing. The surface roughness of as-built products, however, couldn’t exhibit the feasible surface qualities according to our previous work [4]. The surface roughness of the parts, fabricated with the energy density ranging from 70.31 to 97.66 J mm$^{-3}$, was almost the same value: the surface roughness of 15.48, 13.6, 12.8, and 12.94 μm at the laser power of 180, 200, 220, and 250 W, respectively (figure 2(a)); the surface roughness of 12.94, 12.46, 13.68, and 15.92 μm at the scan speed of 800, 830, 950, and 1100 W, respectively (figure 2(a)). The distance between peak and valley point of as-built surface was 0.0609 mm, and the surface morphologies exhibited poor condition because of the overlapped melt pools (figures 2(b)–(d)). The results confirmed that the surface finishing is indispensably required for the as-built parts to be applied to the industrial fields, and the other qualities aside from the roughness were required to be explored for the process optimization.

The distortion of both as-built parts and baseplate is responsible for the process failure or geometric tolerance [11]. Hence, the thermo-mechanical simulation that can previously predict the distortion was conducted using the as-built cubic parts. The simulation predicts that the deformation sharply increases up to 0.642 mm at 85.9 J mm$^{-3}$ (figure 3(a)). A higher energy density will develop a greater peak temperature and higher temperature gradient in the molten pool during manufacturing, resulting in a severe mismatch in thermal strain. This will give rise to thermal residual stress and deformations. Therefore, lower energy density and heat treatment are required to reduce and relieve the residual stress, respectively.

Pores of the as-built parts, which deteriorate the properties, are formed under the inappropriate process conditions where too high or low energy densities lead to keyhole induced pores or lack of fusion [12]. In order

| $P$ [W] | $V_s$ [mm s$^{-1}$] | $E_v$ [J mm$^{-3}$] |
|--------|-----------------|-----------------|
| 250    | 830             | 94.13           |
| 260    | 860             | 90.84           |
| 270    | 890             | 87.78           |
| 280    | 920             | 84.92           |
| 290    | 950             | 82.24           |
| 300    | 980             | 79.72           |
| 310    | 1010            | 77.35           |
| 320    | 1040            | 75.12           |
| 330    | 1070            | 73.01           |
| 340    | 1100            | 71.02           |
| 250    | 800             | 97.66           |
| 240    |                 | 93.75           |
| 230    |                 | 89.84           |
| 220    |                 | 85.94           |
| 210    |                 | 82.03           |
| 200    |                 | 78.13           |
| 190    |                 | 74.22           |
| 180    |                 | 70.31           |

Table 1. Process parameters: $P$, $V_s$, and calculated $E_v$ ($D_t = 0.04$ mm and $D_h = 0.08$ mm).
to obtain the high qualities and properties of products, the porosity of as-built parts was investigated along with the thermal deformation. Porosity and the pore morphology of the as-built specimens fabricated under different processing conditions were examined via the density measurement and OM (figures 3(b) and (c)). It was found that the laser power can play a more predominant role than the printing speed in determining the size of the molten pool, which is a major factor affecting the porosity. As shown in figure 3(b), the energy density ranging from \( E_v = 85 \text{ J mm}^{-3} \) gives rise to a relatively low porosity of 0.7% for the as-built cubic specimens, indicating the feasible manufacturing conditions. Contrastingly, for \( E_v \geq 95 \text{ J mm}^{-3} \), the porosity was up to 2.23%, owing to lack of fusion at low energy densities [13]. The hatch spacing of 0.08 mm and the energy density-dependent melt pools determined the overlap rate of melt pools ranging from 20.09 to 60.57%, when the energy density is 70.02–97.66 \text{ J mm}^{-3}. If the melt pool size becomes smaller under the same hatch spacing and layer thickness, the irregular pores get formed between the melt pools, exhibiting partially melted particles in the pores [14]. Large and irregularly shaped pores sized 21.9–239.0 \mu m were observed by OM (figure 3(c)), owing to insufficient heat energy to completely melt the metal powder during layer-by-layer printing. Similarly, the porosity of up to 1.82% was obtained for \( E_v \geq 95 \text{ J mm}^{-3} \) because a sufficiently high heat energy can induce narrow and deep keyhole molten pools and metal vaporization [12], resulting in the formation of keyhole-induced (vapor entrainment) and metallurgical pores (figure 3(c)). The keyhole induced and metallurgical pores had a round shape, exhibiting a small size.

Figure 3(d) shows the porosity and maximum deformation with respect to the printing speed and power along with the energy density. Considering the low porosity and the maximum deformation resulting from the corresponding residual stress, it can be concluded that 84.92 \text{ J mm}^{-3} (P = 250 W and \( V_s = 920 \text{ mm s}^{-1} \)) is the optimal processing condition for the given specimens along with the metal powder and L-PBF printer.

The microstructures of 17–4PH stainless steel parts, manufactured with the energy density of 74.40, 83.48, and 94.13 \text{ J mm}^{-3}, were examined via OM images after horizontally cutting and etching them with the solution for a few seconds: 30 ml H2O, 5 ml HNO3, 5 ml HCl, and 0.2 g CuCl2. In the case of as-built parts, the microstructures had an average grain size of \( \sim 10 \mu m \) with columnar shape, as shown in figure 4. The shape and size of grains were attributed to the laser power, speed, and path which is responsible for the direction of heat flux [15]. Therefore, the influence of the energy densities on the melt pools dominated the microstructure evolution during the manufacturing. Figure 4 shows the hierarchical microstructures with irregular dispersion of \( \delta \)-ferrite in the matrix of lath martensite, including texture. Since the grain follows the behavior of melt pool, the microstructures exhibited ripple shape with the distance of 80 \mu m: the designed hatch spacing. The copper-rich phases, which were embedded on the matrix, had been reported to precipitate during heating and cooling,

![Figure 2](image-url)
while the primary phase of martensite is supersaturated, and the phase enabled strengthening of 17–4PH stainless steel materials [16]. When the energy density changed from 69.14 to 94.13 J mm\(^{-3}\), the fraction of \(\delta\)-ferrite, distributed at the side of melt pools, decreased, as shown in figures (a)–(c). This is because the higher energy density increases the melt pool size and peak temperature, while decreasing the cooling rate [17]. The \(\delta\)-ferrite will pass the austenite stability region without transformation to austenite under the high cooling rate, and thereby it will produce the dominant ferrite phase under the lower energy density [18]. Even if the change in the microstructures may have an influence on the mechanical properties [19], the above-mentioned

![Figure 3.](image-url)
pores, observed in figures 3(c) and 4, will significantly determine the improvement or deterioration of properties. Therefore, tensile testing and EBSD characterization was conducted using the as-built and post-processed specimens produced with the optimum energy densities of 84.92 J mm$^{-3}$, achieving the densification of as-built parts.

Figure 4. OM images of etched microstructures of as-built 17–4PH stainless steel, manufactured with the energy densities of (a) 74.40, (b) 83.48, and (c) 94.13 J mm$^{-3}$.

Figure 5. (a) EBSD inverse-pole figure map, and (b) grain size distribution. Fractured surface of the 17–4PH SS (c) before, and (d) after HIP treatment. (e) Comparison of specimen properties before and after HIP treatment.

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EBSD analysis was performed (figures 5(a) and (b)) for the microstructural investigations of the cubic specimens fabricated under the optimal condition: \( E_v = 84.92 \ J \text{ mm}^{-3} \) determined in this study. The specimens were horizontally cut in half for examining the microstructures. The black arrow in figure 5(a) indicates the microstructural evolution with printing. Over 95.3% grains (diameter = 0–25 \( \mu \text{m} \)) appeared to grow preferentially along \(<001>\), and equiaxed subgrain or columnar structures were observed along the printing track [20]. The orientations and morphologies of developing grains are determined by the direction of heat transfer in the molten pool, which is mainly dependent on the printing trajectory and the other printing parameters [21].

HIP treatment was employed to improve the relative densities, heterogeneity, and anisotropy of the specimens [22]. Following this, SEM characterization of the fracture surface after the tensile test confirmed that the pore sizes decreased. Microporous calescence was observed in the fracture under higher external pressure and temperature, resulting in a 1.0% increase in relative density (figure 5(d)). From the tensile test results, when compared to those of the untreated printed specimens, the hardness, yield and ultimate strengths of the HIP-treated specimens increased by 10.7, 30.2 and 23.5%, respectively; the elongation at break was decreased by 18.2% (figure 5(e)). This is because the fraction of martensite, which has a 2\( ^\circ \)–15\( ^\circ \) misorientation angle (figure 5(c)), increased from 32.5% to 58.9% and equiaxed subgrains (<5 \( \mu \text{m} \)) were formed in the grains (average diameter = 10.57 \( \mu \text{m} \)) after HIP treatment (figure 5(a)). The microstructures induced an improvement in harness and strength, and decrease in ductility, exhibiting the strength–ductility trade-off effect according to the Hall–Petch effect [23]. The HIP-treated specimen exhibited a more homogeneous microstructure than the as-built parts (figure 5(a)) and no strong \{100\} solidification textures. This indicates that the anisotropic property of the as-built parts changed to isotropic, achieving reliable structural performance in L-PBF parts.

4. Conclusions

L-PBF process and post-processing were investigated with a semi-empirical approach for achieving high-quality 17–4PH SS products. Densification, surface roughness, microstructure, mechanical properties such as hardness and tensile properties were discussed for building high-quality as-built products. Even though the surface roughness of as-built parts decreased at higher \( E_v \), it exhibited the poor condition (12.94–15.92 \( \mu \text{m} \) roughness), which couldn’t be immediately used for the industrial applications. Therefore, the result confirmed that post-processing should be conducted after manufacturing the as-built parts. Next, unavoidable defects (pores and deformations) were optimized since inadequate optimization of L-PBF induces defects, degrading the mechanical properties of products. Lack of fusion- and keyhole-induced pores were generated at low and high \( E_v \) in the range of 70.31–78.13 and 89.84–97.66 \( \text{J mm}^{-3} \), respectively. In the case of thermal deformation, it gradually increased at higher \( E_v \), because high residual stress and distortion accounted for the severe temperature gradient by high \( E_v \) and subsequent mismatch of properties. Then, optimal conditions were obtained considering the porosity and thermal deformation at \( E_v = 84.92 \ J \text{ mm}^{-3} \). Finally, High-quality products fabricated under optimal conditions exhibited enhanced density, hardness, and strength following HIP. On the other hand, ductility decreased due to pore reduction and recrystallization to 58.9% martensitic and equiaxed subgrains (<5 \( \mu \text{m} \)). This work will contribute to improve the stabilization of the L-PBF process by the trial-error-based approach.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Jonghwan Suhr @ https://orcid.org/0000-0003-3491-5738
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