Selective laser melting of 1.2738 mold steel: densification, microstructure and microhardness

Wei Yang, Xiaoxun Zhang, Fang Ma, Sensen Dong and Juze Jiang

1 School of Materials Engineering, Shanghai University of Engineering Science, Shanghai 201620, People’s Republic of China
2 School of Mechanical and Automotive Engineering, Shanghai University of Engineering Science, Shanghai 201620, People’s Republic of China
* Author to whom any correspondence should be addressed.

E-mail: xzxhangsh@163.com

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Abstract

In this study, the effects of laser power and scanning speed on the relative density, microstructure and microhardness of selectively laser melted (SLM) 1.2738 mold steel were systematically investigated. The deposited energy density, which can express the change in these process parameters and the relative density with one curve, were found more reliable than volume energy density (VED) on the design of process parameters. With the same VED value, samples formed by the combination of a high laser power and scanning speed possess a higher densification than that formed by low laser power and scanning speed. High power may lead to keyhole pore formation. In the top of the molten pool, ultrafine cellular structure was formed, causing that the microhardness value of the top section was always higher than that of the side section.

1. Introduction

Additive manufacturing (AM) is a promising technique that enables the fabrication of complex-shaped metal products [1–3]. Selective laser melting (SLM) has become one of the most eye-catching AM technology due to its good processing accuracy. In the SLM process, the thin layer of powder is repeatedly pre-laid and selectively melted by laser irradiation, then realize the manufacturing of parts.

SLM has unique applications in many fields. In order to meet specific requirements, many types of materials have been developed for SLM, such as aluminum alloy [4–6], titanium alloy [7,8] and stainless steel [9–11]. Complicated shape of the conformal waterway significantly improves production efficiency and product quality in industrial production, which cannot be achieved by traditional processing methods. The application of mold steel in the field of SLM has broad application prospects. Zhou et al [12] studied the role of trace element content on SLM forming S316 mold steel. Results showed that the sample fabricated with a higher Si + Mn content exhibited the lower mechanical properties due to a few of pores observed on the cross surface. Katancik et al [13] investigated the effect of different laser volume energy densities on H13 steel formed by SLM and found that when the VED reached 760 J mm⁻³, the density reached 99%. At the same time, it is found that the hardness of the sample will be improved after tempering at 550℃. Mazur et al [14] obtained a best combination of process parameters with porosity less than 0.1% through a series of process parameter optimization, but this will greatly reduce the accuracy of the product. It was found that when the VED was 80 J mm⁻³ and the laser power was 175 W, a compromise between porosity and product geometric accuracy can be achieved. Nowadays, the further application of mold steel that hinders SLM is that there are few types of mold steel materials to choose. In addition, how to achieve the dense SLM-formed (SLMed) parts is still a research hotspot for many researchers [15,16]. 1.2738 mold steel is a medium carbon steel which is widely used as the injection mold materials. The steel has excellent polishing performance, excellent workability and good wear resistance.

In order to obtain high-performance molds, it is very important to understand the density, microstructure and mechanical properties of SLMed mold steels. Porosities are considered one of the major defects in parts.
produced by SLM [17]. Porosity will reduce the mechanical properties of parts greatly and shorten the service life of parts [18, 19]. It is a common optimization method to enhance the performance of SLMed parts by optimizing process parameters. In SLM, there are some influential process parameters such as laser power, scan speed, layer thickness and hatch spacing. These different parameters can be combined as a total volume energy density (VED), according to equation (1) [20]:

$$\text{VED} = \frac{P}{v \cdot h}$$  \hspace{1cm} (1)

where: $p$ is the laser power (W); $v$ is the laser scanning speed (mm s$^{-1}$); $h$ is the hatch spacing (mm); $d$ is the layer thickness (mm). Li et al [21] studied the influence of process parameters on the porosity of 316 stainless steel. In their research, they explored the effect of scanning speed on density by fixing laser power, hatch spacing and layer thickness. They found that a high scanning speed ($v = 180$ mm s$^{-1}$) would result in a poor relative density with the value of 65%, while the scanning speed is reduced to 90 mm s$^{-1}$, the density increased to 95%. Moreover, they found that it is an ideal processing window when the VED is $92 \sim 185$ J mm$^{-3}$. Tomasz et al [22] investigated the effect of different laser power and scanning strategy on SLMed 316 L stainless steel, and found that the laser energy density and scanning strategy will greatly change the composition of austenite and ferrite in the structure, thereby affecting the mechanical properties. Suzuki et al [23] studied the effect of laser power and scanning speed on SLMed martensitic steel, and found that under the same VED, the density, microhardness, and molten pool depth of the part will vary greatly. Ghafoor et al [24] studied the impact of different VED on SLMed 304 stainless steel and found that when the VED was 1400 J mm$^{-3}$, the pores and spheroidization of the sample disappeared, and the density reached 99%.

Although there are some studies on the pore formation mechanism and mechanical properties of different materials under different combinations of process parameters [25–27], the influence of process parameters on relative density, hardness and microstructure of 1.2738 alloy are not clear yet. The proper processing parameters of forming 1.2738 alloy through SLM have not been established. Therefore, the purpose of this paper is to obtain high-quality SLMed 1.2738 parts by changing the process parameters, and explore the pore formation mechanism.

2. Material and methodology

2.1. Material

The powder of 1.2738 alloy was provided by PMG3D Technologies (Shanghai) Co. Ltd. The powder size distribution is 15–53 μm. The morphology of the powder is shown in figure 1; the powder is mostly regular spherical and has good fluidity. In addition, some powders have relatively small satellite spheres on the surface. The chemical composition of the powder is shown in table 1.

![Figure 1. SEM image of 1.2738 powder.](image)

| Table 1. Chemical composition of the used 1.2738-powder. |
|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| Fe | C | Cr | Mn | Si | Mo | Ni | O | N | P |
| Bal. | 0.44 | 2.08 | 1.40 | 0.38 | 0.23 | 1.00 | 0.04 | 0.11 | 0.02 |
2.2. SLM process
The 1.2738 steel specimens in this study was produced by a YLM-120 powder bed machine equipped with a 200 W fiber laser beam. The diameter of laser beam is 70 μm. During the SLM process, nitrogen was continuously introduced to ensure that the oxygen content in the processing chamber was below 100 ppm. In order to match the average size of the powder, the layer thickness was fixed at 30 μm, the hatch space is fixed at 80 μm. The scanning direction was to rotate 67° for each printing layer in order to reduce the residual stress. 8 × 8 × 8 mm³ cubic samples were built for relative density test, microstructure observation and hardness test. The process parameters used in this study is shown in table 2.

2.3. Sample characterization test
2.3.1. Relative density test
The relative density of the SLMed samples was measured by Archimedes' principle, which was considered more reliable and comprehensive than relative cross-sectional microscopic observation. The basic principle of Archimedes is to use an analytical balance to measure the mass of parts in air and in liquid (water in this experiment). The density result is calculated by the following formula, each sample is tested 3 times and the average value is taken as the final density result.

\[
\rho_p = \frac{m_a}{m_a - m_l} * (\rho_l - \rho_a) + \rho_a
\]

Where \(m_a\) and \(m_l\) are the weight measured in the air and liquid, respectively; \(\rho_a\) and \(\rho_l\) are the density of air and liquid, respectively.
2.3.2. Microstructure observation
The as-built samples were polished according to the standard process and, etched by 4% nitric acid alcohol for 10 ~ 20 s. The microstructure was observed using an optical microscopy (OM, CLOOSROMAT350, Germany) and a scanning electron microscopy (SEM, S-3400N, Japan). Both the side section (parallel to the building direction) and the top section (perpendicular to the building direction) were considered in the observation due to the anisotropy of the building process.

2.3.3. Hardness test
The hardness test was carried out by a Vickers hardness tester (HXD-00TMC/LCD) under a load of 300 g for 15 s dwell time. 15 evenly distributed points on the flat surface were tested. In order to study the anisotropy of the SLMed parts, the hardness of both the side section and top section was measured.

3. Results and discussion

3.1. Relative density
Figure 2 shows that the density of as-built 1.2738 samples with different VED values. When the laser power is 180 W, the VED increases from 60 J mm\(^{-3}\) to 90 J mm\(^{-3}\), and the density increases from 94.23% to 99.76%. However, when the VED further increases to 100 J mm\(^{-3}\), the density decreases to 99.29%; When the laser power is 90 W, with the VED increases, the density gradually increases from 90.63% to 93.9%.

When the VED is low, the input energy cannot completely melt the powder. The powder lacking fusion will appear at the bottom of the molten pool or the overlapping area of two adjacent molten pools, which will cause a significant decrease in density. With the VED increases, the powder absorbs enough energy to make the molten pool have a good metallurgical bond with the previous printing layer and the adjacent molten pool, thereby significantly increasing the density. While increasing the input of laser energy may not continue to increase the density, the density may remain relatively stable or even decrease. According to the liquid phase dynamic viscosity formula in the molten pool [28]:

\[
\mu = \frac{16}{15} \sqrt{\frac{m}{k_{B} T}} \gamma
\]

Where \(\mu\) is dynamic viscosity of liquid phase in molten pool, \(m\) is the atomic mass, \(k_{B}\) is Boltzmann’s constant, \(T\) is the liquid temperature, and \(\gamma\) is the surface tension. When excessive energy is absorbed by the powder, the temperature of the molten pool increases significantly, and the viscosity of the molten pool decreases. Excessive energy input will cause spatter and keyholes [29, 30], which will reduce the density of the part. Moreover, some
elements in the powder that have a low melting point are easy to evaporate, generating a recoil force on the molten pool and forming bubbles in the molten pool [17].

It is interesting that under the same VED, the density of the as-built samples formed by the combination of high laser power and high scanning speed is significantly higher than that of low power and low scanning speed. That means when the laser power and scanning speed are doubled at the same time, the same relative density cannot be obtained even under the same VED. Moreover, compare A2 and E1, the laser power is reduced by half and the scanning speed becomes nearly a quarter to get the same density. This means that the influence of laser power on the density is greater than the scanning speed at the same VED. This shows that the VED is inappropriate as an index for evaluating density.

In this study, the deposition energy density ($\Delta H$) is used as a reference for exploring density and process parameters, which is shown in equation (4):

$$\Delta H = \frac{AP}{\sqrt{\pi D v \sigma^2}}$$

Where $A$ is the laser absorption rate, and $D$ is the thermal diffusivity, $\sigma$ is the laser spot size. This parameter was originally proposed by Hann et al [31] and used to investigate the correlation between the melt pool depth normalized by the optical spot size and the laser condition [23]. As $A$, $D$, and $\sigma$ are constants in this study, $Pv^{-1/2}$ was used based on equation (4) to simplify the parameter by removing constants. Table 3 shows the value of $Pv^{-1/2}$ based on deposition energy density under different process parameters.

Figure 3 shows the relationship between density and deposition energy density. When the deposition energy density is lower than 6.236 W mm$^{-1/2}$ s$^{1/2}$, the density is roughly linear with the laser deposition density, and when the deposition energy density is higher than 6.236 W mm$^{-1/2}$ s$^{1/2}$, it can be nearly dense. This result shows that the deposition energy density can be used as an important reference for fabricating dense SLMed parts.

### Table 3. The value of $Pv^{-1/2}$ based on deposition energy density under different process parameters.

| Sample | $p$ (W) | $v$ (mm s$^{-1}$) | $Pv^{-1/2}$ (W mm$^{-1/2}$ s$^{1/2}$) |
|--------|---------|------------------|--------------------------------------|
| A1     | 90      | 625              | 3.600                                |
| A2     | 180     | 1250             | 5.091                                |
| B1     | 90      | 536              | 3.877                                |
| B2     | 180     | 1071             | 5.500                                |
| C1     | 90      | 468              | 4.160                                |
| C2     | 180     | 936              | 5.883                                |
| D1     | 90      | 427              | 4.355                                |
| D2     | 180     | 833              | 6.236                                |
| E1     | 90      | 375              | 4.647                                |
| E2     | 180     | 750              | 6.572                                |
The difference between the deposition energy density and the VED lies in the thermal diffusion model that is assumed. As shown in figure 4(a), the VED converts the energy of the laser into a rectangular region with edge lengths of scan speed, layer thickness, and scan spacing, respectively. The limitation of the model is that the layer thickness is fixed as the depth of heat transfer. However, this is not justified because at different scan speeds it changes the dwell time of the laser at a specific point, and the diffusion distance of this point during this time is the depth. Figure 4(b) illustrates the model of the deposition energy density. The heat will be distributed in a cylinder with the laser spot size $\sigma$ as the diameter of the cylinder and the thermal diffusion distance $(D\tau)^{1/2}$ as the height of the cylinder. The heat-distributed region is a cylinder with a volume of $\pi \sigma^2 (D\tau)^{1/2}$ [32], where $\tau$ is the dwell time, during which the laser is irradiated to a certain point. The laser heat irradiated during $\tau$ is $P\tau$, and the heat absorbed by the material is $A\tau$. The deposited energy density is obtained by dividing $A\tau$ by $\pi \sigma^2 (D\tau)^{1/2}$. Although numerical coefficient is different because Hann et al have taken thermal distribution into stricter
The thermal diffusion length is physically close to the actual melt pool depth, so the deposition energy density model is more appropriate than the VED to explain the density-induced changes.

3.2. Pore formation mechanism

3.2.1. Pore type

The change of density is directly related to the existence of pores in the as-built sample. Figure 5 shows the evolution of pores under different VED values. It can be seen from figure 5 that under the laser power of 90 W, the number of holes gradually decreases with the increase of VED value. However, under the same VED value, the porosity of the as-built sample formed under 180 W laser power is less than that formed under 90 W. Under the high laser power of 180 W, the overall porosity of the formed part is low. Three different types of pore are shown in figure 5, and the formation mechanism of different pores is different. The change of relative density shown in figure 2 can be understood by exploring the formation mechanism of pores:

(1) Lack of fusion pore

Lack of fusion is a typically pore defect due to insufficient energy input, and pores are irregularly shaped and generally distributed between adjacent molten pool or between adjacent layers [34]. As shown in figure 6(a), when the VED is low, the rapid cooling speed does not allow the liquid to wet and spread in time, resulting in a smaller width of the melt pool, which forms a ball-like melt pool to reduce the surface tension. The spherical molten pool will cause obvious undulations on the upper surface of the molten pool, and even large pits. The subsequent laying powder will enter the pits of the unfused area or form a thicker powder layer at the overlap of adjacent molten pools, these locations will be the starting point for the formation of lack of fusion. As shown in figure 6(b), decreasing the scanning speed, the maximum temperature of the melt pool increases, the wettability of the melt pool is better. The width of the melt pool increases, which significantly eliminates the gap between the two melting pool, forming a relatively smooth surface. In addition to the need for a sufficient overlap between adjacent molten pools, good interlayer bonding can reduce the lack of fusion, as shown in figure 6(c). When the penetration depth of the laser cannot achieve a stable combination of the molten metal with the substrate or the previous printing layer, flat arc-shaped pores are easily formed.

Under the same VED value, lack of fusion pore of the as-built 1.2738 sample formed by the combination of high laser power and scanning speed is less than that of low laser power and scanning speed. This indicates that the laser power has a greater influence on the lack of fusion pore of formed part.

Figure 6. Schematic diagram of the change of molten pool size and its effect on fusion with different VED. (a) Low VED; (b) Medium VED; (c) High VED.
Gas pores usually have a round shape with a size below 10um. The melt solubility of gas at high temperature is high, as the temperature of the melt pool decreases, the solubility of the gas decreases. Due to the rapid solidification rate, if gas does not escape from the melt pool in a timely manner will leave spherical pore. Therefore, gas pores are more likely to form at high scanning speed.

The source of gas is mainly attributed to two aspects: (1) Protective gas in the chamber. The loose accumulation of powder will cause gaps between the powders, and part of the gas is between the gaps; the chamber is filled with protective gas, and the molten metal will absorb the gas from the chamber. (2) The metal powder is prepared by gas atomization, generally nitrogen. Therefore, in the process of preparing the powder, all the powder will inevitably undergo the process of absorbing nitrogen from the gas atomization process [35].

Keyhole pore

It is found from the results of the relative density in figure 2 that the relative density decreases at sample E2, it is attributed to the formation of keyhole pore. When the high VED that caused by a high laser power and low scanning speed was applied to the powder bed, some metal elements in the high temperature molten pool will evaporate. Metal evaporation causes the development of a vapor cavity that enhances the laser absorption [33]. Collapse of the cavity can leave voids in the wake of the laser beam. As shown in figure 5, keyhole pore also has a spherical shape. The size of keyhole pore is around 100 um.

3.2.2. Molten pool size

The size of the molten pool is affected by the process parameters. As shown in figure 7, at the same laser power (90 W), it can be seen that at high scanning speed the depth of the melt pool is relatively small. It can be seen that

| VED (J mm⁻³) | Melt pool width (µm) | Melt pool depth (µm) |
|--------------|----------------------|----------------------|
| 60           | 155.34               | 36.71                |
| 70           | 169.26               | 37.92                |
| 80           | 187.58               | 40.27                |
| 90           | 224.21               | 54.96                |
| 100          | 317.48               | 77.30                |

Figure 7. The size change of molten pool under different VED values when $P = 90$ W. (a) VED = 60 J mm⁻³, (b) VED = 70 J mm⁻³, (c) VED = 80 J mm⁻³, (d) VED = 90 J mm⁻³, (e) VED = 100 J mm⁻³. The red dotted line indicates the boundary of the molten pool.
the depth and width of the molten pool significantly increases with the decrease of scanning speed, the overlap area between the adjacent two melt pool increases. The changes in the width and depth of the molten pool with the volume energy density are shown in table 4. As the VED increases, the width of the molten pool increases from 155.34 um to 317.48 um, and the depth of the molten pool gradually increases from 36.71 um to 77.30 um.
For the same VED (80 J mm$^{-3}$), figure 8 show the effect of different combinations of laser power and scanning speed on the molten pool size. Under the combination of low laser power and scanning speed ($P = 90$ W, $\nu = 936$ mm s$^{-1}$), the molten pool shape is shallow and the shape of ripples are elongated, as shown in figures 8(a), (c). While under the combination of high laser power and scanning speed ($P = 180$ W, $\nu = 936$ mm s$^{-1}$), the molten pool shape is much deeper and the ripples are changed to nearly spherical shape. During the SLM process, the oxygen in forming chamber reacts to form an oxide film on the surface of the molten pool, the oxide film will reduce the absorption rate of the molten pool to the laser. However, high laser
power can break the oxide film, increase the energy absorbed by the molten pool, and achieve a greater depth of the molten pool [31].

3.3. Microstructure

During the SLM process, 1.2738 powder was melted by the laser beam to form austenite, and then completes the unbalanced transition at a very high cooling rate to form martensite. The solidly dissolved alloying elements in the austenite will enhance the stability of the supercooled austenite. There is a residual austenite in the microstructure of SLMed parts. Figure 9 shows the x-ray diffraction patterns of the specimens formed by

![Figure 12. Change in Vickers hardness as functions of the volume energy density.](image)

![Figure 13. The relationship between relative density and microhardness.](image)
different VED. It can be seen that the main phase of the SLMed specimens are martensite and residual austenite. Furthermore, changing the process parameters makes no effect on the change of phase.

Figures 10(a)–(e) shows the side section images of the metallographic structure of as-built 1.2738 samples formed under different process parameters, the microstructure is composed of a typical martensite structure. When the scanning speed reaches is low, the microstructure is the fine and uniform. As the scanning speed decreases, the structure shows a coarsening trend. Figure 10(f) shows the metallographic structure of the top section, it can be seen from the top section image that lath martensite grows towards the center of the molten pool.

Figure 11 shows the SEM images of microstructures viewed along the side section of SLMed 1.2738 samples. Two morphologies of microstructure were observed: one is cellular structure represented by region 1, and the other is dendritic structure represented by region 2. The enlarged view of region 1 is shown in figure 11(b), showing the uniform equiaxed grains with a size of 1 ~ 2 μm. The cell structure is usually formed at the top of the molten pool, because the top of the molten pool dissipates heat through air during solidification, which forms a faster solidification rate. Figure 11(c) shows the enlarged view of region 2, represents a typical cellular dendritic structure. The powder was melted to form a semicircular molten pool under the action of laser beam, the heat in the molten pool is mainly conducted through the solidified metal. Therefore, in the solidification stage, the temperature gradient is the largest along the vertical direction of the molten pool boundary. Therefore, the crystal growth direction is generally perpendicular to the molten pool boundary.

3.4. Microhardness

3.4.1. Effects of the VED on microhardness
The influence of different VED values on the microhardness of as built 1.2738 sample is shown in figure 12. When the laser power is 90 W, as the VED increases, the hardness increases from 492.9 HV to 534.13 HV, and the standard deviation of the hardness is getting smaller and smaller. This shows that increasing the VED can effectively increase the hardness. Previous studies generally believed that the hardness will increase with the VED increases [36], which leads to an increase in density. However, in this experiment, when the laser power is 180 W, as the VED increases, the hardness decreases from 536.9 HV to 485.27 HV. While the density of the sample increases from 93.15% to 99.67%, indicating that the density is increases at the same time the hardness value tends to decline.

3.4.2. Effects of relative density on microhardness
Figure 13 depicts the relationship between relative density and microhardness. The microhardness first showed an increase with the increase of relative density and reaches a maximum value of 539.12HV. However, higher
relative density does not mean greater microhardness, when the density exceeds 94.23%, hardness value begins to decrease. This may be related to the microstructure and grain size under different process parameters. According to the Hall-Patch rule, the smaller the crystal structure, the higher the strength of the crystal. In this study, the VED is changed by changing the laser scanning speed. A higher VED means a lower scanning speed and a longer the solidification time, which will make the crystal grow in a certain period of time, and form relatively large grains, thereby reducing the hardness.

3.4.3. Anisotropy of hardness

Figure 14 shows the microhardness on both Top section and Side section of the samples formed under various VED values. The hardness value of the top section is always higher than that of the side section. It is due to that the microstructure in the top is generally small equiaxed grains, while only columnar-dendritic grains can be seen in the side section.

4. Conclusion

Under the same VED, effects of different process parameter combinations on relative density and microhardness were studied. The microstructure and phase composition of SLMed 1.2738 mold steel were also observed. The main conclusions are summarized as follows:

1. The VED cannot be used as an indicator to explain the densification change. An analysis based on the deposition energy density shows that the density is linearly related to the deposition energy density when the value of \( P^{\frac{1}{2}} \), that based on the deposition energy density is lower than 6.236 W·mm\(^{-1/2}\)·s\(^{-1/2}\). The SLMed parts are nearly dense when the value of \( P^{\frac{1}{2}} \) is above 6.236 W·mm\(^{-1/2}\)·s\(^{-1/2}\).

2. Under the same energy density, the influence of laser power on relative density is greater than scanning speed. Samples formed by high laser power and high scanning speed usually possess a high relative density. However, too high laser power will lead to the formation of keyhole pore.

3. The microstructure of SLMed 1.2738 mold steel are composed of martensite and retained austenite. Fine equiaxed structure can be found in the top central area of the molten pool, and dendritic shaped structures that grow along the temperature gradient are formed perpendicular to the molten pool boundary.

4. When the density of SLMed part is low, by adjusting the process parameters to increase density can also effectively increase the microhardness. Besides, when the SLMed part reaches an almost fully densification, the type of microstructure and grain size becomes the dominant factor.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

ORCID iDs

Xiaoxun Zhang https://orcid.org/0000-0003-4326-4893
Sensen Dong https://orcid.org/0000-0003-0320-8758

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