Analysis of morphology and residual porosity in selective laser melting of Fe powders using single track experiments

I V Shutov, G A Gordeev, E V Kharanzhevskiy and M D Krivilyov
Udmurt State University, Universitetskaya str. 1, building 4, 426034, Izhevsk, Russia

E-mail: shutiny@gmail.com

Abstract. Morphology and residual porosity of single tracks obtained by pulse selective laser melting (SLM) of Fe powder have been studied by metallography. Multiple cross sections of the stainless substrate with the single tracks deposited by SLM are examined and classified depending on processing parameters. A sustainable scanning strategy to reduce residual porosity is suggested for pulse laser annealing. The developed method is suitable both for improvement of processing regimes in commercial SLM machines and validation of numerical models in additive manufacturing of metal parts. The effect of the beam radius, pulse energy, its frequency and duration on a shape of the single track and its adhesion to the substrate is revealed.

1. Introduction
Additive manufacturing (AM) based on layer-by-layer fabrication of 3D parts is now one of the most rapidly developing industrial technologies. The methods of selective laser melting (SLM) and sintering (SLS) of metallic powder belongs to a most prominent AM approach in machine construction and engineering [1]. SLM/SLS like other AM methods deliver high-strength metallic parts of arbitrary shape at low level of waste and scrapped components. A vast majority of commercial 3D SLM printers use continuous laser source however in some systems pulse laser systems are implemented [2,3]. Recent studies [1,3,4] shown that pulse SLM machines have advantages in comparison with continuous ones in processing of materials with high reflectivity.

Further improvement of SLM is faced to a problem that good quality of melted or sintered layers for different powders is registered only in a narrow interval of processing parameters. Thus optimization of these parameters by the trail-and-error method is expensive and time-consuming for specific 3D prototypes. Under such conditions, analysis of simplified processing conditions observed in single track experiments allows us to receive useful qualitative dependences and quantitative estimates how the processing parameters effect on the characteristics of SLM components. Hence, the experimental study with single tracks significantly reduces costs of SLM optimization.

Computer simulation is widely used nowadays for SLM processing according to [1,5,6,7]. The developed models cannot account for all underlying physical processes in SLM due to their complexity, otherwise the computing time increases drastically. Moreover, any mathematical model requires proper validation through comparison to the results of laboratory experiments [7]. In this situation single-track experiments are very convenient. It helps the examination of powder melting and consolidation without some auxiliary effects like interference of neighboring tracks and thermal shape distortion. Additionally, morphology and depth of the molten zone, its width and other characteristics
can be directly analyzed. In literature [1,5,6,7], this problem has not been solved completely therefore the aim of this paper is the study of morphology in single track SLM of Fe powder.

The Fe iron powder with low impurity content was compacted via pulse laser annealing under different modes of processing. Iron was selected since it is a well-studied and base component for steels. In what follows, morphology in terms of residual porosity and adhesion to the substrate is first analyzed by metallography of single-track processed samples. Then the effect of processing parameters of SLM on morphology is revealed.

2. Experimental technique

2.1. Processing of single-track samples

Iron powder bed on the surface of a stainless AISI 321 substrate has been processed by the pulse Yb fiber laser LRS AU-300M with a maximum power of 300 W. The ultra-dispersed Fe powder was deposited and flatten on the substrate mechanically. Due to intensive local heating, the powder is melted during processing. The liquid phase consolidates and then rapidly solidifies resulting in a single track, Fig. 1 and 2. The melted track is depicted at different magnification in Fig. 1. Each track was processed with different governing parameters including the energy $E_{imp}$ of a laser pulse, its time duration $\tau_{imp}$, radius of the laser beam $R_b$, frequency $\nu$, and spatial distance $l_{tr}$ between pulses. Then the instantaneous density of power is given by

$$J_{\text{max}} = \frac{E_{\text{imp}}}{2\pi R_b^2 \tau_{\text{imp}}}$$

where $J_{\text{max}}$ is the peak power density of laser emission. The Gauss-type spatial distribution is assumed in which the effective radius $R_b$ holds about 80 % of delivered energy. In experiments, the following parameter intervals were studied: $R_b$ in the range between 200 and 400 μm, $E_{\text{imp}}$ between 0.5 and 5.1 J, $\tau_{\text{imp}}$ between 0.8 and 6 ms. The distance $l_{tr}$ equals to $R_b$ and the frequency varied between 30 and 100 Hz. Accuracy of $E_{\text{imp}}$ determination was 0.1 J.

![Figure 1](image-url) Top view of the molten single tracks processed with the beam radius $R_b = 200 \mu$m and pulse duration $\tau_{\text{imp}} = 1$ ms.
Figure 2. Sketch of morphology and characteristic distances of the molten zone after pulse annealing where $h_m$ is the maximum depth of the remelted zone in the substrate, $d_m$ is the width of molten powder layer, $h_{\epsilon_{\text{max}}}$ and $h_{\epsilon_{\text{min}}}$ are the maximum and minimum thickness of the sintered track correspondingly, and $d_{\epsilon}$ is the width of the sintered track.

2.2. Metallography of single tracks
The processed (Fe powder + stainless substrate) samples have been examined by metallography. The samples were fixed in epoxy resin, mechanically ground and polished. After etching, the samples were analyzed using the optical microscope Neophot-30 Jena Zeiss. Since the samples have different porosity levels their grinding was done gradually to preserve morphology in cross sections, Fig. 2. At some processing parameters, few (between 6 and 9) cross sections were examined along the same track to reach adequate statistical variability. Other single tracks were examined only with a single cross section. Etching was done with the 5\% HNO$_3$ solution in ethanol with the etching time between 2 and 20 seconds.

3. Results
3.1. Classification of track’s morphologies
Laser melting of powder is accompanied by multiple physical phenomena that proceed at different scales and may include chemical reactions. There are the processes of ultra-rapid heating of powder and substrate, melting, solid and liquid phase sintering and consolidation, convective flow in the molten zone, capillary flow, adhesion of the powder bed to the substrate. Additionally, there are intensive evaporation of metal, surface tension, shape distortion of the substrate, rapid solidification of the molten zone and other processes. Contribution of each factor is changing depending on regimes of laser annealing. Hence their variation is a straightforward way to control the residual porosity, pore distribution in the track, shape and size of the track, adhesion to the substrate.

Based on the analysis of obtained cross sections, different morphological groups were classified depending on processing parameters and governing processes acted in separate SLM experiments, Fig. 3. Figure 3(a) corresponds to a typical shape where the pulse energy is small and no good adhesion of the remelted powder with the substrate is observed. A network of small pores is observed and the shape of the track is vague. Black color of this slice is due to the high concentration of iron and weak mixing with the substrate which contains a lot of chromium and nickel. If the pulse energy is increased, Fig. 3(b), a concave shape is registered due to powder consolidation. Melting of the substrate still does not proceed since a transition between the black track and white substrate is clearly
visible. The pores are of different sizes and the black areas correspond to high concentration of small pores which are not well resolved with optical microscopy.

In Fig. 3(c), the beginning of melting of the substrate was detected. Ultimately this is caused by a higher pulse energy although the molten zone in the substrate is too small to provide effecting mixing of powder with the substrate. Large gas pores were found in some cross sections while in other sections they were absent. Finally, at $E_{\text{imp}} = 2.8$ J, Fig. 3(d) stirring of the melt becomes so intensive that good adhesion of the track to the substrate is achieved. Correspondingly, the track’s color becomes lighter and residual porosity significantly decreases. At this stage small pores consolidate in larger ones since they do not have enough time to escape from the melt prior solidification. The same effect was predicted in computer simulation of powder consolidation [9, 10]. The track’s shape is partially concaved that is typical for poor wetting. It was proved experimentally that such situation occurs if a high power density is combined with small pulse duration.

Figure 3. Morphologies of the cross sections obtained under different processing conditions in SLM of Fe powder on top of a AISI 321 substrate. The initial layer thickness before process is 150 µm. The straight-line single-track strategy is applied. The processing parameters are as follows. a) $E_{\text{imp}} = 0.7$ J, $\tau_{\text{imp}} = 4$ ms, $R_b = 200$ µm, $\nu = 50$ Hz; b) $E_{\text{imp}} = 1.1$ J, $\tau_{\text{imp}} = 4$ ms, $R_b = 200$ µm, $\nu = 50$ Hz; c) $E_{\text{imp}} = 1.6$ J, $\tau_{\text{imp}} = 4$ ms, $R_b = 200$ µm, $\nu = 50$ Hz; d) $E_{\text{imp}} = 2.8$ J, $\tau_{\text{imp}} = 3.0$ ms, $R_b = 300$ µm, $\nu = 50$ Hz; e) $E_{\text{imp}} = 3.6$ J, $\tau_{\text{imp}} = 1.5$ ms, $R_b = 400$ µm, $\nu = 50$ Hz; f) $E_{\text{imp}} = 2.4$ J, $\tau_{\text{imp}} = 1.5$ ms, $R_b = 300$ µm, $\nu = 50$ Hz.
Two last morphologies shown in Fig. 3(e,f) are convex. It corresponds to intensive stirring with streamlines visualized by the alternating strips along the flow streamlines, Fig. 3(d,e,f). The strips are formed by the chains of small pores which are carried by convective vortex flow in the molten zone. At such pulse energy $E_{imp}$ and duration $\tau_{imp}$ the lowest porosity is registered with high intensity mixing of material between the powder bed and the substrate. It is logical to conclude that these morphologies comply to complete consolidation [10]. Surface tension at the melt-gas interface forms the convex shape of the track.

In Fig. 3(e), the small pores are observed at the boundary between the track and substrate since the time for gas displacement was not long enough. Comparing Figures 3(e) and (f) one may conclude that shorter pulse duration yields a higher porosity. The case (f) is obtained at a smaller effective radius $R_b = 300 \, \mu m$ and hence at a higher power density. To summarize, the convex morphology is formed only after significant remelting of the substrate where the capillary effects and convection have sufficient time for full consolidation. Small residual porosity provided under the discussed conditions is contradicted by large evaporation of the powder material. As a result, high pulse energy may lead to melt boiling and subsequently to roughness of the processed sample. All samples examined in the study belong to one of the cases described in Fig. 3.

**Figure 4.** Cross section obtained by etching with the HNO$_3$ 5% solution in ethanol after a time of a) 4 s; b) 6 s, c) 10 s, d) 12 s. The magnified image is given in (e). The processing parameters are $\tau_{imp} = 4$ ms, $R_b = 200 \, \mu m$, $\nu = 50$ Hz, $E_{imp} = 2.2$ J.
The results of etching with different times are analyzed in Fig. 4. In Fig. 4(e), a pattern typical for metallographic analysis is observed. At this stage, its interpretation is ambiguous. The first explanation is that this pattern actually represents the traces of small pores which deepen during etching and lead to large roughness of the surface. These areas do not reflect light and are seen as black in optical microscopy. On the other hand, this pattern can be a result of chemical inhomogeneity due to convective mixing of the molten Fe powder and the substrate. The second explanation is supported by the fact that the patterns are observed only in the regimes (c), (d) and (e) in Fig. 3 where melting of the substrate occurs. At high convective stirring where the track is enriched by material from the substrate the pattern was not found, Fig. 3(f). At small and large etching times the pattern was not registered, Fig. 4(a) and Fig. 4(d), correspondingly.

Figure 5. Dependence of the characteristic distances of the track as a function of the pulse energy $E_{\text{imp}}$ in experiments (symbols «» «–» «Δ» in the plot) in different cross sections of the same track. The processing parameters were $R_b = 200 \, \mu m$, $t_{\text{imp}} = 4 \, ms$, $v = 50 \, Hz$, $l_{\text{tr}} = R_b$. The notation of distances $h_m$, $d_m$, $h_c$ and $d_c$ is given in Figure 2. Here «» and «Δ» are the maximum and minimum values of $h_c$, correspondingly.

3.2. Effect of the pulse energy

The results of examination of the samples are shown in Fig. 5. Increase of $E_{\text{imp}}$ results in nonlinear growth of the track depth $h_m$ and quasi linear growth of the track width $d_m$, Fig. 5(a,b). At the smallest pulse energy $E_{\text{imp}} \leq 2.1 \, J$, weak adhesion to the substrate is combined with a wide molten track. The average track height first slightly grows and then starts to decrease if $E_{\text{imp}}$ increases, Fig. 5(d). This observation is explained by combination of two opposite processes. On one side, the higher volume of molten powder gets a larger convex shape due to capillary forces. On other side, the local porosity $\varepsilon$ decreases with higher $E_{\text{imp}}$ hence the relative density grows. Additionally, at high $E_{\text{imp}} > 2.1 \, J$, intensive evaporation of material occurs. Powder burning was registered both experimentally that was predicted in our numerical simulations [8].
Thus, a conclusion was drawn that the increase of pulse energy $E_{\text{imp}}$ yields better adhesion of the track with the substrate and hence good efficiency of SLM processing. However excessive $E_{\text{imp}}$ leads to powder burning. At $R_b = 200$ µm, $\tau_{\text{imp}} = 4$ ms, $\nu = 50$ Hz, $l_{tr}=R_b$, the optimal regime occurs at processing with $E_{\text{imp}}$ between 1.6 and 2.1 J. In other processing regimes at $R_b = 300$ µm, $\nu = 50$ Hz, $l_{tr}=R_b$, the beginning of sintering and good adhesion was registered at $E_{\text{imp}} \leq 1$ J and $1.3 \leq E_{\text{imp}} \leq 2$ J. The recommended pulse duration $\tau_{\text{imp}}$ is between 0.8 and 4 ms. At $R_b = 400$ µm, $30 \leq \nu \leq 50$ Hz, $l_{tr}=R_b$, powder sintering starts at $E_{\text{imp}} \leq 2$ J and good adhesion is provided at $3 \leq E_{\text{imp}} \leq 3.6$ J with $\tau_{\text{imp}}$ between 0.8 and 4 ms. The general tendency is as follows. Increase of $\tau_{\text{imp}}$ requires higher $E_{\text{imp}}$ for initiation of adhesion since heat is effectively removed from the molten zone to the substrate.

Figure 6. Macrostructure of samples processed by SLM with the pulse energy $E_{\text{imp}}$ between 3.4 and 3.5 J, $R_b = 400$ µm, $\nu = 50$ Hz. The pulse durations $\tau_{\text{imp}}$ are a) 0.8, b) 1, c) 1.5, and d) 4 ms.

3.3. Effect of the pulse duration

The pulse duration $\tau_{\text{imp}}$ is also an important governing parameter like the pulse energy $E_{\text{imp}}$. Equation (1) shows that the pulse power density can be increased through both $E_{\text{imp}}$ and $\tau_{\text{imp}}$. The phenomenological model suggested in literature [9,10,11] assumes Arrhenius-type kinetics of powder consolidation. Hence, strictly speaking, powder sintering starts before melting. Around the melting temperature, intensity of consolidation exponentially increases as a function of temperature. High temperature in the annealing zone after pulse heating exists during a time period which covers the pulse itself and a short time immediately after the pulse. Thus it is necessary to account for both opposite effects. Sufficiently large time $\tau_{\text{imp}}$ is desired to complete consolidation. At the same time, small time $\tau_{\text{imp}}$ leads to increase of the power density and processing temperature in the molten zone.

Figure 6 demonstrates the described effect as interplay of two factors. At small $\tau_{\text{imp}}$ the power density is high however powder has not completed its consolidation. Large pores are seen in Fig. 6 (a) which corresponds to the results of numerical simulation [10]. With the increase of $\tau_{\text{imp}}$ in Fig. 6 (b,c), the power density reduces which results in a higher temperature and almost complete consolidation with low residual porosity. Further increase of $\tau_{\text{imp}}$ provides good sintering of the powder but there is no sufficient adhesion to the substrate required in SLM. In [3,12] it was shown that pulse laser annealing allows us to avoid the balling effect. In our study, the balling effect sometimes occurred if the pulse duration was large, Fig. 7(a). Once the pulse duration was reduced, the absence of the balling effect was observed, Fig. 7(b).
Figure 7. Cross sections of the single tracks obtained after processing with $R_b = 200 \mu m$, $\nu = 50$ Hz. a) $\tau_{imp} = 4 \text{ ms}$, $E_{imp} = 2.2 \text{ J}$, the balling effect is registered. b) $\tau_{imp} = 2 \text{ ms}$, $E_{imp} = 1.8 \text{ J}$, no balling effect.

To summarize this discussion on selection of optimal processing regimes, if the parameter $E_{imp}$ is fixed then it is suggested to use a lower pulse duration $\tau_{imp}$. It helps to increase the laser energy power density. However the value $\tau_{imp}$ has a natural lower limit below which powder burning occurs or residual porosity becomes too high. Sometimes it is possible to fulfill both conditions for the high energy density and the duration when the molten zone exists. Although such regime has a risk of balling.

Figure 8. Cross sections of the single tracks obtained after processing with $\tau_{imp} = 4 \text{ ms}$, $\nu = 50$ Hz: a) $R_b = 200 \mu m$, $E_{imp} = 2.2 \text{ J}$; b) $R_b = 300 \mu m$, $E_{imp} = 2.5 \text{ J}$; c) $R_b = 400 \mu m$, $E_{imp} = 2.8 \text{ J}$.

3.4. Effect of the beam radius

The beam radius $R_b$ is another governing parameter of selective laser melting. It influences an area where heat is pumped into the powder. Therefore the width $d\varepsilon$ of the sintered track directly depends on $R_b$, Fig. 8. Decrease of $R_b$ at the fixed $E_{imp}$ facilitate increase of the peak and average power density inside the laser beam and hence improve efficiency of SLM. The discussed tendency was confirmed in experiments, Fig. 8. Deep melting of the substrate is given in Fig. 8(a) at $R_b = 200 \mu m$. Increase to $R_b = 300 \mu m$, Fig. 8(b) results in reduction of the distance $h_m$. At $R_b = 400 \mu m$, Fig. 8(c) no adhesion is registered and the track has high residual porosity and irregular shape.

In some cases, processing with small $R_b$ and high pulse duration $\tau_{imp}$ leads to a controversial morphology. On the periphery of the track porous pattern was etched while closer to the middle of the molten zone good adhesion was found like in Figs. 3 (c,d), 7 (a), and 8 (a). Our explanation is that the effect is due to a significant difference in processing conditions between the edge and center of the laser beam. For efficient and uniform processing of metal powder with a wide laser beam, the applied power should be increased nonlinearly according to Eq. (1). As was discussed before, such scenario
may result to overheating of the surface, shape deformation and long relaxation of the thermal field between impulses.

Thus, the beam radius has to be optimized according to processing conditions and specification of the SLM machine. For higher speed of processing, the beam radius should be increased. In SLM of thin-wall and high precision parts it is recommended to decrease \( R_0 \) to fulfill the imposed technological requirements. At the same time, the pulse duration \( t_{\text{imp}} \) is limited from above in order to prevent sintering at the edge of the track where local porosity is high and adhesion with the substrate is poor.

### 4. Conclusions

Analysis of the impact of governing SLM parameters on morphology of single tracks is performed. The optimal strategy in selection of the beam radius, pulse energy, and pulse duration is developed through the combined experimental and numerical study of Fe samples processed by a pulse laser.

The performed examination of samples shows that the resulting morphology is closely linked to the processing regimes. All images of cross sections of single tracks have been classified depending on the parameters of laser annealing. The high pulse energy combined with a larger beam radius and a short pulse duration leads to uniform convective mixing in the molten zone with a low residual porosity with good adhesion to the substrate.

In processing with the small pulse energy and large pulse duration, powder consolidation is weak and morphology of the track is irregular. The gradual increase of pulse energy and beam radius were combined with the reduced pulse duration facilitates capillary flow and the convex morphology. Increase of the pulse energy alone extends adhesion to the substrate and is considered as useful. However, this parameter has a limit from above beyond which burning of material occurs. For a given pulse energy it is suggested to reduce the pulse duration as far as it possible before burning starts. A key indicator that the pulse duration is too long is the balling effect.

The beam radius has been selected depending on a particular problem and specification of a SLM machine. A large beam radius is better in terms of residual porosity. At the same time, manufacturing of thin wall parts requires a small beam radius and a short pulse duration.

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