Characterization of an iron-based laser sintered material

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Abstract

Direct Metal Laser Sintering (DMLS) is a relatively new rapid tooling technique to fabricate near net-shaped parts. Properties of DMLS parts are governed by their microstructure. Hence, characterization of microstructure is of significant importance. In this study, a new iron-based DMLS material was characterized to unveil its metallic microstructure to support the prediction of end-user performance and the development of new applications. Characteristics of powder particles, roughness and topography of sintered surfaces, and microstructure of sintered body were investigated. The original powder composition, the particle size, particle morphology and processing parameters were discussed relative to the sintered phases, the porosity and the microstructure. We found a microstructural waviness existing in the DMLS material. Some possible reasons are discussed.

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Keywords: Powder metallurgy; Laser sintering; Iron-based material; Microstructure

1. Introduction

Direct Metal Laser Sintering (DMLS) is a rapid manufacturing technology to make intricate and near net-shaped parts from computer aided data. The working principles of DMLS processing, Fig. 1, can be described as the following sequence: (i) Generate the component model from 3D-CAD software and convert it to standard STL format. (ii) Slice the STL model into horizontal layers with a certain thickness (usually 20–50 μm) in the computer control centre of the DMLS facility. (iii) Spread a layer of metal powder on the top of the building platform. (iv) Melt and fuse the powder by a laser beam, as it traces the geometry of the generated slice. (v) Lower the fused layer and spread a new layer of powder with the recoater. (vi) Laser scan the new surface and fuse the metal particles to each other and to the lower layer. (vii) Repeat the process until the component is fully fabricated. (viii) Finally, remove the part from the machine and sieve back the unsintered powder to the powder dispenser for reuse [1–4].

DMLS has significant advantages such as compound geometrical complexity, near net-shape, low energy consumption, high raw material utilization and rapid manufacturing integrated with CAD technique compared to conventional manufacturing techniques [3,4]. During the last decade, the development of DMLS technique has been accelerated exponentially due to the above mentioned advantages. Nowadays, the DMLS technique has successfully developed into the commercial realm and can produce mould inserts, die-cast tools, and functional metal prototypes using bronze-based and iron-based powder by liquid-phase sintering [6,7]. Nevertheless, the performance of DMLS manufactured parts is limited due to low mechanical properties and bad surface quality [8,9], which makes it difficult to supersede the traditional manufacturing methods. Therefore, microstructural characterization of DMLS materials helps to overcome the weakness and extend its area of use.

Recently, the research on the DMLS method was mainly focused on the technique of producing parts with a high accuracy and selection of suitable materials, as for use in rapid tooling. Little effort has been devoted to microstructural characterization. Sun et al. [10] studied iron-base alloy powder laser sintered material and found a very fine dendrite microstructure in the sintered material. Nin and Chang [11] studied liquid phase laser sintered M3/2 high speed steel and showed that a ‘balling’ phenomenon greatly deteriorates the sintered surface roughness. Khaing [12] studied Cu-based DMLS parts and revealed a structure of scanning lines with pores. Irregular shaped pores, nickel and bronze particles were also observed in the sintered specimens [12]. To summarize the previous research results, porosity and heterogeneous microstructure in DMLS material are hard to
avoid based on today’s DMLS technique. The main aim of this investigation is to define the porosity of an iron-based DMLS material and describe important material characteristics, such as powder features, microstructure, sintered surface roughness and microhardness to support prediction of end-user performance and further developments.

2. Material and experiments

2.1. Material

The material was processed using an iron-based powder blend and an EOSINT M250 machine. A carbon dioxide laser (wavelength = 10.6 μm) with a maximum output of 200 W in continuous wave operation is used as a power source. The diameter of the focused laser beam is 0.4 mm.

The chemical composition of the DMLS material was obtained by energy dispersive X-ray spectrometer (EDS) analysis on one specimen, Table 1. The tensile properties at room temperature along layer direction was 424 MPa in 0.2% proof strength (σy), 505 MPa in ultimate tensile strength (σut), and 118 GPa in Young’s modulus (E) [8].

2.2. Powder analysis

Type and size of the powder have a significant effect on microstructure and porosity of sintered body, and consequently they govern the properties of the final products to a considerable extent [13]. In the current study, the powder blend was spread on a double-sided carbon tape and gently shaken to ensure a thin layer of powder being left on the sample. The morphology and the size of the powder were examined using scanning electron microscopy (SEM). Using an image analyzer, about 4700 particles were measured at 1000×, 1500×, 2000× and 8000× magnifications, from which size distribution histograms were obtained.

EDS was utilized to determine elemental composition and distinguish different kinds of particles apart.

Table 1

<table>
<thead>
<tr>
<th>Element</th>
<th>wt.%</th>
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<tbody>
<tr>
<td>Fe</td>
<td>Bal</td>
</tr>
<tr>
<td>Ni</td>
<td>29</td>
</tr>
<tr>
<td>Cu</td>
<td>8.3</td>
</tr>
<tr>
<td>P</td>
<td>1.35</td>
</tr>
</tbody>
</table>

2.3. Surface roughness test

The surface roughness is a characteristic of particle packing and bridging, and as such it is highly related to the shape and size of the sintered particles, packing direction and packing density. In this study, the surface was measured by 3D surface profilometry (Wyko NT 3300) using a vertical scanning interferometer technique where the average surface roughness as well as 3D topography were obtained.

2.4. Microstructure study

Both of microstructure and pore characterization can be carried out on properly-polished cross-sections. The metallographic preparation and interpretation of the DMLS structure were strongly influenced by porosities. In order to get clear polished cross-sections of the sintered body, the samples were carefully polished down to 1 μm diamond paste. During metallographic preparation, the pores can easily carry abrasives and be smeared. Proper polishing should open the smeared pores, then reveal their true shapes and amounts [14]. Excess or lack of polishing would distort and smear pores, giving the appearance of a low porosity [14]. Etching will usually enlarge pores, leading to an overestimate of the porosity [15]. When the specimens is uniformly dense and properly prepared, the area fraction of porosity will equal the volume fraction of porosity, which equals the porosity calculated from the measured and theoretical densities of the part as in Eq. (1) [14]:

\[ S_p = V_p = \frac{\rho_t - \rho_m}{\rho_t} \]  

where \( S_p \) is the area fraction porosity, \( V_p \) the volume fraction porosity, \( \rho_t \) the theoretical density, and \( \rho_m \) the measured density. In this study, optical microscope (OM) aided with Leica microsystems image analysis software was used to determine the area fraction of porosity and other pore parameters, such as the area (S), the length (L) and the width (W) and the aspect ratio (\( a = L/W \)).

Samples for metallographic examination were etched using etchant solution (HCl 75% 10 ml + HNO3 25% 2 ml) and examined in both OM and SEM. The compositional variation within different zones was analyzed by spot EDS analysis. X-ray diffraction (Cr Kα radiation, Seifert XRD 3003 PTS X-ray generator) was utilized to derive the microstructural phases.

2.5. Hardness test

Macro and microhardness tests were performed with measurement on different phases (HV 20kgf and MHV 5 gf respectively) supporting the general characterization of the microstructure.

3. Results

3.1. Powder analysis

In the powder blend, EDS analysis showed the presence of three kinds of particles constituting a mixture of Cu, Fe, and Ni.
Fig. 2. The overall morphology of the powder blend, SEM-image.

Fig. 3. Morphology of copper particle, SEM-image.

according to Table 2. The particles have different morphologic characters, Figs. 2–5, where most of the nickel and iron particles are spherical, but some are aggregated forming irregular morphologies. Copper particles are relatively big and exhibit a crushed morphology and irregular shape.

Table 2

<table>
<thead>
<tr>
<th></th>
<th>Fe (wt.%)</th>
<th>Ni (wt.%)</th>
<th>Cu (wt.%)</th>
<th>P (wt.%)</th>
<th>O (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-particle</td>
<td>99.7</td>
<td>1.3</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Ni-particle</td>
<td>6.0</td>
<td>94.0</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Cu-particle</td>
<td>3.3</td>
<td>1.2</td>
<td>79.0</td>
<td>14.8</td>
<td>1.7</td>
</tr>
</tbody>
</table>

About 2328 particles were randomly selected and measured using SEM. The average particle diameter is 4.3 ± 2.9 μm, Fig. 6.

The size distributions of three sorts of particles were determined individually, showing substantially larger Cu particles than Fe and Ni particles (32 ± 22, 3.6 ± 5.0, and 6 ± 2 μm mean size respectively). The maximum diameter of Cu particles is 178 μm.

3.2. Surface quality

Relative to the building direction the top-view sintered surface (normal to the building direction) and the side-view sintered surface (parallel to the building direction) are schematically illustrated in Fig. 7. Both of the sintered surfaces were observed using SEM and 3D profilometer, Figs. 8–10. The top-view has a coarse topography (R_a 18.2 μm) containing a network of micro-cracks over the surface due to thermal shocks, Figs. 8a and 9a.

On the other hand, the side-view surface parallel to the building direction is finer (R_a 12.6 μm) with layers piling up as observed in Figs. 8b and 9b.

3D topographies of sintered surfaces (2.4 mm × 1.9 mm), Fig. 10, were obtained by using the profilometer in the magnification of 2.5x. The colour bar shows the maximum height of profile peaks (R_p) and the valley depth (R_v) for the top-view and side-view, which are 79 and −197, and 36 and −131 μm respectively. The side-view valleys have elongated shapes oriented along layer interfaces, while the top-view shows no obvious orientation. The spacing between the two adjacent valleys of the top-view is longer than that of the side-view, which is mainly...
3.3. Structure characteristics

The average density of the sintered specimens was 7.73 g cm\(^{-3}\) as measured by the modified Archimedes method [16]. From image analysis the average porosity was determined to 2.6%. The porosities of side-view and top-view sections were 2.1 ± 0.2% and 3.1 ± 0.2%, respectively. The layer and particle interfaces were preferable sites for pores, Fig. 11. In the top-view, Fig. 11a, the pores are fairly round and big, while in the side-view, Fig. 11b, pores emerge as elongated mainly following unmelted particle boundaries in layer interfaces. Combining both two views, one can envision coin-shaped pores mainly existing with a preferential orientation along layers. In addition, a few larger and irregular pores independent of the layer structure can be observed in the side-view, the square area in Fig. 11b. In the top-view, a lot of perfectly spherical pores were gathered in clusters of different pore sizes, originating from local shrinkage during solidification, Fig. 12. To sum up, three sorts of pores have been distinguished according to their location and shape: (a) disk liked pores orientated along layer interfaces; (b) larger and irregular pores regardless of layer interfaces; (c) nearly perfectly spherical solidification pores gathered in clusters. The main pore parameters are given in Table 3.

The metallographic investigation was performed on the cross-sections parallel and normal to the building directions (side-view and top-view, respectively). In the side-view micrograph, Fig. 13, the thickness of structural layers was clearly demonstrated to be around 20 μm, consistent with the sintering layer thickness. It was found that all layers show a regular wave-like form with an average ‘wave length’ of 300 μm. The formation and effect of such microstructural waviness is discussed in Section 4.1.

Meanwhile, in the top-view micrograph, Fig. 14, the mixture of different microstructural phases was clearly demonstrated. The phases, as determined below by EDS and X-ray diffraction

<table>
<thead>
<tr>
<th>Table 3</th>
<th>Main porosity parameters of the iron-based laser sintered material</th>
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<tbody>
<tr>
<td></td>
<td>Top-view</td>
</tr>
<tr>
<td>Avg ± S.D.</td>
<td>Min</td>
</tr>
<tr>
<td>S (μm(^2))</td>
<td>120 ± 280</td>
</tr>
<tr>
<td>L (μm)</td>
<td>20 ± 41</td>
</tr>
<tr>
<td>W (μm)</td>
<td>13 ± 50</td>
</tr>
<tr>
<td>α (L/W)</td>
<td>1.6 ± 0.5</td>
</tr>
<tr>
<td>Porosity (%)</td>
<td>3.1 ± 0.2</td>
</tr>
</tbody>
</table>
analysis are: (i) Fe, Ni, Cu, P eutectic, (ii) Fe, Ni, Cu austenitic, (iii) Cu-rich, (iv) Fe-rich ferritic, and (v) Ni-rich phases, respectively. Their main features are summarized as follows:

1. Fe, Ni, Cu, P eutectic: a dendritic morphology was found consisting of γ-Fe (Ni,Cu) dendrites and Fe₃P. The phosphide forms an interdendrite network as shown in Fig. 15 using a SEM backscatter detector (BSD). The X-ray diffraction spectrum shows Fe₃P peaks at 62.7° and 70.7° diffraction angles, Fig. 16. Through EDS analysis, the content of phosphorus was calculated to 6 wt.% in the dark net-work (Fe₃P) and 1 wt.% in the light bulk (γ). The previous values are just intended for comparison, inasmuch as the EDS measurements only give approximate values.

2. Fe, Ni, Cu austenite: the element of nickel and copper can stabilize γ-Fe phase at low temperature, where Ni and γ-Fe are completelymiscible and the solubility of copper is high. Therefore, most of the iron forms metastable austenite at room temperature mixing with nickel and copper. According to the X-ray diffraction analysis, the austenite peaks η(1 1 1), η(2 0 0) and η(2 2 0) were shifted to the left due to blending with substitutional element Cu and Ni. The quantitative X-ray diffraction analysis present 65% austenite existing in the final sintered material.

3. Cu-rich phase: copper is the low melting point element in the DMLS material and its maximum solubility is 0.3% in α-Fe and 8% in γ-Fe [17] at room temperatures. Although addition of Ni can improve the solubility of copper in iron, the copper-rich phase was still observed adjacent to α-Fe phase, Fig. 17. A variation of the pre-sintered powder distribution may result in local absence of nickel and segregation of copper from α-Fe phase. Due to a small amount of copper-rich phase and the overlap of diffraction peaks of Ni, Cu and γ-Fe, no apparent copper diffraction peaks are illustrated in Fig. 16.

4. Fe-rich ferrite (α-Fe): according to the X-ray diffraction measurement, 31% α-Fe were examined. After etching, α-Fe shows a lot of corrosion pits on the surface, which helps
Fig. 10. 3D topography of sintered surfaces. (a) Top-view surface normal to building direction. (b) Side-view surface parallel to building direction.

Fig. 11. Pore morphology and locations on the polished cross-sections, OM. (a) Top-view normal to building direction. (b) Side-view parallel to building direction.

an easy identification using SEM, Figs. 17 and 18. The ferrite is formed mainly due to two reasons. First, due to the high melting enthalpy of a larger-massed iron particle [18–20], such as in Fig. 4a, the large-massed particle may remain un-melted or partly-melted after laser sintering, and shows a irregular shape and clear particle boundaries in the polished cross-section, Fig. 18. Second, if iron particles were completely melted but not blended with the austenite stabilizers, they will transform to ferrites again upon solidification, since α-Fe has lowest Gibbs free energy in such case. This kind of α-Fe zone exhibits smooth region boundaries.

3.4. Macro and microhardness result

The macro-Vickers hardness of the DMLS material was found to be 230 HV 20 kgf. Microhardness tests were carried out on the dendritic structure and the non-dendritic structure regions. The specific location for the hardness test was required to be free from cavities and inclusions. The average microhardness values were 381 ± 30 and 260 ± 15 HV 5 gf for the dendritic and the non-dendritic regions respectively.

4. Discussion

In the present paper the features of the laser sintered metal are discussed in the terms of layer structure, heterogeneity and solidification. The available information of similar material and processing routes is scarce [4,6–12]. In general, though,
non-porosity and homogenous structure on the layer thickness level are still unsurpassed in DMLS and are described more closely below.

4.1. Layer structure and microstructural waviness

When the laser beam melts a layer of powder to the lower layer, seams between two layers are created. These seams, in the present study, were rich of pores and contaminations giving the evidence of obvious interface boundaries and the layer texture, Figs. 11 and 13. The investigation of DMLS copper alloys [12,19] showed the same phenomenon, but with much higher porosity 30–45% [12] and 23% [19].

The microstructural waviness may be explained by consideration of the interactive zone of the laser beam with the metal powder. The powder melts when the laser beam is absorbed with a highest temperature at the center and a lower
temperature at the edge of the beam spot. Due to the surface tension, the melted liquid attempts to form a liquid ball and merge with the substrate. The subsequent scanning of the laser beam introduces a slight balling line on the working layer illustrated in Fig. 20b. With a continued scanning of the laser beam, more balling lines are set up on the structure surface as shown in Fig. 20c. Those balling lines are connected with each other only at certain points to form sintering necks around its contour. Hence, pores are present between sintering necks. Because of gravity, merging of upper and lower layers is easier with bigger contact area and less porosity. After sintering, the flat powder layer is changed into a wavelike solid layer with a ‘wavelength’ equal to the hatching distance. The surface profilometry measurement also gives the evidence of such a repetitive spacing between adjacent minima on the last sintered layer.

The existence of microstructural waviness is harmful to the dimensional accuracy and the surface roughness of laser sintered components. In addition, the balling wave has a deleterious influence on the material’s mechanical properties due to its effect of inducing pores.

4.2. Microstructural heterogeneity

The blend of three kinds of powder, Figs. 3–5, was sintered into a solid material. Comparing to the other DMLS materials [12,21], the studied material has finer layer thickness and lower porosity which will result in a better mechanical property [22]. However porosity and microstructural heterogeneity are hard to avoid. No matter how evenly the powder is mixed, heterogeneity always exists in the microscale due to the different volume of the particles. Comparing the laser spot size (0.4 mm) with the maximum particle size (0.178 mm), it is recognized that both are of the same order. After a local fusion of particles, the heterogeneity cannot be eliminated for that the diffusion region is too small and the existence time of liquid
phase is too short. Some unmelted particles are often found in the sintered body as in Figs. 18 and 19, which is consistent with the results of DMLS copper-based alloy [12]. In Fig. 21, the heterogeneity was revealed using SEM element mapping analysis. The dark grey zones in the BSD image, Fig. 21a, are indicated iron-rich. The distributions of phosphorus, nickel and copper are equally obviously depending on the distributions of the pre-sintered metal powder. For instance, the nickel rich area in Fig. 21c indicates more nickel particles were located there than elsewhere.

The influence of particle packing on the heterogeneous microstructure could be minimized by refining the metal powder, increasing laser power and decreasing scan speed to provide more fusion heat and keep elemental diffusion faster and longer. Increasing the beam size could also make the material more homogenous, but at the cost of dimensional accuracy.

Fig. 21. Element mapping showing heterogeneity of DMLS material. (a) BSD image. (b) Iron mapping. (c) Nickel mapping. (d) Copper mapping. (e) Phosphorus mapping.
4.3. Solidification

Comparing with selective laser sintering [4], the current DMLS has the same working principle using a low melting compound to merge powder particles. In this case, Cu particles have a lower melting point than the others and melt first then flow into the gaps between the particles acting as a binder. Then thermal conduction and elemental diffusion take place between the liquid and the solid phase, which lowers the melting points of Ni and Fe particles. And more liquid phase formed. During cooling, solidification of the Fe–Ni–Cu–P system is complicated, and in particular for laser sintering where the content of every component is very localized. In Fig. 22, the Cu–Fe–Ni ternary miscibility gap is described and the intersection of the three dashed lines denotes the average chemical composition of DMLS material from Table 1. Around 800°C above, the three elements are completely miscible to form one phase. As the temperature is lowered below 600°C, the two phases γ1 (FeNi) and γ2 (Cu) become stable. Consistently, a Cu-rich segregation phase was observed in Fig. 17. A severe design limitation is induced in the current DMLS material due to the fact that the low melting component (Cu) is rich in phosphorus, an undesired element which deteriorates mechanical properties of most metals [17].

The three-dimensional microstructure of the DMLS material is illustrated in Fig. 23, made from etched optical micrographs. There, the dark parts indicate dendrite structures, and the light parts indicate iron nickel or copper rich zones. The characteristics of the microstructure can be summarized as follows: (i) dendritic eutectic discs are distributed randomly in the layers, (ii) irregular pores are located at particle interfaces, (iii) coin-like pores are located at layer interfaces, (iv) the layer matrix is a mixture of a Cu-rich phase, a Ni-rich phase, a Fe-rich ferritic phase and a dendritic phase.

5. Conclusion

Based on the study presented in this paper, the following points are stressed:

1. The iron-based laser sintered material has a density of 7.73 g cm⁻³ and an average porosity of 2.6%. The porosities of sections evaluated parallel and normal to the building directions were 2.1 ± 0.2% and 3.1 ± 0.2% respectively.
2. The presence of three kinds of particles, iron, nickel and copper particles with different morphologies and size distributions affects the laser sinter process optimization.
3. Due to the relatively small thickness of the sintered layers and the large laser spot size, the top-view sintered surface normal to the building direction has a coarser topography ($R_a 18.2 \mu m$) than the side-view surface parallel to the building direction ($R_a 12.6 \mu m$).
4. Building layers piling up and the layer thickness were clearly demonstrated in the polished side-view cross-section, consistent with the thickness of the dispersed powder layers.
5. The location and morphology of pores are sensitive to the laser building direction. The pores on the top-view have a bigger mean pore area ($120 \pm 280 \mu m^2$) and a smaller aspect ratio ($1.6 \pm 0.5$) than the pores on the side-view with the mean pore area of $57 \pm 114 \mu m^2$ and the aspect ratio of $2.1 \pm 0.9$. The layer interfaces were mainly preferable sites for pores.
6. Microstructural waviness with an average ‘wavelength’ of about 300 μm was observed on the polished side-view cross-section and the last sintered top-view surface. It can be explained by the laser hatching distance and a balling phenomenon.
7. The sintered material is heterogeneous with the mixture of different microstructural phases: (i) Fe, Ni, Cu, P eutectic, (ii) Fe, Ni, Cu austenitic, (iii) Cu-rich, (iv) Fe-rich ferritic, and (v) Ni-rich phases. The corresponding microhardness of different phases also shows its heterogeneity with a mean microhardness $381 \pm 30 \text{ HV } 5 \text{ gf}$ of the dendritic regions and $260 \pm 15 \text{ HV } 5 \text{ gf}$ of the non-dendritic regions.
Acknowledgement

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References