Metallurgical Evaluation of Laser Sintered M3/2 HSS Powder

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Keywords: High speed steel, M3/2 steel powder, Rapid Tooling, DMLS.

Abstract. Direct Metal Laser Sintering (DMLS) is one of the leading commercial rapid tooling (RT) technologies that produce 3D fully functional parts and tools directly from a CAD model. High Speed Steels (HSS) have the desired combination of hot hardness, wear resistance and toughness needed for applications such as cutting tools, special tools, inserts and dies that can be successfully developed in a near net shape by DMLS.

This paper presents a study of DMLS of commercial water atomized M3/2 powder with different apparent densities. The effect of apparent density and DMLS process parameters on the surface morphologies and microstructure of laser sintered M3/2 powder was analysed and the optimum processing conditions to reduce balling effect were identified.

Laser sintered M3/2 HSS with energy densities between 2.4 and 12 J/mm² produced a coarse roughness (Rz) ranging from 135 to 234µm. The lowest roughness (65µm) was obtained with 36J/mm², the highest laser energy density value used. The microstructure of laser sintered M3/2 HSS consisted of austenite, martensite and a fine carbide structure.

Introduction

Direct Metal Laser Sintering (DMLS) is a technology that enables the fabrication of true net-shape parts in just a few hours. In this process, a CO₂ laser is scanned across the surface of a loose powder bed, sintering and/or melting the powders into the shape of the required cross section, launched by CAD model. The part is built up layer by layer from the bottom to the top. Thus, it is a sequential layered approach to manufacture any desired 3D part that may have a simple or complex shape [1].

The research on the DMLS method was mainly focused on the technique of producing parts with a high accuracy and selection of suitable materials. Some efforts have been devoted to the surface morphologies and microstructural characterization. The iron-based laser sintered material showed a microstructural waviness and a heterogeneous microstructure [2]. A fully dense surface of gas atomized (GA) M2 high speed steel (HSS) sintered was obtained with laser power ranging from 40 to 80W, 0.15mm of scan line spacing and laser scan rates ranging from 1 to 25mm/s. The sintered microstructure of GA M2 HSS consists of a ferritic matrix with grain boundary carbides of M₆C and M₄C₃ [3].

The surface morphology of laser sintered GA M2 HSS powder at a higher laser power (200W) was almost fully dense for specimens processed at 75mm/s and columnar agglomerates parallel to the scan direction were formed when the scan rate is increased to 125mm/s. Additionally, the formation of metallic balls at columnar surface and large pores between these columns were observed. The microstructure of laser sintered at scan rate of 75mm/s consisted of austenite, martensite and fine carbides structure. Fine cells (or dendrites) delineated by an intercellular (or interdendritic) network of carbides were observed [4]. DMLS of water atomised (WA) M3/2 HSS powder with different particle sizes tends to produce poor surface density with large agglomerates and inter-agglomerate pores [5]. The agglomerate size increased with increasing laser power or decreasing scan rate. These
two process parameters have a large effect on the sintered M3/2 microstructure. The carbides change from the angular carbide at the three grain junction to a necklace along the grain boundaries [5].

This study describes a way to enhance the apparent density and grain size distribution of the commercial WA M3/2 powder. The modified powders were processed by DMLS technology to evaluate the effect of these properties on the surface morphologies and microstructures of sintered material. Different levels of laser power, laser scan speed and hatch spacing (distance between scanning lines) were used to identify the values that could lead to final surfaces with good continuity of melted material. Final surface evaluation was made with the measurement of surface roughness.

**Experimental Procedure**

The laser sintering experiments were carried out on EOSINT M250 Xtended equipment, using a CO₂ laser with 250W of maximum power and 0.40mm spot size. Further information about this machine and DMLS process is available in another paper [6]. The loose powder was manually placed on a steel plate attached to the building platform and then leveled with the machine recoater. Laser sintering of each layer was performed using the “single exposure” feature in X direction in areas with 15X15mm. The DMLS process parameters and the calculated energy density used in these experiments are shown in Table 1 [7].

<table>
<thead>
<tr>
<th>Trials</th>
<th>T1</th>
<th>T2</th>
<th>T3</th>
<th>T4</th>
<th>T5</th>
<th>T6</th>
<th>T7</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser power (W)</td>
<td>154</td>
<td>180</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Scan speed (mm/s)</td>
<td>50</td>
<td>150</td>
<td>250</td>
<td>50</td>
<td>150</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>Hatch spacing (mm)</td>
<td>0.30</td>
<td>0.10</td>
<td>0.30</td>
<td>0.30</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Layer thickness (µm)</td>
<td>4 x 60</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Laser energy density (J/mm²)</td>
<td>10.3</td>
<td>3.4</td>
<td>2.1</td>
<td>36.0</td>
<td>12.0</td>
<td>4.0</td>
<td>2.4</td>
</tr>
</tbody>
</table>

Commercial M3/2 HSS powder (Höganäs, Sweden) was produced by water atomisation and annealed. Chemical composition of M3/2 HSS powder is in Table 2 [7].

<table>
<thead>
<tr>
<th>Composition</th>
<th>C</th>
<th>Mo</th>
<th>Si</th>
<th>Mn</th>
<th>W</th>
<th>Cr</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>M3/2 HSS</td>
<td>1.11%</td>
<td>6.26%</td>
<td>0.30%</td>
<td>0.11%</td>
<td>5.00%</td>
<td>3.77%</td>
<td>2.68%</td>
</tr>
</tbody>
</table>

The M3/2 powder was milled at room temperature in a planetary high energetic Fritsch mill at 200rpm during 11h. The next step consisted of blending the sieved powder (<63µm) with 10 and 30 wt. % of the milled M3/2 powder in a Turbula mixer during half an hour. The trials were identified as 10T, 30T and 100T, when mixtures have 10, 30 or 100 wt. % of the milled M3/2 powder, followed by a number that identify a set of DMLS process parameters (Table 1). The details of the preparation of the M3/2 powders are reported elsewhere [7]. Apparent density and particle size distribution of the powders are shown on Table 3.

Laser sintered surfaces were characterized using roughness Rz and the tests were performed on a Perhometer S2 Mahr apparatus using the following conditions: a traversing length (Lt) of 13mm and a cut-off (Lc) of 1.875mm, since the maximum length of the samples were 15mm. The roughness was measured normal to the building direction.

The laser sintered samples for metallographic examination were prepared using standard techniques and etched with 5% Nital. Microstructural evaluation was made by optical and
stereoscopic microscopy, supported by other techniques like Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS) analysis and X-Ray Diffraction (XRD) using Cu-Kα radiation.

Table 3 - Apparent density and particle size distribution of M3/2 powder [7].

<table>
<thead>
<tr>
<th>Material</th>
<th>Apparent density (g/cm³)</th>
<th>D₁₀ (µm)</th>
<th>D₅₀ (µm)</th>
<th>D₉₀ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M3/2 commercial</td>
<td>2.53±0.02</td>
<td>27</td>
<td>62</td>
<td>116</td>
</tr>
<tr>
<td>M3/2 &lt; 63µm</td>
<td>2.43±0.02</td>
<td>24</td>
<td>42</td>
<td>79</td>
</tr>
<tr>
<td>M3/2 Milled 11h</td>
<td>3.25±0.01</td>
<td>9</td>
<td>27</td>
<td>57</td>
</tr>
<tr>
<td>90%M3/2&lt;63µm+10%M3/2 Milled 11h</td>
<td>2.43±0.02</td>
<td>22</td>
<td>44</td>
<td>76</td>
</tr>
<tr>
<td>70%M3/2&lt;63µm+30%M3/2 Milled 11h</td>
<td>2.61±0.02</td>
<td>17</td>
<td>42</td>
<td>73</td>
</tr>
</tbody>
</table>

Results and Discussion

It is clear that the powder size and powder size distribution have a significant role on apparent density (Table 3). On this work a higher relative apparent density was achieved by mixing different sizes of powder. The principle involves using finer particles to fill the voids formed by the larger powders [8]. The M3/2 HSS D₅₀ (27µm) was reduced after milling and consequently the apparent density increased. The mixture of these fine particles (10 and 30 wt. %) with M3/2 HSS sieved at 63µm allowed the production of new powders with a lower D₅₀, a narrow particle size distribution and a higher apparent density. These properties are more favorable to the DMLS process than those of the commercial M3/2 powder [7].

The areas built with the mixture 30T (30 wt. % M3/2 milled) need at least 3.4 J/mm² to get a sintered surface. The measured roughness Rz was in the range between 227µm and 234µm (Fig.1). When the laser energy density was increased to 12 J/mm², Rz decreased to 135 µm. In this trial the gaps between the “balls” were almost filled by the melted material, which lead to a lower roughness (Fig.2a). There were no attempts to produce sintered surfaces with the lower energy densities (30T3 and 30T7).

Fig.1 – Roughness Rz of the sintered surfaces as a function of laser energy density.

The morphology of the 30T4 sintered surface (36J/mm²) with a hatch spacing of 0.10mm is quit different from those produced with a hatch spacing of 0.30mm. The measured roughness Rz was
65µm (Fig.1). This lower value is a result of the high laser energy density used in this trial, which produced a sintered surface with fewer and smaller gaps (Fig. 2b).

The energy density needed to sinter the powders increases with the increase of apparent density and decrease of D_{50} and also D_{10}. In fact, the greater surface area of finer powders leads to higher sintering activity and thereby faster sintering rate (Table 3). The behavior of 30T follows this reasoning.

The mixture 10T (10 wt. % M3/2 milled) needs at least 2.4J/mm² to have a sintered surface. The measured roughness Rz was in the range between 199 and 233µm. The sintered surface in trial 10T3 (2.1J/mm²) presents some areas without any melted material.

The trials with M3/2 milled (100T4 and 100T5) produced sintered surfaces with a high roughness (Rz: 218-224µm). However, sintered surface of 100T5 shows a lower balling effect than 100T4 (Fig. 3a vs Fig. 3b). The higher energy density used in trial 100T5 (12J/mm²) can explain this effect. The trials with less than 4J/mm² of energy density produced uncompleted sintered layers.

The M3/2 milled had the best apparent density and the finer D_{50} and also D_{10} but its behavior was not the expected due to agglomeration [7] and probably to powder oxidation during the milling.

Laser sintered 30T4 (36J/mm²) shows a typical microstructure consisting of austenite and martensite detected by X-Ray Diffraction [7] and a very fine carbide structure that delineates the grain boundaries (Fig.4a). These carbides are rich in chromium, tungsten, molybdenum, iron and vanadium and were identified by EDS (Fig.4b). The grain size was 1.9±0.3µm, measured on the micrographs.

Similar microstructures were obtained by other researchers in laser sintered M2 HSS [9]. The microstructure of commercial M3/2 laser sintering (T5: 12J/mm²) comprised a fine “as cast” structure consisting of M₂C and M₆C eutectics at the cellular-dendritic grain boundaries and a matrix with martensite and austenite [7].
Conclusions

M3/2 HSS powders with different apparent densities have been sintered by DMLS using a single-direction scan. The findings can be summarized as follows:

- The M3/2 milled powder had the lowest $D_{50}$, the highest apparent density and the narrow particle size distribution among the powders studied.
- The M3/2 milled and the mixture 30 wt. % M3/2 milled sintered surfaces with laser energy density of 12J/mm$^2$ were continuous with reduced balling effect.
- The highest laser energy density (36J/mm$^2$) produced a surface with a smoother morphology (Rz: 65µm).
- The typical microstructure consisted of very fine carbides around the grains and a matrix with austenite and martensite.
- The higher Rz values restrict the building of volumetric samples.

Acknowledgments

Project INATEC – Innovation, Agility and Technology in Moulds supported by PRIME Program. The authors are grateful to INETI and to all the persons involved in technical support.

References