Direct selective laser sintering of iron–graphite powder mixture


a Department of Mechanical Engineering, Indian Institute of Technology (IIT), Kharagpur 721302, India
b Department of Metallurgical and Materials Engineering, Indian Institute of Technology (IIT), Kharagpur 721302, India

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Abstract

In the present work, laser sintering of a powder mixture of iron and graphite (99.22 and 0.78%, respectively) was carried out using a pulsed Nd-YAG laser. The paper reports experimental details with evolution of the microstructures and evaluation of some of the physical and mechanical properties of the resultant sintered material. The obtained results indicate suitability of the laser sintered material for some special applications.

Keywords: Laser sintering; Rapid prototyping; Iron powder

1. Introduction

Rapid prototyping (RPT) has been termed by many manufacturing scientists and engineers to be the ultimate solution to manufacturing whereby complex, convoluted and near-impossible shapes can be modeled and developed within a very short period. Selective laser sintering (SLS) [1,2], micro-cladding [3,4] and ballistic particle manufacturing (BPM) [5,6] are among the recently developed technologies that offer the possibility of direct fabrication of metallic parts of arbitrary geometry in a single-step process.

SLS is the process of melting a layer of powder by a laser beam, which scans selected regions of the layer. The powder in those regions melts and gets consolidated. After consolidation of one layer, another layer of powder is applied and the cycle is repeated. In such an exercise, the successive layers of solidified material formed in each cycle get joined to each other during the process and ultimately a solid form emerges, which can be used for form visualization, design feedback, functional tests and other applications.

The materials which can be used for developing laser sintered solid freeform fabricated (SFF) products are thermoplastics, polymer coated low carbon steel powder, bronze–nickel blend, pre-alloyed bronze powder etc. [7–9]. The products developed with these materials suffer from low mechanical strength and durability. They can hardly, in most cases, be employed for functional prototypes of engineering components. In some cases, post-processing operations are necessary for achieving full density [10]. Single metal powders have been proved to be difficult materials for SFF by laser sintering since in molten form, they tend to ‘ball up’ [2,7,11,12]. Solutions to this problem have been tried out by either pre-alloyed single phase powder system or a powder mixture of two phases with different melting temperatures instead of one so that one phase melts while the other remains solid. This actually means that liquid sintering takes place where one metal acts as the binder [7].

In this investigation, it was attempted to produce and study an innovative metallic microstructure from a mixture of iron (99.22%) and graphite (0.78%) powder by laser sintering employing a pulsed Nd-YAG laser. The evolved microstructure and some physical and mechanical properties were studied and are reported here.

Not much work has previously been reported specifically on the laser sintering of an iron and graphite powder mixture. However, a substantial amount of work has been carried out in the field of laser sintering of metal parts, some of which are as follows.

An extensive review has been made by Agarwala et al. [2] of producing parts by direct SLS of metals. Past efforts had been concentrated on SLS of Cu–Sn, Cu–solder, Ni–Sn, etc. Best results were obtained in the cases of metal powder blends with constituents of varying melting behavior. It has been observed that presence of partial liquid and solid phases during processing results in smooth and strongly sintered layers with minimum balling. Some experimental
work has also been reported by Song [7] on direct sintering of pre-alloyed bronze as a low melting metallic powder on a laboratory test facility. The experimental investigations with single spots, lines and layers on the powder bed indicate successful direct sintering of bronze powder without the use of polymer binder or preheating. It was concluded that laser beam power, scanning speed and hatching distance (the side step taken by the laser after every scan line, Fig. 1) exert an influence on the part quality characteristics.

Kathuria [8] has discussed some novel features of the fabrication of metal or metal matrix composite parts using CO₂/Nd-YAG lasers. The basic process of beam interaction with the metallic powder was investigated and results concerning the microstructure and its shape factor have been interpreted. According to O’Neill et al. [13], in processing metallic materials, porosity is still a major problem although a number of notable solutions, such as infiltration with low melting point alloys have been proposed. The work examined the feasibility of using low energy but high peak power laser pulses from a Q-switched Nd-YAG laser to melt stainless steel powder fractions whilst examining the melt displacement and the effect of rapid vaporization of the powder layer.

Schueren and Kruth [12] have performed experiments on sintering of Fe–Cu powder mixture using an Nd-YAG laser and have proposed the same melting/densification mechanism of using a lower melting point component. They have also made an extensive study of the influence of powder properties on deposition.

Pham et al. [14] has focused on the applications of some of the parts developed by the SLS process and, by means of case studies, has explained the technological capabilities of the process. Abe et al. [15] has reported that laser sintering of nickel base alloys (die materials) does not exhibit balling up phenomenon (like aluminium and stainless steel powders) but defects like deflection and cracking are evident. These defects can be reduced by employing a dual laser system scanning system.

Niu and Chang [17–20] have reported on the SLS of HSS powder with a carbon di-oxide laser of 25 W. They concluded that the dominant mechanism of the SLS process changes from solid phase sintering to liquid phase sintering to full melting/solidification with increasing laser power. Marangoni flow in the melt pool affects the morphology of the sintered tracks produced at high laser powers. The agglomerate size was also seen to increase with increasing laser power or decreasing scan rate. It was also found that powder size less than 38 μm tends to give less dense structure due to the presence of high oxygen level. They are of the view that surface tension gradient is the key factor responsible for balling up or break up the liquid cylinder.

2. Experimental details

The set-up consists of an Nd-YAG laser (pulsed, average power 100 W) retrofitted to the table of a CNC milling machine where the head of the milling machine has been replaced by the laser. An inert gas chamber (IGC) made of Perspex, which houses the powder bed, has been mounted on the table of the CNC machine (Fig. 2). The laser enters through a quartz window mounted on the top surface of the chamber. Argon gas is fed into the IGC through a brass nozzle at a rate of 5 l/min in order to drive out the air from the chamber and maintain an inert atmosphere. The powder bed is in the form of a drawer that can be slid out of the box for depositing the layer.

![Fig. 1. Laser sintering nomenclature.](image)

![Fig. 2. Inert gas chamber.](image)

<table>
<thead>
<tr>
<th>Parameters selected for experimentation</th>
<th>Selected values</th>
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</thead>
<tbody>
<tr>
<td><strong>Process parameters</strong></td>
<td></td>
</tr>
<tr>
<td>Hatching distance (μm)</td>
<td>500</td>
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<tr>
<td>Layer thickness (μm)</td>
<td>500</td>
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<tr>
<td>Step size (μm)</td>
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<tr>
<td>Inert gas flow rate (l/min)</td>
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<td><strong>Laser parameters</strong></td>
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<tr>
<td>Spot size (μm)</td>
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<tr>
<td>Wavelength (μm)</td>
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<tr>
<td><strong>Material parameters</strong></td>
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<tr>
<td>Mean size of iron powder (μm)</td>
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</tr>
<tr>
<td>Percentage of iron powder</td>
<td>~99.2</td>
</tr>
<tr>
<td>Percentage of graphite powder</td>
<td>~0.78</td>
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</table>
After the metal powder is deposited on the powder bed, the bed is placed inside the IGC. Before starting processing of the metal powder, argon gas is continuously fed through the inlet nozzle to create and maintain a non-oxidative environment. Thereafter, sintering is allowed to take place by scanning powder along a predetermined path with the laser beam. Table 1 lists the values of the process, laser and material parameters which have been used in the present investigation.

2.1. Procedure

A homogeneous mixture of 99.22 wt.% of iron powder (with mean particle size of 53 μm) and 0.78 wt.% of graphite powder was prepared. A powder layer (the first layer) of the required thickness was applied over a mild steel substrate of 10 mm × 10 mm cross-section with the help of scraper and slip gauges. In order to level the powder, the scraper was slid on top of two stacks of slip gauges (equal in height to layer thickness) kept on two sides of the powder. After that the bed is put back into the Perspex chamber exactly below the quartz window as shown in Fig. 2.

After a laser pulse was fired at a particular location, the table was moved to a new position in the XY plane and the operation was repeated (point-to-point CNC control). In this way the entire cross-section was completed. After one such layer was sintered, the bed was taken out for another layer to be deposited over it. The vertical distance of the powder bed with respect to the laser was retained by lowering the platform by the distance of the layer thickness. The sintering process was then repeated. The process continued until the specified number of layers was built up to form a body of desired thickness. Fig. 3 shows sintered samples in different views. The top surface was ground, as shown in Fig. 3(c), for conducting various tests.

The following tests were carried out on the laser-sintered sample:

- Microstructural observations,
- Measurement of density,
- Macrohardness test (HVN),
- Microhardness test,
- Wear test.

3. Results and discussion

3.1. Microstructural observations

The laser-sintered samples were polished, etched and examined under the optical microscope. The samples were also observed under scanning electron microscope (SEM) in order to identify the constituent phases with better resolution.

In the optical micrographs (Fig. 4(a) and (b)), there are circular traces, which represent the boundaries of small metallic balls formed from melting of the powder. Due to high surface tension, the molten metal balls up. These balls solidify very quickly due to high cooling rates. However, the micrographs show that they have probably undergone
remelting during subsequent laser passes (during the sintering of the upper layers) and have apparently solidified as a single solid body with only the traces of their spherical boundaries in evidence. The SEM micrograph at low magnification (Fig. 5) shows similar sintered globules overlapping each other.

On further examination of the structure at higher magnification under SEM, different morphological features of martensite (Fig. 6) were evident at the core and the periphery of the globule. Though martensite had formed both at the core and the periphery, it varied in the size of the needles at the two locations. It is further expected that the volume fraction of retained austenite at the periphery would be higher than that at the core. Such differences are reflected in the differential etching characteristics of the two regions. Needle-shaped martensitic structures were clearly visible on both the boundary as well as in the core of a globule (Figs. 7 and 8).

The process of laser sintering by pulsed laser is characterized by localized heating with short interaction time of a few milliseconds. Due to the action of laser pulse, iron powder, which is having much lower melting point
(1537 °C) than graphite (>3500 °C), melts and balls up due to high surface tension and viscosity. It is unlikely that substantial amount of graphite, if at all any, gets melted down by the laser pulse. Thus, the iron globules so formed are having graphite particles at their periphery. Some graphite particles could, however, be engulfed inside the iron globules. Due to the high temperature and associated high diffusion rate, graphite tends to diffuse into the iron globules. The degree of diffusion of graphite depends on the temperature, the laser pulse width and the graphite particle size. As most of the graphite would be outside the globules, diffusion of graphite will be more near the periphery and less at the core of the globule. Rapid quenching due to the high cooling rate leads to formation of hard martensite. Auto-tempering of martensite (by overlapping of laser pulses and the effect of laser pulses for the formation of next layer) is also evident at some locations from the micrographs (Fig. 9).

The martensite matrix is found to be interspersed with several black near-round and oval shaped patches. These patches appeared to be undissolved graphite but they could also be voids or cavities. In order to clear this confusion, further examination under SEM revealed that some of them were in fact voids. This suggests that the sintering process that has been carried out did not yield a pore-free matrix. Subsequent measurement of density substantiates this observation. However, the voids were small in size (20–150 μm).

When the particle size of graphite powder is substantially large, only a portion of it gets diffused during the small time of interaction and the rest remains embedded in a tempered martensite matrix (Fig. 10(a) and (b)).

Fig. 11 is a SEM micrograph, which reveals both the martensitic structure and the boundary of some pores present in the matrix. It is observed with sufficient clarity that the pores or cavities contain some inclusions. By energy dispersive X-ray analysis, it is confirmed that such inclusions are basically various oxides and silicates of aluminium, calcium and magnesium, which originate from the trace amounts of impurities present in the iron and graphite powders. These solid inclusions remain non-reactive and get trapped in the molten matrix. The cavity is created due to non-uniform shrinkage when the molten metal solidifies. Under intense thermal stress these inclusions crack up in the cavity itself during fast cooling (Fig. 12).

The iron–graphite powder mixture, which has been used for the investigation, contained 99.22% iron and 0.78% carbon. After sintering, the carbon content of the resultant solid product was found to be only 0.3%. It is possible that some amount of graphite gets oxidized inside the sintering chamber due to the presence of trace amounts of oxygen (present in the inert gas and dissolved in iron powder) or gets vaporized off. Graphite powder might even get ejected due to explosive pressure of emanating metal vapour or simply

Fig. 9. SEM micrograph showing tempered martensite.

Fig. 11. SEM micrograph showing cavity formation.

Fig. 10. Optical micrographs showing graphite inclusions in the sintered specimen at two different magnifications.
due to radiation pressure. Graphite, being lighter as compared to iron powder, is more prone to this expulsion from a homogeneous mixture of iron and graphite powder.

3.2. Density

The density of the sintered sample formed out of an iron and graphite powder mixture was found to be 7.127 g/cm$^3$ using Archimedes’s principle. This value appears to be low compared to the density of pure iron metal powder (7.84 g/cm$^3$). This observation suggests that the sintering process probably led to the formation of some voids inside the sample.

3.3. Macrohardness test

The hardness test was performed with the help of hardness testing machine, H.P.O-250, using a load of 5 kg and an indentation time of 15 s. The HVN of the sintered sample was found to be 254, which corresponds to HRC 24.

3.4. Microhardness test

Microhardness values measured at different regions of the sample are tabulated in Table 2. The microhardness values are taken with a load of 100 g and indentation time of 15 s. The locations at which the hardness tests have been carried out are shown by symbols in the micrograph (Fig. 13).

It can be easily seen that the microhardness values are much higher than the macrohardness value. This further suggests the existence of voids inside the sintered sample. Microhardness at specific locations (free from cavities and voids) might be very high but the macrohardness, which would be affected by cavities and voids, is naturally lower. Microhardness values are higher at the periphery of the globule than that in the core. This may be explained from the fact that high and low carbon martensite has formed at the periphery and core of the globule, respectively.

Although martensite formation was evident, hardness of the sample could not be expected to be very high as final carbon percentage was as low as 0.3%. In addition to this, during subsequent laser passes, this martensite has tempered, resulting in further reduction in the hardness of the sintered mass.

3.5. Wear test

The wear tests were carried out on a standard pin-on-disk machine with a continuously rotating low alloy steel plate of hardness 63 HRC as the counter surface, without a lubricant. All the experiments were carried out at loads of 6 and 2 kg and at sliding speeds of 0.58 and 4 m/s. The mass change was measured after careful cleaning of the specimen before and after each test. The specific wear rate = volume abraded/(sliding distance × load applied) is tabulated in Table 3 for different conditions.

The wear rate data are found to be comparable to those reported by Sudhakar et al. [16] on material similar to the

<p>| Table 2 Microhardness values |</p>
<table>
<thead>
<tr>
<th>Location in micrograph</th>
<th>Hardness value, HVN (HRC)</th>
<th>Location in micrograph</th>
<th>Hardness value, HVN (HRC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>348.3 (36)</td>
<td>5</td>
<td>304.7 (31)</td>
</tr>
<tr>
<td>2</td>
<td>389.6 (40)</td>
<td>6</td>
<td>540.5 (51)</td>
</tr>
<tr>
<td>3</td>
<td>352.0 (36)</td>
<td>7</td>
<td>524.0 (50)</td>
</tr>
<tr>
<td>4</td>
<td>334.2 (34)</td>
<td>8</td>
<td>488.6 (48)</td>
</tr>
</tbody>
</table>

| Table 3 Results of wear tests |
|-----------------------------|----------------|
| Velocity                    | 0.58 m/s | 4.0 m/s |
| Load = 2 kg                 |           |         |
| Weight loss (g)             | 0.0069   | 0.0200  |
| Sliding distance (m)        | 3723.6   | 14400   |
| Specific wear rate (mm$^3$/N m) | 7.9E−06 | 6.25E−06 |
| Load = 6 kg                 |           |         |
| Weight loss (g)             | 0.0337   | 0.0385  |
| Sliding distance (m)        | 2088     | 14400   |
| Specific wear rate (mm$^3$/N m) | 0.17E−06 | 3.1E−06 |

Fig. 12. SEM micrograph showing cracking up of an impurity in the cavity.

Fig. 13. Location of the microhardness measurements.
one developed as regards raw material (powder material) and hardness. Sudhakar carried out wear tests on high density Fe–2Ni–0.2C P/M alloy (hardened and tempered, 27 HRC) at a constant load of 5 kg and a sliding speed of 2 m/s. The specific wear rate of this alloy (Fe–2Ni–0.2C) is reported to be 1.95E–06 mm³/N m.

4. Conclusion

Laser sintering, like conventional powder metal sintering, does give rise to porosity. These pores are a consequence of ball formation and entrapment of air in melting powder mass. Remelting by subsequent pulses reduces this porosity. Due to the presence of porosity and presence of graphite in the sintered specimen, its macrohardness is less than its microhardness. Laser sintering, as carried out in this investigation, shows carbon content in the sintered sample to be as low as 0.3% in comparison to initial carbon content of 0.78%. Hence in order to achieve a targeted carbon content, a powder mixture of higher graphite content must be used. Hardness of the sample cannot be expected to be very high as carbon percentage was quite low. In addition to this, martensite gets tempered at some zones by subsequent laser pulses, which would further reduce the hardness of the sample. To have graphite inclusions in the sintered material, the particle size of graphite has to be substantially large so that only a portion of it gets diffused and the rest gets entrapped into the matrix.

It can be concluded that laser sintering of iron and graphite powder produces a material, which is substantially different from the same produced by conventional sintering. The sintered material with tempered martensite matrix has moderately high hardness. The presence of graphite inclusions suggests the possibility of formation of graphitic steel with good wear characteristics. Lastly, the inherent disadvantage of SLS (i.e., porosity) may be a point of advantage for making porous bearing-like materials. The pores, which are inherently formed in SLS, can act as oil retainers and help in damping vibration. Further research work in this direction is necessary to explore these possibilities.

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