Optical absorption and conduction of copper carbon nanotube composite for additive manufacturing

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Abstract. The applications of nanotechnology are growing widely as nanoscale structures provide unique properties such as high surface area unique plasmonic response and excellent conductivity that can be utilized for a wide range of applications. Optical absorption expansion of copper using carbon nanotube composite is one of the applications of nanotechnology for additive manufacturing of metals, particularly copper. Cu-CNTs mixtures at different percentage concentrations will be prepared via Resodyn, an acoustic mixer machine. Pure copper powder will be used with a spherical powder shape. The evaluation of the samples will be performed via spectroscopy to determine the reflection and thermal absorption of the light by the Cu-CNTs composition. The enhancement in the thermal absorption of Cu powder via additions of CNTs leads to the improvement in the bonding of particles by absorbing laser power. Due to the lower thermal expansion coefficient, sintering is possible at lower laser powers < 40%.

Introduction

Copper is one the unique metals having ductility and exceptional electrical and thermal conductivities of 58×10^6 S/m and 400 W/m.K [1-3] Therefore, many engineering applications are possible due to these properties such as heat exchangers and heat sinks, thermal energy storage systems, etc., [4,5]. Moreover, heat transfer is further enhanced by making some geometrical changes by making fins, extended structures, and porous structures[6]. Metal Additive manufacturing (AM) has made it possible to manufacture complex structures with high accuracy and less material loss. Therefore, several complex components are being manufactured using different metals [7,8]. Despite having favorable properties, copper has the disadvantage of having high reflectivity which makes it difficult to manufacture using AM techniques such as L-BPF. The reflectivity of the copper can be reduced by increasing the absorption of the copper powder.

Considerable work is noted in the literature on additive manufacturing of copper powder. Lykov et.al [9] performed the additive manufacturing of pure copper powder using selective laser melting with a laser power of 200W. They manufactured cuboidal shape geometries and measured the maximum achieved relative densities. A maximum of 88% relative density was reported at 200W of the laser power. Jadhav et.al [10] also used pure copper to produce parts with a laser power of 300W. In their work lower relative densities were achieved due to insufficient diffusion of heat within the copper particles. Due to very high thermal conductivity, the heat was dissipated instantly from the melt pool. The issue of the lower laser power was addressed in numerous studies using high laser power up to 1kW which resulted in 96% relative densities of manufactured parts. However high laser power resulted in significant reflectivity due to that damage to the optical mirror [11–13]. In solution to this problem, the green and blue lasers were also by various authors

[14]. The remedy to address this problem is either by playing with laser parameters such as laser power and scan speed etc. or by modifying the absorption rate of the copper powder.

In the present work, Cu-CNTs powder mixture with CNTs having percentage concentrations in the range of 0.1%-0.3% CNTs was prepared using a Resodyn, an acoustic mixer machine. The cold isostatic hydraulic pressing method was used to fabricate the copper pellets. The copper powder was used with a spherical shape[15]. The evaluation of the samples was performed via spectroscopy to determine the reflection and absorption of the light by the Cu-CNTs composition. The enhancement in the thermal absorption of Cu powder via additions of CNTs leads to the improvement in the bonding of particles by absorbing laser power. Due to the lower thermal expansion coefficient, sintering is possible at lower laser powers < 40%.

Material and Methods

The experimental setup for the laser sintering of carbon nanotubes composite mixed copper powder consisted of Resodyn, an acoustic mixer machine to mix Cu-CNTs in different concentrations. Cu-CNTs pellets were produced via compaction by using Autotouch 40Ton Hydraulic Press. Sintering was performed via fiber laser (I.P.G) having a wavelength of 1064nm and 200W power in a custom made sintering rig capable of maintaining an inert environment to avoid any oxidization and contamination.

The copper powder and CNTs were mixed using Resodyn, an acoustic mixer machine, and compacted using the hydraulic press. The effects of mixing ratios on the quality of samples are investigated experimentally. From Sigma Aldrich, a copper powder with spherical-shaped powder particles was used. Different weight percentage ratios of CNTs were added to achieve higher laser absorption and less reflection. In mixing and compaction, CNTs weight percentage was the operating parameter. To get the same size pellets compaction parameters were the same for all sets of experiments. During the compaction process, a die with a diameter of 20mm is used, which results for each sample, the diameter remains constant.

The Cu-CNTs composite spectroscopy was performed with Perkin Elmer Spectrum Two FTIR a well-known and easy-to-use equipment for spectroscopy.

Table 1. Physical properties of Copper and CNTs.

Product Form	**Particle Size**	**Purity (%)**	**Melting/Boiling (C)**	**Density (kg/m^3)**
Powder (spheroidal)	10-25 μm	98%	1083.4/2567	8960
Carbon nanotube (Multiwalled)	10-20 nm	95%	3652-3697	2200

Compaction of Cu-CNTs.

The present work demonstrates cold isostatic pressing using hydraulic press technology of Cu-CNTs mixtures. Spherical-shaped copper powder is received from Sigma-Aldrich with a purity of 98% having a particle size of 10-25 μm and multiwalled carbon nanotubes are received from Cheap Tubes with a purity of 95% and length of 10-30 μm. Further technical and physical properties of copper powders and carbon nanotubes are listed in Table 1. Cold isostatic hydraulic pressing is a well known method for powder compaction. The compaction process is illustrated in Fig. 1, where the compressive force is applied by moving the piston from the bottom of the compaction die. 20mm diameter Cu-CNTs pellets were produced using this die. The compaction pressure for each pellet was 793 MPa and the compaction holding time was 10 mins.

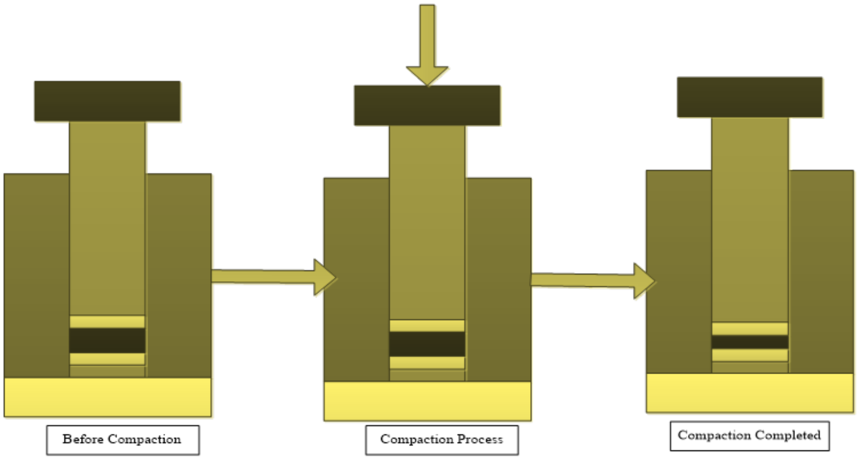


Fig. 1. The Schematic Diagram of Autotouch Hydraulic press compaction process.

Laser sintering.

The laser sintering of the Cu-CNTs composite pellets was carried in controlled temperature and air pressure, using a specially designed laser sintering rig shown in Fig. 2. The laser sintering rig contains two Calex PMU21 USB infrared temperature sensors to record the sintering temperature, UVFS Broadband Precision Window, -B Coated from Thorlabs to allow the laser beam to the sample and Argon gas connection to avoid oxidation and contamination during laser sintering. Fiber laser from IPG of 200W is used for laser sintering having wavelength of 1064nm. The laser sintering parameters are shown in Table 2. The laser spot size is fixed at 5µm.

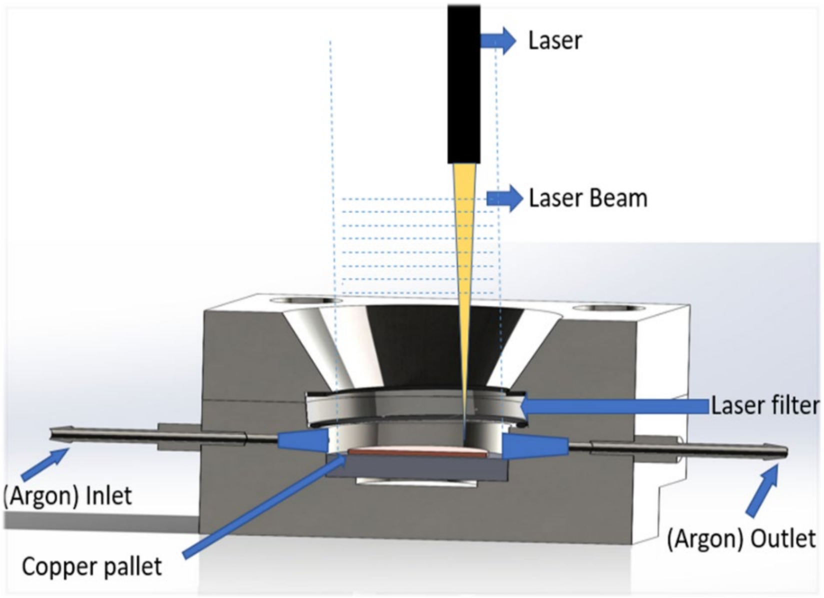


Fig. 2. Schematic of laser sintering setup demonstrating the passage for laser beam, argon inlet. Outlet, laser filter and copper pellet in the inert environment.

Table 2. Laser sintering parameters of sintering which include the laser power, laser spot size, and laser scan speed.

Power Percentage (%)	**Spot size** (μm)	**Scan Speed** (mm/s)
15	5	5
20	5	5
20	5	2
25	5	2
40	5	1
40	5	1
40	5	1
40	5	1
40	5	1

Results and Discussion

Spectroscopy of Cu-CNTs Composition.

Before performing the spectroscopy on Cu-CNTs samples, the reflectance of the pure copper pellet was measured and compared with the broadband mirror.

Fig. 3 presented the reflectance of pure copper where the reflectance of the copper pellet was measured laser wavelength of up to 1070 nm. The reflection and absorption of the Cu-CNTs and the pure copper are shown in Fig. 4. From the spectroscopy of the pure copper powder, it is observed that reflection is 100% at 1064 nm of wavelength. This high reflection of copper powder causes difficulties in laser sintering at low laser power and the reflection of the laser beam can damage the laser source. The addition of multiwalled CNTs reduces the reflection of the copper and increases the laser absorption. From Fig. 4 it can be analysed that the reflection of the Cu-CNTs gradually decreases with the increase of CNTs. The addition of 2% (mass) of CNTs reduced copper powder reflection at 1064 nm of wavelength by 8% compared to the pure powder.

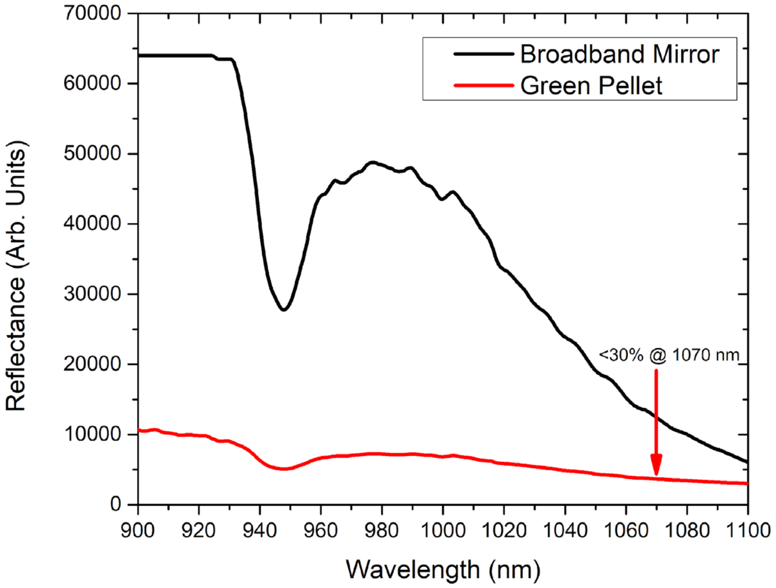


Fig. 3. Comparison of the spectroscopy results of green pellet and broadband mirror.

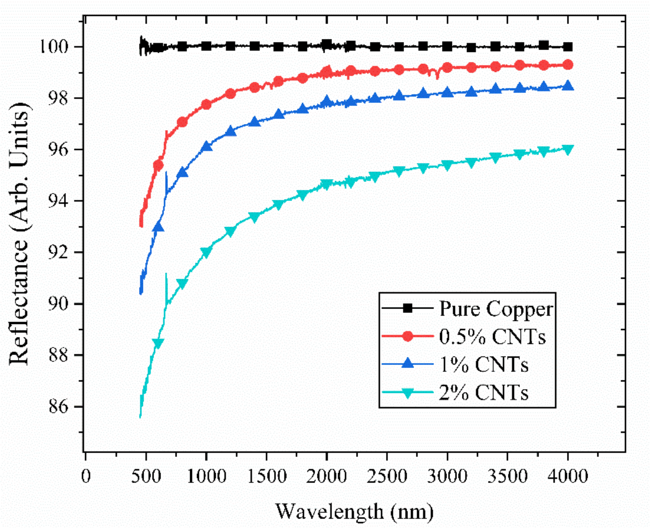


Fig. 4. The results for the reflectance of pure copper powder and Cu-CNTs powder with 0.5%-2% CNTs by mass percentage.

Laser sintering of Cu-CNTs Composition.

The pure copper compacted pellet was first laser sintered in the specially designed laser sintering rig shown in Fig. 2. The pure copper powder compacted pellet was first sintered to adjust the laser parameters. Fig. 5(a) shows the laser sintered pure copper pellet in the laser sintering rig with an Argon gas environment to prevent contamination and oxidation. Fig. 5 (b) shows the laser sintered pure copper pellet in an open atmosphere environment which clearly shows the contamination and oxidation. The laser parameter for pure copper sintering is shown in the Table 2. Fig. 6 (a), (b), (c), and (d) present the maximum pellet temperature for different scan speeds and laser power percentages. Fig. 6 (a) indicates that increasing the power percentage up to 40% and lowering the scan speed of the laser from 5 mm/s to 1 mm/s resulted in the pellet temperature being up to 570°C. Similarly, Fig. 6 (b),(c), and (d) present the temperature rise as a function of laser power and scanning speed for the Cu-CNTs powder containing 0.5%-1.5% of the CNTs. The same sintering parameters were used for the copper pellet having CNTs. Cu-CNTs pellet having 0.5% (mass%) of CNTs achieved the maximum temperature of 594 °C while with 1% and 1.5%, CNTs raised the pellet temperature up to 618°C and 657°C. Comparing to pure copper, with 1.5% CNTs 13% higher temperature was achieved which shows better absorption of the laser beam power into copper pellets.

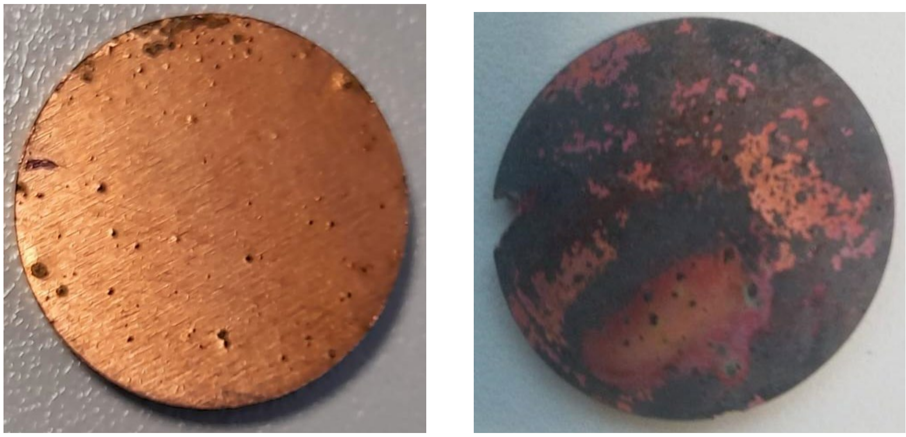
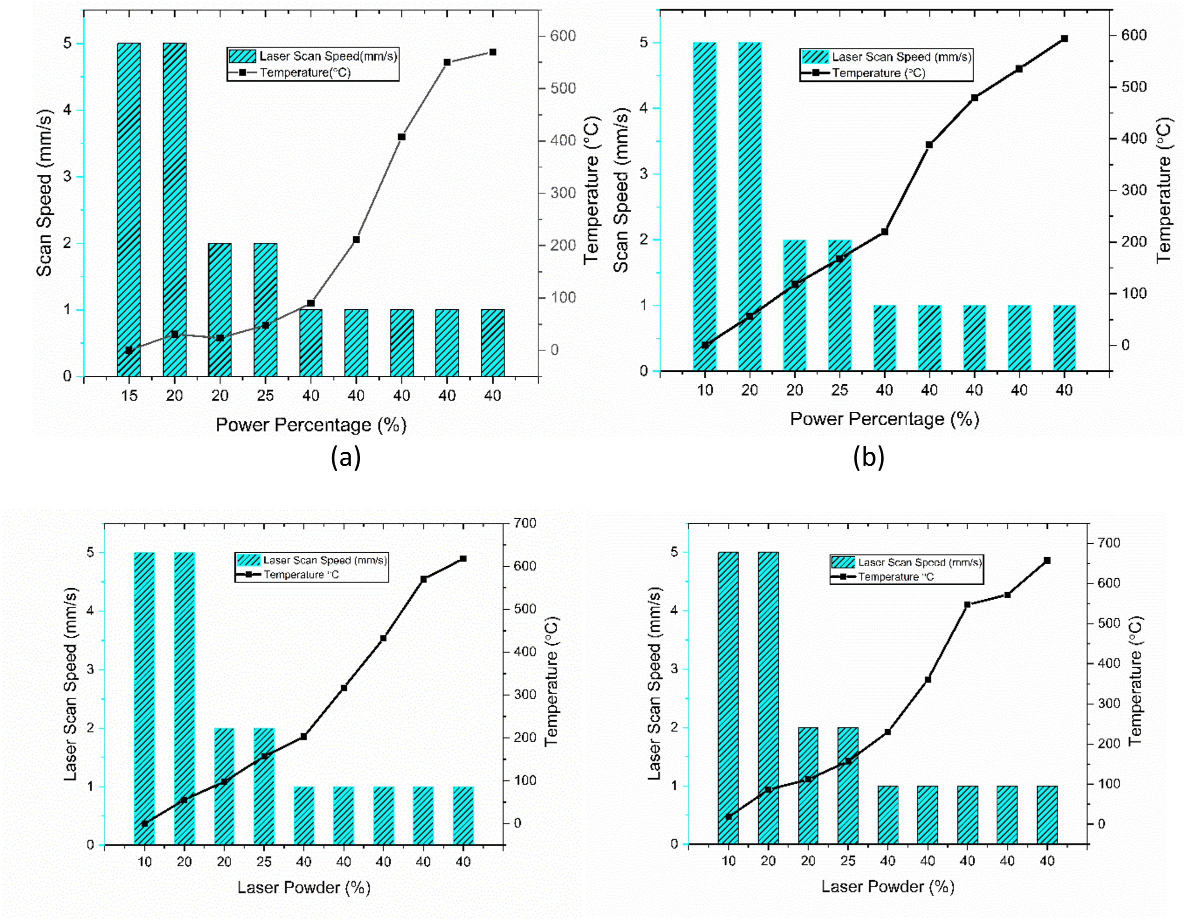

(a) (b)

Fig. 5. (a) Laser sintered pure copper pellet in sintering rig (b) laser sintering of copper pellet in open atmosphere environment.



(a) (b)

(c) (d)

Fig. 6. The sintering temperature of pellet as a function of laser power percentage and laser scan speed (a) Pure copper powder (b) 0.5% CNTs mixed copper composite (c) 1% CNTs mixed copper composite (d) 1.5% CNTs mixed copper composite.

Microstructure of laser sintered pellets.

Cu-CNTs compacted laser sintered samples have mechanical properties that are determined by the microstructure distribution. The microstructure of the laser sintered pellets were analysed using Zeiss EVO LS-15 SEM. Fig. 7 shows the SEM images of laser sintered pellets without using polishing. Fig. 7 (a) shows the microstructure of the laser sintered pure copper pellet. The higher reflection of pure copper causes the loss of mechanical bonding of the powder particles leaving small pores on the surface. The addition of 0.5% of CNTs in the copper powder increased the bonding of the powder particles. Fig. 7 (b) shows the overlapping of the powder particles. In Fig. 7 (c), the addition of CNTs 1% of copper resulted in good consolidation of the particles and increases small pores on the surface comparing 0.5% of CNTs. The increase of CNTs percentage of 1.5% in copper resulted in high density in the pellet. Fig. 7 (d) shows the melted particles due to heat expansion in the pellet. The higher persistence of CNTs in the copper caused coarse particles on the surface of the pellet.

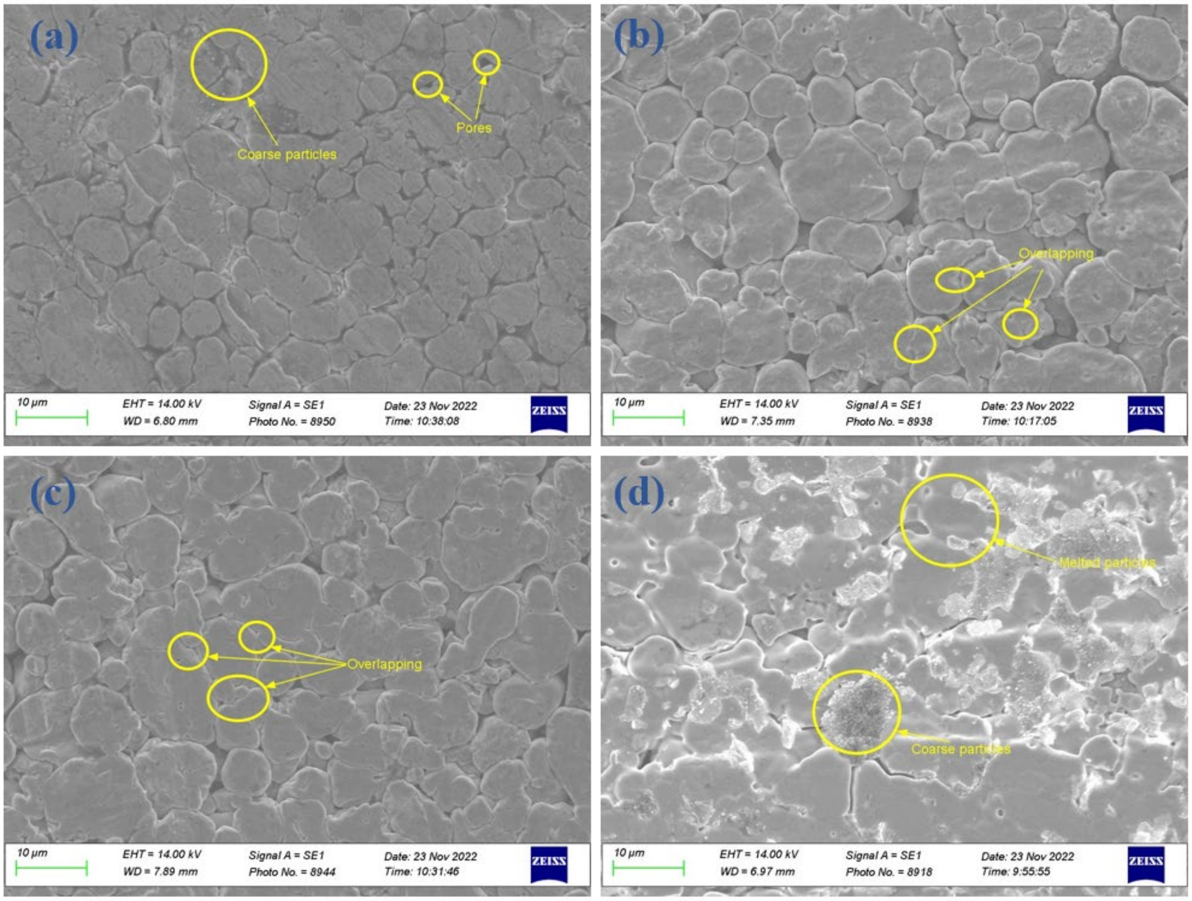


Fig. 7. SEM image of laser sintered samples (a) Pure copper pellet (b) 0.5% Cu-CNTs (c) 1% Cu-CNTs (d) 1.5% Cu-CNTs.

Summary

In this paper, the copper powder absorption was modified by adding different percentages of carbon nanotubes (CNTs). CNTs were added up to 1.5% to increase the laser power absorption ability of the copper powder. Spectroscopy was performed and results revealed that the reflectance of the copper powder by the addition of CNTs was decreased by 8%. Lesser contamination and oxidization were observed by using a specially designed laser sintering rig. The temperature of the pellet during sintering was controlled by adjusting the laser operating parameters. Improved consolidation of powder particles was observed by using a sintering rig as compared to the laser sintering in the open atmosphere environment.

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