



**HAL**  
open science

## Laser sintering of cold-pressed Cu powder without binder use

Loic Constantin, Lisha Fan, Bruno Mortaigne, Kamran Keramatnejad, Qiming Zou, Clio Azina, Yong Feng Lu, Jean-François Silvain

► **To cite this version:**

Loic Constantin, Lisha Fan, Bruno Mortaigne, Kamran Keramatnejad, Qiming Zou, et al.. Laser sintering of cold-pressed Cu powder without binder use. *Materialia*, 2018, 3, pp.178-181. 10.1016/j.mtla.2018.08.021 . hal-01136406

**HAL Id: hal-01136406**

**<https://hal.science/hal-01136406>**

Submitted on 4 Feb 2021

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

# **Laser sintering of cold-pressed Cu powder without binder use**

*Loic Constantin, Lisha Fan, Bruno Mortaigne, Kamran Keramatnejad, Qiming Zou, Clio Azina, Yong Feng Lu, and Jean-François Silvain*

**ABSTRACT:** In this work, laser sintering (LS) of cold-pressed copper (Cu) powder without using a binder was demonstrated. To promote the densification of the final layers, the Cu powder was cold pressed, thereby forming a densely packed powder bed for the laser sintering. This densification and microstructural evolution study shows that the cold-pressed powder led to an increase of up to 10% in the relative density and a decrease of 10 times in the surface roughness. The influence of the scan speed on the densification and sintering quality was studied. A relatively slow scan speed, 25  $\mu\text{m/s}$ , allowed sufficient atomic diffusion during the laser sintering process and produced dense Cu layers without cracks and open pores. The introduction of the cold-pressing step into the LS process is critical for achieving high-density powder metallurgy parts without using binders.

**Keywords:** Laser sintering (LS); copper powder; densification; cold pressing

## Introduction

Laser sintering (LS) of metal-based powder metallurgy parts has many prominent advantages over conventional sintering (CS), such as short sintering cycles, high-density powder metallurgy parts, easy integration with other operations, and flexibility in structural and functional design. LS of metal-based powder uses a laser as the power source to heat the powder above the softening temperature. In the CS process, bonding occurs when the metal particles form interlinked necks through atomic diffusion at elevated temperatures [1,2]. However, solid-state diffusion is a slow process, which is difficult to initiate during LS due to the very fast heating rates and short sintering times [3]. To accelerate the binding process, liquid sintering is widely used in LS by adding a binder that turns to liquid during the sintering [4,5]. Liquid sintering occurs as a result of liquid flow and atomic rearrangement [6]. Many research groups have reported the manufacturing of copper (Cu)-based metal parts by LS using various systems (copper-tin (Cu-Sn), copper-iron (Cu-Fe), copper-tin-nickel (Cu-Sn-Ni)) with properties comparable to those obtained by CS [7–9]. Although fast sintering for fabrication of metal matrix composites or alloys can be achieved by adding a binder to the powder mixture [10–12], single-metals remain a challenge due to the necessity of using a high number of binders [13].

In this work, we demonstrate the binder-free LS of Cu powder to fabricate dense, high-quality Cu parts. To realize LS of pure Cu powder without binders, a 532 nm Nd:YAG laser was chosen because Cu has a high optical absorptivity (about 40%) at 532 nm, which ensures a high sintering temperature and sufficient atomic diffusion [14]. To enhance the atomic diffusion between metal particles and the density of the final parts, the Cu powder was cold pressed before the LS step, as shown in Figure 1(a)-(b). Finally, dense and smooth layers were obtained by LS, as shown in Figure 1(c).

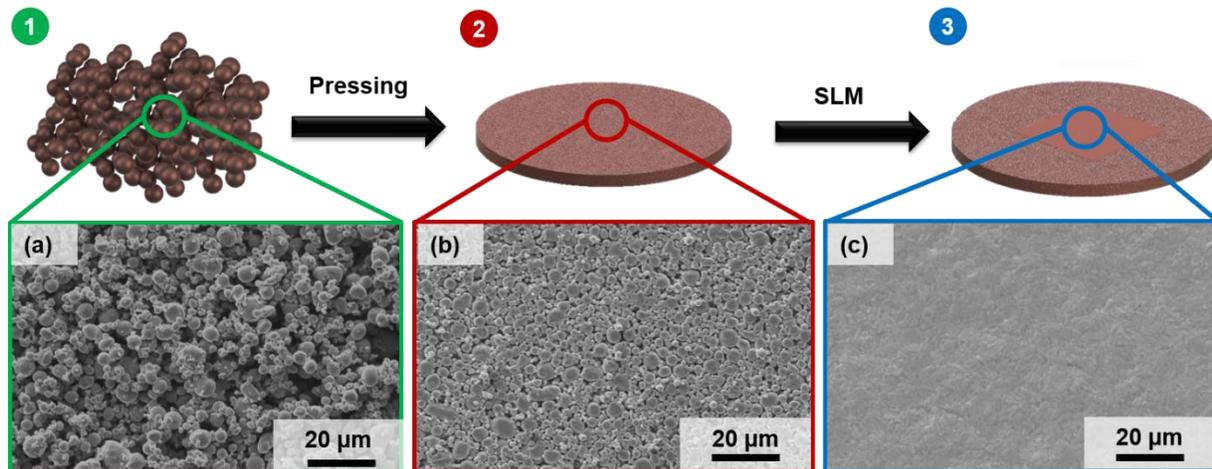


Figure 1. Powder metallurgy steps to obtain dense Cu layers: (a) raw powder, (b) after cold pressing, and (c) after the LS step.

## Experimental Section

Figure 2(a) schematically shows the home-built LS setup used in this work. A continuous-wave (CW) Nd:YAG laser (Coherent Verdi-G Series), with a wavelength of 532 nm and a maximum output power of 5 W, was used as the heat source. An optical objective (5× Mitutoyo M Plan Apo) was used to focus the laser beam into a spot with a diameter of 20 μm. A charge-coupled device (CCD) camera was used to monitor the printing process. The Cu powder was placed in a chamber which was filled with nitrogen (N<sub>2</sub>) gas on a 3D moving stage with a step resolution of 2.5 μm. The chamber pressure was kept at 130 Pa under a N<sub>2</sub> flux of 10 sccm for all of the experiments. A simple line scanning pattern was used to sinter the Cu powder. A line spacing of 10 μm (50% overlap) was used to promote the atomic diffusion on each Cu layer, as illustrated in Figure 2(b).

Spherical Cu powder (Eckart Granulate Velden GmbH, 99% purity) with a diameter of 5 μm was used as the initial raw material. The Cu powder was cold pressed using a hydraulic press with a

pressure of 100 MPa for 5 min. The thickness and the diameter of the prepared disc were about 50  $\mu\text{m}$  and 50 mm, respectively. Scanning electron microscopy ((SEM) XL 30, Philips Electronics) was performed to study the microstructures and the fracture behaviors of the LS materials. Sample density was estimated based on the SEM micrographs using ImageJ software. An optical surface profiler (Zygo NewView™ 8300) was used to evaluate the surface roughness.

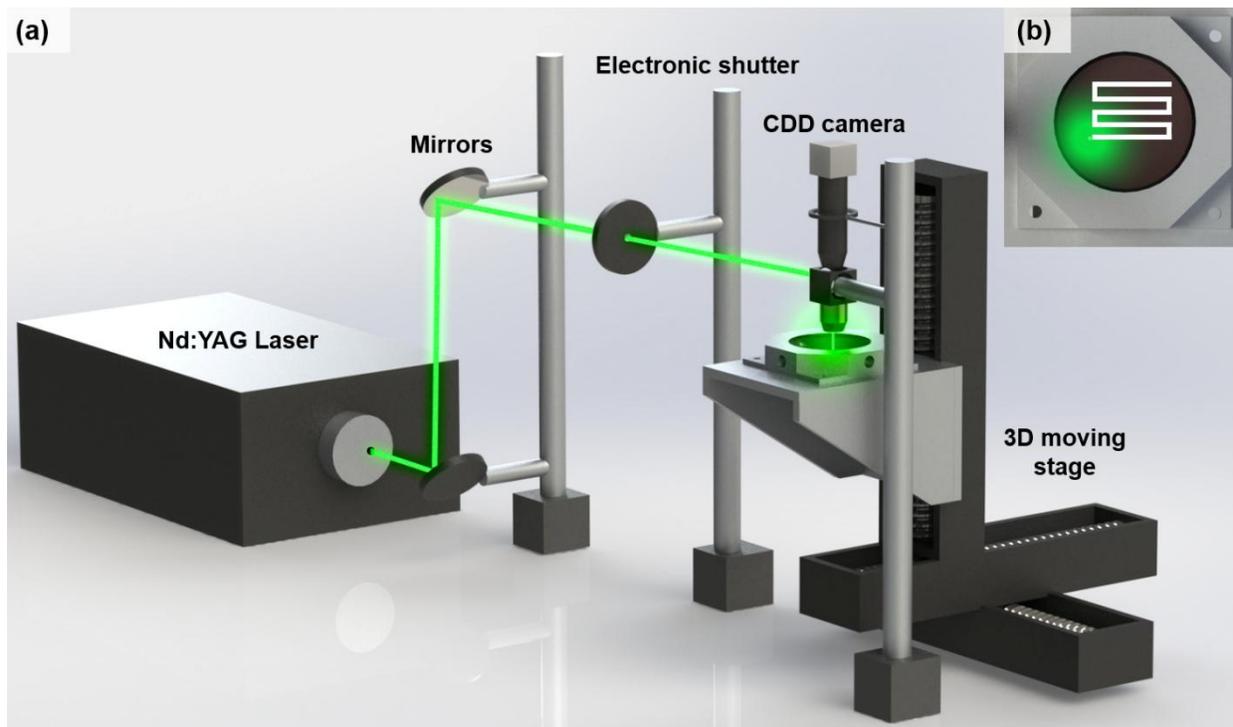


Figure 2. (a) Schematic of the homemade LS system and (b) line scanning strategy of the laser.

## Results and Discussion

To enhance the particle contact and thus the surface diffusion, the effect of cold-pressed powder was investigated during LS. Copper powder with a diameter of about 5  $\mu\text{m}$  was sintered on loose and cold-pressed powder using an Nd:YAG laser at a constant laser scan speed and power of 125  $\mu\text{m/s}$  and 5 W, respectively. Figure 3 (a) shows the SEM micrographs of parts fabricated from loose and cold-pressed Cu powders. The part obtained from loose Cu powder has a porous

surface with discontinuous laser scan tracks, and Cu drops, which formed at the surface. This phenomenon is attributed to poor wetting of the molten pool due to incomplete heating of the loose powders and the splashing of Cu particles during laser heating [15,16]. The part density was estimated to be around 85%. In contrast, a dense part was fabricated from the cold-pressed powder with an estimated density of around 95%, as shown in Figure 3(a). The number of pores and their sizes decreased significantly. Balling "globules" were not observed, and laser scan tracks became continuous. Cold-pressed powder achieved better packing and more intimate contact between the powder particles, thereby avoiding particle ejection during the laser scan, as can be observed in Figure 1(a) and (b).

The surface roughness of loose and cold-pressed Cu powder was analyzed using a three dimensional (3D) optical surface profiler. As can be seen in Figure 3(b), sintering of loose powder resulted in a rough surface containing a large quantity of voids and balling globules. A large average surface roughness value of 6.5  $\mu\text{m}$  resulted, which was even higher than the powder diameter. In contrast, the sample surface fabricated from the cold-pressed powder had a smooth surface with no obvious voids. A significant decrease in the average surface roughness to 0.5  $\mu\text{m}$  was identified.

It was noted that the porosity and the average surface roughness of the final Cu parts were strongly affected by the cold-pressing step. Sintering is a diffusion mechanism driven by the reduction of the surface energy. Fick's and Jost's laws describe the diffusion mechanism mathematically and determine its dependence on the temperature as well as the material [17,18]. In solid diffusion, different diffusion paths occur at the same time, such as surface, phase boundaries, grain boundaries, and volume diffusion. The faster path is surface diffusion, which depends on the particles' contact. Next, in order of decreasing speed, is phase boundaries

diffusion, which varies depending on the number of phases; then, grain boundaries diffusion, which is influenced by the grain sizes; and, finally, volume diffusion, which is affected by the density of dislocation [1,19]. The contact between powder particles was enhanced by cold-pressed powders which led to easier neck formation and thus, raising the surface diffusion during LS. Furthermore, it is well known that, pressing of powder particles engenders dislocation formation, thus the cold-pressing treatment promoted volume diffusion as well [20].

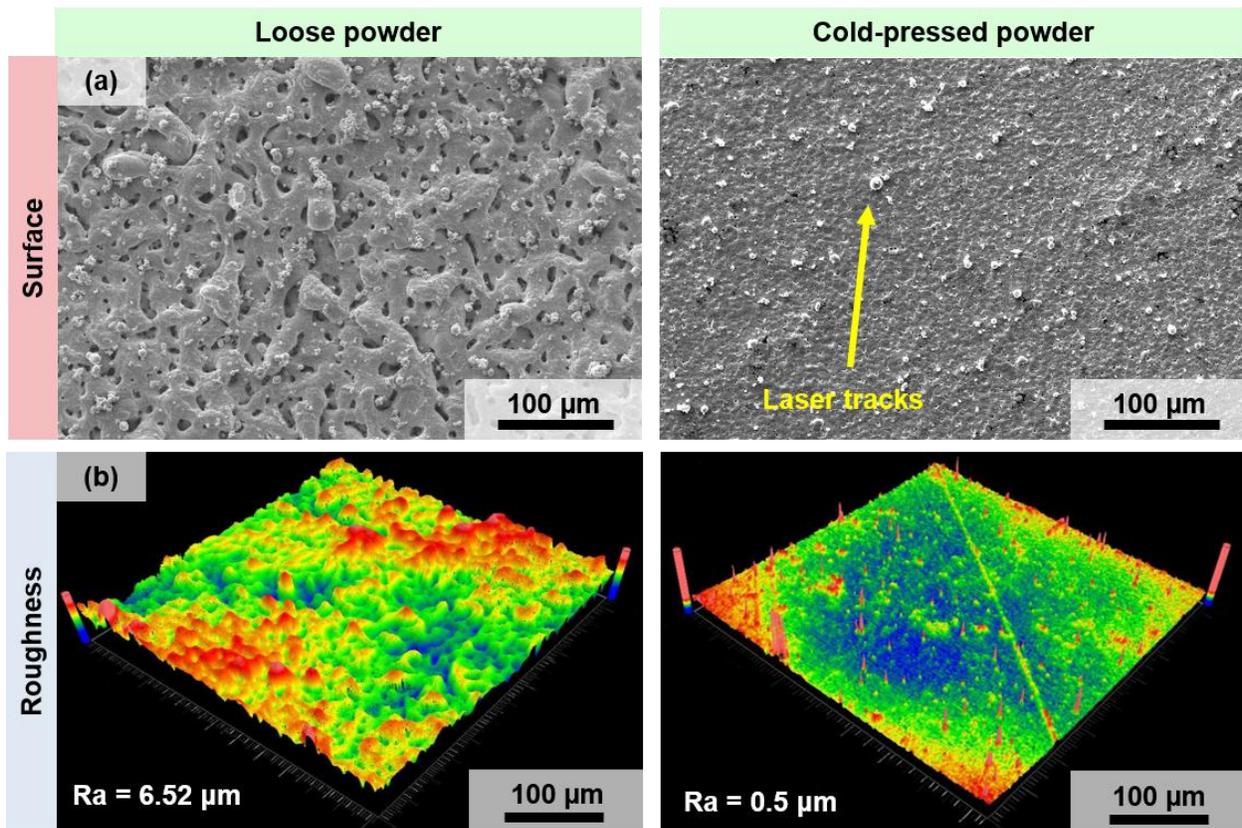


Figure 3. (a) SEM micrographs and (b) 3D surface mapping of Cu layer fabricated using LS from loose and cold-pressed Cu powders, respectively.

Although cold-pressed powder achieved a faster diffusion by enhancing surface and volume diffusion, solid diffusion remained a slow process and all pores were not suppressed. To obtain a fully dense part with a density close to the theoretical value, the sintering time had to be

increased; and thus, the laser scan speed was varied from 125 to 25  $\mu\text{m/s}$ . Figures 4(a) and (b) show the surface morphologies and the cross-sectional micrographs of 50- $\mu\text{m}$ -thick parts fabricated by LS at different scan speeds. As shown in Figure 4(a), smooth surfaces without balling globules were obtained for all cases; and the number of pores decreased as the laser scan speed decreased. The cross-sectional micrographs, shown in Figure 4(b), revealed the consolidation behaviors for different scan speeds. At a scan speed of 125  $\mu\text{m/s}$ , necks formed between Cu particles. By reducing the scan speed to 62  $\mu\text{m/s}$ , the sintering of the powder was enhanced. At a scan speed of 25  $\mu\text{m/s}$ , complete densification of the Cu powders was attained. The resulting sample surface showed dimple-like features, which are a typical characteristic of ductile fracture [12,21]. As the scan speed decreased, the sintering time increased, which means more energy was delivered and absorbed by the powder; and thus the atomic diffusion was promoted, which led to dense parts [22].

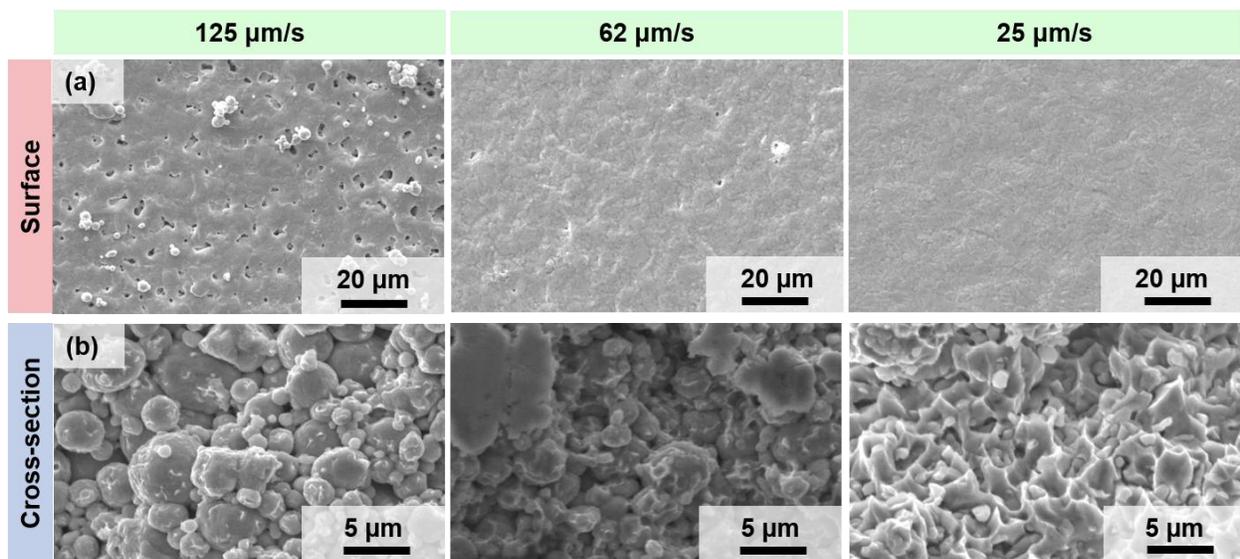


Figure 4. SEM micrographs of Cu sample surface fabricated by LS at different scan speeds:

(a) surface morphologies and (b) cross-sectional microstructures.

## Conclusion

This paper reports on the investigation of the effect of cold-pressed powder in LS. It was demonstrated that cold-pressed powder allows high density Cu parts with low surface roughness without the use of binders. Due to better contact between particles, the surface diffusion during LS was promoted and powder ejection out of the laser tracks vanished. Furthermore, by decreasing the laser scan speed (thereby increasing sintering time), fully dense Cu parts were manufactured by LS.

#### ACKNOWLEDGMENT

The authors would like to express their appreciation to Dr. D.R. Alexander in the Department of Electrical and Computer Engineering at the University of Nebraska-Lincoln for providing convenient access to the SEM.

#### References:

- [1] H.E. Exner, E. Arzt, in: *Sinter. Key Pap.*, Springer, Dordrecht, 1990, pp. 157–184.
- [2] B.H. Alexander, R.W. Balluffi, *Acta Metall.* 5 (1957) 666–677.
- [3] D.L. Johnson, in: *Ultrafine-Grain Ceram.*, Springer, Boston, MA, 1970, pp. 173–183.
- [4] M. Agarwala, D. Bourell, J. Beaman, H. Marcus, J. Barlow, *Rapid Prototyp. J.* 1 (1995) 26–36.
- [5] Y. Tang, H.T. Loh, Y.S. Wong, J.Y.H. Fuh, L. Lu, X. Wang, *J. Mater. Process. Technol.* 140 (2003) 368–372.
- [6] J.-P. Kruth, P. Mercelis, J.V. Vaerenbergh, L. Froyen, M. Rombouts, *Rapid Prototyp. J.* 11 (2005) 26–36.
- [7] H.J. Niu, I.T.H. Chang, *Scr. Mater.* 39 (1998) 67–72.
- [8] L. Lü, J. Fuh, Y.-S. Wong, *Laser-Induced Materials and Processes for Rapid Prototyping*, Springer US, 2001.
- [9] H.H. Zhu, L. Lu, J.Y.H. Fuh, *Mater. Sci. Eng. A* 371 (2004) 170–177.
- [10] A. Popovich, V. Sufiiarov, I. Polozov, E. Borisov, D. Masaylo, A. Orlov, *Mater. Lett.* 179 (2016) 38–41.
- [11] D. Gu, Y. Shen, *Mater. Lett.* 60 (2006) 3664–3668.
- [12] L. Ren, K. Memarzadeh, S. Zhang, Z. Sun, C. Yang, G. Ren, R.P. Allaker, K. Yang, *Mater. Sci. Eng. C Mater. Biol. Appl.* 67 (2016) 461–467.
- [13] H.H. Zhu, L. Lu, J.Y.H. Fuh, *Mater. Sci. Eng. A* 371 (2004) 170–177.
- [14] A. Hess, R. Schuster, A. Heider, R. Weber, T. Graf, *Phys. Procedia* 12 (2011) 88–94.
- [15] X. Zhou, X. Liu, D. Zhang, Z. Shen, W. Liu, *J. Mater. Process. Technol.* 222 (2015) 33–42.

- [16] Y.F. Shen, D.D. Gu, Y.F. Pan, Balling Process in Selective Laser Sintering 316 Stainless Steel Powder, n.d.
- [17] W. Jost, Diffusion in Solids, Liquids, Gases: W. Jost., Academic Press, New York, 1960.
- [18] D.A. Fick, Lond. Edinb. Dublin Philos. Mag. J. Sci. 10 (1855) 30–39.
- [19] C.H. Kühl, N. Straße, (n.d.) 8.
- [20] A. Eksi, M.K. Kulekci, Metalurgija 43 (2004) 129–134.
- [21] H. Zhang, H. Zhu, T. Qi, Z. Hu, X. Zeng, Mater. Sci. Eng. A 656 (2016) 47–54.
- [22] R.M. German, in: Sinter. Empir. Obs. Sci. Princ., Butterworth-Heinemann, Boston, 2014, pp. 183–226.