A STUDY ON POROSITY IN MICRO-SELECTIVE LASER SINTERING OF COPPER POWDER

[1]Srijan Paul, [2]Nagahanumaiah and [3]Souren Mitra

[1]Research Scholar, School of Laser Science and Engineering, Jadavpur University, Kolkata
[2]Director, Central Manufacturing Technology Institute, Bangalore
[3]Professor, Production Engineering Department, Jadavpur University, Kolkata
[1]E-mail address: srijanpaul@yahoo.co.in

Abstract: *In Selective Laser Sintering metal powder particles are consolidated layer-by-layer through laser heating. In recent years, process has been improved that addressed the defects such as porosity, micro-cracks etc., resulting its wider applications in industries. However, there is a promising opportunity exists to use this process in micro manufacturing, which has not been exploited to a great extent. In this work, an attempt has been made to understand the porosity defects formed in micro-selective laser sintering (Micro-Sintering) of copper powder. Molecular Dynamics based simulation study performed for the Micro-Sintering of nano-scale powders has shown insight into the mechanism of neck growth formation and resulting porosity on their joining with adjacent particles. The other defects such as cracks formation, balling effects, presence of residual binders contributing to porosity and micro-cracks observed during experimental study on Micro-Sintering of 5µm copper powder has also been discussed.*

Key words: *Selective Laser Sintering, Copper, Defects, Porosity, Molecular Dynamics Simulations.*

1. INTRODUCTION

In recent years, the interest in Additive Manufacturing is increased to use this as an alternative process to produce complex functional parts in shorter time. It is also getting popular in manufacturing microparts using micro-nano scale metallic and non-metallic powders. Among others, Selective Laser sintering (SLS) is one of the novel techniques in which metal powder particles are consolidated layer-by-layer through laser heating. In the process of selective laser sintering, powder particles are scanned selectively by laser beam spot. It is observed that sintering submicron sized powder particles selectively using laser radiation is advantageous and cost effective (Ko et al., 2007). Review of past literatures reveal the promising commercial acceptance of SLS for metal powders in the order of few tens of microns (Regenfuss et al., 2007a) (Regenfuss et al., 2007b) (Duan and Wang, 2011). The commercial SLS systems could sinter a powder >10µm and capable of creating features size in the order of 1mm or larger (Regenfuss et al., 2007c).

In recent years, process has been improved to address the process defects such as porosity, micro-cracks etc. Kasperovich et al. studied various defects such as balling effect, cracks during SLS of TiAl6V4, alloy of Ti and Al. They mentioned cracks are mostly influenced by balling effects as well as inadequate melting of powder particles. They also mentioned that the pores created due to surface tension of molten materials (Kasperovich et al., 2016). Porosity is the empty spaces, created during SLS, which make the part weak. Strength of the produced part can be improved by reducing porosity. SLS is a technique in which powder particles are heated at below melting temperature of the material to avoid full melting of material. Porosity arises during SLS due to the reasons like inadequate melting, shrinkage, gas bubble entrapment. The studies on the influence of process parameters on porosity in SLS process have concluded that laser power, scanning speed, hatch spacing, hatch style have significant influence (Kasperovich et al., 2016) (Read et al., 2015). Further, particle size is another key parameter and it is found that the porosity increases with the increase in particle size (Yan et al., 2011) (Koo et al., 2017). Wei et. al. as it is a function of sintering time (Wei et al., 2015). Shaw et. al. (Shaw and Dirven, 2016) investigated porosity during SLS of Nylon and studied the mechanical properties. With such significant process improvements, SLS process with its variants has been used in industries in producing relatively smaller projects having features in the order few millimetres. However, use of this process in micro manufacturing has not been exploited to a greater extent. In one such study, SLS process has been used to develop 3D micropart of porous silica structures in the presence of polymer binder (Chang et al., 2017).

In the current research authors are investigating on feasibility of using selective laser sintering technique to sinter submicron size metal powder and to generate micro parts having few tens of micron size features, which is called as 'Micro-Sintering' process. However, in this paper discussion is focused on understanding the sintering mechanisms and the parameters contributing to the porosity of sintered micro parts. In the present paper, two different sets of experiments have been conducted. In the first phase, virtual experiments of micro-sintering of 0.72nm, 1.82nm and 3.64nm grain sized copper powder are performed using Molecular Dynamics simulations and in the second phase, practical experiments of micro-sintering of copper powder of average grain size of 5μm are performed using nanosecond pulsed diode pumped Q-switched Nd:YAG laser and Continuous Wave (CW) laser sources.

3. MATERIALS AND METHODS

In the first part, virtual experiments have been conducted using Molecular Dynamics (MD) simulations technique, where influence of particle size on porosity has been investigated by simulated process for sintering different sized copper nanoparticles. During conduction of virtual experiments using MD simulations, three different atomistic models of identical spherical copper nanoparticles are created considering periodic boundary conditions. In the different models, size of the particles is different. Each of the atomistic model of particles are heated continuously for same duration of time in the non-isothermal consideration to study the effect of particle size on porosity created during micro-sintering. During virtual experiments of micro-sintering, copper nanoparticles are h eated continuously for 30 picoseconds of time at the traditional heating rates of 4.00×10^{13} K/s. It is considered that no heat is lost during micro-sintering of copper nanoparticles.

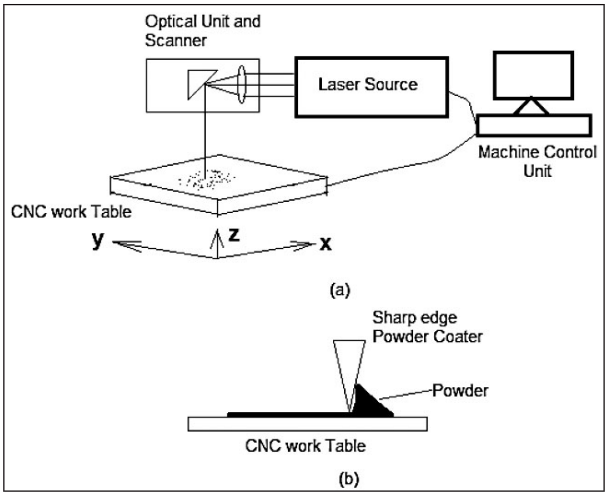
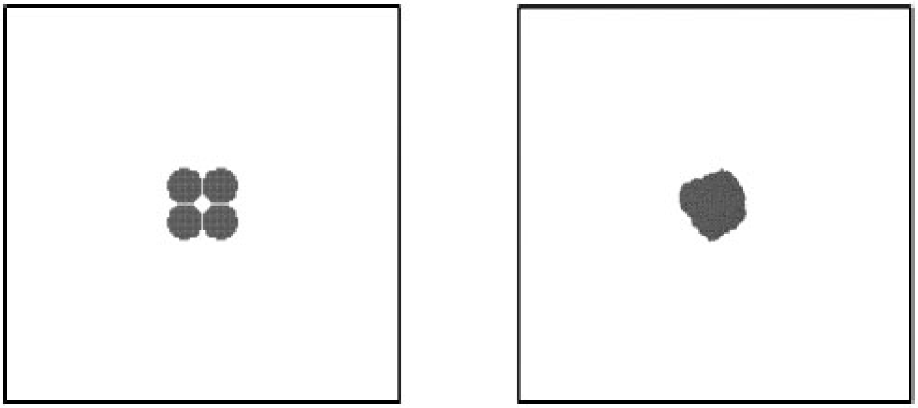


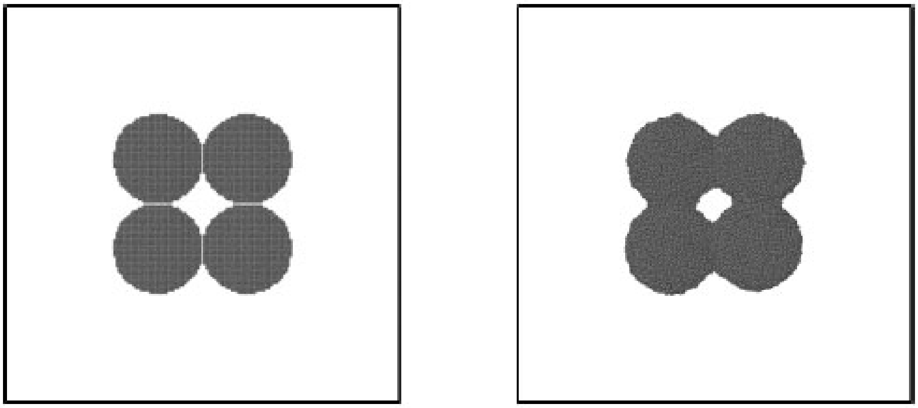
Fig 1. (a) Schematic Diagram of the Laser Sintering Machine (Song et al., 2007); and (b) Powder Coater

In the second part, experiments were performed for Micro-Sintering of copper powder of average grain size of 5μm. The layers of copper powder of average particle size of 5μm is sintered selectively using different laser sources: Nanosecond pulsed diode pumped Q-switched Nd:YAG laser and Continuous Wave (CW) Diode Laser. Preliminary investigations of micro-sintering of copper powder are conducted to study the influence of different process conditions of micro-sintering using laser irradiation on various defects. The laser power, scanning speed and hatch spacing have been used as variable process conditions.

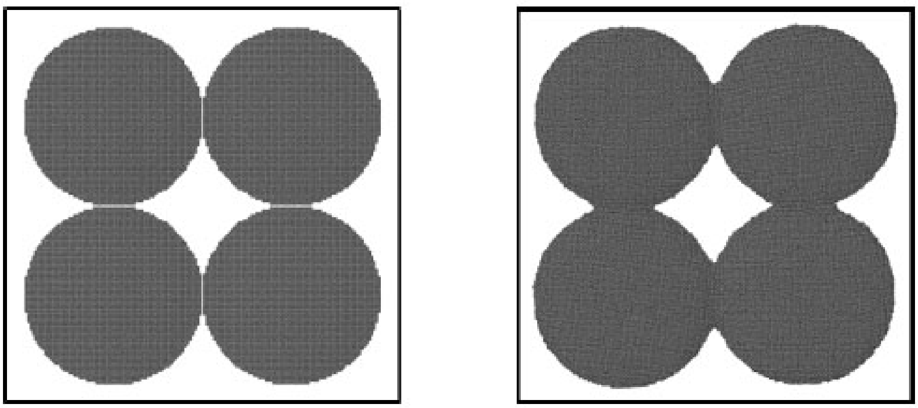
The schematic representation of SLS systems used in these experimental studies is shown in Fig. 1. The machine developed inhouse consists of a laser source, an optical scanning unit, a CNC work table, a machine control unit and a powder coater. The CNC controlled work table controls the laser scanning as per the CAD data. The laser system control unit facilitates the setting of various process conditions such as laser power, scanning speed, hatch spacing and hatch style. Layer of powder is coated on the top of the platform of CNC controlled work table layer by layer. Optical scanner controls the



(a) Copper nanoparticles of size 0.72 nm (before and after sintering)



(b) Copper nanoparticles of size 1.82 nm (before and after sintering)



(c) Copper nanoparticles of size 3.64 nm (before and after sintering)

Fig 2. Snapshots of Sintered Copper Nanoparticles in Non-Isothermal Considerations at 4.00×10^{13} K/s

focus distance and spot size of laser beam via control unit.

One 3D simulation box is created for each model. Inside each of the simulation box the atomistic model of four copper nanoparticles are developed. The spherical Copper nanoparticles of face cantered cubic (FCC) crystal structure are created inside the simulation box. Using Verlet algorithm the velocities of atoms are defined to achieve control over temperature. Time step have been used as 1.0femtosecond and list of neighbouring atoms is updated during sintering. To simulate the sintering process, four identical spherical particles are kept into contact and the centre distance of the two adjacent particles are kept diameter of the particle at approximately in room temperature (300 K).

After development of initial models, the MD simulations are conducted. The atomistic models are kept in 300K temperature at the beginning and the atoms are randomized with a velocity following Gaussian distribution with the interatomic EAM potential gradients (Paul et al., 2018). The atoms are equilibrated at the temperature 300 K with a time step of 0.001ps. After equilibration, the heat is supplied to the particles. During sintering simulation particles are heated continuously and temperature is changed from 300 K to 1500 K with the heating rate of 4.00×10^{13}K/s. To simulate the micro-sintering the copper nanoparticles are subjected to continuous laser heat with no heat loss. Fig. 2 is showing the snapshots of initial model of copper nanoparticles and snapshots of copper nanoparticles after sintering.

While more details of this work are reported in Paul et al., 2018, during non-isothermal sintering simulation, it is observed that at the end of sintering the 0.72nm particles melted and joined with each other and tend to become a single spherical particle. This phenomenon reveals full melting of particles, which was representing the condition of Selective Laser Melting (SLM). During sintering particle should not be fully melted, particles should be fused at less than its melting temperature and joined together. Whereas, in same heating rate 3.64nm particles and 1.82nm particles fused and joined with each other but not fully melted.

3.2 Experiments using Dry Copper Powder

Dry copper powder of average particle size 5µm, are micro-sintered using different laser power and scanning speed. Copper powder was preheated inside the oven at 200ºC temperature for about six hours to remove the moisture. The layer of this dried copper powder then sintered using two laser sources.

In the first sets of experiments, the layer of dry copper powder is scanned by Nd:YAG laser source at scanning speed of 960 mm/min with different laser power such as 1.25W, 1.75W and 2.25W. In this process, the balling effects were significant before those particles gets solidified and formed the lumps. Further, it is also noted that during laser scanning the dry powder particles displaced from the track of laser scan due to high energy under laser irradiation. However, those sintered lumps shown high porosity between the sintered particles was observed under microscope (Fig. 3).

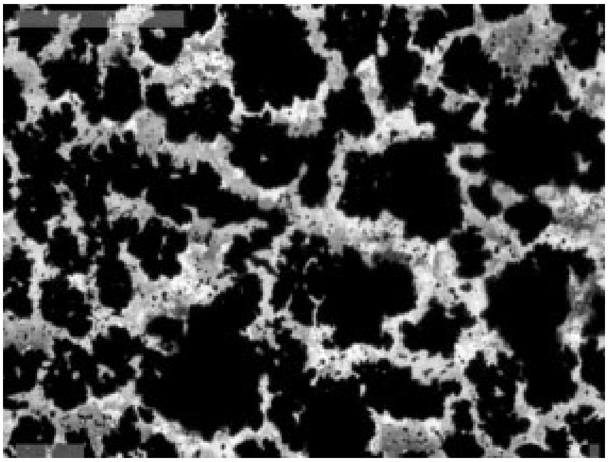


Fig 3. Microscopic view of Laser Sintered Single layer of dry Copper Powder after Scanning with Nanosecond Pulsed Nd:YAG Laser Source

In the second set of experiments, CW Diode Laser was used as source to sinter layer of same dry copper powder, scanned at laser power of 14W and with variable scanning speed 720mm/min and 840mm/min. In the different scanning speed, ten thin layers of dry copper powder is sintered to develop a 3D micropart of square cross-section of 10mm×10mm. Every layer of dry copper powder is scanned for five times. After sintering, the micropart is found very fragile and it is broken just after getting separated from the substrate.

With these experiments, it is noted that sintering of layer dry copper powder leads high porosity and sintered part is fragile does not retains its shape. After sintering multiple layers, the part was still fragile and it was decided to use the binder to perform further investigations.

3.3 Micro-sintering of Copper Powder with Binder Material

These experiments were continued, where the same 5µm copper powder was blended with silicon oil as binder material and it was found to be feasible to sinter multiple layers to create 3D parts. With the use of binder material, flying out of particles from the track of laser scan and balling effect could be avoided. Layers of Copper powder blended with Silicon Oil is scanned by again by two different laser sources: Nd:YAG Laser source and CW Diode Laser scanned at different laser power and scanning speeds and 3D micropart has been successfully built. In laser scanning using nanosecond pulsed Nd:YAG Laser source, 5.6W laser power and 960mm/min scanning speed are used. In scanning by CW Diode Laser source, laser power and scanning speed are kept as shown in Table 1. After sintering, residual of silicon oil is observed in the microparts in both the cases and they are post-processed by keeping in heating oven at 250ºC for several hours. The SEM images of the samples are shown in Fig. 4.

4. RESULTS AND DISCUSSION

4.1 Sintering Simulation

From the MD simulations of Micro-Sintering it is observed that in case of the 0.72nm particle size copper powder is fully melted; four particles joined with each other at sintering simulations performed with non-isothermal considerations from 300K to 1500K at the heating rate of 4.00×10^{13} K/s. However, under the same sintering conditions, the MD simulation results did not show the complete melting for larger particle size 1.82nm and 3.64nm. The MD simulations models have shown circular shaped pores between the adjacent particles on their joining and the pore size is found to be increased with increase in particle size for the same heating rate.

Table 1: Laser Power and Scanning Speed during Sintering using CW Diode Lase

Sample	Power (W)	Scanning Speed (mm/min)
1	13	500
2	10	500
3	10	800
4	12	180
5	13.65	180
6	15.2	180

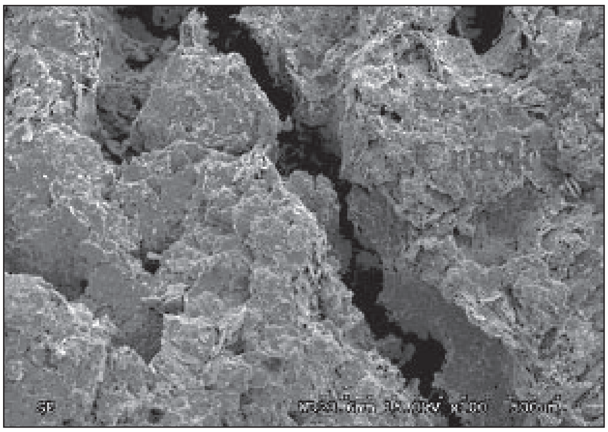


Fig 4. SEM Views of Sintered Copper Powder Blended with Silicon Oil using Nanosecond Pulsed Nd:YAG Laser

4.2 Defects in Micro-Sintered Parts

Initially, sintering of dry copper powder is attempted in various process conditions but sintering of dry powder is found unsuccessful. Due to shrinkage of powder particles spherical balls are created, this is attributed the balling effect as reported in (Regenfuss et al., 2007b). Though bonding with adjacent particles is found but a large gap is also observed in the sintered first layer as shown in the Fig. 3. The sintered micropart is so fragile that it is collapsed when it is separated from the substrate.

On the other hand, copper powder blended with binder material is sintered using pulsed Nd:YAG and CW Diode laser and built 3D micro part. It is observed that sintering with nanosecond pulsed Nd:YAG laser at 5.6W laser power and 960mm/min scanning speed is quite successful. SEM images shows promising result of successful sintering with good presence of layer serrations which is favoured for sintering of successive layers. However, still pores and microcracks are observed in various zones of the sintered part. Due to high intensity of laser and low frequency pulses rough surface is observed.

The SEM micrographs for three samples (1,2, and 3) shown in Fig. 5(a), 5(b), and 5(c) have indicated good and dense bonding of copper particles. However, due to high intensity of laser with higher scanning speed rough surface is observed.

The SEM images of Sample 4, 5 and 6 (Fig. 5(d), 5(e), and 5(f)) reveals full melting of the particles with large pores on the surface. Due to low scanning speed the energy density of laser irradiation is quite high. High energy density leads the particles to full melting. Due to rapid melting and solidification shrinkage in

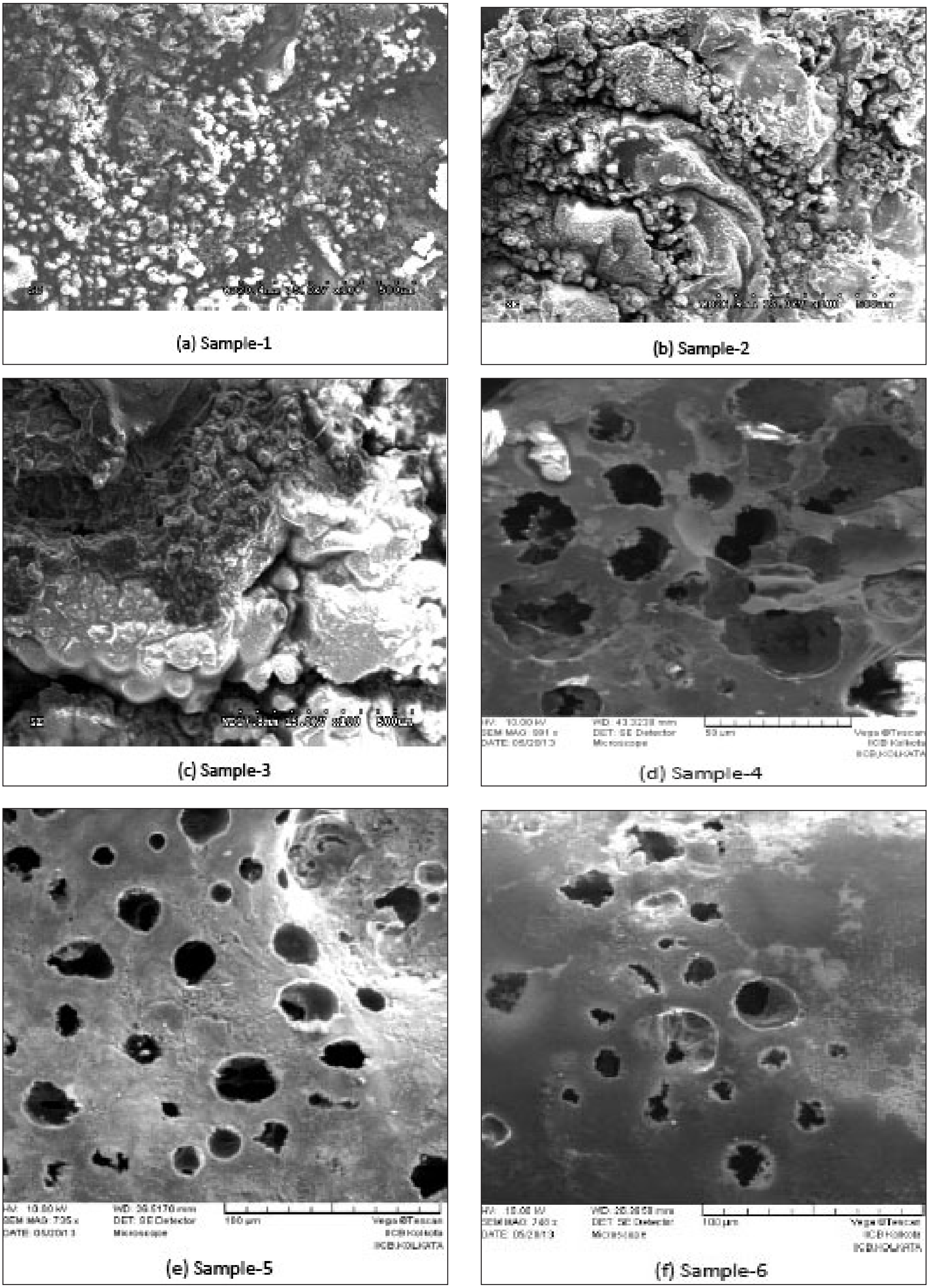


Fig 5. SEM Views of Sintered Copper Powder Blended with Silicon Oil Using CW Diode Laser Source

material take place and large pores are created. These preliminary investigations have shown various limitations like balling effects, porosity, microcracks in micro-sintering using different process conditions. Further investigations can be undergone to improve performance to minimize defects through optimizing the process conditions and choosing of proper binder materials.

5. CONCLUSIONS

In the present paper the MD simulations of copper nanoparticles are conducted successfully to investigate the effect of particle size in minimizing porosity level. Experiments of micro-sintering of dry copper powder and with binder material are conducted. Micro-sintering of dry copper powder was unsuccessful in different process conditions. Using silicon oil as a binder material, the balling effect could be avoided and showed promising micro-sintering mechanism under various process conditions. Defects like microcrack and porosity are observed. Further, in case of scanning with CW Diode laser at low scanning speed and high laser power results in melting. Microscopic view of dry sintered powder is showing balling phenomenon with large gaps in shrunk sintered powder particles. However, SEM micrographs of microparts sintered using Q-switched nanosecond pulsed Nd:YAG laser shows microcracks with low porosity.

6. REFERENCES

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Srijan Paul is presently working in Asansol Engineering College, Asansol, West Bengal, India as Assistant Professor. He is graduated in Mechanical Engineering from C. V. Raman College of Engineering, Bhubaneswar and M.E. in Production Engineering from Jadavpur University, Kolkata. He is PhD (Engineering) research scholar of School of Laser Science and Engineering, Jadavpur University, Kolkata. His research topic is in the area of Selective Laser Sintering of submicron scale metallic powder. He has published three technical papers in international conferences and one international book chapter. He is a Member of Institution of Engineers (India). (E-mail: srijanpaul@yahoo.co.in)

Dr. Nagahanumaiah is presently working in Central Manufacturing Technology Institute, Bengaluru, Karnataka, India as Director. He received PhD from IIT-Bombay. He has more than 23 years of professional experience. He worked as Chief scientist and Head of Micro Systems Technology Lab in CSIR-CMERI, Durgapur for more than 20 years. He supervised several PG and PhD scholars. He has more than 100 research papers in national and international journals and conferences. His research areas are micro-nano manufacturing, additive manufacturing, non-conventional machining. He was principal investigator of several national and international institutional projects. (director.cmti@nic.in)

Prof. (Dr.) Souren Mitra is working in Jadavpur University, Kolkata as Professor of Production Engineering Department. He worked as Head of Production Engineering Department for couple of years. He has more than 25 years of professional experience. He supervised more than 50 PG and PhD scholars. He has more than 100 research papers in various national and international journals and conferences. His area of interest in Teaching and Research are Product Design, Manufacturing Process Mechanics, Non-conventional Machining processes, Additive Manufacturing, Micro-nano Machining. He successfully completed several govt. sponsored projects.