Consolidation Behavior of Metal Powder in Additive Manufacturing

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学位論文要旨 Consolidation Behavior of Metal Powder in Additive Manufacturing

積層造形法による金属粉末の結合特性に関する研究

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Abstract

Additive manufacturing (AM) is a relatively new and emerging manufacturing technology that is able to revolutionize the manufacturing industry. This is due to its high flexibility in processing different types of material under various conditions. However, capability of a product to have desirable quality comparable to traditional processing techniques is still not achievable. Consolidation behavior and influences of processing parameters are important in determining the part quality. Therefore, in this research, consolidation behavior of metal powder was examined by monitoring the real time consolidation process and surface temperature. A high-speed camera was utilized with telescopic lenses in order to monitor interaction of laser and material within the fusion zone (FZ). In order to investigate the consolidation temperature, a two-color pyrometer was used. The influences of processing parameters were examined. It was found the temperature and consolidation behavior were affected by the processing parameters. The line consolidation characteristics were analyzed according to the line consolidation width, FZ, melt pool and splattering behavior. Based on the study, the line consolidation can be classified into five different consolidation types. These types are continuous, discontinuous, ball shaped, weak and very little consolidation. The consolidation mechanisms that occurred during line and area consolidation were also reported. Other than that, the properties of the consolidated material were studied, and its potential for the development of a permeable structure was investigated. It was found that the properties of the structures developed via AM relatively good and feasible to be used for the manufacturing of injection mold.

1. Introduction

Additive manufacturing (AM) is a relatively new and emerging manufacturing technology that able to revolutionize the manufacturing industry. Initial development of the technology dated back approximately 30 years ago with the introduction of an automatic method for fabricating a threedimensional plastic model by Kodama [1]. This was followed bv the first commercialized stereolithography based AM by 3D Systems [2]. Various types of materials being used in AM including metallic material [3]. Therefore, realizing its high potential, AM also had been speculated to be the third industrial revolution after mechanization of the manufacturing industry in 18th century and introduction of the assembly line in 20th century [4].

Among AM processes, Selective laser sintering/Selective laser melting (SLS/SLM) has a high potential for development of functional product. In the SLS/SLM process, a thin powder layer is deposited, and the laser is irradiated to powder surface successively until the final part is produced based on CAD data. During the iterative process of powder deposition, laser irradiation and molten powder solidification, the powder metals are consolidated to form a solid metal part based on the CAD data transferred to the AM system. The product part is a consolidated material where its properties and appearance are influenced by powder materials and fabrication parameters. The SLS/SLM involves the consolidation of metal powder to solid under the influence of high heat that is generated from laser irradiation within very short time. As a result, the powder and the consolidated structure experience repetitive microstructural changes. This is because sintering/melting and solidification alternately occurred during layer-by-layer laser irradiation.

The nature of the SLS/SLM where the consolidation occurred at a micron-sized scale with very high temperature and intensity of energy has caused earlier research on real time observation of the SLS/SLM process using various imaging systems at the irradiation spot was not clear. Latest study also suggested that an in-depth study on the mechanisms of single laser-melted tracks formation and instabilities of the molten pool is required so that the application of a wider range of commercially available powder is feasible [5]. Hence, a clear and real time monitoring of the consolidation process in the SLS/SLM is essential.

As a relatively new process with a wide variety of materials being used in the SLS/SLM, behavior and performance of the consolidated material are utmost important. Furthermore, since the SLS/SLM process is based on the transformation of the metal powder to the consolidated structure under high temperature, information on temperature during the SLS/SLM is crucial. During the SLS/SLM processing, there are continuous heating and solidification of metal powder within a very short period. Hence, temperature evolution and profile in the SLS/SLM has a significant effect on the final quality of the consolidated part. Temperature measurement enhances better understanding of the interaction between the laser beam and powder bed [6].

A lot of research tries to understand consolidation behavior through visualization of the process. However, only limited studies are available, and some of the images were not clear enough to allow observation on the transformation process of the metal powder particles to the consolidated structure. Process visualization and monitoring of the SLS/SLM is crucial as it allows observation on the thermo-fluid behavior of the melt pool region and its surrounding during the transformation of the metal powders to the consolidated structure. Since the movement of the laser beam in the SLS/SLM can be relatively fast, utilization of a high-speed camera is essential so that evolution of the melt pool and the consolidated structure formation during the SLS/SLM process can be monitored.

Process visualization of the SLS/SLM with high speed imaging using various methods has been made by many researchers. Visualization of the SLS/SLM was made by Hauser to view track formation revealed a periodic melting of the powder ahead of the main track. It was reported that the tracks did not grow in length steadily but as a series of steps [7]. Series of images was also recorded using high speed infrared camera by Bayle [8]. Based on the research, temperature evolution, phenomena in laser-powder interaction and dynamics of droplets emitted from the molten pool were reported. The author suggested it was important to apply very precise temporal and spatial scales. Other than that, other studies also attempted monitoring of the melt pool during the SLS/SLM. Some of these observations were reported by [9], [10] and [11]. Nevertheless, most of the images were not clear enough. Differ to other studies, this study used filter and orientated the laser in order to enhance the melt pool observation.

The research concentrates on understanding the behavior of the consolidated structure manufactured via the SLS/SLM process. In order to understand this, thermal conductivities of the metal powder and the consolidated material are essential because they affect the heat transfer process. Although the SLS/SLM process has successfully developed the consolidated structure from the metal powder, the consolidated structure formation and irregularities of droplets known as balling, porosity and surface quality still not currently understood. Deen understanding and triggering mechanism that causes the metal powder to consolidate are the main interest in the SLS/SLM as it can assist further understanding of the process. This research concentrates on the consolidation behavior of metal powder in AM. Hence, the objectives of the research are:

a. To study the thermal conductivity of the metal powder and the consolidated material.

b. To analyze the sintering/melting temperature during the SLS/SLM process

c. To develop a methodology that enables monitoring of the consolidation process of metal powder during the SLS/SLM so that the characteristics and mechanism during laser irradiation on metal powdered surface can be understood.

e. To investigate the properties of the consolidated material and its feasibility for the development of a permeable structure.

2. Experimental Procedure

2.1 Material

SEM image of the material used in the study is shown in Fig. 1. The material is a mixture of 70% chromium molybdenum steel, 20% copper and 10% nickel with the average particle diameter of 25 μ m.



Fig. 1 SEM image of the material

2.2 Thermal Conductivity Measurement

Thermal conductivity measurement was performed using thermocouple principle and laser flash method for the metal powder and the consolidated material respectively. In determining the porosity percentage, SCION Image was used as illustrated in Fig. 2. The porosity percentage was calculated based on the equation (2).

Porosity,
$$\emptyset = \frac{A_c - A_{Non-pore}}{A_c} \times 100$$
 (2)

 \emptyset = porosity

 A_C = area of the consolidated surface $A_{Non pore}$ = area of the non-pore surface



Fig. 2 Porosity determination using Scion Image

The influence of laser power and scan speed on the thermal conductivity and the porosity were investigated. The influence of scan speed and laser power can also be further illustrated by referring to the combined effect of laser power (P), scan speed (V) and hatching size (H) known as energy density (E). The relation of energy density to the parameters used in the SLS/SLM is given by equation (1). Therefore, the porosity of the consolidated material at various energy density values was determined.

Energy density,
$$E = \frac{\text{Laser power}}{\text{Scan speed x hatching size}}$$
 (1)

2.3 Temperature Measurement

Schematic of the experimental setup for temperature measurement is shown in Fig. 3. The temperature measurement was performed on AM facility manufactured by Matsuura Machinery Corporation (Japan) Model 25C. The laser used in the study is Yb:fiber laser. The chalcogenide optical fiber was placed at 45 degrees from the substrate surface. The distance between the optical fiber to the consolidated line structure was set at 4.2 mm.

The technique used two-color pyrometer, which integrates chalcogenide optical fiber, condenser lens, germanium (Ge) filter and detectors. The optical fiber is used to transmit the signal from the target area to the detectors. The detectors were Indium Arsenide (InAs) and Indium Antimonide (InSb). The detectors were mounted in a sandwich configuration in order to cater different range of the acceptable wavelengths. InAs was used to detect radiation from 3 to 5.5 μ m.

The infrared rays emitted during consolidation of metal powder within target spot size area was captured by chalcogenide fiber, which then converted from infrared energies into the electric signal after being amplified. The ratio between the voltage outputs from the detectors can be correlated to the temperature during consolidation with reference to the calibration curve.



Fig. 3 Temperature measurement setup

2.3 Consolidation Behavior and Mechanism



Fig. 4 Setup of consolidation monitoring apparatus

		Laser 1	Laser 2
Model		SUNX	IPG Photonics
		Ltd.	(YLR-3000-
		(LP-F10)	AC-Y11)
Туре		Yb:fiber	Yb:fiber
Wavelength [nm]	λ	1070	1070
Beam diameter at		45	50
focal spot [µm]			
Power [W]	Р	1 - 40	50 - 150
Scan speed	V	1 - 200	100 - 500
[mm/s]			

Table 2 Experimental conditions

High speed camera			
Model	FASTCAM SA5		
Shutter speed [fps] f	10,000		
Resolution	768 x 648		
Light source	Metal halide lamp		
Filter			
Model	Sigma Koki (YL- 500P-Y1)		
Material	Polymethyl metaacrylate		
Thickness [mm]	3.5		
Size [mm]	50 x 50		

In order to monitor the consolidation behavior of metal in the SLS/SLM, the experimental setup as shown in Fig. 4 was prepared. The Yb:fiber laser beam was placed at 45 degree angle from the surface. Metal halide lamp was used to ensure sufficient light was given to the laser irradiation region and its surrounding. Consolidation process was done in a closed test chamber with nitrogen-controlled atmosphere.



Fig. 5 Line consolidation characteristics

The laser source and condition used in the experiment are tabulated in Table 1 and Table 2 respectively. The influence of laser power and scan speed on line consolidation characteristics were analyzed. The analysis was made with respect to characteristics defined in the study. These characteristics are consolidation width, melt pool, powder fusion zone (FZ) region, splattering behavior Consolidation consolidation mechanism. and characteristics are illustrated in Fig. 5.

2.5 Properties of the Consolidated Structure

Properties of the consolidated material with respect to surface quality, permeability, hardness and strength were evaluated. The permeability was studied in order to investigate its feasibility in developing a permeable structure through the SLS/SLM. The relation of the properties to the porosity and the processing parameter were analyzed.

A sectional view of the permeability measurement equipment is shown in Fig. 6. The equipment was designed and fabricated to measure the permeability of the prepared specimen. The compressed air from the compressor flowed through a control valve and then travelled through a pressure regulator in order to control the amount of the air that flowed to equipment. The flow meter was used to confirm and maintain the flow rate of the compressed air prior to its flow inside equipment. When the specimen was mounted inside the equipment, the specimen separates the equipment into two different chambers. The chambers contained the pressured air before and after flowing through the porous consolidated material. In order to measure the permeability of the specimen, the pressure at each chamber was measured with a pressure transducer. The pressure transducer was also connected to the oscilloscope for pressure measurement.



3. Results and Discussions

3.1 Thermal Conductivity

0 0



Porosity Fig. 8 Effect of porosity on thermal conductivity

Ø %

20

30

40

50

10

Fig. 7 indicates the relation of energy density on the porosity of the consolidated structure. The figure shows decreasing porosity with the increase of energy density. The graph shows before energy density of 3 J/mm², the porosity is decreasing abruptly to a small change in energy density. However, after 3 J/mm², the porosity does not decrease significantly with the increase of energy density.

Table 3 Thermal conductivity result

Powder,	Consolidated,	Solid,
K_{powder} W/(m·K)	<i>K_{consolidated}</i> W/(m·K)	<i>K_{solid}</i> W/(m·K)
0.15	2.2 to 8.3	42.60

Fig. 8 shows the effect of percentage of porosity on thermal conductivity. The result indicates that thermal conductivity was decreasing with the increase of porosity. This is due to the increasing percentage of scattered pores. The pores were subjected to convective heat transfer mechanism, which reduced the overall heat transfer rate to measuring point. Average thermal conductivity of the consolidated material was compared with thermal conductivity of the metal powder and the same material in the form of solid material. The comparison is shown in Table 3.

Based on the result, it can be deduced that air gap among metal powders particles contributed to low thermal conductivity of the metal powder. Whereas the scattered pores significantly affect the thermal conductivity in the consolidated material. High difference in thermal conductivity among the metal powder, the consolidated material and the solid material was attributed to distinctive the heat transfer mechanism and porosity in each material state.

3.2 Temperature Measurement





Fig. 10 Influence of processing parameter on temperature

Fig. 9 shows the temperature calibration curve which was obtained in the study. The line denotes the theoretical curve, which can be calculated based on the sensitivity of various material and parts used in the developed pyrometer system. On the other hand, the points indicate the experimental result. It can be observed that the experimental points approximately fit the theoretical curve. Therefore, throughout the experiment, conversion of ratio output to temperature was made using this calibration curve.

Fig. 10 indicates the influence of laser parameter on the consolidation temperature during the SLS/SLM process. Based on the relationship, the temperature was increased with the rise of laser power. In contrast, the temperature was decreasing with the increase of scan speed. This behavior was contributed by the amount of heat radiated on the powdered surface during consolidation. When energy density within the localized heating region was relatively high, the heat that was radiated initially liquefied the outer layer of the metal powder and subsequently melted the metal powder. Due to the very thin layer of metal powder thickness of 50 µm, the heat was transferred to the substrate surface. The heat was then transferred radially through the substrate material circumferentially mainly by conduction. This was due to a high difference in the thermal conductivity of cold-rolled substrate surface (56W/(m•K)) in comparison to the thermal conductivity of the metal powder (0.14 W/(m•K)). Higher laser power caused an increase of energy density per unit area around the laser irradiation spot, and the energy was not fully transferred to its surroundings. As a result, the increase of the temperature was observed with the increase of the laser power.

3.3 Consolidation Behavior

The influence of laser power on the consolidation behavior was monitored at different laser power ranging from 1 to 150 W, scan speed of 50 mm/s and 50 micron thickness. Based on the analysis, it was found that the increase of laser power had affected the line consolidation width where it was increasing with the rise of laser power. The influence of laser power on the consolidation width is presented in Fig. 11

In contrast, the increase of scan speed resulted in narrower line consolidation width, smaller FZ and smaller consolidated agglomerate compared to when the laser power was increased. The influence of scan speed on the line consolidation width before solidification, and after solidification is illustrated in Fig. 12.



consolidation width



consolidation width

Influence of laser power on the FZ at the scan speed of 1 mm/s and 100 mm/s is graphically depicted in Fig. 13. The size of the FZ was increasing with the increase of laser power for both scan speed of 1 mm/s and 100 mm/s. It was observed that as the laser beam irradiated on metal powder, a larger region of the FZ was formed. The influence of scan speed on the FZ at the laser power of 40W and 150W when the scan speed was varied from 1 to 200

mm/s is illustrated in Fig. 14. The figure shows at both laser power of 40W and 150W, the FZ is decreasing with the increase of scan speed.



Fig. 13 Influence of laser power on powder FZ



Fig. 14 Influence of scan speed on the powder FZ

In this research, the process map of the metal powder consolidation was also determined. The laser was varied from 1 to 150W, and the laser scan speed range was from 1 to 250 mm/s. The line consolidation quality is shown in Fig. 15. The process map represents the general consolidation characteristics that can be attained from the SLS/SLM process when the metal powder is irradiated with the laser beam in the range of 1 to 150W and scan speed of 1 mm/s to 250 mm/s.

As shown in the figure, changes in the line consolidation characteristics were observed in term of the line consolidation width, FZ, consolidated structure and splattering behavior. The consolidation types can be classified as continuous consolidation, consolidation, discontinuous ball-shaped consolidation, weak consolidation and very little consolidation. Continuous consolidation is preferable compared to other types. This form of consolidation is considered as good because the structure is connected to each other in a straight line and formed relatively dense surface, which penetrate to the substrate surface.









Fig. 16 Consolidation mechanism of ball shaped type consolidation

Laser scan direction



consolidation

Images from the high-speed camera are useful in understanding the consolidation mechanism. The consolidation process can be schematically illustrated in the Fig. 16 and Fig. 17. The figures show the top view of the consolidation process of the ball-shaped type consolidation and the continuous type consolidation respectively. For the ball-shaped type consolidation, the laser was set at 40 W, whereas the scan speed was 50 mm/s. As the laser beam was irradiated on powdered surface, the powder was heated. When the energy was sufficiently high, the melt pool was formed. Surface tension caused movement the molten powder from the circumferential powder to the center of the laser beam as illustrated in Fig. 16(b). Further heating caused the molten powder to grow up gradually. This is depicted in Fig. 16(c). However, the melt pool was relatively very small. As a result, the melt pool transformed to the ball shaped directly. When the laser beam moved away, heat was dissipated from the molten powder to the surrounding. This caused solidification of the molten powder. In contrast, in the continuous type consolidation, a bigger melt pool was formed. This is indicated in Fig. 17. The bigger melt pool enhanced stability of the liquid cylinder. Hence, mixing of molten powder at a high temperature occurred, which allowed the formation of the continuous consolidated structure.

Investigation of surface temperature allowed segregation between SLS and SLM. Fig. 18 is sintering condition of metal powder. The images of the molten powder at melting condition are depicted in Fig. 19 and Fig. 20. The main component of the powder mixture was steel, which the melting temperature was 1540 °C. Therefore, when the temperature is below than the melting temperature, only sintering of metal powder occurred. This is shown in Fig. 18. The steel powder was partially melted, and the shape of the ball-shaped structure was relatively rough. In contrast, when the melting of metal powder was achieved, spherical shaped structure was formed. The shape was obtained due to the effect of surface tension on the molten powder.



Fig. 18 Sintering condition at P = 10 W, V = 88 mm/s



Fig. 19 Melting condition at P = 40 W, V = 88 mm/s



Fig. 20 Melting condition at P = 150W, V = 250mm/s

The consolidation mechanism of the metal powder is illustrated in Fig. 20. During the laser beam movement, transformation of the metal powder to the consolidated structure occurred in sequence of stages. The arrows indicate powder movement direction, splattering direction and shrinkage direction throughout the consolidation process.

Based on the study, it can be summarized that the transformation process of the metal from powder state to the consolidated structure occurred as the laser was irradiated powder surface. Formations of the consolidated structure were highly influenced by the amount of heat on the irradiation spot, which then affected surface tension, and flowability of molten powder. When the laser was beamed on metal powder, localized high temperature occurred at the irradiation spot within a very short time. Sufficient thermal energy liquefied outer layer of metal powder first. Further heating further liquefied the successive outer layer and eventually changed the powder to molten powder. Rapid increase in temperature has caused high-temperature gradient in the consolidated region. The high temperature stimulated physical changes and chemical reaction. The difference in temperature between the edge and center of the liquid/melt pool also contributed to the consolidation characteristics where the flow molten powder was observed. It is generally accepted that surface tension is dependent on temperature. As a result, hightemperature gradient has induced surface tension on the irradiated surface. Since molten powder spread and coexisted simultaneously on the FZ, gradient of surface tension between these molten powders had initiated the movement of molten powder within the FZ. Movement of the metal powder has induced collision between the metal powders, which contributed to the metal powder splattering. The melt pool was formed as metal powder fully melts. Chaotic movement of the powder particle and laser beam on the melt pool has also caused sudden momentum on the melt pool. Furthermore, the entrapped gases between powder particles were under the influence of high temperature. As a result, small explosion occurred that contributed to the splattering of the molten metal. When the laser beam moved

forward in laser direction, the melt pool cooled down, and semi-molten metal formed and then solidified to form the consolidated structure.

3.4 Properties of the Consolidated Structure

Properties of the consolidated material are shown in Table 4. The consolidated permeable structure produced via the SLS/SLM was compared to the commercialized materials. The comparison was made based on the permeability, porosity, surface roughness, hardness, tensile strength and thermal conductivity. It was found that the permeable consolidated structure have comparable properties to the commercialized materials that being produced using other manufacturing techniques and to other mold materials.

Based on the study, it was also found that the property of the consolidated material is highly dependent on the processing parameter used during the SLS/SLM. The porosity is an important property of the consolidated material fabricated via the SLS/SLM. The porosity of the consolidated material is a function of laser power, scan speed, hatching size, spot size and part orientation during manufacturing. The porosity significantly affects other properties of the consolidated material such as surface roughness, permeability, hardness and strength.

Other than that, it was found that the permeable consolidated structure is feasible to be manufactured through the SLS/SLM process. Comparison of its properties to the commercialized permeable insert indicates that the permeable structure manufactured through the SLS/SLM has approximately comparable properties. Selection of the processing parameter is important for its application in developing a permeable mold.



Fig. 21 Influence of porosity on permeability

Therefore, the SLS/SLM is a potential process as the final product can be manufactured relatively fast, and its properties can be controlled through optimization of the processing parameter. This reveals the consolidated material potential as an alternative material in a wide variety of engineering applications and the SLS/SLM as a credible technique that able to revolutionize the manufacturing industry in the near future.

Properties	Consolidated structure	
Permeability (m ²)	3 x 10-11 to 25 x 10-14	
Porosity (%)	5 to 30	
Pore Size (µm)	0.5 to 120	
Surface Roughness (µm)	0.5 to 10	
Hardness (HV)	140 to 250	
Tensile Strength (MPa)	200 to 600	
Thermal Conductivity (W/m·K)	8.3	

Table 4 Properties of the consolidated material

4. Conclusions

Based on the study, it can be concluded that the consolidation process of the metal powder is a very complex process where it involves changes of the metal powder to the consolidated structure at a very high and localized temperatures approximately 2200°C. Laser processing parameters such as laser power, scan speed, layer thickness, underneath surface, hatching size, and others significantly affect the properties of the consolidated structure. Nevertheless, the quality of the consolidated material manufactured via the process is relatively good. This is reflected in the properties of the final consolidated material. Based on the research the following conclusion can be made.

1. The porosity is an important property of the consolidated material. The porosity is highly dependent on the processing parameter used during the SLS/SLM, which finally affect other properties of the consolidated material such as surface roughness, permeability, hardness and strength. Among the most important processing parameters that affect the properties are laser power, scan speed, hatching size, spot size and part manufacturing forming strategy.

2. Within the FZ, all microstructural, changes and transformation of the metal powder to the consolidated structure occurred. Therefore, the area is one important characteristics in the SLS/SLM process, which affect overall part quality.

3. The melt pool, which is located inside the FZ, is the most important behavior since the flow and spread of molten metal occurred within this region. The melt pool formed during laser irradiation relatively larger than the size of the spot size of the laser beam. The size was increased with energy density. Stable and large melt pool size contributed to the formation of good and continuous consolidated structure.

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学位論文審査報告書(甲)

1. 学位論文題目(外国語の場合は和訳を付けること。)

Consolidation Behavior of Metal Powder in Additive Manufacturing

積層造形法による金属粉末の結合特性に関する研究

2. 論文提出者 (1)所 属 <u>システム創成科学専攻</u>機能開発システム講座 もはまど りざる びん あるかはり (2) 発 落 <u>MOHD RIZAL BIN ALKAHARI</u>

3. 審査結果の要旨(600~650字)

平成26年1月31日に第1回学位論文審査委員会を開催し、平成26年2月3日に 口頭発表ならびに第2回審査委員会を開催して慎重に審査した結果、以下のように判定し た.積層造形法は、金属粉末の薄い層にレーザ光を照射して結合し、その層を積層するこ とによって3次元体を造形する方法である、本研究では、粉体にレーザを照射したときの 温度変化を精密に測定し、瞬間点熱源の理論を用いて金属粉体の熱伝導率を実験的に求め ている、次に、金属粉末が結合する過程において、レーザ照射部の温度を赤外線輻射温度 計を用いて測定している。また、粉体が溶融して金属層を形成する様子を高速ビデオによ り観察している。その結果、適正なエネルギー密度の領域があり、溶融した粉体は連続的 に結合され、積層に適した金属層が形成される、エネルギー密度が高くなり過ぎると、溶 融した金属が表面張力によって大きなボールを形成して不連続な金属層を形成する、逆に エネルギー密度が低ければ結合の弱い金属層が形成され、いずれの状態も積層していくこ とはできない、以上のように、本論文は積層造形における金属粉末の熱伝導率及び金属粉 末の結合特性について、これまで得られなかった多くの新しい知見を得ることに成功して おり、博士(工学)論文に値するものと判定した。

4. 審査結果 (1) 判 定 (いずれかに〇印) 合格 · 不合格

(2) 授与学位 <u>博士(工学)</u>