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Laser-assisted Selective Infiltration of Low Melting-point Metal Powders

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Abstract

Laser-assisted selective infiltration is a new method of building metal layers to make metal parts layer by layer, in which superheated microscopic metal droplets are infiltrated into a laser-preheated layer of microscopic metal powders. In this work, the selective infiltration of a low melting-point metal, Sn-37Pb wt%, was conducted to investigate the effects of such dominant parameters as superheating temperature, Nd:YAG laser power for preheating, substrate temperature, etc. The optimal conditions for successful selective infiltration of a single layer of microscopic metal powder were experimentally obtained

Key Words: Rapid Prototyping (쾌속조형), Pulsed Nd:YAG laser (펄스형 Nd:YAG 레이저), Superheated metal droplets (과열된 금속 액적), Laser-preheating (레이저 예열)

1. Introduction

A variety of solid freeform fabrication (SFF) or Rapid Prototyping (RP) processes for making metal parts in a layer-by-layer fashion directly from the CAD solid models have been developed. In comparison with the conventional manufacturing processes, these have distinctive capabilities primarily stemming from the layer-by-layer addition of metals. For example, they are capable of fabricating injection molds equipped with conformal cooling channels that geometrically conform to the mold cavity surface and thus help to reduce product cycle times considerably and to improve defects; and functionally gradient metal parts that comprise two or more different kinds of metals; and smart metal structures that contain sensors or other electronic devices at intended locations¹⁻²⁾. In spite of these unique advantages, however, most of them are suffering from such drawbacks as dimensional inaccuracy, long building time, and residual stress-induced deformation³⁾. This is mainly because each layer undergoes an overall phase change that results in severe residual stress in the fabricated metal parts.

This paper presents a new method of building metal layers, selective infiltration, in which superheated microscopic metal droplets are infiltrated into a laser-preheated layer of metal powder as shown in Fig. 1⁴⁾. Experiments on the selective infiltration of a low melting-point metal are conducted to investigate the effects of

the dominant parameters, such as superheating temperature, laser power for preheating, substrate temperature, etc. and also to obtain the optimal conditions for successful selective infiltration of a layer of metal powder.

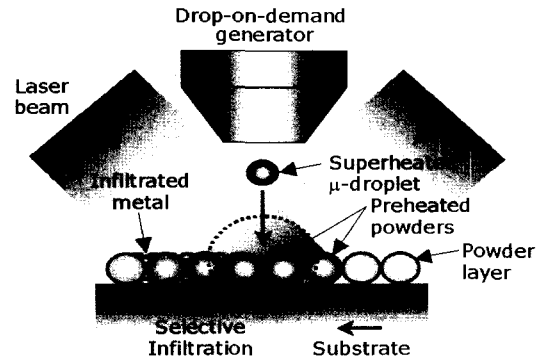


Fig. 1 Schematic of the SIM process.

2. Experimental setup

The experimental setup comprises a drop-on-demand (DOD) generator, an Nd:YAG laser system for preheating, and a substrate which is mounted on an X-Y positioning system. The DOD generator employs a precision solenoid vibrator as a source of impulse whose frequency is synchronized with that of droplet ejection. Since a tin alloy (Sn-37Pb wt%) was used for both droplets and powder, all the components of the DOD generator was made of stainless steel (SUS303) to avoid the molten metal from dripping at the nozzle orifice.

The aspect ratio of the nozzle orifice, which has a significant influence on droplet ejection, must be equal to or smaller than unity⁵⁾. This ensures that

the liquid coming out of the nozzle orifice can more easily break into separate droplets. The nozzle orifice was micro-drilled to be 130 μm in diameter and was ground to be 100 μm in length, so that the aspect ratio was kept less than unity (Fig. 2). The average diameter of superheated droplets ejected from the orifice was about 300 μm (Fig. 3).

For successful selective infiltration, the inside temperature of laser-preheated powders must be kept as low as possible, while that of the powder surface as close to its melting point as possible. Thus, a pulsed Nd:YAG laser was employed whose energy can be concentrated on the powder surface in that the pulsed energy input offers insufficient time for conduction of the absorbed energy into inside of the powders. Due to the working principle of selective infiltration, the laser beam should be delivered to powders at an angle of incidence (Fig. 5). Experiments showed that the amount of energy input from an inclined laser beam to a surface depends on the scanning direction⁶⁾. To minimize the effect of the non-uniform intensity distribution, twin laser beams at a right angle to each other were used for laser-preheating. They were defocused to minimize the thermal gradient in the preheated area. The incidence angle of the twin laser beams was set at 25° with respect to the powder layer surface. To ensure the complete infiltration of superheated microscopic metal droplets into a metal powder layer, which is being

laser-heated, an aluminum plate secured on a hot plate was used as a substrate. The hot plate controls directly the temperature of the aluminum substrate, which has a good wettability to tin-lead alloys.

Table 1 Chemical composition of Sn-37Pb wt%

	wt%
Sn	63.340
Pb	balance
Bi	0.0006
Cu	0.0003
Fe	0.0002
Sb	0.0300
As	0.0001

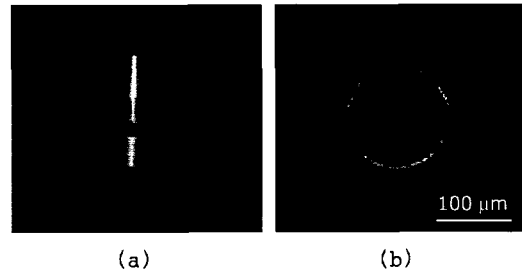


Fig. 2 The μ -nozzle (a) and its μ -drilled orifice (b), which is made of stainless steel.

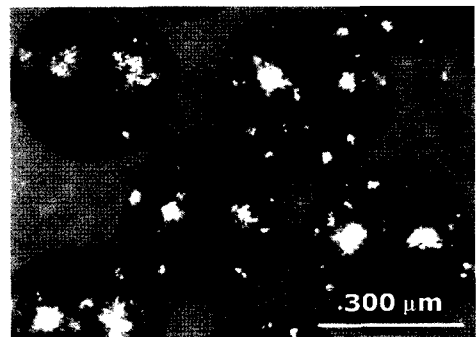


Fig. 3 Generated microscopic droplets of Sn-37Pb wt% at temperature 260 °C.

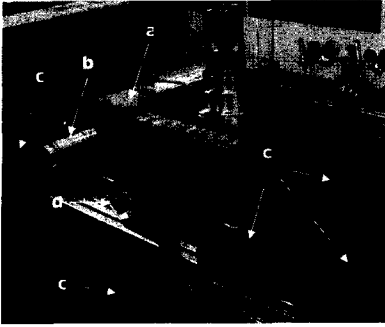


Fig. 4 Optical device arrangement for the laser beam delivery: (a) laser head, (b) collimator, (c) reflectors, and (d) beam splitter.

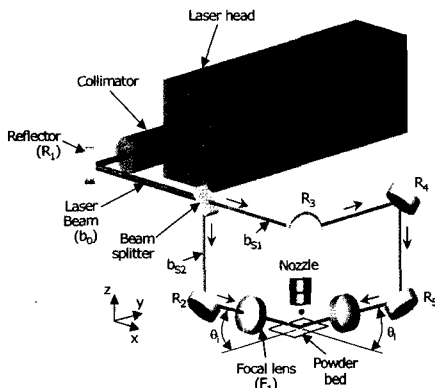


Fig. 5 Schematic of the laser beam delivery for preheating via optical devices (incidence angle of laser beams, $\theta = 25^\circ$)

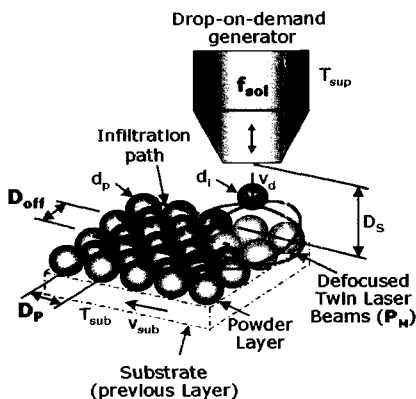


Fig. 6 The dominant process parameters for the selective infiltration of a single layer.

As shown in Fig. 6, the dominant process parameters include the superheating temperature of microscopic metal droplets T_{sup} , the laser power for preheating P_M , the substrate temperature T_{sub} , the frequency of the solenoid vibrator f_{sol} , the standoff distance D_s , and the substrate velocity v_{sub} and its temperature, T_{sub} . The melting temperature of Sn-37Pb wt% is 183°C. Since at or slightly above the melting point the alloy flows still sluggishly, the superheated temperature T_{sup} of the alloy was set at 260, 290, and 320°C in the experiments. Laser energy input to a surface usually depends on various factors, such as surface topology, initial surface temperature, laser power density, incidence angle, etc. For this reason, it is difficult to control directly the preheating temperature. The mean laser power P_M was thus used to indicate the degree of laser preheating. The substrate temperature, which is controlled by a hot plate, also affects the preheating temperature.

3. Selective infiltration of a single droplet

To effectively heat a target surface with a pulsed Nd:YAG laser, it is indispensable to control the pulse duration time t_p and the repetition rate f_L , because these dominate the amount of heat to be absorbed into the surface. Two laser beams, superimposed on the powder surface, can transfer a different amount of heat to the surface according

Table 2 Experimental conditions for the selective infiltration of a single droplet of Sn-37Pb wtx

T_m ($^{\circ}\text{C}$)	T_{sup} ($^{\circ}\text{C}$)	P_M (W)	T_{sub} ($^{\circ}\text{C}$)	v_{sub} (mm s^{-1})	d_i (μm)	d_p (μm)	f_{sol} (Hz)	D_s (mm)
183	260	0	0	1.25	301±10	500±25	2	5
	290	-	-	-	299±10			
	320	43	160	2.50	301±12			

to the combination of the two factors. As general known, it is more favorable to increase t_p than f_L to enhance the heat transfer, especially when the reflectance of a target surface is high.

The mean power of the pulsed Nd:YAG laser at its maximum pulse duration time (1.73 ms) was quantified (Table 3). The results showed that the pulse energy has its maximum value of 1.27 J, when the mean power is set at 33 W and the frequency 26 Hz. This indicates that among the cases in Table 3 where t_p was set at a constant of 1.73 ms, this case can convey the largest effective amount of heat energy to a target surface.

Table 3 Pulse energy at each combination of the pulse duration time and the pulse repetition rate of the pulsed Nd:YAG laser

Duration time (ms)	Repetition rate (Hz)	Measured mean power (W)	Pulse energy (J)	Pulse energy (J)
t_p	f_L	P_M	EP_1	EP_2
at $f_L _{\text{max}} = 35 \text{ Hz}$	at $t_p _{\text{max}} = 1.73 \text{ ms}$		P_M/f_L	$PM/f_L _{\text{max}}$
1.13	14	17	1.21	0.48
1.26	19	23	1.21	0.66
1.37	23	28	1.22	0.80
1.53	26	33	1.27	0.94
1.61	30	37	1.23	1.06
1.66	34	42	1.24	1.20
1.73	35	43	1.23	1.23

■ CASE 1: $T_{\text{sup}} = 260^{\circ}\text{C}$

When $T_{\text{sub}} 140^{\circ}\text{C}$, powders started to collapse at the bottom because of excessive heat input from the hot plate. Thus, the substrate temperature was limited below 140°C . In all cases where $v_{\text{sub}} \geq 1.67 \text{ mm/sec}$ or $T_{\text{sub}} < 120^{\circ}\text{C}$, no complete infiltration occurred because the surface temperature of the powders was too low. In other words, the excessive heat transfer from the droplet to the powders immobilized the molten droplet. Experiments on the effect of laser beams with low pulse energy, i.e., with high frequency and short duration time, were carried out. When the mean laser power varied from 28–43 W, no selective infiltration was observed. Only when the mean power was set at 43 W, the droplet was partially infiltrated. This means that laser beams with high frequency and short duration time are unsuitable for heating a metal surface. This is similar to the cases of laser welding and laser surface treatments where laser beams with low frequency and long duration time are in use to maximize the heat input.

Based on the previous results, experiments were conducted under the conditions that the pulse duration time was set at its maximum of 1.73 ms, the

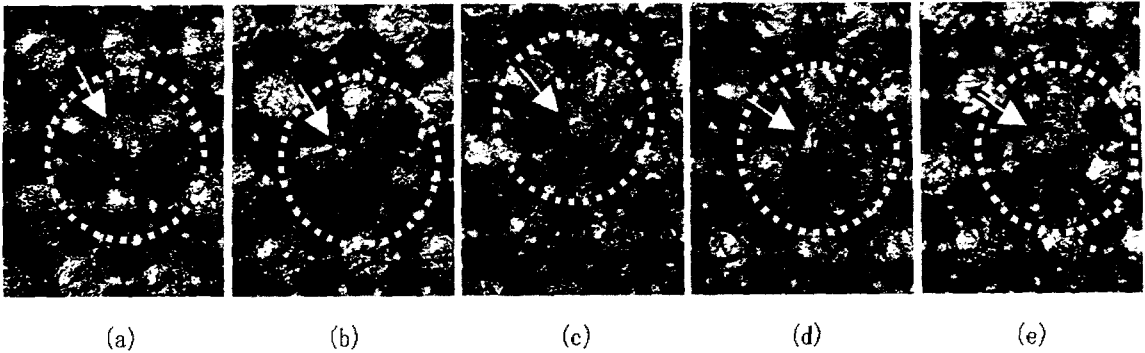


Fig. 7 Selective infiltration of a single 300- μm diameter droplet of Sn-37Pb wt% in case of $T_{\text{sup}} = 260^\circ\text{C}$, $T_{\text{sub}} = 120^\circ\text{C}$, $v_{\text{sub}} = 1.25 \text{ mm/sec}$ and $t_p = 1.73 \text{ ms}$ with varying laser pulse energy and pulse repetition rate: (a) $P_M = 28 \text{ W}$, $E_P = 1.22 \text{ J}$, $f_L = 23 \text{ Hz}$, (b) $P_M = 33 \text{ W}$, $E_P = 1.27 \text{ J}$, $f_L = 26 \text{ Hz}$, (c) $P_M = 37 \text{ W}$, $E_P = 1.23 \text{ J}$, $f_L = 30 \text{ Hz}$, (d) $P_M = 42 \text{ W}$, $E_P = 1.24 \text{ J}$, $f_L = 34 \text{ Hz}$, and (e) $P_M = 43 \text{ W}$, $E_P = 1.23 \text{ J}$, $f_L = 35 \text{ Hz}$. 500- μm diameter powders were used. Only when $P_M = 33 \text{ W}$, $E_P = 1.27 \text{ J}$, $f_L = 26 \text{ Hz}$, a successful infiltration occurred (b).

substrate velocity at 1.25 mm/sec and the substrate temperature at 120°C . As shown in Fig. 7, only when $P_M = 33 \text{ W}$ and $f_L = 26 \text{ Hz}$, i.e., in case when the combined laser beam has its maximum pulse energy (Table 3), the selective infiltration occurred successfully.

■ CASE 2: $T_{\text{sup}} = 290^\circ\text{C}$

When $v_{\text{sub}} \geq 1.67 \text{ mm/sec}$, $T_{\text{sub}} < 120^\circ\text{C}$, or $P_M < 28 \text{ W}$, no infiltration occurred. When $P_M > 33 \text{ W}$, selective infiltration did not occur at any combinations of the substrate temperature and velocity. Even though the superheating temperature of droplets rose by 30°C , a complete infiltration occurred only under the condition where $T_{\text{sub}} = 120^\circ\text{C}$, $P_M = 33 \text{ W}$, and $v_{\text{sub}} = 1.25 \text{ mm/sec}$.

■ CASE 3: $T_{\text{sup}} = 320^\circ\text{C}$

No selective infiltration was observed in this case. Even with the maximum pulse energy, only incomplete infiltration could be achieved. In case that P_M was set as low as 17 W, a droplet at a temperature of 320°C melted down the surfaces of the three laser-preheated powders. The excessive superheating temperature of the droplet caused the top surfaces of the powders to partially collapse.

4. Selective infiltration of a series of droplets

There are two geometrical factors related to the deposition of superheated droplets: pitch and offset. The pitch D_p is defined as the center distance between

two adjacent droplets successively ejected from the nozzle orifice, while the offset D_{off} indicates the distance of the centerlines of two neighboring rows of droplets (Fig. 6). These two factors determine the density and thickness of a selectively infiltrated layer

4.1. Pitch of selective infiltration

The pitch of droplet deposition can be controlled in two ways, either by changing v_{sub} or f_{sol} . It is easier to control f_{sol} , because in case of changing v_{sub} , the laser power needs to be accordingly adjusted to keep the heat input to the powder layer constant.

v_{sub} was set constant at 1.25 mm/sec, which indicates no change of heat input from the laser beams to the powder layer. To control D_p , f_{sol} was changed within a range of 2-4 Hz; so, for each case, the number of droplets to fall within a length of 1.25 mm is 2, 3, and 4, respectively. It is possible to calculate D_p for each case assuming the average droplet diameter d_i is approximately 300 μm . D_p at each f_{sol} is 625, 417, and 312 m, respectively. Since each D_p used in the experiments is larger than d_i ejected from the nozzle, no collision between falling droplets occurs. The diameter of powder d_p was about 500 μm .

■ Pitch: $D_p = 625 \mu\text{m}$ ($f_{sol} = 2 \text{ Hz}$)

Since D_p was too large compared to d_i , a dotted line instead of an intended solid line was formed. In both cases, however, selective infiltration of each droplet occurred successfully with an intended pitch.

■ Pitch: $D_p = 417 \mu\text{m}$ ($f_{sol} = 3 \text{ Hz}$)

As shown in Fig. 8, at both superheating temperatures, the selective infiltration of a solid line was achieved successfully. In other words, the deposited droplets were fully infiltrated into the powder layer to the bottom. The width of the selectively infiltrated line is wider when $T_{sup} = 290^\circ\text{C}$. This is due to the relatively decreased viscosity of droplet (or increased fluidity) at a superheating temperature of 290°C .

Unlike the selective infiltration of a single droplet, successive infiltration of droplets could raise the temperature of the powders on which the next droplet lands. The extra heat input accumulated from the previous infiltration caused the partial collapse at the bottom of the powders, which was more severe at $T_{sup} = 290^\circ\text{C}$. This reveals the importance of the infiltration path design for practical parts to minimize the heat concentration. Without preheating (Fig. 10(a)), no infiltration occurred and the powders on

Table 4 Experimental conditions for the selective infiltration of a series of droplets of Sn-37Pb wtx to build a metal powder layer

T_{sub} ($^\circ\text{C}$)	d_i (μm)	P_M (W)	T_{sub} ($^\circ\text{C}$)	v_{sub} (mm/sec)	d_p (μm)	D_{off} (μm)	f_{sol} (Hz)	D_s (mm)
260	301±10	33	120	1.25	500±25	300	2	5
290	299±12					400	3	
						500	4	

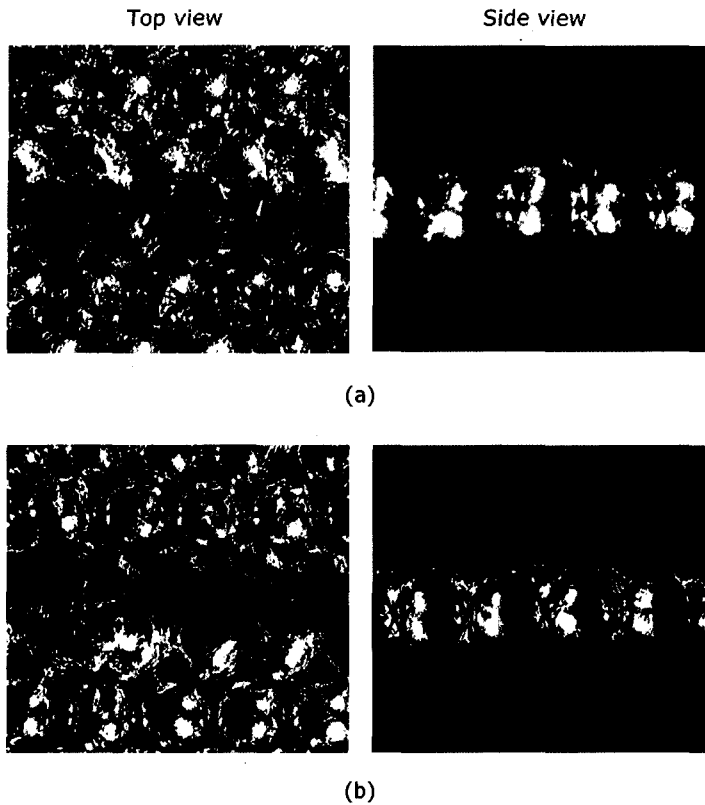


Fig. 8 Selective infiltration of a line of superheated 300- μm diameter droplets of Sn-37Pb wt% into a layer of 500- μm diameter powders of Sn-37Pb wt% under the conditions that $f_{\text{sol}} = 3 \text{ Hz}$, $v_{\text{sub}} = 1.25 \text{ mm/sec}$, $DP = 417 \mu\text{m}$, $PM = 33 \text{ W}$, and $T_{\text{sub}} = 120\text{C}$: (a) $T_{\text{sup}} = 260\text{C}$ and (b) $T_{\text{sup}} = 290\text{C}$.

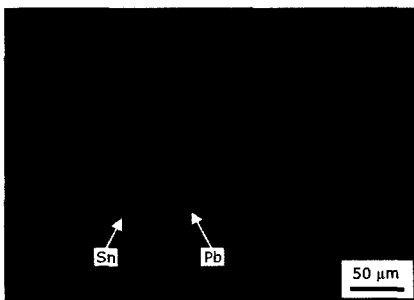


Fig. 9 Photo of microstructure of a selectively infiltrated solid line.

the substrate still maintained their spherical shape. On the contrary, with preheating successful infiltration occurred and the local collapse at the bottom of the powders was observed.

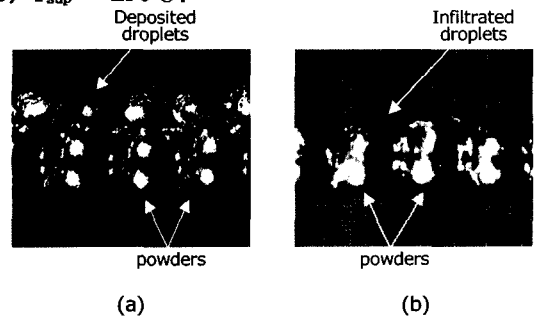


Fig. 10 Selective infiltration of a line of superheated 300- μm diameter droplets of Sn-37Pb wt% into a layer of 500- μm diameter powders of Sn-37Pb wt% under the conditions that $f_{\text{sol}} = 2 \text{ Hz}$, $v_{\text{sub}} = 1.25 \text{ mm/sec}$, $D_p = 417 \mu\text{m}$, $T_{\text{sub}} = 120\text{C}$, and $T_{\text{sup}} = 260\text{C}$: (a) without laser preheating and (b) with laser preheating ($P_H = 33 \text{ W}$).

■ Pitch: $D_p = 312 \mu\text{m}$ ($f_{\text{sol}} = 4 \text{ Hz}$)

The width of the solid line at $T_{\text{sup}} = 290^\circ\text{C}$ was the wider than that of at $T_{\text{sup}} = 260^\circ\text{C}$. The oversupply of molten droplets caused the powders to collapse at the bottom. Also, the thickness of the selectively infiltrated solid line was larger than the powder diameter, which is undesirable for the infiltration of next layer.

3.2. Offset selective infiltration

Under the conditions obtained from the experiments for the optimal D_p , experiments for the optimal D_{off} were conducted in which D_{off} ranged from 300-500 μm . To control D_{off} , the feed of the X-Y table perpendicular to the infiltration path was adjusted. The previous experiments showed that the optimal D_p was 417 μm , i.e., three droplets over a length of 1.25 mm. To obtain the optimal feed to guarantee the layer thickness equal to the powder diameter, two-line selective infiltration was carried out.

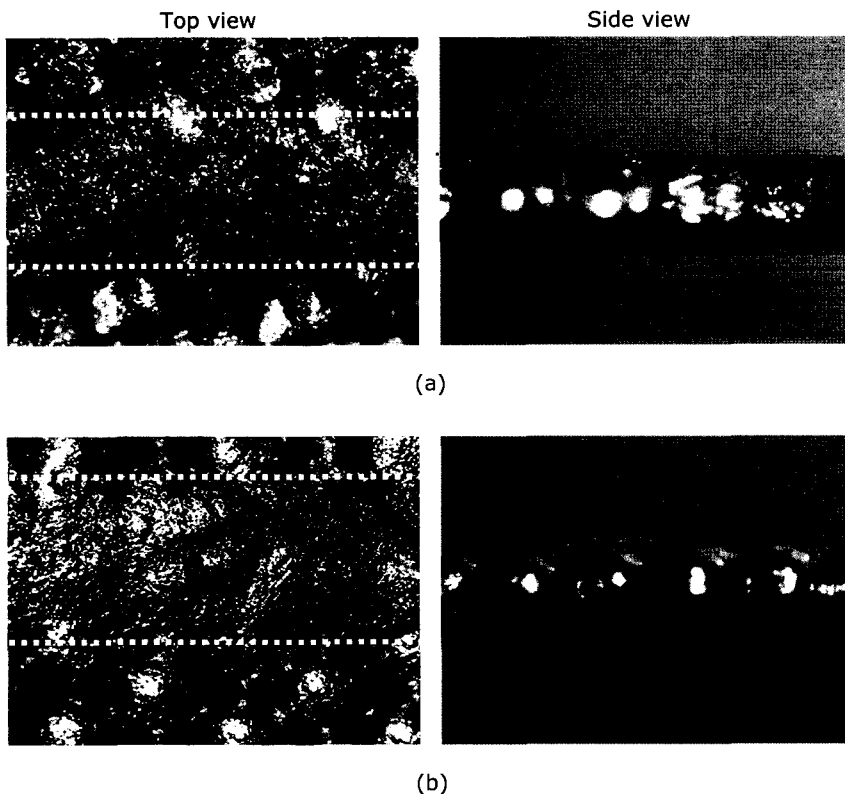


Fig. 11 Selective infiltration of two lines of superheated 300- μm diameter droplets of Sn-37Pb wt% into a layer of 500- μm diameter powders of Sn-37Pb wt% under the conditions that $f_{\text{sol}} = 3 \text{ Hz}$, $v_{\text{sub}} = 1.25 \text{ mm/sec}$, $D_p = 417 \mu\text{m}$, $P_H = 33 \text{ W}$, $T_{\text{sub}} = 120^\circ\text{C}$, and $D_{\text{off}} = 400 \mu\text{m}$: (a) $T_{\text{sub}} = 260^\circ\text{C}$, $W = 850 \mu\text{m}$, and (b) $T_{\text{sub}} = 290^\circ\text{C}$, $W = 900 \mu\text{m}$. Successful selective infiltration.

■ **Offset: $D_{\text{off}} = 300 \mu\text{m}$**

In this case, the thickness of the selectively infiltrated line is greater than the powder diameter, the optimal value. This is due to the excessive supply of droplets, i.e., the offset is too small. Even though the molten metal infiltrated the desired region completely, the superfluous material solidified above the powder layer would prevent the preparation of next layer.

■ **Offset: $D_{\text{off}} = 400 \mu\text{m}$**

In case of $D_{\text{off}} = 400 \mu\text{m}$, at both superheating temperatures, the results were successful. As shown in Fig. 11, the thickness of the selectively infiltrated line is approximately equal to the powder diameter. The width of the selectively infiltrated line at $T_{\text{sup}} = 290^\circ\text{C}$ is the wider than that at $T_{\text{sup}} = 260^\circ\text{C}$, because the viscosity of molten metal decreases as temperature increases. The widths of the lines at $T_{\text{sup}} = 260, 290^\circ\text{C}$ measured to be 850 and 900 μm , respectively. The dotted lines in Fig. 11 were used to measure the width.

■ **Offset: $D_{\text{off}} = 500 \mu\text{m}$**

In this case, incomplete selective infiltration occurred because of the insufficient supply of molten metal.

Sn-37Pb wt% have drawn the following:

1. Experiments on the selective infiltration of a single droplet with various dominant parameters, such as the superheating temperature T_{sup} , the substrate temperature T_{sub} , the substrate velocity v_{sub} , and the mean laser power P_m , were carried out. Results have shown that the selective infiltration occurred successfully under the following conditions: $T_{\text{sup}} = 260^\circ\text{C}, 290^\circ\text{C}$, $P_m = 33 \text{ W}$ ($t_p = 1.73 \text{ ms}$ and $f_l = 26 \text{ Hz}$), $T_{\text{sub}} = 120^\circ\text{C}$. When $T_{\text{sup}} = 320^\circ\text{C}$, no complete selective infiltration was observed, because the excessive superheating temperature of the droplet caused the top surfaces of the powders to collapse.

2. To examine the effect of the geometrical factors of droplet deposition, i.e., pitch, D_p and offset, D_{off} , experiments on selective infiltration of a line(s) of superheated microscopic droplets have been conducted. Experiments have shown that when D_p and D_{off} were set at 412, 400 μm , respectively, the thickness of a selectively infiltrated solid line was virtually equal to the powder diameter, the optimal layer thickness, at both T_{sup} of 260°C and 290°C . In the two-line selective infiltration, the width was wider at $T_{\text{sup}} = 290^\circ\text{C}$ than at $T_{\text{sup}} = 260^\circ\text{C}$.

4. Conclusions

The experiments on the selective infiltration of superheated microscopic droplets of Sn-37Pb wt% into a layer of the Nd:YAG laser-preheated powders of

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