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Pulsed laser sintering of metallic powders

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Abstract

We have investigated the interaction of near infrared pulsed laser radiation with metallic powders both theoretically and experimentally at 1.064 μm with a pulsed Nd:YAG laser. The lower average power requirement (below 100 watts) in the case of pulsed interaction, predicted by calculations, has been confirmed. Temperature measurements show that a lower average temperature occurs whereas consolidation takes place at much lower average power compared to continuous wave sintering. The sintered material has been analysed and the results are compared to the calculation predictions: material analysis has shown that only a narrow surface layer of the individual powder particles has been affected by the interaction. Therefore, significant material modifications only take place in the mentioned surface layer of the single grains whereas the main part of the material remains unchanged. Furthermore, surface finishing of the work piece by means of a pulsed Nd:Glass laser have been performed, which leads to a bulk-like density at the surface.

Keywords: Laser irradiation; Titanium; Sintering

1. Introduction

Traditionally, laser sintering of metallic powders is accomplished at high average powers by means of a CO_2 laser source [1]. In this case the powder is nearly homogeneously heated and sintered, with reported lateral precision of several tens of micrometres. Using pulsed Nd:YAG radiation bears several advantages: on the one hand is the possibility of a higher lateral precision due to its smaller wavelength, where the lateral precision is then given by the used grain sizes of the powder. On the other hand, pulsed laser sintering can be achieved at moderate laser powers, typically below 10 watt. Upon the interaction of a laser pulse with a powder grain, a temperature distribution in the grain builds up, where the surface is at a much higher temperature (skin temperature) than the rest of the grain. Within several microseconds, temperature homogenisation takes place and the whole grain is at a much lower average temperature. Therefore, balling effects can be minimised and finally less residual stresses in the work piece are expected. By adapting the pulse length to the grain size of the used metal powder, a totally different sintering mechanism can be achieved.

2. Theoretical background

On the basis of the interaction model of near infrared pulsed laser sintering of metallic powders, the temperature evolution can be calculated [2].

A rough estimation based on the thermal diffusion length δ_{th} shows that the amount of heated material depends strongly on the pulse duration τ.

$$\delta_{th} = 2\sqrt{\kappa\tau} \qquad (1)$$

For a titanium grain with a thermal diffusivity of $\kappa = 9.3\times10^{-6}$ m^2/s and a diameter of 20 μm, irradiated with pulses longer than 10 μs, the entire grains becomes uniformly heated. Upon the interaction of a 1 μs pulse, the heat diffusion length reduces already to 6 μm and only a shell of approximately 20% of the volume of the grain is heated during the interaction. Less than 1% of the entire grain volume is heated upon the interaction with 100 ns pulses, as the thermal diffusion length is then less than 2 μm. The same ratio of reduction of needed energy for starting sintering is a consequence of short time interaction.

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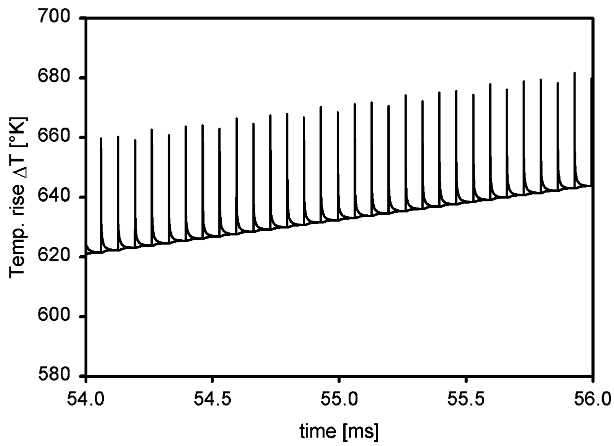

Fig. 1. Thermal evolution after 50 ms upon the interaction of titanium with a pulsed Nd:YAG laser (150 ns pulses at a repetition rate of 15 kHz).

The temperature rise after a small number of pulses is sufficient to melt the surface layer of the single powder grain (described by the skin temperature rise ΔT_{Skin}), whereas the centre of the individual grain remains at nearly room temperature and acts subsequently as an efficient heat sink. The surrounding powder, approximated as a continuum with the measured physical properties (such as thermal diffusivity, optical penetration depth and powder density), remains at the average temperature, which is much lower than the peak skin temperature. Thus, only the surfaces of the powder grains are molten and connected. As shown in Ref. [2], the average temperature rise $\Delta T_{\text{average}}$ of one grain after the interaction time t can be approximated as:

$$\Delta T_{\text{average}}(t) = \alpha t \tag{2}$$

where $\alpha = 1.148 \times 10^4$ K/s in the case of the considered titanium powder, a beam diameter of 100 μm, an average laser power of 3 watts, a repetition rate of 5 kHz and a scan speed of 1 mm/s. Eq. (2) describes the baseline in Fig. 1, whereas α is the slope. The temperature evolution is pictured in Fig. 1, whereas the peaks represent the skin temperature rise at a repetition rate of 15 kHz. Upon the interaction at a repetition rate of 5 kHz at the same average power, the slope (α) remains the same but the skin temperature rise after one pulse is three times higher. For a repetition rate of 5 kHz and an interaction time of 0.1 s (which corresponds to the time required for the beam to pass over its own diameter) the peak temperature rise then becomes $\Delta T_{\text{Peak}} =$ 1314 K (=1041 °C) when the average power is 3 watts. This value is well under melting temperature but sufficient for liquid phase sintering.

3. Materials and methods

3.1. Powder

We used a commercially pure spherical titanium powder (from Pyrogenesis) with grain sizes smaller than 30 μm. The grain size has a Gaussian distribution with a peak at 8 μm, whereas most of the mass is contained in the grains with a diameter of 22 μm. The layer density of the Pyrogenesis titanium powder is 2931.5 kg/m^3, which equals to a volume ratio of 64.6% compared to bulk titanium (density 4540 kg/m^3). The optical penetration depth (OPD, defined as the depth where the intensity of the radiation is at $1/e$ of the non-back-scattered intensity of the Nd:YAG radiation) was measured to be 63 μm. The thermal conductivity of the titanium powder was found to be $k_{\text{Titanium powder}} = 1.45$ W/m K, which allows calculation of the thermal diffusivity $\kappa = 1.481 \times 10^{-6}$ m^2/s [3].

3.2. Experimental set-up

The used equipment consists of a Q-switched Nd:YAG laser with 3 watts average power in fundamental mode emitting 150 ns (FWHM) pulses at a repetition rate between 1 and 30 kHz. The laser beam was focused to a diameter of approximately 100 μm on the powder bed surface which was placed in argon protective atmosphere. With a scan speed of 1 mm/s the beam requires 0.1 s to travel over its own diameter. We used a Raytheon Radiance HS infrared camera with a spectral band pass filter with transmission between 3 and 5 μm. The objective allowed a picture size of approximately 2.8×2.8 mm^2 to be obtained, with a resolution of 256×256 pixels at a frame rate of 10 frames per second and an integration time of 0.01 ms per frame. The calibration was performed with a pyrometer.

3.3. Analysis

The samples were metallographically prepared, thus mounted in a Duroplast with electrical conductivity, SiC ground, diamond polished and etched. Optical and scanning electron microscopy allowed the different microstructures to be distinguished.

4. Experiments

Experiments with two different lasers at different parameter sets, pulsed and continuous wave, have been performed.

4.1. Temperature measurements

The infrared camera used (Raytheon Radiance HS) was driven at a frame rate of 10 fps and was thus not

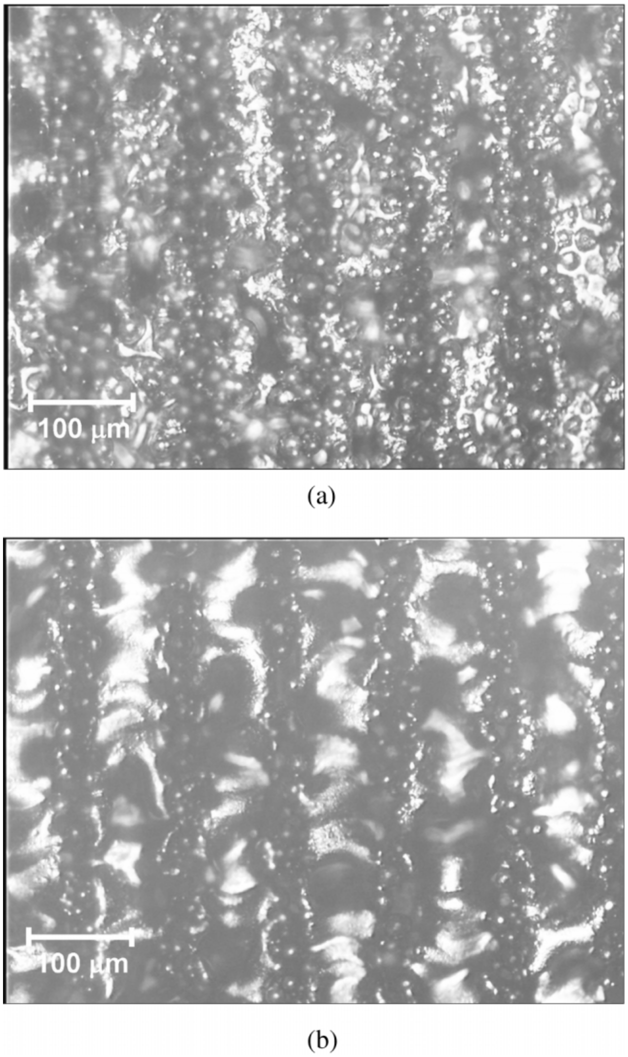

Fig. 2. Optical micrograph of (a) continuous wave (3 W average power) and (b) pulsed sintered titanium (2 W average power). The observed difference is due to the presence of the temporarily higher skin temperature in the case of pulsed interaction.

able to resolve the temperature rises upon the interaction of the single laser pulses. Therefore, the obtained temperature field represents the average temperature [4].

4.1.1. Continuous wave interaction

With continuous wave radiation consolidation occurred already starting from an average power of 3 watts. Fig. 2a shows an optical micrograph of the surface of the continuous wave-sintered titanium powder. The path of the laser beam has been chosen in such a manner that a separation from line to line can be observed. Furthermore, the single powder grains are also still visible in Ref. Fig. 2a.

Fig. 3a shows the measurement of the temperature distribution during the sintering process when irradiating with continuous wave radiation. It shows an area of approximately 1 mm^2 which is at a temperature of approximately 2000 °C and an area of approximately 0.1 mm^2 where the temperature clearly overcomes the melting temperature. This latter area is in the close neighbourhood of the interacting laser beam.

4.1.2. Pulsed interaction

Pulsed laser sintering was achieved at an average power of 2 watts with 150 ns pulses at a repetition rate of 5 kHz. Fig. 2b shows an optical micrograph of the sintered tracks on the powder bed. Here again a scan strategy was chosen which clearly separates the consolidated lines. The single powder grains are no longer visible in the track of the laser beam (the light tracks) in Fig. 2b. Fig. 3b shows the measurement of the temperature distribution during the sintering process when interacting with pulsed radiation at a repetition rate of 5 kHz. It shows an area of approximately 0.1 mm^2 where the temperature is approximately 1500 °C (the close neighbourhood of the laser beam) whereas the rest of the powder surface is at a temperature clearly below 1000 °C. In Fig. 3b one can also see the path of the beam by the darker lines: Where consolidation took place, the thermal conductivity increased and therefore the cooling is faster. The frame rate of the camera is too low for resolving the single pulses and is thus not able to measure the peak skin temperature rises. Thus the measured temperature field represents the average temperature of the powder bed after homogenisation of the temperature within the single grains has taken place.

4.2. Material analysis

The liquid phase sintering takes place in the narrow skin layer (as estimated in Section 2) where the temperature clearly overcomes the melting point, whereas the inner part of the grains remain at a much lower temperature. This molten layer can be seen in Fig. 4. The layer thickness is between 700 nm and 1 μm.

The microhardness of the centre of the grain was found to be 200 HV 0.01, which is the value given by the provider for the initial, unsintered powder. On the other hand, the microhardness of the binding material (which consists of the totally molten smaller grains and fills the void between the bigger grains) is approximately 800 HV 0.01.

4.3. Different porosities and surface finishing

By the change of the laser parameters, the same powder can be sintered with different porosities (as shown in Fig. 5a,b). In this case, only the repetition rate was changed whereas the average power was kept at a comparable level of 3 and 3.2 watts, respectively. Surface finishing was obtained by a second processing step using 40 pulses of a Nd:Glass laser with 10 ns

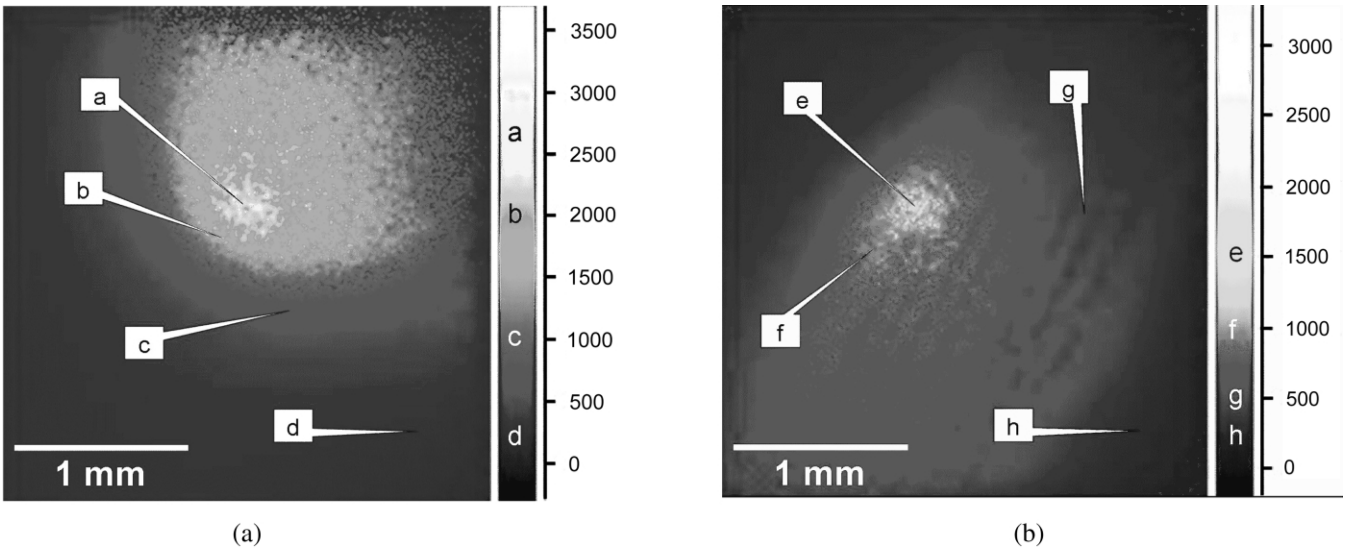

Fig. 3. Measured average temperature field during the (a) continuous wave and (b) the pulsed sintering process. The average temperature rise upon continuous wave sintering is much higher due to the higher average power (3 watts) compared to pulsed sintering (2 watts). The camera is not able to resolve the temporarily higher skin temperature in the case of pulsed interaction. Nevertheless, sintering is stronger upon pulsed sintering, as can be seen from Fig. 2.

pulse duration, a spot diameter of 2 mm and an energy of 200 mJ/pulse.

5. Discussion

During continuous wave interaction, the grains are homogeneously heated. This can be compared to a 'cooking' of the grains. No significant distinction between the peak skin temperature and the average temperature can be made [2]. The single powder grains are still visible in Fig. 2a. A mixture between solid state sintering (where the individual grains are still visible, thus the melting temperature has not been reached during the interaction) and liquid phase sintering (visible as the light tracks) can be observed. Melting only takes place where the average temperature reaches or overcomes the melting temperature of the titanium, which is 1933 °K (=1660 °C) [5].

During pulsed laser interaction, the grains are no longer homogeneously heated. In reference to the interaction model described in Ref. [2], a clear distinction between the peak skin temperature and the average temperature can be made. This can be compared to a 'roasting' of the grains. Liquid phase sintering has taken place. The peak skin temperature is expected to be much higher compared to the case of continuous wave sintering, but only exists within a very short time. The average temperature, after consolidation has taken place, is then much lower. The maximum average temperature reduces with the same amount as the required average power as a consequence of the pulsed interaction.

From the material analysis, it can be seen that in the case of pulsed laser beam interaction, liquid phase sintering is spatially limited to a very narrow surface layer of the individual grains with a thickness of approximately 1 μm. Furthermore, the smaller grains are totally molten and act as a binder between the bigger grains, filling the void spaces in between them. As the inner part of the grain, which stays at much lower temperature acts as an efficient heat sink, the cooling rate is high and the liquid lifetime is thus short. This high cooling rate leads to an increased microhardness.

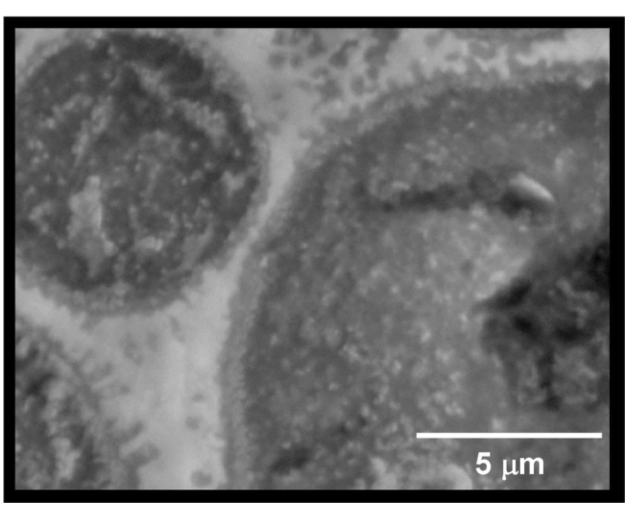

Fig. 4. SEM micrograph of a cut sintered plate. The different regions in the microstructure are clearly distinguishable: core, surface layer and binding material.

6. Conclusions

Even though the average power required to achieve

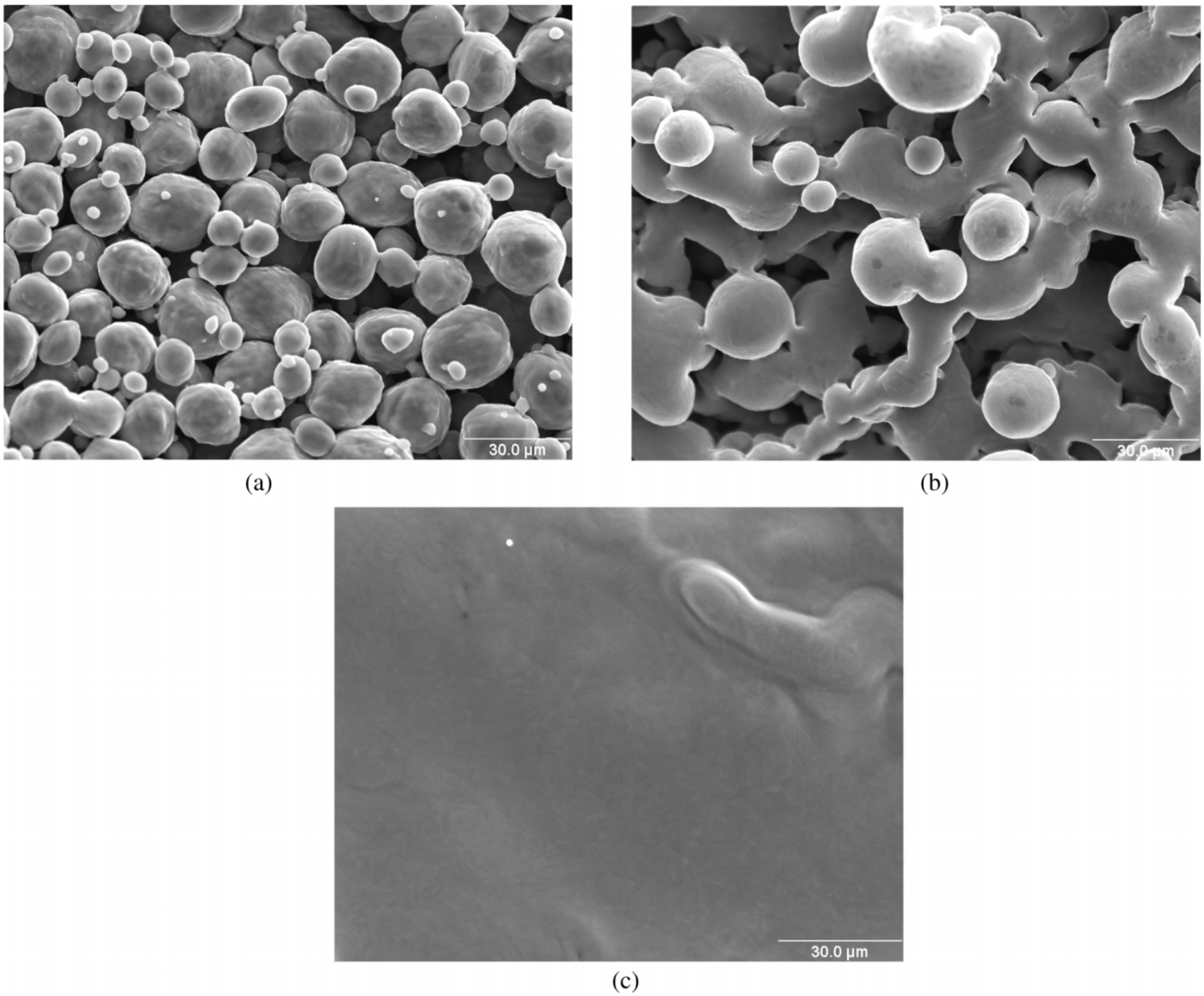

Fig. 5. Different morphology of the sintered structures by changing the laser parameters: (a) Nd:YAG with 150 ns pulses at 3.2 watts av. Power, 25 kHz repetition rate, (b) Nd:YAG with 150 ns pulses at 3 watts av. Power, 5 kHz repetition rate and (c) second processing step with a Nd:Glass laser, 40 pulses with 200 mJ/pulse.

consolidation with pulsed laser radiation is 30% lower, the consolidation of the powder due to liquid phase sintering is much more efficient (compare Fig. 2a,b), which yields a stronger connection between the grains. The camera is not able to resolve the temporarily higher skin temperature rise, but measures the average temperature. In the case of continuous wave sintering, no distinction between skin and average temperature can be made. However, in the case of pulsed interaction the average temperature is much lower but the sintering is stronger. Therefore, a significant higher skin temperature rise must have existed during the sintering process. The area of the temperature elevation (Fig. 3b) during the continuous wave interaction is much larger and the achieved peak temperature nearly 1000° higher than during the pulsed interaction, which scales quite well with the higher average power. The measurements of the temperature field confirm the predicted advantages of the pulsed laser sintering: lower average power and thus lower average temperature yield stronger consolidation due to the much higher peak skin temperature. This is also verified by the metallurgical analysis of the sintered samples.

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References

[1] J.-P. Kruth, X. Wang, T. Laoui, L. Froyen, Proceedings of the

3rd Laser Assisted Net-shape Engineering Conference LANE, 6, 2001, pp. 3–24.
[2] P. Fischer, N.P. Karapatis, V. Romano, R. Glardon, H.P. Weber, Appl. Phys. A 74 (2002) 467–474.
[3] P. Fischer, V. Romano, H.P. Weber, Near Infrared Pulsed Laser Sintering of Metal Powders, Scientific Report, University of Bern, 2000.
[4] P. Fischer, M. Locher, V. Romano, H.P. Weber, S. Kolossov, R. Glardon, Temperature Measurements During Selective Laser Sintering of Titanium Powder, Scientific Report, University of Bern, 2003.
[5] J. Emsley, The Elements, Oxford University Press, 1990, pp. 200–201.