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On thermal properties of metallic powder in laser powder bed fusion additive manufacturing



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ABSTRACT

Keywords: Laser powder-bed fusion Laser flash Finite element modeling Inverse method Powder thermal conductivity Powder thermal properties play a critical role in laser powder-bed fusion (LPBF) additive manufacturing, specifically, the reduced effective thermal conductivity compared to that of the solid significantly affects heat conduction, which can influence the melt pool characteristics, and consequently, the part mechanical properties. This study intends to indirectly measure the thermal conductivity of metallic powder, nickel-based super alloy 625 (IN625) and Ti-6Al-4V (Ti64), in LPBF using a combined approach that consists of laser flash analysis, finite element (FE) heat transfer modeling and a multivariate inverse method. The test specimens were designed and fabricated by a LPBF system to encapsulate powder in a hollow disk to imitate powder-bed conditions. The asbuilt specimens were then subjected to laser flash testing to measure the transient thermal response. Next, an FE model replicate the hollow disk samples and laser flash testing was developed. A multi-point optimization algorithm was used to inversely extract the thermal conductivity of LPBF powder from the FE model based on the measured transient thermal response. The results indicate that the thermal conductivity of IN625 powder used in LPBF ranges from 0.65 W/(m·K) to 1.02 W/(m·K) at 100 °C and 500 °C, respectively, showing a linear relationship with the temperature. On the other hand, Ti64 powder has a lower thermal conductivity than IN625 powder, about 35% to 40% smaller. However, the thermal conductivity ratio of the powder to the respective solid counterpart is quite similar between the two materials, about 4.2% to 6.9% for IN625 and 3.4% to 5.2% for Ti64

1. Introduction

In laser powder-bed fusion (LPBF) metal additive manufacturing (AM), compacted metallic particles in a powder bed play a significant role in the heat transfer phenomenon during the localized laser melting process, because heat dissipation to the surrounding influences the rate of solidification of the molten metal and subsequent cooling, and therefore, the mechanical properties of the built part. In addition, accurate information of thermal properties of metal powder in LPBF is essential for high-fidelity process modeling and predictions. While there are numerous publications regarding to the thermal properties of common solid materials, little research has been reported regarding to powder thermal properties in AM.

Many researchers estimated the thermal conductivity of powder in LPBF using numerical approaches. Early work on evaluating the thermal conductivity of composite media (e.g., powder and gas) can be derived from the Maxwell approach [1–4], which has been improved by the consideration of contacts between neighboring particles and gas in

the pores. Some models have been developed to investigate the heat transport mechanism of a powder bed in AM and simulate the effective thermal conductivity. For example, Gusarov et al. claimed that the thermal conductivity of gases at ambient pressure is substantially lower than that of metals and considered less important than other factors such as contacts between particles [5]. However, one of the authors later showed that the effect of interstitial gases on the effective thermal conductivity of a powder bed can be significant in some conditions [6]. Siu et al. and Slavin et al. both incorporated contact effects, such as the contact angle and the neck area between the neighboring particles for heat transfer in a powder bed, and conducted an analytical study to compute the powder thermal conductivity [7,8]. Moreover, Singh et al. utilized an artificial neural network approach to predict the effective thermal conductivity of a porous system, which may contribute toward LPBF studies [9]. Gong et al. incorporated the powder thermal conductivity obtained from hot-disk based measurements and an analytical means into a 3D finite element (FE) thermal model to simulate the thermal field in the powder-bed electron beam additive manufacturing

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[10].

Currently, there exist various transient techniques to measure thermal conductivity, such as the transient hot-wire method, the transient plane source method and the laser flash method. Each method has its specific apparatus to measure some kind of thermal response, from which, the thermal conductivity or diffusivity of a material is derived. In the transient hot-wire method, a thin metal wire acts as both a resistive heat source and a thermometer. Then the thermal transport properties are determined by the rate of temperature-dependent voltage across the wire versus the time changes [11,12]. Using this technique, Richard et al. measured the thermal conductivity of gases [13]. The transient hot-wire method was studied and used by Wei et al. to measure the thermal conductivities of commercial metal powder in a pressurized inert gas chamber, and the authors reported that the heat dissipation of a powder bed is influenced by gas infiltrating [14]. Similar to the transient hot-wire method, the transient plane source method utilizes a plane instead as the temperature sensor to determine the thermal conductivity and thermal diffusivity of the mediums by recording the voltage variation as a function of time [15,16]. Apart from the applications to common materials (solids, liquids or gases), this method has been used in extensive objects in many different engineering fields, such as food and agriculture [17], medical engineering [18,19], and architecture [20,21], etc. Additionally, a modified transient plane-source method has been employed in some commercial devices to quickly measure the thermal conductivity of small samples, such as fluids [22] and building materials [23].

Among different techniques for thermal diffusivity measurements, laser flash analysis, which was first developed by Parker et al. [24], is a widely used method for a wide variety of materials with a high precision. It uses the transient thermal response of a sample after a short heating pulse by a laser, then utilizes various heat transfer models to extract the thermal diffusivity from the measured response. For heterogeneous or anisotropic materials, more complex models may be required. Inverse heat transfer methods, in conjunction with laser flash technique, have been used to evaluate the thermal properties of thin coated films [25-29]. The solution of the analysis in these studies was based on the minimization of the least-squared errors between numerical model predictions and experimentally measured data, which was detailed in a publication from Ozisik [30]. Parker's theory of the flash method assumes one-dimensional heat transfer, without heat losses, and the homogeneity of the tested specimen. On the other hand, it is difficult to measure the thermal conductivity of metal powder, particularly with the size (approximately $< 50 \,\mu$ m) used in powder-bed fusion. With the inverse method approach, Cheng et al. developed and validated a combined experimental-numerical method to evaluate the powder thermal conductivity using laser flash testing and numerical heat transfer simulations [31]. The authors used additively fabricated hollow samples, with specially designed internal geometry, to enclose powder from LPBF. The internal geometry was designed to overcome an issue in which a gap occurred between the top shell and the internal powder, as reported in [32], which resulted in thermal insulations and complicated heat transport in the testing sample.

Continued from the previous work [31], the objective of this study is to analyze the temperature-dependent thermal conductivity of powder used in LPBF additive manufacturing. The test specimens of different designs with enclosed powder were laser-flash tested at different temperatures to obtain experimental thermal response, and the developed inverse methodology was employed to evaluate the temperature-dependent thermal conductivity of both nickel super alloy 625 (IN625) and titanium alloy (Ti64) powder materials.

2. Experimental details

2.1. Specimen design and fabrication

The test specimens were designed thin hollow disks to encapsulate

powder during fabrication. Internal cone features, either on the top or both the top and bottom sides of the hollow disks were included to ensure the contact between powder and the solid shells, preventing a large-area gap caused by powder settling observed in [32]. As an example, Fig. 1(a) is a photo of a fabricated two cones (0.5 mm height) sample. The radial cross-section of the sample model is shown in Fig. 1(b). The overall dimensions of hollow disks are 25 mm in diameter and 3 mm in height with a shell of 0.5 mm thickness. The internal geometric feature had three different cone features: (1) both cones with a height of 0.5 mm (noted as 2Cone-0.5 throughout the paper), (2) both cones with a height of 0.25 mm (2Cone-0.25), and (3) one cone with a height of 0.5 mm on the top (1Cone-0.5). The dimensions of the conefeature designs are shown in Fig. 1(c).

An EOS M270 LPBF system¹ was employed for sample fabrications. The powder materials used included both IN625 and Ti64. The specimens were built vertically by the LPBF process (shown in Fig. 1(d)), as such to avoid support structures beneath. Additionally, to achieve a full-density build, the process parameters suggested by the manufacturer were adopted for the solid shells. For IN625, the process parameter set was 195 W laser power and 800 mm/s scan speed [33] and the layer thickness was set as 40 µm. For Ti64, a laser power of 170 W and a scan speed of 1250 mm/s [34] were used, with a layer thickness of $30 \,\mu\text{m}$. For both materials, the hatch spacing was $100 \,\mu\text{m}$. No laser exposure was applied to the internal hollow section, as it was intended to encapsulate powder. Fig. 1(e) shows the scan methods applied at the radial cross-section. As inert gases were inflated to minimize the oxygen levels for both material fabrications, the encapsulated powder into the hollow specimens were resultant surrounded by the gas. Therefore, in this study, the powder thermal properties restore the powder-bed status, including the inert gas environment.

2.2. Laser flash testing

Thermal diffusivity testing of both solid and different powder-enclosed samples were carried out using a DLF-1200 from TA Instruments¹, shown in Fig. 2(a). In this system, the test specimens are held in a furnace chamber, purged with either nitrogen or argon gas, which has environment temperature control that can be increased up to 1200 °C. A laser pulse with a variable energy up to 25J was applied uniformly in a concentrically circular area with a diameter of about 22 mm at the bottom surface of the specimen. The laser power is adjusted and set automatically by the system to create an adequate thermal response and resulting signal from the pyrometer. The duration of laser irradiations was approximately 0.003 s. An infrared pyrometer collects thermal response from a 9.6 mm diameter circular region on the top surface of the test specimen and converts to digital signal output (Fig. 2(b)). To reduce laser reflection, the test specimen was coated with liquid graphite, and dried completely before loading onto a sample holder in the furnace chamber. During testing, the furnace heats to different programmed setpoint temperatures. Once steady state environment temperature is reached, the laser pulses, which increases the sample temperature only enough to enable a measurement from the pyrometer. The procedures and settings of specimen testing suggested by the manufacturer (TA Instruments) were followed.

The laser flash instrument generates a set of thermal radiation measurements collected over time via an infrared pyrometer. The experimentally acquired data is given as the voltage output and then

¹ Certain commercial entities, equipment, or materials may be identified in this document in order to describe an experimental procedure or concept adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the entities, materials, or equipment are necessarily the best available for the purpose.



Fig. 1. (a) An LPBF fabricated sample; (b) a geometric model of LPBF sample (e.g.: 2Cone-0.5); (c) dimensions of the 2Cone-0.5 powder-enclosed samples (unit: mm); (d) vertically built specimens on build plate; and (e) scan methods in LPBF.



Fig. 2. (a) DLF-1200 laser flash apparatus; (b) Schematic of laser flash method; (c) Specimen loading system; (d) Specimen holder; (e) Dimensions of IR detection and laser irradiation areas. Note that pyrometer spot size is not to scale as shown.

transferred into a normalized response ranging from 0 to 1 which corresponds to the output at lowest and highest signal values. The response vs. time result is termed as a "thermogram". Since the diffusivity is related to the time response (e.g., rise time of the thermogram), knowledge of the absolute temperature rise due to the laser pulse is not necessary. The experimental results of IN625 and Ti64 are discussed in the following two sections.

2.3. IN625 powder samples

Fig. 3 shows the experimentally obtained thermograms of a solid sample as well as an example of the 2Cone-0.5 specimen with

encapsulated powder. Compared with the solid sample, the heating rate of specimens with encapsulated powder is much slower; the maximum temperature is reached at between 10 s and 20 s vs. less than 3 s for the solid sample. It can also be noticed that as temperature increases, the heating period in the thermogram shifts to the left gradually due to increased thermal diffusivity of the IN625 material with the temperature.

Furthermore, at a given testing temperature, the thermograms of specimens with 2Cone-0.25 and 1Cone-0.5 features exhibit similar results in the heating period, and on the other hand, the 2Cone-0.5 specimen has a slightly higher heating rate than the specimens with the 2Cone-0.25 feature. An example of the comparison between the three



Fig. 3. Experimental time-response thermograms of IN625 at various temperatures: (a) solid specimen measurement and (b) specimens with encapsulated powder (2Cone-0.5). Averages are taken from three separate measurements at each temperature.



Fig. 4. Comparison of laser flash thermograms of IN625 with three cone features at 100 $^{\circ}$ C. The light-color bars in the plot indicate the range of measurement results.

cone features at 100 °C is shown in Fig. 4.

2.4. Ti64 powder samples

Same as the IN625 samples, thermograms from laser flash testing of Ti64 specimens show an increased thermal diffusivity as the testing temperature increases. Fig. 5(a) shows the results of the 2Cone-0.5 specimen at various temperatures. Fig. 5(b), on the other hand, compares thermograms from laser flash testing of the IN625 and Ti64 specimens, both with encapsulated powder and with the 2Cone-0.5 feature. It is noted that the Ti64 specimen has slower heating compared to IN625 specimen, indicating a smaller diffusivity value because of the inherently lower thermal diffusivity of Ti64 alloy; in addition, the difference in thermograms between the two materials becomes smaller at higher temperatures.

3. Inverse method for powder thermal property evaluation

To analyze the thermal conductivity of the powder inside the LPBFbuilt specimens, the laser flash system was modeled and simulated by a finite element (FE) method using ABAOUS software. The specimen and its holder were modeled using the measured physical dimensions, with a mesh size of 0.5 mm and 0.7 mm, respectively. The laser heat source was simplified as a uniformly-distributed surface heat flux applied on the bottom side of the specimen. Convection and thermal radiation heat loss were included as the boundary conditions with the ambient temperature set as the testing temperature. The encapsulated powder together with the interstitial gas was treated as a continuum and assumed to have the following unknown properties: density (p) and conductivity (k). Besides, two contact conductance values: (1) between the powder and the top solid shell (k_t) , and (2) between the powder and the bottom solid shell (k_b) , needed to be determined as well. Additionally, the specimen-holder contact conductance (k_p) at testing temperatures was obtained by analyzing the thermal response of the solid sample testing using the same laser flash system and the FE simulations, and then included in the laser flash simulation for the specimens with encapsulated powder.

Fig. 6 illustrates two examples of temperature contours of the cut-off sectional area at different times for a 2Cone-0.5 IN625 powder-enclosed sample at 200 °C and 500 °C with assumed material properties. At the beginning, the heat flux is applied at the bottom surface of the sample, and the irradiation time period is 0.003 s as in testing. The temperature of the irradiation region of the sample can increase by about 29 °C for the 500 °C testing case, for instance. Then, the heat flows upward through the sample. It is found that the heat dissipates into the internal powder zone slowly, because the finite contact conductance imposed to represent limited powder contact with the solid shell. It can be observed that there exists a temperature difference (about 18.0 °C for 200 °C and 16.6 °C for 500 °C) at the interface between the sample and the powder



Fig. 5. (a) Laser flash thermograms of Ti64 (Ti64) 2Cone-0.5 specimen; (b) Comparison of thermograms between IN625 and Ti64 specimens with encapsulated powder, both with 2Cone-0.5 feature.



Fig. 6. Simulated thermal contours of IN625 2Cone-0.5 sample tested at (a) 200 °C and (b) 500 °C at different times.

at t = 1.183 s. Meanwhile, the heat spreads faster through the solid shell. Subsequently, the heat flow passes through the internal powder to the top surface eventually, and the temperature changes at the top surface of the sample are acquired for the analysis purpose.

Because of multiple unknowns, a multivariate inverse method with a multi-point optimization algorithm was utilized to fit the simulation results to the experimental results (the thermograms shown in Figs. 3–5), and eventually to extract the powder thermal conductivity as one of the optimization variables through iterations. The methodology of the inverse approach uses the Levenberg-Marquardt method, which has been used in a variety of inverse problems [30]. In this study, 20 points are selected on the experimental thermogram, including 12 points in the heating period and 8 points in the cooling period, which will be compared against the FE simulation data at the same time intervals. A sum-squared error (S) is calculated based on the difference between the measured thermogram and the FE simulation thermogram from the current iteration. For each iteration, a damping factor (u) is introduced to adjust the selection of optimal property variables for the next step iteration. The newly-calculated variable values are then reincorporated in the FE model to perform the thermal simulation for the next iteration and acquire a new thermogram. The error (S) value is evaluated at each iteration to make sure that it is smaller than previous iteration. If current S value is larger than previous iteration's S value, the variables are calculated again from previous iteration. When the S value is smaller than a user-defined criteria or cannot be further reduced, the iteration will stop and the result is considered optimal. Fig. 7 shows the schematic of multivariate inverse method. The detailed approach can be found in [31].

3.1. IN625 powder study

In the thermal simulation of laser flash testing, temperature-dependent material properties of solid IN625 [35,36] and alumina [37], which are applied for the solid capsule of the sample and the sample holder, respectively, are given in Fig. 8. In addition, the density of alumina is assumed as 3800 kg/m^3 [38]. Moreover, the emissivity (unitless) for IN625 and alumina is 0.12 to 0.16 [39] and 0.7 [37], respectively. The convection coefficient was estimated to be 10 W/ (m²·K) [40]. The uncertainty of these parameters is assumed to have an insignificant effect on the evaluation of the unknown parameters determined by the inverse method (e.g., powder thermal conductivity), although the sensitivity to parameter uncertainty is yet to be studied.

3.1.1. Example of powder-enclosed sample analysis

Fig. 9(a) shows the thermograms from three shots of laser flash testing of an IN625 specimen (2Cone-0.5) at 100 °C. To illustrate iterative results from the inverse method, Fig. 9(b) shows the simulated thermogram from each iteration, with the third and fourth approaching the experimental curve. Table 1 below lists the simulations output as well as the overall error (S), calculated as the sum-squared error between the measured and simulated thermogram, calculated at each iteration. The initial values for the four unknowns were set as 10% of the solid IN625 density and thermal conductivity at the testing temperature, and $100 \text{ W}/(\text{m}^2 \cdot \text{K})$ for the contact conductance. The initials were purposely set far away from the possible actual values to ensure no effects of initials to the final solution. By adjusting the damping factor in each iteration [31], an optimal set of the four unknown properties was selected for the next step. By calculating the S value (overall error), it can be determined if the simulation for the next iteration is necessary to proceed. In this case, the result from the 3rd iteration is considered the optimal solution, because the error increases at the 4th iteration. Moreover, a few additional iterations were conducted to verify that the final solution was indeed the local optimum. The S values for the three iterations were calculated and showed to be increasing continuously. The increasing S value can confirm that the optimal solution has been reached at the 3rd iteration. Furthermore, it is noticed that the obtained thermal conductivity of IN625 powder is only 6.9% of that for solid In625, whereas the density appears 43% approximately. In addition, the k_b (bottom contact conductance) is more effective than the k_t (top contact conductance) owing to possibly gravity-induced additional contact areas, for example, $927 \text{ W}/(\text{m}^2 \text{K})$ vs. $352 \text{ W/(m}^2 \text{ K})$ in the case of 100 °C testing.

To evaluate the reliability of experimental testing of the powderenclosed samples, repeated tests were conducted using three different 2Cone-0.5 samples. The three samples were fabricated in one batch using the same LPBF process parameters. The thermal response comparison of the three 2Cone-0.5 samples at 500 °C is shown in Fig. 10(a). The three thermograms exhibit close heating and cooling curves, and are considered to satisfy the experimental repeatability. For each thermogram from the three tests, the inverse method was utilized to evaluate the thermal conductivity and the density of the internal powder, which are compared in Fig. 10(b). It can be noticed that the difference of powder conductivity and density in the three samples are not significant. At 500 °C, the powder conductivity is about 1.01 W/ m·K, and the density is about 4655 kg/m^3 , with the variation between



Fig. 7. Schematic illustration of multivariate inverse method.







Fig. 9. (a) Experimental results, including three shots and the average thermogram, and (b) Simulated thermograms from each iteration.

 Table 1

 Results from inverse method for IN625 2Cone-0.5 sample at 100 °C.

n	и	<i>k</i> , W/(m·K)	k_t , W/(m ² ·K)	k_b , W/(m ² ·K)	ρ , kg/m ³	S
0		0.1	100	100	841	0.512417
1	-2	0.4314	609.90	566.95	902.63	0.275936
2	-0.02996	0.7347	372.60	738.03	3682.66	0.015569
3*	0.05	0.7955	351.77	926.54	4775.56	0.003393
4	-9.2	0.8000	351.62	934.50	4740.68	0.003789
5	-8.56	0.8102	356.17	953.96	4684.94	0.005510
6	-9.45	0.8281	363.75	988.95	4594.48	0.010547
7	-78.6	0.8304	364.71	993.54	4583.60	0.011362

* Optimal solution.

the three samples of approximately 1% and less than 5%, respectively.

3.1.2. Cone configuration effect

As reported in [32], for the powder-enclosed specimen designed with flat top and bottom surfaces (named as No-cone), a gap exists between the internal powder and the top shell, and consequently affect the heat transfer dramatically through the specimen. Fig. 11(a) shows thermograms of different samples (No-cone, 2Cone-0.5 and 1Cone-0.5), from laser flash testing at 500 °C. It is noted that the thermogram of the No-cone sample shifted to the right side of the other two, indicating a slower heating rate as expected. Moreover, a heat transfer simulation was conducted for the No-cone model. However, the FE simulation was unable to approach to the experimental curve unless the internal powder was removed (Fig. 11(b)). This implies a gap between the internal powder and the top shell, making it virtually insulated with no heat flow through. On the other hand, the designed simple cone features are effective in mitigate the insulating issue and appropriate to the overall combined experimental-numerical method for powder thermal conductivity analysis.

To determine the possibility of powder settling and air gaps several

samples were measured using x-ray computed tomography (XCT). Air gaps would inhibit heat transfer into and out of the powder and elicit greater effective contact conductance $(k_t \text{ and } k_b)$ values in the FE model. Fig. 12 below shows the XCT images of a 2Cone-0.25 sample scanned, positioned vertically and horizontally, using a Bruker SkyScan 1173 micro XCT scanner¹. The sample, fabricated using Ti64 powder, has a small diameter of 12.5 mm in order to be fully transmitted by the x-ray of the scanner, limited by the voltage capacity (130 kV). From the vertical position scan (Fig. 12(a)), a gap (dark area) at the top, between the powder (gray area) and the shell (light area), is clearly noticed (in coronal and sagittal views). On the other hand, when the sample is at a horizontal position during the scan, the contact between the powder and the top shell appears continuous, without noting dark areas except around the very outer circumference (corresponding to gas/void). This again demonstrates (1) the significance of a potential gap that resulted from fabricating the cone structure and may result in thermal insulation, and (2) the effectiveness of the internal cones for powder and solid contacts that improve heat transfer through the powder and ensure the testing and the simulation are meaningful.

Nevertheless, laser flash testing results from the samples with three different cone configurations were studied using the inverse method to attain the powder thermal conductivity, which shows minor differences among the three cone configurations, shown in Table 2 (for 500 °C). It appears that the 2Cone-0.5 sample shows a higher thermal conductivity than the other two samples (2Cone-0.25 and 1Cone-0.5), which have a similar thermal conductivity. On the other hand, the powder porosity of the 2Cone-0.5 sample is approximately 9% lower than the other two models. Though it is not anticipated that the internal cone features would affect the analyzed powder thermal conductivity, the deviation from design to fabrication may result in a small difference that may be within the measurement uncertainty.

3.1.3. Thermal conductivity and porosity at different temperatures The laser flash results of the IN625 specimens with encapsulated



Fig. 10. (a) Repeatability of experiments and (b) Statistics of powder conductivity and density.



Fig. 11. Comparison of thermograms between (a) No-cone, 1Cone-0.5 and 2Cone-0.5 samples, and (b) No-cone experiment and FE simulation that assumes no powder.



Fig. 12. CT images of a 2Cone-0.25 Ti64 sample (12.5 mm diameter) positioned differently in the micro-CT scanner: (a) vertically and (b) horizontally.

Table 2Comparison of analytical thermal conductivity and porosity of In625 powder at500 °C.

Cone configuration	Thermal conductivity (W/m·K)	Porosity (%)
2Cone-0.5	1.020	42.32
2Cone-0.25	0.857	51.60
1Cone-0.5	0.828	51.30

powder, and three different cone features, at various temperatures were analyzed to inversely calculate the temperature-dependent powder conductivity. The results are summarized in Fig. 13. The powder conductivity obtained ranges from 0.65 W/(m-K) to 1.00 W/(m-K), and generally, the powder conductivity is nearly linear to the temperature.

However, the results extracted from the samples of three different cone features are slightly different. The models of the 2Cone-0.25 and 1Cone-0.5 give a similar powder thermal conductivity, while the powder conductivity analyzed from the 2Cone-0.5 model is about 0.1 W/(m·K) to 0.2 W/(m·K) higher than that from the other two models for all testing temperatures. The calculated density of IN625 powder exhibits a general descending trend, a decrease from about 5000 kg/m³ to about 3700 kg/m³, when the temperature increases from 100 °C to 400 °C, then a slightly increase at 500 °C. Besides, the powder density is similar among three cone configurations, except that the 2Cone-0.5 sample has a slightly higher density at a higher temperature, greater than 300 °C, shown in Fig. 13(b).



Fig. 13. (a) Thermal conductivity and (b) porosity of IN625 powder.



Fig. 14. Thermal properties of solid Ti64.

3.2. Ti64 powder study

The FE model for Ti64 specimens with encapsulated powder was established also based on the actual geometry of fabricated specimens and Ti64 material properties. Fig. 14 shows the material properties of solid Ti64 [41] that were incorporated in FE modeling. The same simulation approach and the inverse method used in the IN625 powder study were employed to the Ti64 powder-enclosed samples with three different cone configurations, same as in the IN625 powder study.

Fig. 15(a) compares Ti64 2Cone-0.5 thermograms between simulations and experiments at 100 °C and 500 °C. It can be noted that the simulated thermal responses agree well with the experimental results well during the heating period, though along the temperature decay, there is a minor deviation between the simulation and the experiment. Fig. 15(b) shows the fitting curve comparison between Ti64 and IN625 for the 2Cone-0.5 model at 100 °C. It can be observed that the thermograms of Ti64 has a lower heating rate than that of IN625.

The analyzed thermal conductivity values of Ti64 powder at various temperatures (100 °C to 500 °C) are plotted in Fig. 16(a). It can be observed that the simulated Ti64 powder thermal conductivity linearly increases with temperatures and ranges from 0.30 W/(m K) to 0.65 W/(m K), for 100 °C and 500 °C, respectively. Also, similar to the IN625 powder study, the result from the 2Cone-0.5 sample gives a higher thermal conductivity than the other two (1Cone-0.5, 2Cone-0.25), which show close thermal conductivity values. Moreover, when normalized by the solid thermal conductivity, it is noted that the Ti64 powder conductivity is approximately only 3.4% to 5.2% of the solid Ti64 conductivity at all testing temperatures, Fig. 16(b), and different cone configurations result in an insignificant difference. This finding is similar to the results of IN625 powder, which shows a slightly higher ratio, 4.2% to 6.9%.

Furthermore, similar to IN625 powder, Ti64 powder exhibits a

descending temperature-dependent density from 100 °C to 500 °C, Fig. 17(a). Also, the porosity of Ti64 powder-bed is ranging from 43.5% to 57.0%, while 40.3% to 55.7% for IN625, shown in Fig. 17(b).

4. Conclusions

The LPBF specimens with encapsulated powder were designed and fabricated, to imitate powder-bed conditions in LPBF, by an EOS M270 system using two different powder materials: IN625 and Ti64. Different internal cone features were incorporated in the specimens to ensure contact between the powder and the top solid shell. To evaluate the powder thermal conductivity, laser flash experiments and a numerical approach using FE thermal simulations and an inverse method were conducted to analyze the powder thermal conductivity. The major findings are concluded as follows:

- (1) The combined approach using laser flash experiments, FE heat transfer simulations and a multivariate inverse method demonstrated the feasibility of indirectly measuring and analyzing the thermal conductivity and density of metal powder in similar to powder-bed conditions.
- (2) The significance of this study is to prove that the thermal conductivity of powder from LPBF is much lower than the solid conductivity, e.g., 0.65 W/(m·K) to 1.02 W/(m·K) for IN625, and 0.30 W/(m·K) to 0.65 W/(m·K) for Ti64, in the range of 100 °C to 500 °C with a linear temperature dependence.
- (3) The powder thermal conductivity of IN625 is approximately 4.2% to 6.9%, independent to temperature, of the corresponding solid thermal conductivity. On the other hand, such a ratio for Ti64 powder is slightly lower, 3.4% to 5.2%.
- (4) The calculated powder porosity is in the range of about 40% to about 55% between 100 $^\circ C$ and 500 $^\circ C.$
- (5) The internal cone features in the sample appear to be effective to ensure a proper contact between the internal powder and the solid top shell, making the problem otherwise unsolvable due to thermal insulations caused by a gap resulted from powder settling. Interestingly, different cone configurations in the samples result in minor different results, which may be attributed to variations in fabrications.

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Fig. 15. Comparison of the analytical results for (a) Ti64 2Cone-0.5 at different temperatures, and (b) 2Cone-0.5 model at 100 °C using different materials.



Fig. 16. (a) Thermal conductivity of Ti64 powder at different temperatures, and (b) ratio of powder to solid thermal conductivities for IN625 and Ti64.



Fig. 17. (a) Density of Ti64 powder and (b) porosity of Ti64 and IN625 powder-bed at different temperatures.

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