

## THERMAL PROPERTY MEASUREMENT METHODS AND ANALYSIS FOR ADDITIVE MANUFACTURING SOLIDS AND POWDERS

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### Abstract

Thermal properties of additive manufacturing (AM) solids and precursor materials are important factors for build process and part performance. However, measured thermal properties are not well documented, despite being used extensively in AM modeling and simulation. The National Institute of Standards and Technology (NIST) and the University of Louisville are developing the measurement science for AM material thermal properties. New measurement methods, sample preparation techniques, and results are demonstrated for laser flash (LF) thermal diffusivity measurements on metal AM solids and powders. Due to the complexity of these materials, new sample fabrication methods are developed and demonstrated. Results from LF measurements are presented and compared.

**Keywords:** laser powder bed fusion; thermal properties; thermal diffusivity; thermal conductivity

### 1 Introduction

As additive manufacturing (AM) gains popularity and becomes widely used, so does the demand for accurate and reliable AM processes. Of the many factors that play a role in the quality of AM parts, the thermal properties of the material arguably play one of the most important roles. These properties dictate the rate of solidification, amongst other things, which in turn controls the phase composition and therefore the mechanical properties. While there is, for the most part, sufficient published empirical data on the thermal properties of the common materials used in AM, there is very little in regards to the thermal properties of these materials in a powder form and even less in terms of sintered or melted states. There is a need for accurate AM thermal property data.

The laser flash technique, developed and first used by Parker et al. in 1961 [1], allows the direct measurement of thermal diffusivity,  $\alpha$ . The technique consists of a pulsed thermal energy source (i.e., laser or flashlamp) which heats one side of the sample being analyzed. The heat conducts through the specimen and is detected by a pyrometer on the opposite side. This signal vs. time

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result is called a thermogram. Based on a solution to the Fourier heat equation assuming semi-infinite boundary conditions, the thermogram of this heat is used along with the specimen's thickness to obtain the thermal diffusivity of the material. This relationship, derived by Parker in [1], is

$$\alpha = \frac{1.37L^2}{\pi^2 t_{1/2}}, \quad (1)$$

where  $L$  is the sample thickness and  $t_{1/2}$  is the time from the laser pulse to half-maximum of the thermogram. Note that derivation of diffusivity from Equation 1 depends on the relative amplitude of the thermogram to determine  $t_{1/2}$ , and not absolute temperature. A schematic of the process is shown in Figure 1. For this relationship to hold true, there are several assumptions that need to be made. The derivation of Parker's diffusivity calculation assumes the heat to be instantaneously and uniformly absorbed by a thin portion of the sample, the sample to be homogeneous and isotropic, and the heat transfer in the radial direction to be negligible. Testing thermally conductive materials at optimal thicknesses will allow the original Parker solution to be used with minimal error assuming the conditions of the test are similar to the assumptions made, but this is often not the case. Since Parker's work was published there have been quite a few pieces of work aimed to improve on the originally proposed solution by accounting for these assumptions. The heat losses are considered in [2] and [3], while the finite heat pulse is accounted for in [2, 3-5], and the lack of uniform of heating in [2,3,6]. Some of these can be avoided with appropriate experimental apparatus and conditions, including the use of a high-quality fiber laser to avoid nonuniform heating and integration of the measured temperature over the sample's surface.

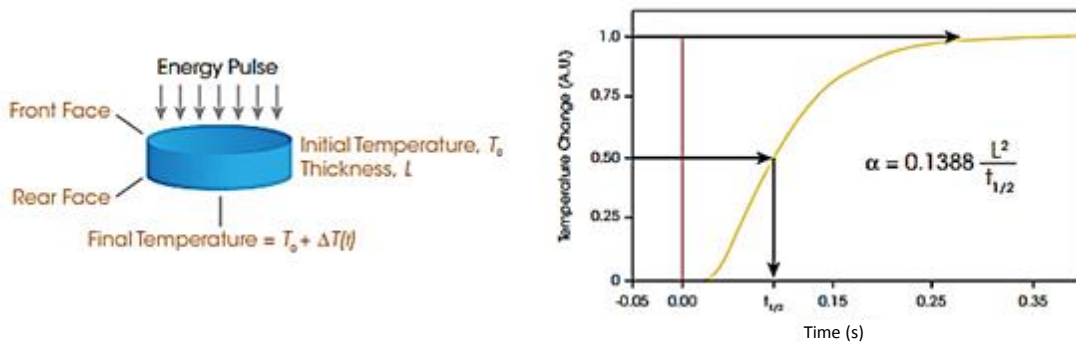


Figure 1: Schematic of the laser flash technique

The work detailed here focuses on measuring the thermal properties of solid and powder AM materials. While the powders evaluated here are comprised of particles with a distribution of sizes optimal for laser powder bed fusion (LPBF), there is no reason these methods could not be applied to powders optimized for other AM processes.

## 2 Experimental Procedure

The work shown here uses a commercially available laser flash system that features an environment module capable of heating samples to 1600 °C, a six-sample carousel that holds 12.5 mm cylindrical samples, an Nd:glass laser, and a InSb pyrometer (shown in Figure 2). The manufacturer claims the device provides thermal diffusivity measurements accurate to  $\pm 2.3\%$  with a repeatability of  $\pm 2\%$ . Prior to testing, each sample is coated with a thin layer of graphite as described in ISO 22007-4:2017 [7] to optimize laser energy absorption on the top surface, and emission towards the pyrometer on the bottom surface. Each test consists of a series of ‘test segments’ in which the samples are raised to specific setpoint temperatures within the environment chamber. During each segment the samples undergo three ‘shots’ with ample time in between for the sample to cool to the current segment setpoint temperature.

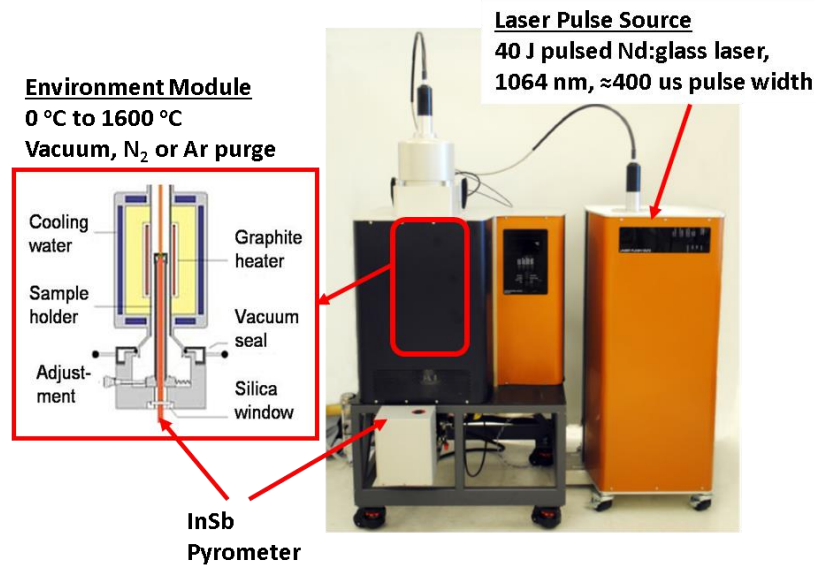


Figure 2: Schematic of the laser flash system

### 2.1 Solid AM and Wrought

Solid AM samples were built in a commercial laser powder bed fusion system using nickel alloy 625 and 17-4 stainless steel powder using the original equipment manufacturer’s suggested settings. In order to ensure geometric accuracy, the samples were taken from a 25 mm tall cylinder with a 12.5 mm diameter using an electrical discharge machining (EDM) operation. Wrought nickel alloy 625 and 17-4 stainless steel were procured in 12.5 mm rods and were machined using EDM in a similar fashion. The nickel alloy was heat treated at 1010 °C for an unspecified duration then water cooled and the 17-4 stainless steel was heat treated at  $\approx 482$  °C for 1 hr then air cooled. Both groups (AM and wrought) were sliced into similar thicknesses ( $\approx 1.6$  mm).

### 2.2 AM Powder Capsules

The laser flash technique requires two sides of the sample to be accessible, and, as is the case for the system used at the National Institute of Standards and Technology (NIST), the cylindrical

sample's flat surfaces are horizontally oriented. This prevents loose powder from being tested without some sort of container.

In order to imitate the conditions in which the powder is used, additively manufactured capsules were fabricated with loose powder trapped inside. This allows a volume of powder to be extracted that has the same density as in the LPBF machine and for the powder sample to be taken with the environmental gas used in the process (nitrogen in this case). The thermal conductivity of the gas has been shown to have a significant impact on the effective conductivity in [8]. The as-built configuration is shown in Figure 3. The samples are built in groups of five and subsequently machined to size and removed from the others with an EDM operation. The powder was removed from one sample by means of a small ( $\approx 0.5$  mm) hole to differentiate the powder's contribution to the thermogram's shape and calculated diffusivity from the solid capsule shell. While there will be a portion of overhanging solid geometry in the samples, they are oriented so that this region's size is minimized and in a location in which it should not significantly alter the heat transfer.

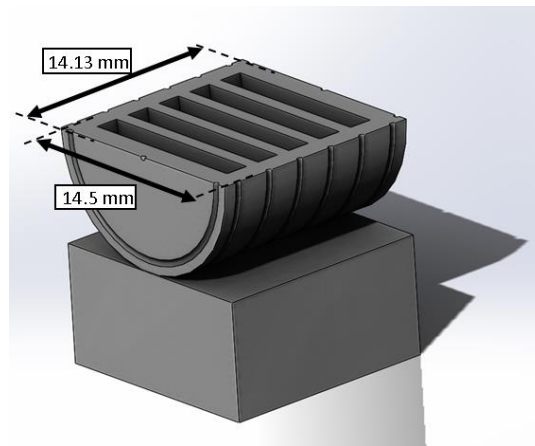


Figure 3: Cross-sectional view of model of five-sample group

### 2.3 Copper and Yttria Stabilized Zirconia

A powder container was fabricated using a top and bottom with high thermal conductivities, and a thermally insulating surrounding tube to ensure a substantial portion of the laser's thermal energy passes through the powder. Figure 4 shows a cross section of the powder capsule design and the actual assembled capsule can be seen in Figure 5. Copper was chosen for the top and bottom, and yttria stabilized zirconia (YSZ or Zr) for the tube. The powder and holder materials will be subjected to temperatures up to 400 °C and will move with respect to one another due to their different thermal expansion coefficients. Therefore the lid is fabricated with a diameter slightly less than that of the tube's inner diameter so that it can move freely in the vertical direction to ensure it remains in contact with the powder throughout the test. The bottom is press-fit into the ceramic tube, then powder is poured in and the excess is scraped away with a razor blade and the lid is gently placed on the powder.

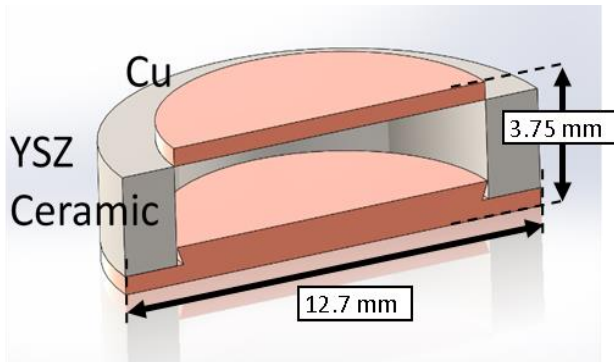


Figure 4: Cross-sectional view of computer-aided design model of Copper and YSZ powder container



Figure 5: Powder filled copper and YSZ powder capsule

### **3 Results and Discussion**

All thermal diffusivities reported are calculated using the Parker model and each of the reported values for diffusivity is an average of three ‘shots’ of the laser flash system. As previously mentioned there are some assumptions made in Parker’s derivation that may not be appropriate, therefore the results reported here should serve as comparisons to one another and are not necessarily absolute values. This is especially true for the more complex powder capsules. Finite element modeling will help extract the effective thermal properties of the powder from the aggregate (i.e., what is empirically measured). If error bars are present, they are the range of values over the three ‘shots’.

#### **3.1 Solid AM and Wrought**

Results from the laser flash testing of nickel alloy 625 and 17-4 stainless steel are shown in Figure 6. The calculated thermal diffusivity of the wrought nickel alloy 625 samples and the AM counterparts are very similar with a maximum percent difference of 1.5 % at 600 °C. Conversely, the wrought and AM stainless steel specimens are quite different from one another. The wrought samples show a nonlinear trend especially at temperatures over 200 °C while the AM samples are linear. The wrought 17-4 stainless steel purchased was H900 conditioned. Since the laser flash system holds all the samples at elevated temperatures that are near this H900 heat treatment (i.e., 483 °C ) for often over several hours, this material is likely undergoing phase transformations that are altering the thermal properties of the wrought material. As shown in [9], the nitrogen gas used during the atomization process of this 17-4 stainless steel powder seems to play a role in retaining the austenitic phase during heat treatment. This may explain the discrepancy between the two materials’ measured thermal diffusivities.

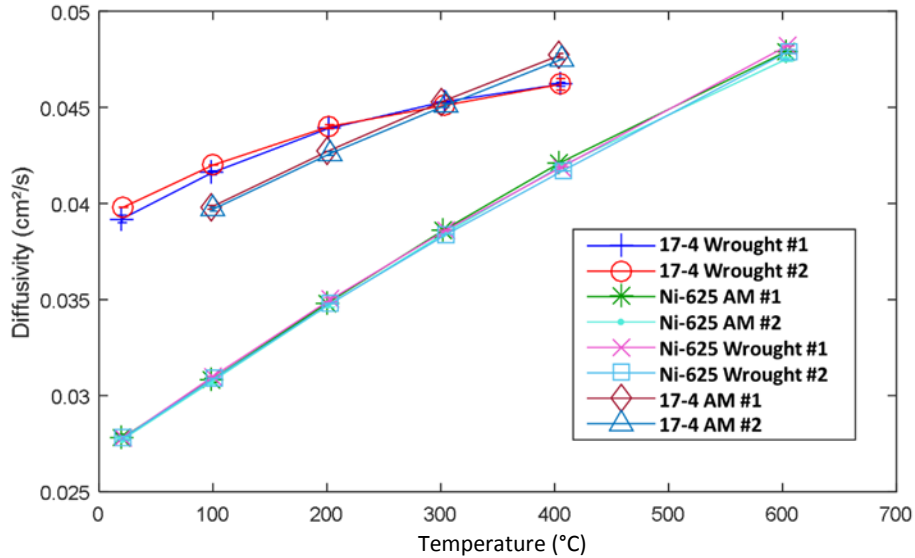


Figure 6: Thermal diffusivities of solid AM and wrought 17-4 stainless steel and nickel alloy 625. Diffusivity is calculated using the Parker model.

### 3.2 AM Powder Capsules

The results from the laser flash testing of both full and empty 17-4 stainless steel AM powder capsules are shown in Figure 7. Each sample was tested at 100 °C intervals from 100 °C to 400 °C, and again at 100 °C to test for the presence of hysteresis. This entire process was repeated upon the first test's completion. While the thermal diffusivities of the hollow sample are repeatable and linear with respect to temperature, the powder filled specimens showed substantial deviation from one another, were not linear with temperature, and showed hysteresis in regards to the measured thermal diffusivities. This indicates some sort of change in the powder likely either due to a density change in the powder caused by settling or a phase transformation as seen in the solid wrought materials. The entire test process was conducted a total of three times with only the initial results showing the presence of hysteresis. Additionally, the large range in the diffusivity of the shots seen in Test #1 Capsule #1's first segment (conducted at 100 °C) are not seen elsewhere indicating the sample was changing in between shots. The results from the second test are shown in Figure 8, and show that whatever was causing the variability is not a factor after the initial test segment. The x-ray computed tomography image in Figure 9 shows settling near the top corners of the powder container which may be the cause of the deviation. The powder undergoes thermal expansion and contraction during testing which causes the powder to settle. Also, the thickness of the side walls of the sample is also variable ranging from  $\approx .861$  mm to  $\approx .342$  mm. This is attributed to a combination of errors incurred during the EDM operation and the portion of the sample that is built as an over-hanging structure. Since the temperature of the bottom surface is integrated by the pyrometer, this will likely not significantly alter the measurement, but more work is needed to confirm this.

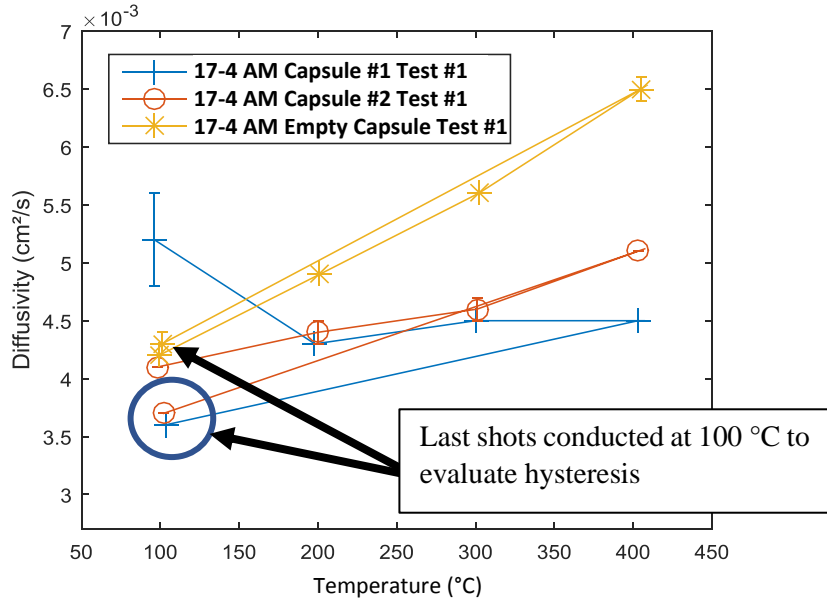


Figure 7: Thermal diffusivities of powder-filled AM capsules compared to the emptied, hollow capsule

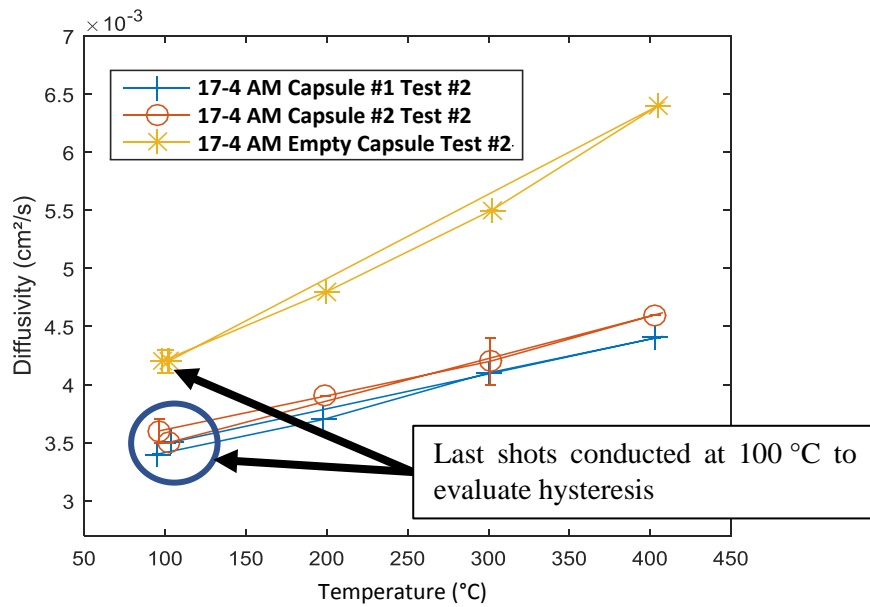


Figure 8: Repeat of test conducted in Figure 7 comparing powder-filled AM capsules to the emptied, hollow capsule.



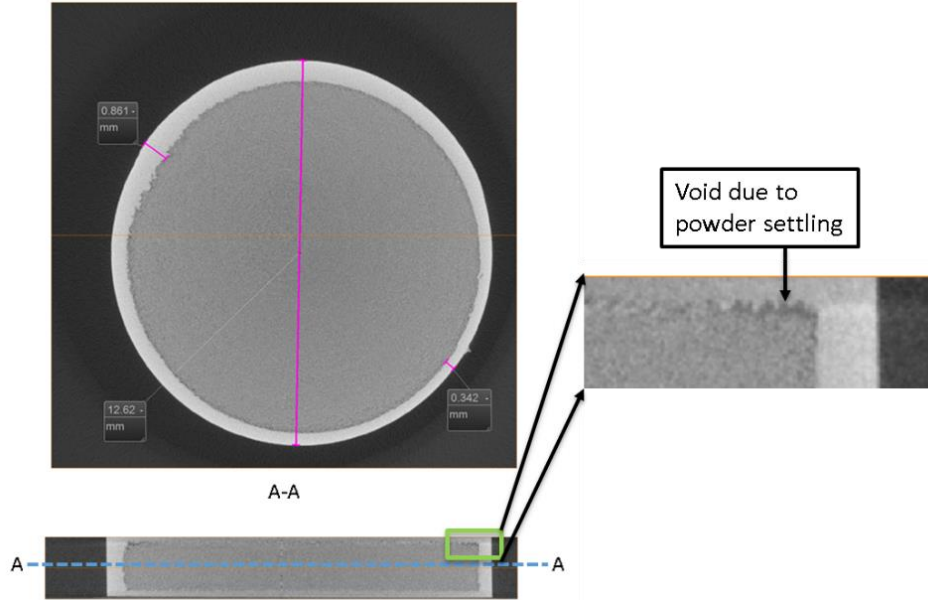


Figure 9: XCT image of 17-4 stainless steel AM capsule showing asymmetry and powder settling

### 3.3 Copper and Ytria Stabilized Zirconia

Calculated thermal diffusivities from the Cu and YSZ sample holders are shown in Figure 10. The hysteresis previously seen in the AM capsules is not present indicating the copper lid is maintaining contact with the powder throughout the tests. While this apparatus seems to be the most promising given the aforementioned constraints, more work needs to be done to extract the powder's effective thermal properties before they can be evaluated. This will involve comparison with theoretical powder bed heat transfer as well as finite element method modeling.

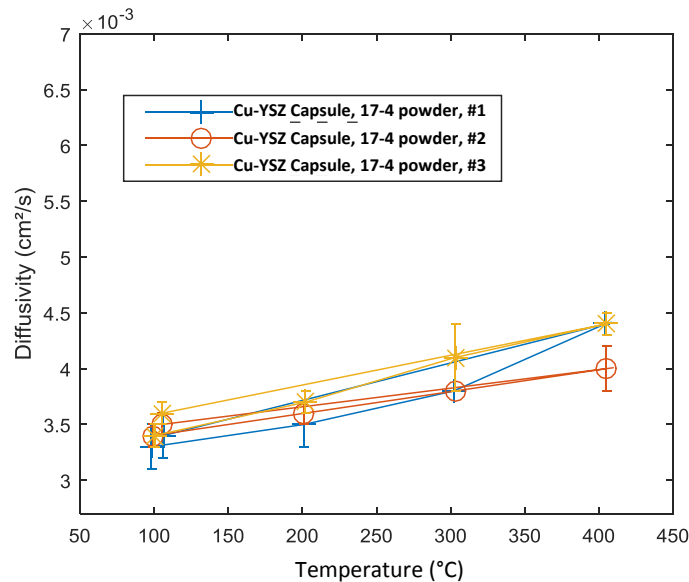


Figure 10: Thermal diffusivities of 17-4 stainless steel powder filled Cu-Zr capsules. Parker model is used to extract diffusivity values from thermograms.



#### **4 Conclusions**

The thermal diffusivities of LPBF solid nickel alloy 625 and 17-4 stainless steel have been tested using the laser flash technique. While the AM nickel alloy specimens proved to have very similar thermal diffusivities (within 1.5 % of their wrought counterparts), the stainless steel specimens showed a nonlinear relationship between temperature and thermal properties compared to the linear nature of the AM samples which is likely due to phase transformation. To evaluate the thermal properties of precursor AM materials, work has been done to design and fabricate various sample holders. While the additively manufactured powder capsules allow the powder to be captured in a state similar to its use in the LPBF process, several drawbacks were identified including inconsistent contact between powder and solid phase and the observed asymmetry of the capsules. A copper and yttria stabilized zirconia holder was employed and has shown to provide repeatable results. Comparisons to modeling and theoretical thermal properties will be conducted in the future.

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