

ORNL/TM-2015/563  
CRADA/NFE-14-05102

**Demonstration of thermal control, microstructure control, defect mitigation and process parameter database generation for Ti-6Al-4V Direct Digital Manufacturing - *Understanding defect mitigation and process parameter database generation for direct digital manufacturing***



Ryan Dehoff

September 22, 2015

**CRADA FINAL REPORT  
NFE-14-05102**

**Approved for Public Release.  
Distribution is Unlimited.**

## DOCUMENT AVAILABILITY

Reports produced after January 1, 1996, are generally available free via US Department of Energy (DOE) SciTech Connect.

**Website** <http://www.osti.gov/scitech/>

Reports produced before January 1, 1996, may be purchased by members of the public from the following source:

National Technical Information Service  
5285 Port Royal Road  
Springfield, VA 22161  
**Telephone** 703-605-6000 (1-800-553-6847)  
**TDD** 703-487-4639  
**Fax** 703-605-6900  
**E-mail** [info@ntis.gov](mailto:info@ntis.gov)  
**Website** <http://www.ntis.gov/help/ordermethods.aspx>

Reports are available to DOE employees, DOE contractors, Energy Technology Data Exchange representatives, and International Nuclear Information System representatives from the following source:

Office of Scientific and Technical Information  
PO Box 62  
Oak Ridge, TN 37831  
**Telephone** 865-576-8401  
**Fax** 865-576-5728  
**E-mail** [reports@osti.gov](mailto:reports@osti.gov)  
**Website** <http://www.osti.gov/contact.html>

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Material Science and Technology Division  
Advanced Manufacturing Office

**Demonstration of thermal control, microstructure control, defect mitigation and process parameter database generation for Ti-6Al-4V Direct Digital Manufacturing - Understanding defect mitigation and process parameter database generation for direct digital manufacturing**

Authors

Ryan Dehoff ORNL  
Ralph Dinwiddie ORNL  
Brian Jordan ORNL  
S.S.Babu UTK/ORNL  
Alan Robson UTK  
Niyanth Sridharan UTK  
Anil Chaudhary Applied Optimization

Date Published:  
September 22, 2015

Prepared by  
OAK RIDGE NATIONAL LABORATORY  
Oak Ridge, Tennessee 37831-6283  
managed by  
UT-BATTELLE, LLC  
for the  
US DEPARTMENT OF ENERGY  
under contract DE-AC05-00OR22725

Approved For Public Release



# CONTENTS

	Page
CONTENTS.....	v
LIST OF FIGURES .....	vi
ACKNOWLEDGEMENTS .....	vii
ABSTRACT.....	1
1. PHASE I SCOPE: UNDERSTANDING DEFECT MITIAGTION AND PROCESS PARAMETER DATABASE GENERATION FOR DIRECT DIGITAL MANUFACTURING .....	1
1.1 BACKGROUND .....	1
1.2 TECHNICAL RESULTS.....	2
1.2.1 Characterization of Powder Stream: .....	2
1.2.2 Effect of Process Parameters on Deposit Characteristics .....	5
1.2.3 Modelling of the Track Characteristics: .....	6
1.3 IMPACTS .....	7
1.4 CONCLUSIONS AND SUGGESTIONS FOR PHASE 2 WORK.....	7
2. PARTNER BACKGROUND .....	8
3. REFERENCES .....	9

## LIST OF FIGURES

Figure 1: Processed images from the high-speed camera measurements on powder flow characteristics shows the drastic changes in the powder stream flow characteristics as a function of the distance from the nozzle.....	3
Figure 2: Powder size distribution as a function of vertical distance from the outlet (a) overall data and (b) expanded view of the distributions in the range of 15 to 20 mm shows small distinct differences between different conditions. ....	4
Figure 3: Detailed analyses of expanded powder size distribution in the range of 0 to 20 mm distance from outlet: (a) Conditions 1-3; and (bb) Conditions 4-7.....	4
Figure 4: Variation of single track bead cross-section shape as a function of different conditions (a) C-1, (b) C-2; (c) C-3, (d) C-4, (e) C-5, (f) C-6, (g) C-8, and (h) C-9 .....	6
Figure 5: Typical plots of calculated (a) thermal gradient, (b) liquid-solid interface velocity, (c) temperature and (d) Peclet number for heat and mass transfer for a default laser direct energy deposition of Ti6Al4V alloys.....	6

## ACKNOWLEDGEMENTS

This CRADA NFE-14-05102 was conducted as a Technical Collaboration project within the Oak Ridge National Laboratory (ORNL) Manufacturing Demonstration Facility (MDF) sponsored by the US Department of Energy Advanced Manufacturing Office (CPS Agreement Number 24761). Opportunities for MDF technical collaborations are listed in the announcement “Manufacturing Demonstration Facility Technology Collaborations for US Manufacturers in Advanced Manufacturing and Materials Technologies” posted at <http://web.ornl.gov/sci/manufacturing/docs/FBO-ORNL-MDF-2013-2.pdf>. The goal of technical collaborations is to engage industry partners to participate in short-term, collaborative projects within the Manufacturing Demonstration Facility (MDF) to assess applicability and of new energy efficient manufacturing technologies. Research sponsored by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Advanced Manufacturing Office, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.





## ABSTRACT

Researchers from Manufacturing Demonstration Facility (MDF) at Oak Ridge National Laboratory (ORNL) worked with Applied Optimization (AO) to understand and evaluate the propensity for defect formation in builds manufactured using DM3D-POM laser direct metal deposition. The main aim of this collaboration was to understand the character of powder jet behavior as a function of the nozzle parameters such as cover gas, carrier gas, and shaping gas. In order to evaluate the sensitivities of the parameters used in model, various experiments were performed with in-situ monitoring of the powder stream characteristics using a high speed camera. A wide variety of conditions while keeping the hopper motor rpm constant, including laser power and travel speed were explored. The cross sections of the deposits were characterized using optical microscopy.

### 1. PHASE I SCOPE: UNDERSTANDING DEFECT MITIGATION AND PROCESS PARAMETER DATABASE GENERATION FOR DIRECT DIGITAL MANUFACTURING

This phase 1 technical collaboration project (NFE-14-05102 and MDF-TC-2013-029) started during June, 2014 and was completed September, 2015. The collaboration partner Applied Optimization Inc. is a small business.

#### 1.1 BACKGROUND

Additive manufacturing is a shaped deposition process, which progressively adds material to the substrate. The material fed could be in either solid or liquid or a mixture of both while it reaches the substrate and then fuses with the substrate. Laser additive manufacturing is a free form fabrication technique that involves the deposition of the material in the form of powder into a melt pool created by a laser source. Despite offering great flexibility in manufacturing, the mechanical properties of additively manufactured samples are lower than that of their wrought counterparts [1]. This difference in performance has been attributed to defects and a graded microstructure formation [1]. Hence defect elimination is of primary importance. The majority of these defects are interlayer lack of fusion and process induced porosity formation. Both these defects are closely related to the melt pool and track geometry [2]. Hence it is important to have a capability to predict the track geometry a-priori as a function of process parameters. The melt pool and the track geometry are closely related to the powder stream characteristics since this governs the process efficiency [3-5].

Powder delivery for laser additive manufacturing can be achieved by using a co axial nozzle (DM3D-POM machines at MDF) or an off axis process such as those present in the LENS process. The major advantage of the off axis systems is from the fact that it reduces powder melting and sticking to the nozzle but suffers from what is called an “over hill cladding”. This technique of deposition where the powder is deposited from the side (i.e. off axis) leads to an “against hill condition” cladding while powder is applied from the side the substrate moves [3, 5]. Delivering the powder co-axially with the laser beam can offset this issue since all the directions of the substrate movement in a plane perpendicular to the laser beam become equivalent.

The major components of the coaxial nozzle are

1. The central hole through which the laser beam and the shielding gas pass
2. The surrounding annular zone into which the powder is blown out by the carrier gas
3. Outer chamber where the shaping gas focuses the powder into a conical beam

The other process gas that is used in the process but not covered in the nozzle arrangement is the cover gas, which is used to push the powder from the hopper into the delivery system. After the powder particles exit the nozzle they are drawn to the melt pool by the action of gravity and the momentum of the gas used to push the powder [6]. Thus it is not surprising that the process gases have been reported to play a major role in the shape of the powder cone and the powder velocity. The most widely studied process gases are the carrier gas and the shaping gas.

It has been reported that a higher carrier gas velocity increases the powder velocity and produces a smoother build [7]. It has also been reported that the gases interact with each other creating compound jets contributing to turbulent flow [5].

Normally the operational window is defined in terms of laser power, scanning speeds and the powder feed rate and the effects are well studied and well known. However the additional parameters such as the flow rates and velocities of the process gases are not very well understood. There are several simulation studies that either look at understanding the effect of gas flows on the powder stream or the effect of process parameters on the builds [6]. However, the effect of change in the powder stream on the build quality is not well understood. The shape of the powder stream (controlled by the process gas flow rate and velocity) and the size of the melt pool, (controlled by the energy density) are two important parameters that determine the efficiency and the deposit quality [4]. Thus any study aimed at understanding the nature of the process needs to integrate all the above process parameters into the study and systematically evaluate the quality of the deposit as a function of the process parameters. It has been reported that the smaller the diameter of the powder stream at the point of impact with the substrate, the greater the powder catchment efficiency [4].

A complete deposit can be treated as single beads deposited adjacent to each other. Hence to make a defect free deposit the single tracks should fulfill certain geometrical characteristics as stated earlier. As stated earlier the major defects occurring in all AM parts are porosity and lack of fusion. Lack of fusion occurs primarily because the previous layer does not melt. This incomplete melting is a consequence of insufficient laser power. Porosity formation can be related to the surface roughness of the builds. In the case of builds with increased surface roughness where the adjacent tracks do not wet each other, during the deposition of the subsequent pass the liquid metal is unable to fill in the cavities caused, resulting in residual porosity. The other source of porosity could be from trapped gas. Thus the shape of the single track is important from the standpoint of eliminating both the lack of fusion and the creation of porosity. In this study we focus on how the changes caused to the powder stream manifest itself on the deposit quality.

Initial trials were made with Ti-6Al-4V. However, due to the lack of a standard Ti6Al4V powder size with good flow characteristics and high oxygen levels of the powder (> 20 wt. PPM), additional experiments were performed with 316 stainless steel powder. The process models developed by Applied Optimization, is indeed capable of predicting steel alloy 316 powders. In both cases, one of the important model validations is the ability to characterize the power flow as a function of a variety of nozzle parameters during directed energy deposition.

## **1.2 TECHNICAL RESULTS**

### **1.2.1 Characterization of powder stream:**

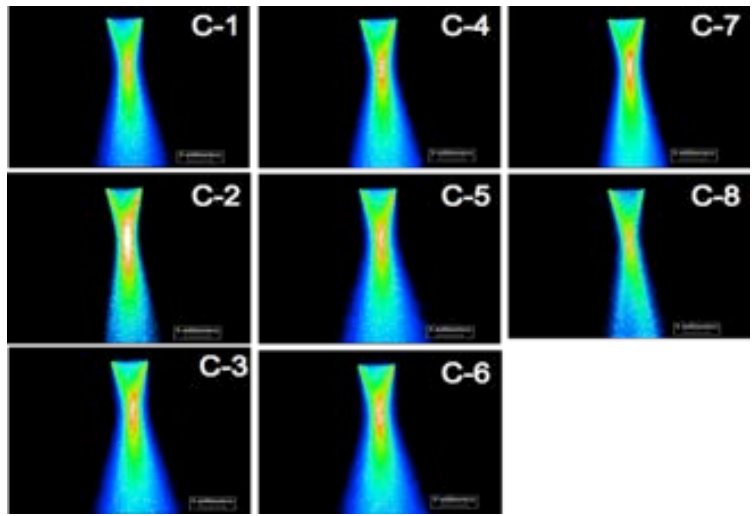
The effect of change to the powder stream was evaluated using a high-speed camera. The powder stream was evaluated as a function of change to the mass flow rate, shaping gas, cover gas, carrier gas and nozzle gas to understand the effect of those parameters on the builds. The above parameters were varied systematically and the changes to the powder flow patterns were monitored. The analysis

includes particle velocity, powder mass flow and the distribution of the powder across the nozzle. All three parameters have a direct influence on various aspects of the deposit. The change to the velocity of the powder stream is measured as a function of the process gases. The velocity increases significantly with changes to the cover gas. An increase in the cover gas flow rate from 6 lpm to 12 lpm shows that the velocity increases from 2674 mm/s to 4338.3 mm/s. An increase in the cover gas from 3 lpm to 6 lpm raised the particle velocity from 2674.7 mm/s to 3484.8 mm/s. The fact that there is decrease in the particle velocity with a decrease in shaping gas is intriguing since with increasing the gas flow rates should only increase the powder velocity. This however could be rationalized by looking at the nozzle gas values.

**Table-1:** Shows the changes to the powder flow characteristics as a function of the process gases

Case	Cover Gas (lpm)	Carrier gas (lpm)	Shaping gas (lpm)	Nozzle (lpm)	Mass flow (g/min)	Motor rpm	$v_{nozzle}$ (mm/s)
1	6	3	18	18	4.13	1850	2648.2
2	6	3	12	18	4.10	1850	2743.4
3	6	3	12	14	4.07	1850	2628.6
4	6	3	12	14	6.00	2376	2674.7
5	12	3	12	14	6.27	2376	4338.3
6	6	6	12	14	6.13	2376	3484.8
7	3	3	12	14	2.53	2376	1791
8	3	1.5	12	14	0.8	2376	1488.7

As stated in the previous section the shaping gas and the nozzle gas are in the inner and the outer regions of the nozzle. By decreasing the flow rate of the shaping gas we are actually setting up a strong compound jet. This sets up a complex shear stress, which increases with an increase in the velocity difference between the inner and the outer stream. This is a consistent observation since with a decrease in the nozzle gas velocity in case 3 the powder velocity drops down again. The increase in the mass flow rate does not contribute to any major change in the particle velocity. The increase in the mass flow rate from 4.0 g/min to 6.0g/min results in the change in the powder velocity of only of 46.1 mm/s. Such small changes may not be significant.

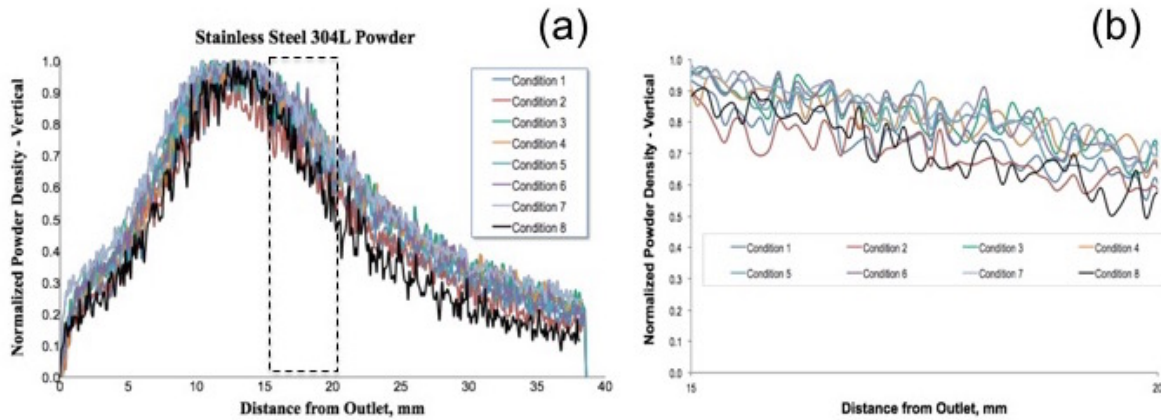


**Figure 1:** Processed images from the high-speed camera measurements on powder flow characteristics shows the drastic changes in the powder stream flow characteristics as a function of the distance from the nozzle.

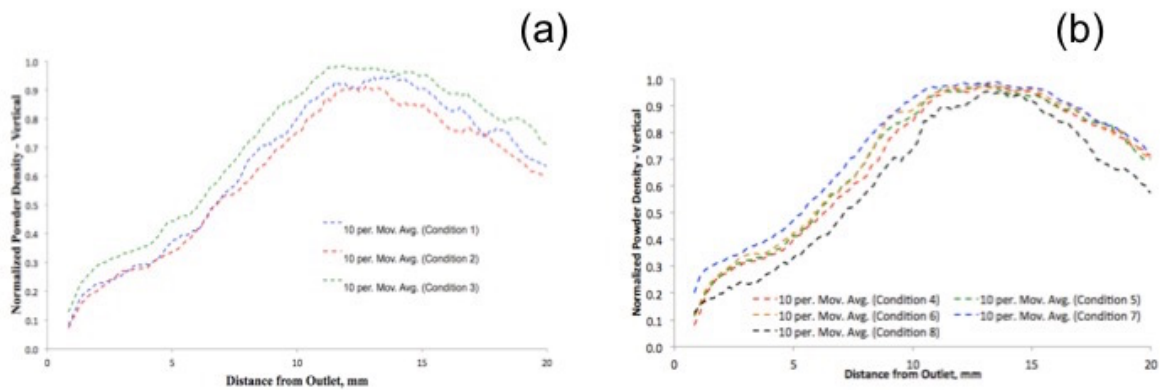
There are no major changes to the powder mass flow rates with

changes to the process gas. They seem to remain constant at close to 6 g/min. The 0.27g/min and 0.13g/min are not significant since the standard deviation of these values during testing is close to 0.15g/min. We now quantitatively analyze the changes in the powder stream.

The images (Fig. 1) from the high-speed camera that have all the frames were averaged. The regions with a higher concentration of powder are represented in red and the regions that are leaner in powder are in blue. The results of the changes to the powder flow as a function of process conditions are summarized (Figs. 2 and 3) below.



**Figure 2: Powder size distribution as a function of vertical distance from the outlet (a) overall data and (b) expanded view of the distributions in the range of 15 to 20 mm shows small distinct differences between different conditions.**



**Figure 3: Detailed analyses of expanded powder size distribution in the range of 0 to 20 mm distance from outlet: (a) Conditions 1-3; and (bb) Conditions 4-7.**

From the images the distribution of the powder is extracted and presented graphically (see Figs. 2 and 3). Note that the powder distribution is normalized and hence the actual particle density is not measured but rather only the changes between conditions. The most salient observation is that the powder stream is most focused at 12.5 mm below the nozzle. Interestingly, the machine operates at a stand off distance close to 20 mm! This is a significant finding and small changes in the stand off distance will significantly affect the build characteristics.

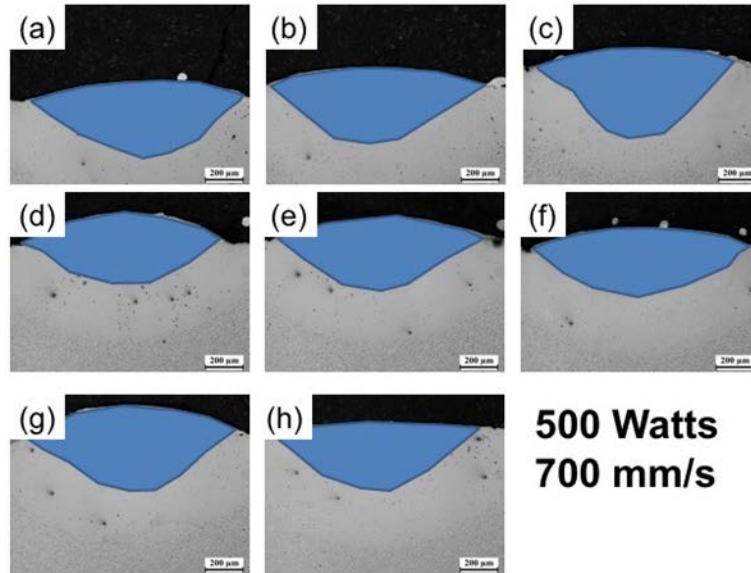
With data from Figs. 2 and 3, it is clear that the powder distribution changes as a function of the process gases (see Table 1) and the distance from the outlet. The plots show that the number of particles, which is converted to intensity and plotted as a function of the distance from the nozzle. It is seen that the powder density rises, rapidly peaks close to 12 mm and then drops off. We are more interested in the profile of the drop since stand off distance is close to 20 mm. Figure 2 shows significant changes in the powder particle density. There are several studies that have related the differences to the flow behavior to the shear stresses set up due to the action of the jet streams set up as a consequence of the differences in the velocities of the inner and the outer jets. To illustrate and quantify these effects the powder distribution for these two cases mentioned above (see Table 1) were analyzed further.

The results are interesting. For example, case-2, where the differences in the flow rates between the shaping gas (outer gas) and the nozzle gas (inner stream) is high (6-lpm), turbulent flow is expected near the exit of the nozzle. We relate this phenomenon to the measured drop in the particle distribution and the reduction in overall particle density. In contrast, with case-3, where the difference in the flow rate is 2-lpm, the particle density increases. One reason may be due to the excessive flow rates of both the nozzle gas and the shielding gas that are set at 18-lpm. The higher flow rates may actually contribute to the powder particle loss leading to the decrease.

The conditions 4 and 8 were obtained at a higher motor speed corresponding to a higher mass flow rate. However, the ratio of the shaping gas and the nozzle gases were not varied and the ratio was kept constant. Only the cover gas and carrier gas were changed. In conditions 4 and 5 the cover gas was altered and this increased the powder velocity, but the particle density seems to be constant. Comparing the conditions 4 and 6, where the carrier gas was altered from 3-lpm in C-4 to 6-lpm in C-6, keeping the shaping and the nozzle gas constant. In C-8 the carrier gas was decreased to 1.5 lpm and the flow of cover gas was reduced by 50%. These conditions lead to a drop in the powder mass flow and also a drastic reduction in the powder particle density. Thus, there is an optimal range of cover gas and carrier gas flow rates. Nevertheless, the upper limits for these parameters were not detected.

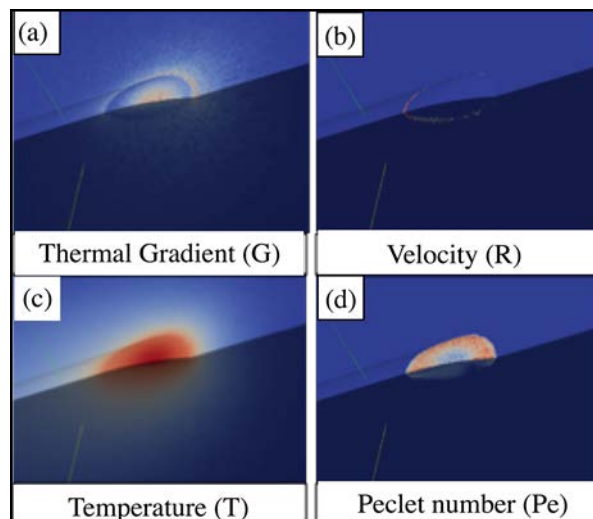
### **1.2.2 Effect of process parameters on deposit characteristics**

We can conclude from the previous section that, by just controlling the process gas the powder velocities and the shape of the powder stream can be modified. In order to understand the effect of these differences in the flow rates on the deposit shapes, different sets of parameters were used to manufacture single-track deposits. The laser power (500 to 700W in steps of 100W) and the travel speed (600 to 700 mm/min in steps of 100 mm/min) were varied for each condition. A detailed experimental matrix with seventy-two builds were completed. Figure 4 compares sub-set of 72 samples with cross-section of eight builds made with different conditions shown in Table 1, but with same laser power and travel speed. The builds after analysis showed that the melt pool shapes were altered due to the difference in the powder stream distribution. The height of the deposits was also different for each case. The height of the deposit directly depends on the powder catchment efficiency of the system. The powder catchment efficiency is directly related to the powder stream diameter that is controlled by the process gas flow. A narrow stream corresponded to a higher powder catchment leading to larger track heights. This way the differences in the track shapes and sizes can be rationalized.



**Figure 4: Variation of single track bead cross-section shape as a function of different conditions (a) C-1, (b) C-2; (c) C-3, (d) C-4, (e) C-5, (f) C-6, (g) C-8, and (h) C-9**

Figure 5: Shows the variation of the builds as a function of the shaping gas. All the builds were made with 500W laser power and a 700mm/s travel speed (a) Case-4 (b) C-5 (c) C-6 (d) C-7 (e) C-8. Note the changes to the track shape despite being made with identical laser power and travel speed.



**Figure 5: Typical plots of calculated (a) thermal gradient, (b) liquid-solid interface velocity, (c) temperature and (d) Peclet number for heat and mass transfer for a default laser direct energy deposition of Ti6Al4V alloys.**

### 1.2.3 Modelling of the track characteristics:

The track characteristics such as track width height and wetting angle are important from the perspective of eliminating defects. Such detailed experiments using high speed cameras followed by powder deposition and characterization is not feasible practically. Hence knowing the track shape and

width a priori as a function of the processing parameters could help select the process parameters to eliminate defects. AO has developed software (PARAGEN) that is capable of predicting the above characteristics. The software is currently available for MDF researchers to perform “what-if” evaluations of laser track size as a function of processing parameters.

Currently, there is a gap in understanding the interactions between the laser and the particle cloud which in turn leads to changes seen in Fig. 4. When the characteristics of the powder stream changes we expect a change in the fraction of laser energy absorbed. We need to understand if these effects are significant and can lead to a drastic improvement in process efficiency. Indeed, even if most of the laser power reaches the work piece, only a fraction is captured by the powder particles, thus heating them. Moreover, only the powder particles striking to the molten pool will eventually be captured, whereas particles hitting the solid region may ricochet and may be lost. Thus, there is need for further work to characterize these effects.

### **1.3 IMPACTS**

This research shows that by using analytical models and by selecting process parameters we are able to predict the track geometry. The experiments and track depositions shows that by changing the process gas parameters the deposit geometry changes rapidly, which cannot be predicted by the existing model. Though the model is currently capable of performing calculations up to reasonable accuracy for given set of conditions with normal flow conditions, it is clear that there is a need for fine tuning based on the experimental data shown in Figs. 1 to 4. In the next step, by understanding the track geometry as a function of process flow and powder flow rate, the defect formation in the deposit can be estimated. This would be helpful, if deployed on an industrial scale to select process parameters and eliminate defects in complex geometrical parts for wide range of iron, titanium and nickel base alloys, thereby improving the Technology Readiness Level (TRL) of additive manufacturing simulation technology. The current results and expected results from Phase II are consistent with the goals and the vision of the Department of Energy Advanced Manufacturing Office (AMO).

### **1.4 CONCLUSIONS AND SUGGESTIONS FOR PHASE 2 WORK**

The Phase I project experiments confirmed the hypothesis that the relative difference in flow of cover, carried and shaping gas flow rate for a given powder flow rate do affect the powder distribution as a function of distance from the outlet. Furthermore, we have demonstrated that these subtle changes also change the molten bead cross sections. It is, however, necessary to understand the interaction of laser with powder cloud as a whole as well. Therefore, in the Phase II proposed work, experiments need to be performed to understand the powder - laser interaction and will be correlated to geometry of single and multi-track. We proposed that these experiments be performed under in situ monitoring of powder and temperature with high speed camera and thermography equipment, respectively. The capabilities to perform such experiments are readily available at the MDF at ORNL. Preliminary experiments show the feasibility of performing such experiments.

## **2. PARTNER BACKGROUND**

Applied Optimization, Inc. (AO) was founded in 1995 by Dr. Anil Chaudhary. From our original focus on advanced manufacturing to our recent aerospace engineering projects, our mission since day one has been to offer clients the right balance of non-conformity and critical thinking. As a company of engineers and scientists, our passion for all things mathematics and science has literally opened doors for our clients in multiple industries and disciplines. Dr. Anil Chaudhary and the AO-Team develop algorithms for the control of materials processing equipment as well as for the collection, reduction, and analysis of materials data for structural and engineering components.

As a principal scientist at Applied Optimization, Dr. Anil Chaudhary's core specialty is manufacturing process design, computational mathematics, materials and mechanics. Anil is principle investigator for several projects in additive manufacturing, which include: (1) Physics and probability based design of processing parameters for the SLM and EBM process; (2) Characterization of uncertainty in the directed energy deposition process for the purpose of rapid qualification of Ti-6Al-4V components; (3) Collection of in-situ data for nondestructive characterization of power bed process. He has demonstrated the use of physics-based modeling for the prediction of deposition parameters for a variety of aerospace alloys.

<http://www.appliedo.com>



### 3. REFERENCES

- [1] P. Kobryn, S. Semiatin. Mechanical properties of laser-deposited Ti-6Al-4V. Solid Freeform Fabrication Proceedings: Austin, 2001. p.6-8.
- [2] A. Chaudhary. Modeling of laser-additive manufacturing processes, ASM Handbook 22 (2009) 240-252.
- [3] U. De Oliveira, V. Ocelik, J.T.M. De Hosson. Analysis of coaxial laser cladding processing conditions, Surface and Coatings Technology 197 (2005) 127-136.
- [4] J. Lin. A simple model of powder catchment in coaxial laser cladding, Optics & Laser Technology 31 (1999) 233-238.
- [5] J. Lin, W. Steen. Design characteristics and development of a nozzle for coaxial laser cladding, Journal of Laser Applications 10 (1998) 55-63.
- [6] S. Zekovic, R. Dwivedi, R. Kovacevic. Numerical simulation and experimental investigation of gas-powder flow from radially symmetrical nozzles in laser-based direct metal deposition, International Journal of Machine Tools and Manufacture 47 (2007) 112-123.
- [7] H. Tan, F. Zhang, R. Wen, J. Chen, W. Huang. Experiment study of powder flow feed behavior of laser solid forming, Optics and Lasers in Engineering 50 (2012) 391-398.