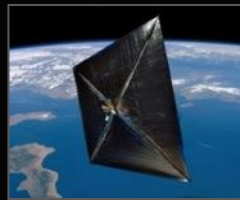
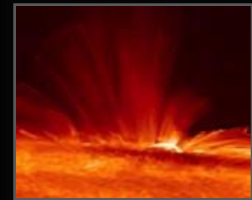




Study of Material Consolidation at Higher Throughput Parameters in Selective Laser Melting of Inconel 718

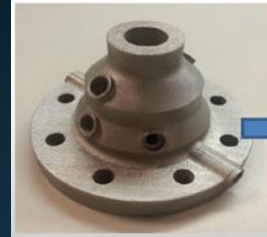
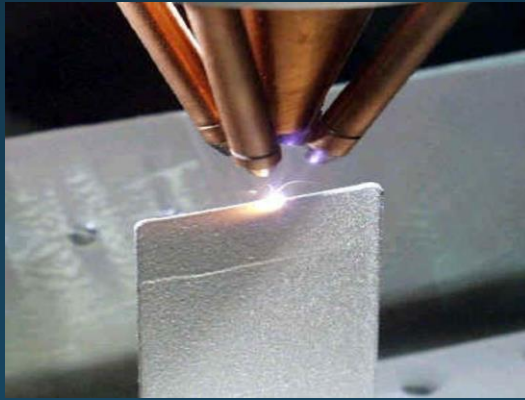
*The Materials Society (TMS) Annual Meeting
Additive Manufacturing: Building the Pathway towards Process and Material
Qualification
Nashville, TN*

marshall



Additive Manufacturing at MSFC

20+ Years of Experience



1991

2000

2010



Additive Manufacturing at MSFC



Near-Term

For Space



Long-Term



In-Space



Why Additive Manufacturing?



J-2X Gas Generator Duct



Pogo Z-Baffle



Turbopump Inducer



RS-25 Flex Joint

Part	Cost Savings	Time Savings
J-2X Gas Generator Duct	70%	50%
Pogo Z-Baffle	64%	75%
Turbopump Inducer	50%	80%

RS-25 Flex Joint	Heritage Design	SLM Design
Part Count	45	17
# Welds	70+	26
Machining Operations	~147	~57

Background

- The additive manufacturing team (AMT) at NASA MSFC has three Concept Laser machines (M1, M2, and X-Line)
- Current primary focus of the AMT is process development and generation of material property design curves for materials used in propulsion applications
 - In625
 - In718
 - Ti-64
- Most previous NASA MSFC studies on selective laser melting (SLM) of In718 centered around vendor default parameters



Purpose of work and objectives

- investigate the effect of higher throughput settings (higher laser power, higher scan speeds) on material consolidation
 - knowledge of critical importance to mass production industries (ex. automotive) who use powder bed fusion AM processes
- fill knowledge gaps and expands results of previous In718 studies, which focused on roughly +/- 20% of vendor default build parameters and did not consider higher speeds and powers
- provide insight into establishing acceptable/rejectable parameter limits for density and porosity of In718 produced by SLM
- consider density as a “first-gate” indicator of material quality
- study is also designed to assess the impact of heat treatment and deposition pattern on material consolidation

Design of Experiments

- Three parameters were varied in this study:
 - a) Hatch spacing: distance between each parallel vector of the laser
 - b) Laser power: power provided by the laser to melt the powder
 - c) Speed: rate that the laser is moved at
- Design of experiments was a 27 sample full factorial DOE (3 factors at 3 levels)

Factor	Level I	Level II	Level III
Power (W)	180	256	332
Scan speed (mm/s)	500	1030	1560
Hatch spacing (mm)	0.0675	0.105	0.1425

- Specimens produced for evaluation were blocks measuring 3/8" on each side

Design of Experiments

- Ancillary objective of study was to evaluate effect of heat treatment and deposition pattern on material consolidation at higher throughput parameters
- Four block sets produced (27 blocks in each set) for a total of 108 specimens

Block set	Deposition pattern	Heat treatment
A	Continuous	No
B	Continuous	Yes
C	Chess	No
D	Chess	Yes

- B and D class specimens underwent the following postprocess heat treatment regime:
 - stress relief at 1950 F for 1.5 hr, 2-4 bar Argon quench to remove specimens from build plate
 - HIP (hot isostatic press) at 2050-2125 F/14750 psig for 3-4 hr, solution heat treat at 1900-1950 F
 - aging at 1400 F holding at heat for 10 hrs, furnace cooling to 1200 F, holding at 1200 F until a total precipitation heat treatment time of 20 hours was reached, and cooled

Density testing

- Density testing was performed by Southern Research Institute (SRI)
- Four density measurements were collected for each block
 - **Gravimetric density:** uses dry mass of specimen and volume calculated directed from measurement of length between specimen faces
 - **Open porosity:** percentage of the volume enclosed by the specimen that is made up of voids open to or communicating with the surface of the specimen
 - **Bulk density:** uses data taken during the open porosity measurement process to calculate a volume*
 - **Apparent density:** volume used in calculation does not include open porosity (but internal porosity is reflected in this value). Apparent density is a closer representation of the material that composes the specimen.

*Ideally, bulk density should equal gravimetric. However, this is not the case for these specimens, possibly due to deviations in the outer contour line of the blocks which impacts the length measurement.

Summary of Block Set Comparisons

- Table summarizes mean porosity and density reported for each block set
- Each value represents mean of 27 specimens produced using same deposition pattern and subject to the same postprocessing conditions
- Density also expressed as a percent of theoretical density (textbook value) for In718 (appears in parentheses) for gravimetric values

What is the overall impact of deposition pattern and heat treatment across all parameter sets in the higher throughput regime?

	A	B	C	D
Open porosity (%)	0.7	0.9	0.4	0.7
Apparent density (g/cc)	8.158	8.239	8.155	8.245
Bulk density (g/cc)	8.104	8.166	8.119	8.191
Gravimetric density (g/cc)	8.074 (98.2%)	8.134 (98.9%)	8.086 (98.4%)	8.171 (99.4%)

A – continuous, no heat treatment
B – continuous, heat treatment
C – chess, no heat treatment
D – chess, heat treatment

Summary of Block Set Comparisons

- No statistically significant differences in densities or open porosity detected when comparing groups A and C or B and D
 - suggests that for this data set, deposition pattern has no engineering significant impact on material consolidation
- Heat treatment imparts a slight improvement in density (about 1%) and improvement is of similar magnitude for both chess and continuous deposition groups
 - The statistical significance of differences in density between groups as a result of heat treatment is not consistent
 - Based on ANOVA:
 - Groups A and B have statistically significant differences in apparent density at the $p=.05$ level
 - Groups C and D have statistically significant differences in apparent and gravimetric density at the $p=.05$ level

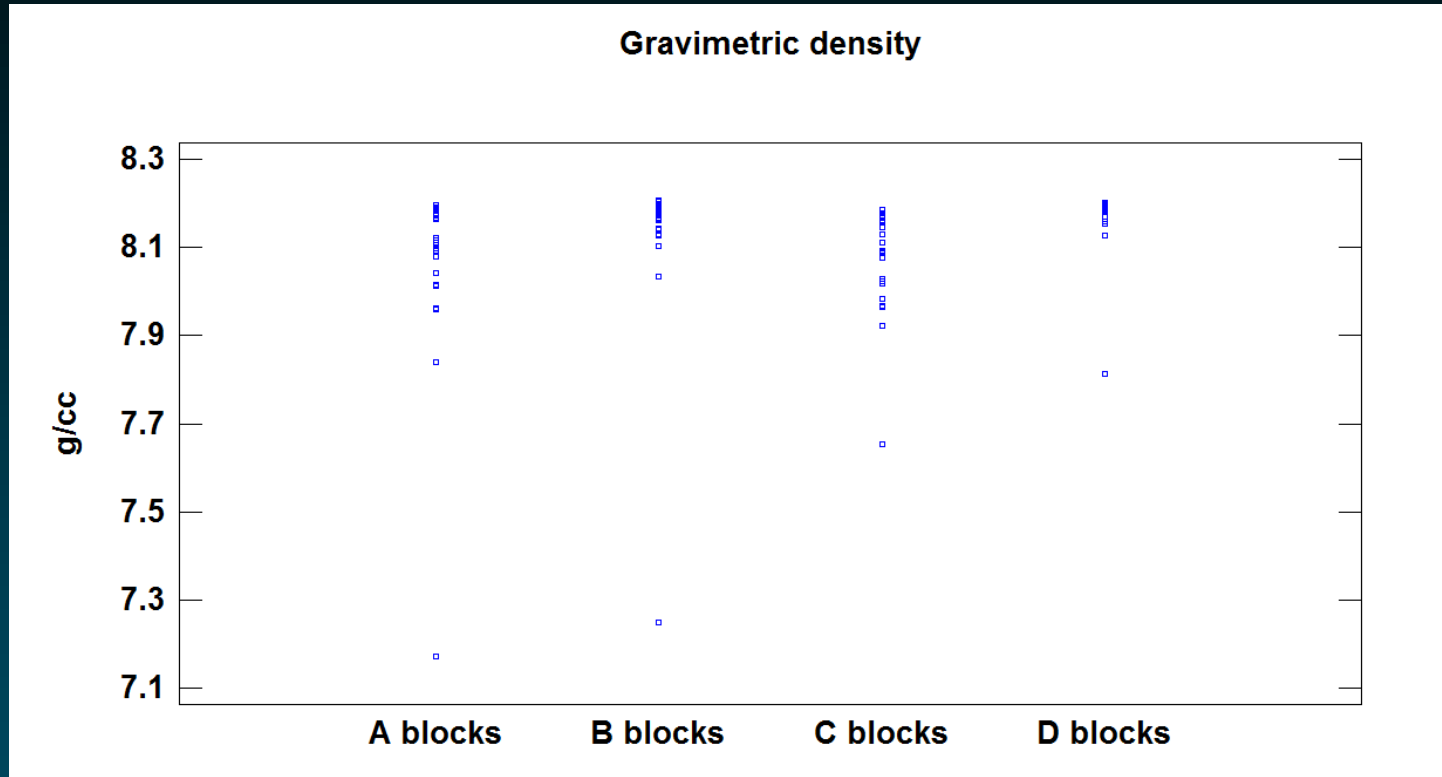
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Summary of Block Set Comparisons

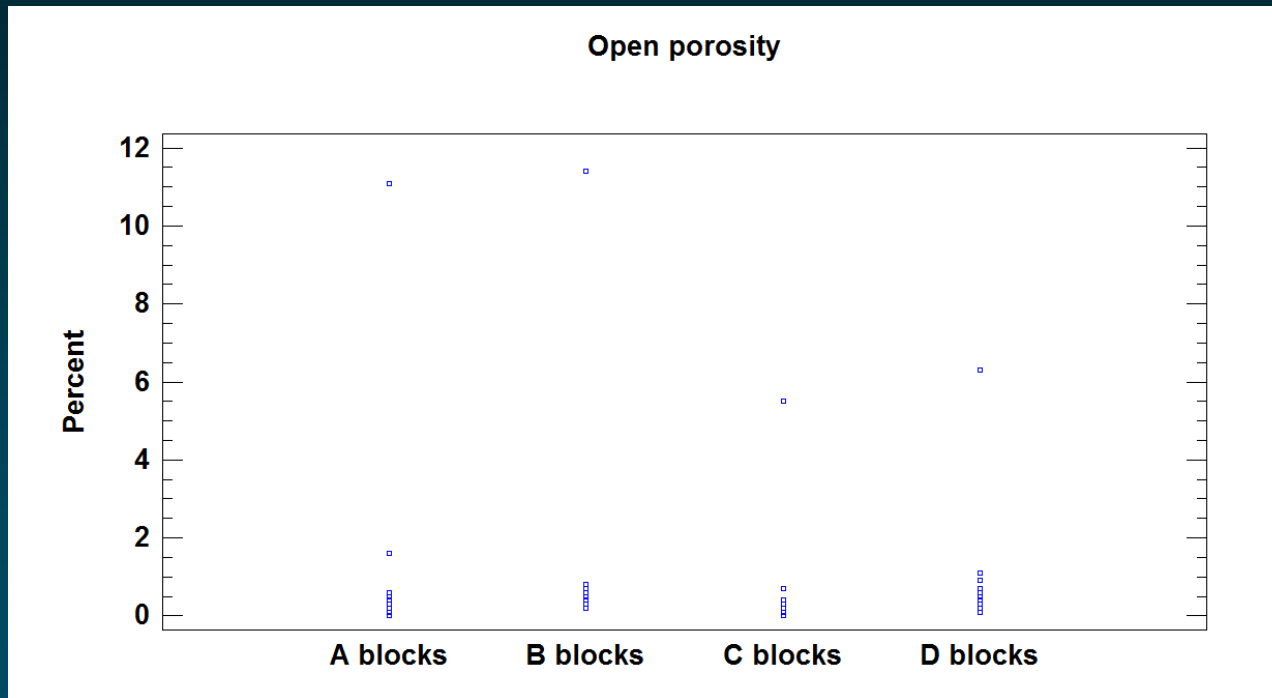


Scatterplot of reported gravimetric density measurements for all block sets

- A – continuous, no heat treatment
- B – continuous, heat treatment
- C – chess, no heat treatment
- D – chess, heat treatment

Summary of Block Set Comparisons

- While density improves with heat treatment, average open porosity seems to increase with postprocessing
 - May be a tradeoff in size vs. frequency of pores for as-built and HT samples
 - Microstructural evaluation needed
 - Apparent increase is more likely an artifact of outliers in open porosity data



A – continuous, no heat treatment
B – continuous, heat treatment
C – chess, no heat treatment
D – chess, heat treatment

Exploring the Relationship Between Density and Process Parameters in the Higher Throughput Regime

- Multiple regression models for each of the following density measurements (with the build parameters as input variables) were constructed:
 - Gravimetric density
 - Bulk density
 - Apparent density
 - Open porosity
- Regression analyses provide insight into the following open questions:
 - 1) What is the bias and strength/significance of the relationships between scan speed, hatch spacing, laser power and density?
 - 2) Can the build parameters be used to reliably predict the degree of material consolidation?
 - 3) How much of the variability in the density and open porosity values can be accounted for by variation in build parameters?

Exploring the Relationship Between Density and Process Parameters in the Higher Throughput Regime

- Summary of observations from 16 multiple regression models
 - Density (apparent, gravimetric, and bulk) is positively correlated with power, but negatively correlated with hatch spacing and scan speed*
 - Open porosity is negatively correlated with laser power and positively correlated with speed and hatch spacing
 - Statistically significant correlations:
 - Hatch spacing and apparent density for B blocks
 - Hatch spacing and apparent density for D blocks
 - Statistically significant regression models:
 - Apparent density (function of power, scan speed, and hatch spacing) for B blocks
 - Apparent density for D blocks

Summary of bias of correlations

	Apparent density	Bulk density	Gravimetric density	Open porosity
Power	+	+	+	-
Scan speed	-*	-	-	+
Hatch spacing	-*	-	-	+

*Exceptions to this are the A block set (positive correlation for scan speed and apparent density) and C block set (positive correlations for scan speed, hatch spacing, and apparent density)

Exploring the Relationship Between Density and Process Parameters in the Higher Throughput Regime

- Relationships indicated by multiple regression models are largely intuitive
 - Increased densification observed with increased laser power and decreased scan speed and hatch spacing
 - As hatch spacing decreases, material consolidation should improve
 - Relationship between hatch spacing and density is only statistically significant correlation
- Little of the variability in the data sets can be attributed to changes in process parameters
 - R^2 values are generally <0.30 and majority of regression models are not statistically significant at the $p=0.05$ level*
 - Models do not impart a predictive capability for density based on process parameters in this operational envelope

*Exceptions to this are the models of apparent density for the B and D block sets

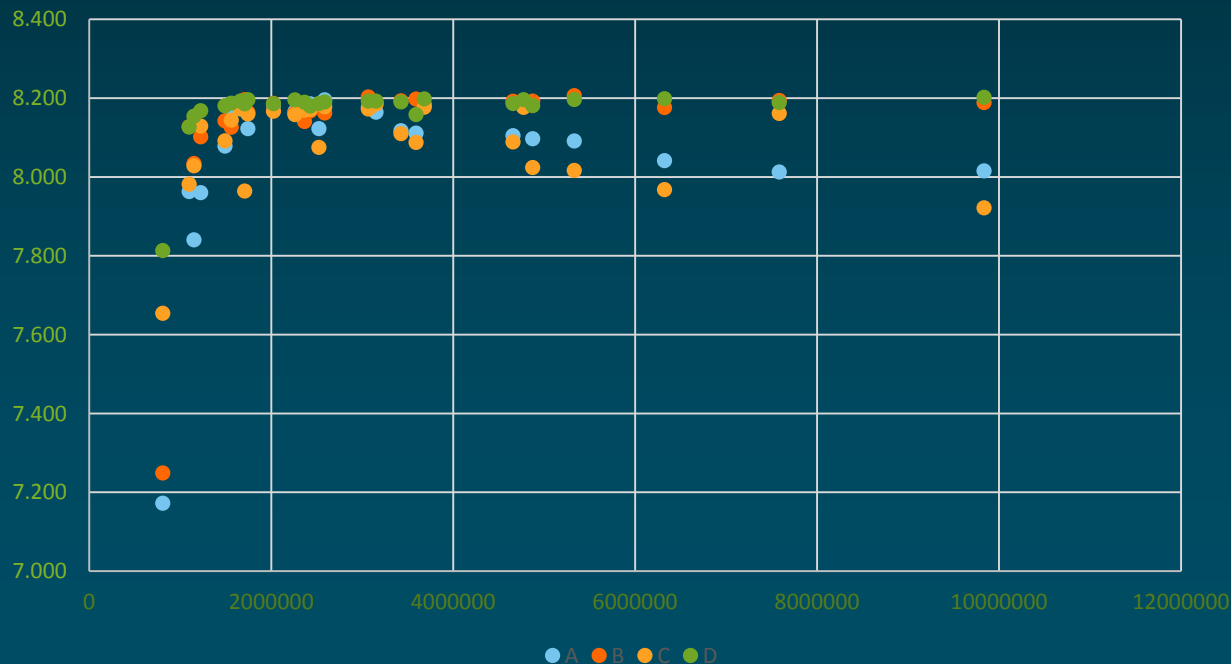
Regression Analysis Based on Energy Density and Scan Rate

- Relating physical properties of materials to a parameter (sometimes dimensionless) that represents the product and/or ratio of several other variables can improve the predictive capability (and scalability) of a model
- Density data were re-analyzed using the Andrew number (A_N) and the scan rate (SR) as input variables.
- The Andrew number A_N [J/m^2] is a measure of relative energy density
$$A_N = P/V*HS$$
- The scan rate SR [m^2/s] is a measure of build speed
$$SR = V*HS$$
- Both A_N and SR are indirect indicators of thermal input.
 - The temperature of the melt pool plays a key role in material consolidation.

P = laser power [W]
V = scan speed [m/s]
HS = hatch spacing [m]

Regression Analysis Based on Energy Density and Scan Rate

- None of the regression models relating density or open porosity to Andrews number or scan rate are significant at the 0.05 level
 - Data fit to linear, logarithmic, and exponential models
- Models can be used to define a region of laser power/scan speed/hatch spacing that correspond to values of density that are above a target value (similar to a machinability map)
 - Example: target density of 98% theoretical for corresponds to Andrews numbers of approximately 2E6 and above



Discriminant Analysis

- Analysis seeks to construct a function to discriminate between parameters (expressed in their native form or in terms of the Andrews number or scan rate) that result in acceptable densification and those that produce a deficient material
 - For propulsion applications, currently require measured density value to reach 99.7% of theoretical
- Table summarizes the classification accuracy of the discriminant functions constructed based on process parameters
 - Discriminant functions significant at the 0.05 level are in bold

	Apparent density	Bulk density	Gravimetric density	Open porosity
A	66.7%	81.0%	100%**	81.0%
B	70.3%	71.4%	76.2%	66.7%
C	66.7%	85.7%	100%**	71.4%
D	100%*	76.2%	61.9%	76.2%

A – continuous, no heat treatment
B – continuous, heat treatment
C – chess, no heat treatment
D – chess, heat treatment

*100% of samples meet or exceed density criteria

**100% of samples do not satisfy density criteria

Discriminant Analysis

- Discriminant functions which classify materials as acceptable or rejectable based on Andrews number, scan rate, or both have classification accuracies that are less than functions constructed using native process parameters as inputs
- Discriminant analysis illustrates the improvement in material consolidation that can be facilitated by postprocessing
 - Example: for gravimetric density, 100% of specimens in groups A and C fail minimum density criteria
 - With heat treatment, 15% and 23% of specimens in groups B and D, respectively, pass
 - Heat treatment can be used to transition a material that is rejectable in the as-built state to an acceptable material
 - Specimens that this statement applies to generally have at least one parameter (laser power, scan speed, or hatch spacing) at the default setting

Percent of specimens failing density criteria by block set

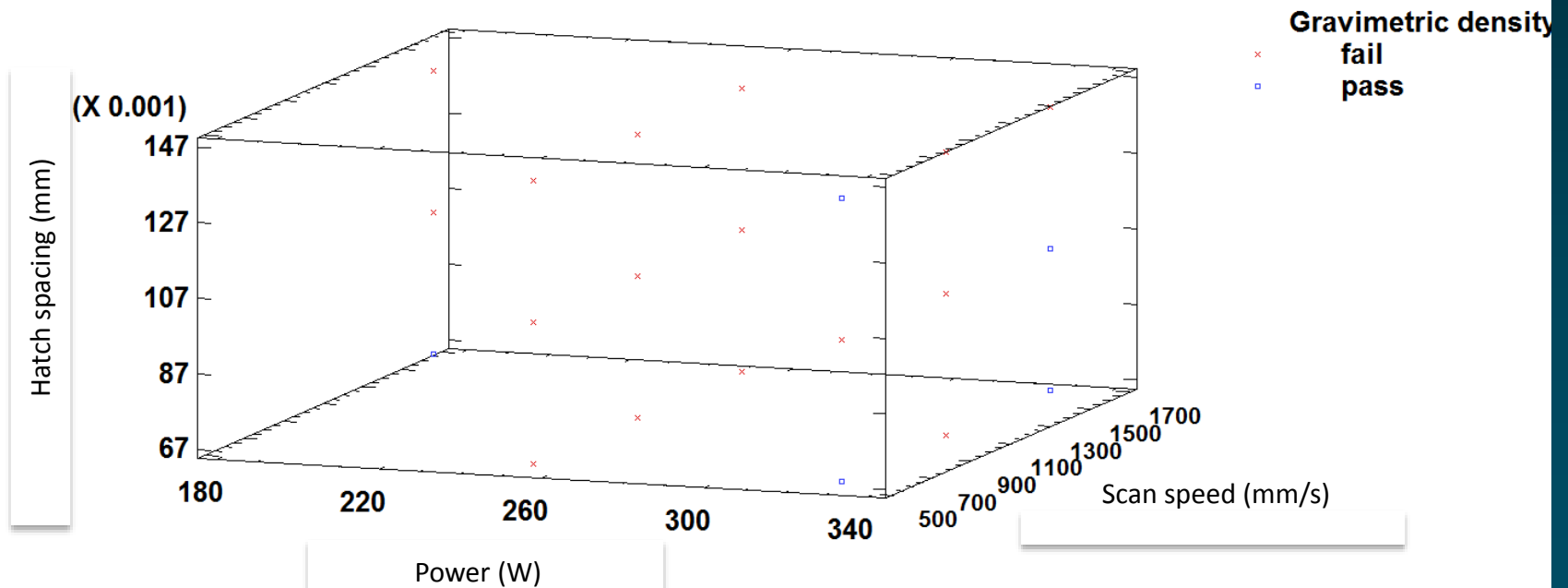
	A	B	C	D
Apparent density	51.9%	3.7%	48.1%	0%
Bulk density	59.3%	25.9%	70.3%	22.2%
Gravimetric density	100%	85.2%	100%	77.8%

A – continuous, no heat treatment
B – continuous, heat treatment
C – chess, no heat treatment
D – chess, heat treatment

Discriminant Analysis

- Parameters with hatch spacing of 0.0675 mm are 5 times more likely to satisfy density criteria than parameters with larger hatch spacing
- Production of an acceptable material is less sensitive to laser power than other parameters
- Parameters at highest scan speed (1560 mm/s) generally do not produce an acceptable material unless the laser power is also operating at the highest level and/or the hatch spacing is at the lowest level

Plot of acceptable and rejectable material for block set D



Other general observations

- Highest values of open porosity were observed for the following parameters:
 - 180 W/1560 mm/s/0.105 mm
 - 180 W/1560 mm/s/0.1425 mm
 - 256 W/1560 mm/s/0.1425 mm
 - 332 W/1560 mm/s/0.1425 mmAll of these specimens have one or more parameters which lie at the extremes of the design matrix.
- Specimens built at 256 W/1030 mm/s/0.1425 mm had among the highest calculated density values, but also one of the larger values for open porosity (~0.4%) in the data set.
- Best (recommended) higher throughput parameters based on density alone:
 - 180 W/1030 mm/s/0.0675 mm
 - 180 W/1030 mm/s/0.105 mm
 - 180 W/1560 mm/s/0.0675 mm
 - 256 W/1030 mm/s/0.0675 mm
 - 256 W/1030 mm/s/0.105 mm
 - 256 W/1560 mm/s/0.0675 mm
 - 332 W/1030 mm/s/0.0675 mm
 - 332 W/1030 mm/s/0.105 mm
 - 332 W/1560 mm/s/0.105 mm



Takeaway: can increase scan speed and still produce acceptable aerospace material in terms of density as long as hatch spacing is at level I or II.

Other general observations

- Parameters associated with lowest density:
 - 180 W/1030 mm/s/0.1425 mm
 - 180 W/1560 mm/s/0.105 mm
 - 180 W/1560 mm/s/0.1425 mm
 - 256 W/1560 MM/S/0.1425 mm

Irrespective of other parameters, general rule that a larger hatch spacing is more likely to yield a deficient material.

- Specimen 9B (built at 180 W/1560 mm/s/0.1425 mm) had approximately 10 times the open porosity of the mean samples in its data set. This large difference is consistent across data sets.
- Set D (chess, heat treatment) generally had higher densities and lower open porosity. Set D also had a 1% smaller coefficient of variation than set B. With the exception of 9D, measurements in set D were fairly uniform.

Conclusions and Further Work

- For aerospace applications, key driver is material quality rather than build time.
 - The data in this investigation is also applicable to other industries who seek to transition AM from the custom, low-rate production sphere it currently operates in to high-rate mass production applications.
 - Threshold density values for acceptable material may be less stringent for these sectors, making operation in the higher throughput regime more feasible
- Few significant relationships between build parameters and density in the higher throughput regime were found
 - In general, recommendation is that when operating in the higher throughput regime, at least one process parameter should remain at the level I (default) value.
 - Densification for In718 produced by SLM is highly sensitive to hatch spacing. This parameter should be maintained at the smallest reasonable spacing to ensure sufficient material consolidation.
- Data is most useful when a target (minimum acceptable) value of density has been identified
 - Functions can then be constructed to define a processing envelope for acceptable material consolidation
 - Target value of density will be application specific

Conclusions and Further Work

- While deposition pattern seems to have a negligible effect on densification, the data from block set C and D (chess) is more consistent (lower standard deviation) than block sets A and B (continuous).
- This data set demonstrates that it is possible to transition a material from deficient to acceptable (in terms of density) with postprocessing (HIP and heat treatment).
 - However, microstructural evaluation is necessary to assess the size of pores and the type of porosity.
 - Open porosity will generally heal with HIP, but linear porosity is more difficult to eradicate with postprocessing
 - Whether or not a material can reach acceptable densification with postprocessing is governed by build parameter settings.
 - Parameters with one or more levels at the extremes of the envelope are less likely to be brought into specification with heat treatment.

Conclusions and Further Work

- Based on empirical observations and provides little insight into process physics that create an acceptably dense material
 - Opportunity for collaboration
- Microstructural evaluation of specimens that:
 - Are considered “fully dense” (>99.7% theoretical density)
 - Have levels of porosity and/or density that are “out of family” with the rest of the data set
 - Are deficient in the as-built condition, but rendered acceptable with further processing
- Correlation of density with mechanical properties for these parameter sets (phase III)
- Need for a “true” AM In718 target density value
 - Textbook value may not be best representation of AM material

Questions



Test firing of rocket engine with 3D printed parts
NASA Marshall Space Flight Center
December 2015