

WHITE PAPER

Titanium Process Capability Using Laser Powder Bed Fusion



Introduction

Laser powder bed fusion (LPBF) enables manufacturing of highly optimized metal parts. Over the past decade, the LPBF technology has matured significantly. Direct metal printing (DMP) is the brand name of 3D Systems' LPBF technology. High-end industries such as healthcare and aerospace have embraced this technology for producing metal components with enhanced functionality and performance or significant weight savings for their critical applications. DMP components for such demanding applications are often manufactured in Ti6Al4V because of the alloy's compelling material properties, such as biocompatibility, high strength-to-weight ratio, and excellent fatigue properties. Aerospace and healthcare are heavily regulated industries, in which components are subjected to rigorous validation/qualification procedures and quality assurance requirements. These industries require stable and capable manufacturing processes to allow the adoption and industrialization of any given manufacturing technology. Therefore, equipment and process architecture, combined with proofing capability, are vital for bringing DMP technology beyond introduction to fully qualified serial manufacturing of critical applications.

The DMP process capability study performed on LaserForm Ti Gr23 (A), and summarized below, shows that the DMP system architecture is a key contributor to the DMP process robustness. Namely, the tight control of the inert atmosphere in the build chamber results in stable powder feedstock quality (chemistry and deposition quality), and reliable printed part quality, yielding excellent and repeatable density, chemical, and mechanical properties.^{1,2} Moreover, the robustness and reliability of the current DMP machine platforms and the preceding generation (DMP ProX 320) have been demonstrated over the decade by delivering a qualified DMP production process flow and reproducible product quality to 3D Systems' healthcare and demanding industrial customers.

Process Overview

FROM POWDER FEEDSTOCK TO PRINTED PART QUALITY, A CRITICAL ASSESSMENT

DMP qualification relies on a risk-based assessment of the DMP process flow, going from powder feedstock to printed part. A simplified DMP flow chart covering the powder feedstock, the DMP process, and the DMP product is presented in Figure 1. This flow chart includes, but is not limited to, critical parameters and characteristics of the powder feedstock, the DMP process, and the DMP end-product. These critical parameters are among the variables considered for the risk-based approach of the DMP qualification.

Powder Feedstock

- Powder (deposition) quality**
 - Chemistry
 - Particle size distribution
 - Morphology
 - Flowability

- Powder management**
 - Sieving
 - Recycling & blending
 - Storage

DMP Process

- System architecture & process control**
 - Inert atmosphere control
 - Gas flow
 - Laser focus
 - Powder deposition (recoater)
- DMP parameter set**
 - Laser power
 - Scan speed
 - Layer thickness
- DMP build file**
 - Digital workflow
 - Part design and build strategy

DMP Product

- Printed part quality**
 - Chemistry
 - Density
 - Mechanical properties
 - Surface roughness
 - Dimensional accuracy

Figure 1. Simplified DMP process flow including, but not limited to, variables and characteristics of the powder feedstock, the DMP process, and the DMP product.

POWDER FEEDSTOCK

Quality assurance of the powder feedstock is the first prerequisite for obtaining high-quality and reproducible DMP products. The feedstock quality relies on the powder chemistry and intrinsic powder properties such as particle size distribution (PSD), powder morphology, and the associated flowability. The powder deposition quality is affected by these powder properties and the powder surface chemistry (moisture, oxygen content).

The chemical composition of the Ti6Al4V feedstock material must meet the requirements of ASTM F3001, ASTM F2924, and ASTM F3302. It should be noted that Ti grade 23 has a maximum allowable oxygen content of 0.13 wt%. The reactive nature of titanium with oxygen forms a risk to the powder chemistry. Namely, poor control of the inert atmosphere in the build chamber (oxygen, moisture) will lead to a gradual oxygen increase in the Ti grade 23 feedstock material, thereby affecting the feedstock's reusability and conformity.

Powder feedstock management and handling such as sieving, recycling, blending, and storage can affect these feedstock quality characteristics when poorly controlled. Moreover, the extent of powder reuse affects the economic viability of DMP manufacturing when considering the material cost of Ti6Al4V powder feedstock. Therefore, quality assurance requirements and traceability of reused powder batches are vital when scaling up to industrial DMP production. This white paper explains the powder management approach in a subsequent section.

DMP PROCESS

Exhibiting a stable DMP process is essential to pass the strict DMP validation process, thereby delivering reliable DMP products with excellent and reproducible part quality. DMP process stability can be affected by three main process variables: DMP machine condition, DMP parameter set, or DMP build file, as illustrated in Figure 1. These main process variables can be further broken down into more tangible and controllable DMP process variables. Mind that many more DMP process variables exist, but their risk or impact is limited, and they are therefore not covered in the simplified DMP flow chart.

ROBUST DMP MACHINE ARCHITECTURE

A stable DMP process with a low build-to-build and machine-to-machine variability starts with a robust machine architecture and appropriate machine calibration procedures. The robust DMP machine platform architecture (ProX DMP 320, DMP Flex/Factory 350, DMP Factory 500) allows for a vacuum pre-cycle prior to the printing job and actively removes air and moisture from the build chamber and the powder feeds and fills it afterwards with high-purity argon gas. The event of extracting oxygen and moisture from the powder feed is called the "boiling effect" and is shown in Figure 2. The efficient/effective vacuum pre-cycle helps achieve an extremely low oxygen environment. Furthermore, the vacuum chamber's leak-tight design ensures that no oxygen can leak into the build chamber and results in exceptionally low argon consumption during printing. This vacuum chamber concept is key for achieving the desired tight control of the build chamber's inert atmosphere during printing. The vacuum chamber concept helps to eliminate the risks for oxygen pick up by the powder feedstock, resulting in stable powder chemistry and a significant enhancement of the powder reusability of Ti Gr23.¹

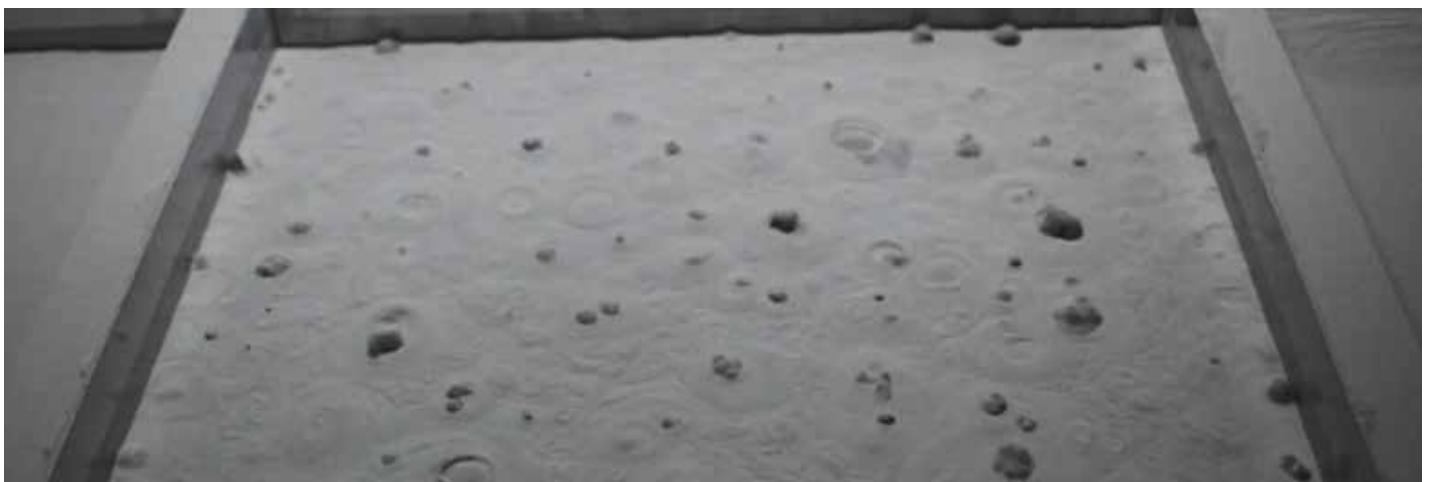


Figure 2. Boiling effect showing the extraction of oxygen and moisture from the powder feed.¹

DMP MACHINE CALIBRATION

Periodic maintenance practices and bi-annual machine calibration checks are necessary for the quality assurance of the DMP process. Therefore, calibration on the gas flow, laser focus, laser power, and laser scanner are required to ensure the DMP machine's health condition. Moreover, these calibrations ensure that the material is printed within the acceptable energy density window defined by the DMP parameter set (laser power, spot size, scan speed, hatch spacing, layer thickness).

The gas flow across the powder bed has a significant effect on the print quality of the material. Direct metal printing is a localized and energy-dense welding process, fusing loose powder particles at high scanning speeds. The energy-intense laser-material interaction creates vapor plumes and spatters above the meltpool. The interaction of these vapor plumes and spatters with the laser beam is undesired and can lead to incomplete welding and lack-of-fusion defects in the printed material. Moreover, an insufficient extraction of the vapor may cause optics instability (e.g., thermal lensing) during printing. Therefore, a stable gas flow across the powder bed is required to ensure a stable meltpool and an efficient extraction of vapor and spatters resulting from the meltpool. A detailed study on the effect of an optimized gas flow for achieving homogeneous, reliable, and repeatable density and mechanical properties of DMP Flex 350 LaserForm Ti Gr23 (A) across the build envelope is described by Beckers et al.¹

Lastly, it is important to note the powder deposition is not purely an intrinsic property or quality measure of the powder feedstock. Besides the powder properties and powder surface chemistry, the powder deposition quality is also affected by atmospheric control in the gas chamber (oxygen, moisture) and the coater type and its settings. Therefore, careful machine preparation and coater installation need to be performed by trained machine operators to ensure a good powder deposition quality during the build.

DMP BUILD FILE

Digital data exchange through software workflows is an essential element of the additive manufacturing process. Securing digital data exchange and validating software workflows are part of the quality assurance requirements and the DMP qualification process. 3DXpert is an all-in-one integrated software package, allowing part design including supporting, assignment of build parameters, and scan strategy.

As such, the 3DXpert job file contains all DMP parameter data and dimensional data for the part, which can be transferred entirely across machines of the same type with no further slicing or manipulation, nor machine-specific compensations or calibrations.

DMP PRODUCT

Printed part quality can be assessed on many distinct aspects such as the chemistry, density, mechanical properties, surface roughness, or dimensional accuracy, shown in Figure 1.

The initial chemical composition of the Ti6Al4V grade 23 (< 0.13 wt.% O) printed material must comply with the same material standard requirements, as is the case for the feedstock material. Ti6Al4V grade 23 imparts better ductility and fatigue properties than other Ti6Al4V grades with a higher concentration of interstitials (O, N, H).^{3,4} For that reason, standards prescribe strict maximum oxygen limits for Ti6Al4V grade 23. Poor control of the inert atmosphere (oxygen, moisture) during the build will lead to an oxygen increase in the printed material. This oxygen enrichment forms a substantial risk for the deterioration of quasi-static and dynamic mechanical properties of Ti grade 23. Therefore, tight control of the inert atmosphere in the build chamber is a prerequisite for extended powder reusability and for obtaining excellent and repeatable quasi-static and fatigue properties of Ti grade 23 printed parts,^{1,2} as discussed previously.

In conclusion, quality assurance and monitoring of powder feedstock, combined with a robust DMP process, well-documented machine calibration protocols, and trained machine operators all play a vital role in achieving reliable and repeatable print-to-print quality, yielding nearly dense and excellent mechanical properties and surface roughness.

DMP QUALIFICATION

The DMP qualification process relies on a risk-based approach and follows a phase-gated flow, passing through process validation, equipment validation, and product validation in order to reach a qualified production. The DMP qualification process complies with ASTM F3434. Figure 3 represents the DMP qualification flow chart:

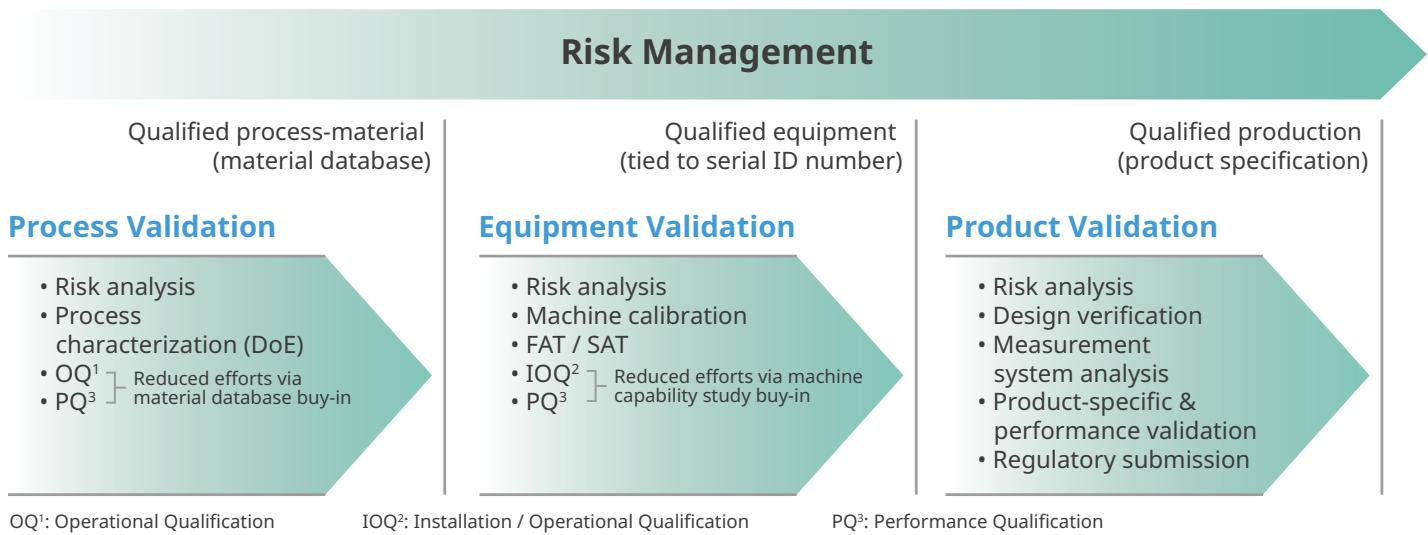


Figure 3. DMP qualification flow chart

PROCESS VALIDATION

DMP qualification starts with process validation, in which qualifying the DMP process and the processed material go hand in hand. Risk analysis allows the definition of the significant process variables, which have been covered in Figure 1 and discussed in the previous section (DMP Process). A Design of Experiments (DoE) on these DMP process variables is applied to define an acceptable process window. After this process characterization, where worst case process parameters and combination of parameters are defined, an operational qualification at the extents of the process window (OQ) and performance qualification at nominal conditions (PQ) are run to prove the DMP process's capability. Building such material databases, proofing the process capability can be costly and time-consuming. Therefore, a material database buy-in can be applied at this stage of the process validation. With this, a reduced and cost-efficient testing campaign can be set up, proofing the equivalency with the existing material datasheet. A qualified process and material are obtained when passing this process validation. The process validation allows setting material's acceptance criteria on chemistry, density, mechanical properties, surface roughness, etc.

EQUIPMENT VALIDATION

The second phase of the DMP qualification focuses on validating the equipment (i.e., DMP machine). Unlike the process validation, the equipment validation is always tied to the equipment's unique serial number. Machine calibration must be performed before each factory acceptance testing (FAT) and site acceptance testing (SAT) campaigns. The acceptance criteria, defined from the process validation, are often used as a pass/fail criterion for the FAT and SAT. Upon successful FAT and SAT, the equipment validation continues with a series of test builds to assess the robustness and reliability of the DMP equipment, known as Installation / Operational Qualification (IOQ) and the Performance Qualification (PQ). Likewise to the process validation, the aim is to collect data proofing the process capability (Cp, Cpk) and process performance (Pp, Ppk) of the equipment (serial ID number). The scope and cost of the IOQ and PQ testing campaign can be reduced through a buy-in of existing equipment capability studies of the same DMP platform family. In the latter case, equivalency between the serial numbered DMP machine and the DMP platform family needs to be proven.

PRODUCT VALIDATION

The final phase of DMP qualification focuses on the product specific qualification. At this phase, the process and equipment have been duly qualified to meet the material and process standards and the form, function, and performance of the part must be verified or validated. The acceptance criteria of such test campaigns will vary depending on the application. With a well-executed process, material, and equipment validation, the scope and breadth of the validation can be limited while leveraging objective evidence of the process' capability. Other aspects of the processes required to attain desired characteristics are integrated in the test campaign to assure that the entire workflow is adequately controlled and can produce to specification capably. Ultimately, the collection and evaluation of this data, from the process design stage through commercial production, establishes scientific evidence that a process is capable of consistently delivering quality products.⁵

Deep Dive into Process Characterization

The previous section outlined the different steps it takes to achieve and demonstrate a capable and validated process and product. The next sections explain in more detail how the process characterization is conducted based on establishing a Design of Experiment (DoE) resulting in an acceptable process window.

PROCESS CHARACTERIZATION INTRODUCTION AND PRINCIPLE

Process characterization begins with the development of a Critical-to-Quality versus Critical Process Parameter (CTQ/CPP) matrix. The goal of this matrix is to determine which CPPs influence each CTQ, and ultimately create a pathway to develop studies and establish objective evidence before moving into full process validation (OQ/PQ). The objective is to make sure that CPPs that have an influence on the CTQs are well tested and validated, while limiting time and incurred cost by ruling out CPPs which do not significantly influence the CTQs. The output of the CTQ/CPP matrix is used as input to a process characterization DoE, and eventually flows into an Operational Qualification (OQ), providing challenge variables and a process window.

CTQ/CPP MATRIX

The framework of the matrix begins with establishing CTQs, which are generally based on material properties and relevant material standards. Next, CPPs are established based off the process flow and risk analysis, then listed and categorized into one of four groups of variables. CPP variable groups are defined as follows for early-stage development, pre-validation:

- **Monitored:** Variable that influences the process, but monitors whether process is 'in control'; may have action limits (e.g., oxygen concentration while printing)
- **Fixed:** Variable that influences the process, has known range or set points, but has a standard operating procedure or work instruction in place to control (e.g., file preparation)
- **Challenge:** Variable that influences the process, has range which has not been established at current phase of characterization (early on), requires challenge in DoE to define range, plus challenge within OQ if main effects are present (e.g., laser power)
- **Non-critical:** Does not need process control, supported by justification and/or objective evidence for why it is not critical

Once CPPs are categorized, the matrix is used to understand what data/process controls are needed in order to fully characterize the process.

DOE SETUP/DEFINITION

For the 3D Systems DMP process of printing, Ti6Al4V ELI components, the CTQ/CPP matrix, and process characterization culminates into a DoE study. The study's test configuration is based on a four-factor (CPP), two-level, full factorial DoE, and is comprised of 16 runs (builds).

This experiment configuration allows for full resolution statistical regression analysis, examining the main effects and higher order interactions between the inputs and outputs of the study, as illustrated by Figure 4. Once the factorial regression analyses are performed, the results are evaluated to determine the practical significance of the effects.

The DoE inputs (four CPPs) include Laser Power (LP), Feed Rate (FR), Argon Flow (AF), and Build Time (BT), and were determined throughout various engineering studies within the CPP/CTQ matrix phase. Each variable is challenged at either the low or high end of its process window during each build throughout the DoE. The outputs (three CTQs) include mechanical properties, chemical composition, and alpha case, and are based off ASTM F3001, F2924, and F3302. Specific mechanical property CTQs included in the evaluation are the following:

- 0.2% Yield Strength (YS)
- Ultimate Tensile Strength (UTS)
- Elongation (EL)
- Reduction of Area (R/A)

The entire DMP manufacturing process, from build preparation to outside processing/testing (including HIP heat treatment), is used throughout execution of the study. For equipment and parameters that do not directly affect the output for which a test configuration is intended, or that cannot be manually controlled, nominal operating conditions are used (e.g., build chamber pressure).

In order to gather quantitative data for the outputs, build plates are prepared with a series of coupons which are subjected to testing per the applicable and aforementioned ASTM standards. All test samples are printed at both 30 μm and 60 μm layer thicknesses across the entire build envelope of the machine, which maximizes verification of in-process variability in order to challenge the process capabilities. In addition, the mechanical test samples are printed in both horizontal (XY) and vertical (Z) orientations.

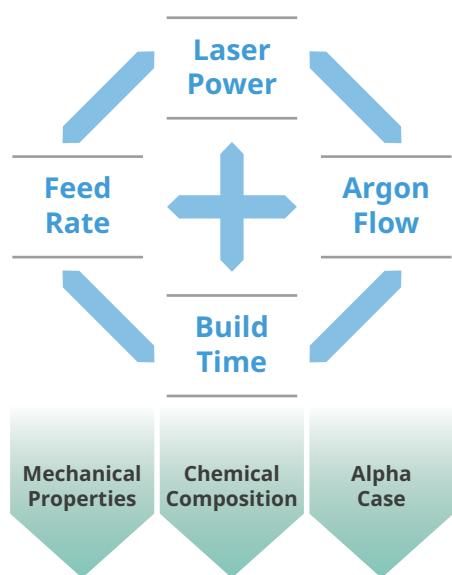


Figure 4.

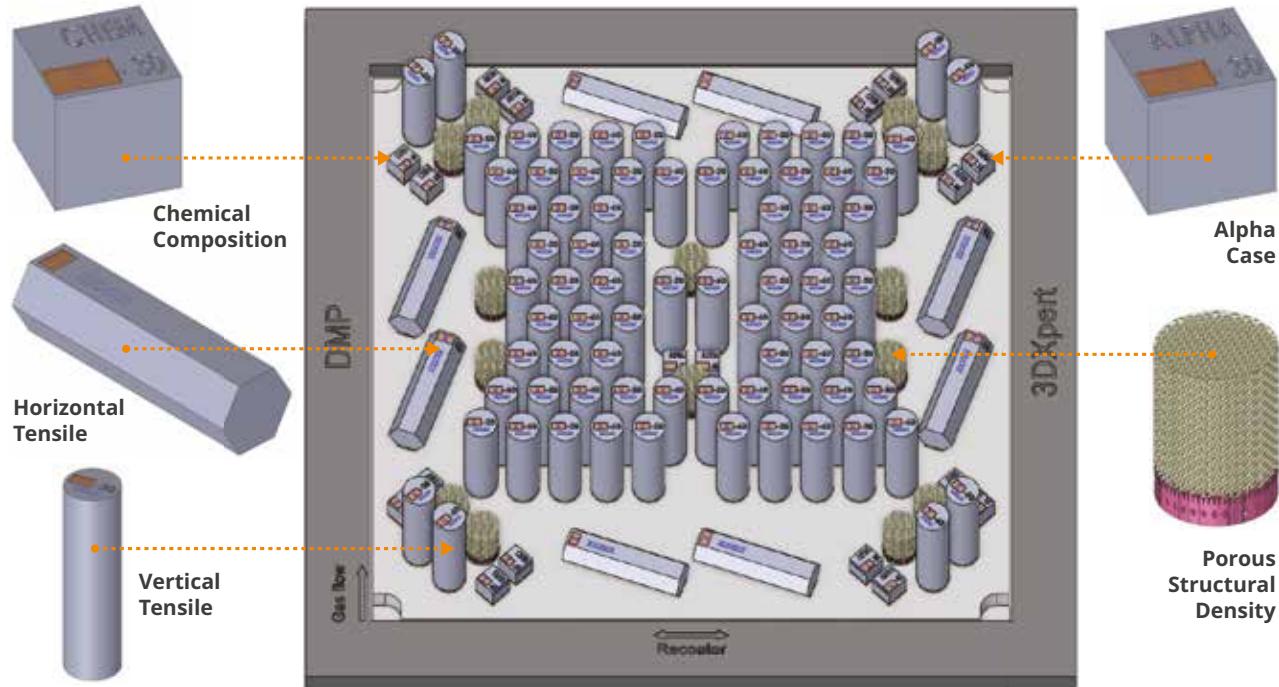


Diagram 1.

DOE DATA ANALYSIS METHODS

The data gathered from the DoE is analyzed in two steps for each CTQ, such that valid conclusions can be made on the effects of the chosen CPPs on the considered CTQs.

First, Analysis of Variance (ANOVA) studies are conducted to determine whether the population means of the continuous CTQs (chemical and mechanical test data) differ when segregated into groups based on layer thickness (30 µm/60 µm) and sample orientation (XY/Z for mechanical test data).

A statistically significant difference between populations is considered when the P-value output by the ANOVA studies is ≤ 0.05 . A statistically significant difference ($P \leq 0.05$) results in splitting the data into the respective populations, regardless of whether a practical significant difference is shown or not. This increases the sensitivity of the factorial regression analysis. When the difference in population means is not considered statistically significant, data is pooled into a single population.

Secondly, effects of the four CPPs are assessed against statistical and practical significance limits. The absolute values are used to compare against these significance limits. For statistical significance, the limit is calculated for each parameter per population by converting the p-value limit into an absolute effect limit. For practical significance, the standard deviation is used, calculated from historical data.

Pareto charts are used to visualize the absolute effects. When the absolute effect is greater than both significance limits, the effect is considered significant.

DOE EXECUTION AND ANALYSIS

A total of 16 builds, each with their respective set of challenged conditions for the four identified CPPs, are executed consecutively on a single machine. The coupons are removed from the build platform and post-processed according to the process flow depending on the foreseen test purpose: mechanical, chemistry, and alpha case. Tests are performed at accredited labs compliant to applicable standards.

MECHANICAL PROPERTIES

Four mechanical properties are analyzed within the factorial regression analysis for both vertically (Z) and horizontally (XY) oriented tensile samples, each built in 30 µm and 60 µm:

- 0.2% Yield Strength (YS)
- Ultimate Tensile Strength (UTS)
- Elongation (EL)
- Reduction of Area (R/A)

For both sample orientations and layer thicknesses, a statistically significant difference is found for at least one mechanical parameter. This results in splitting the data in four populations: 30XY, 60XY, 30Z and 60Z. However no practical significance is observed.

The analysis and pareto charts (Figures 5–8) show that multiple factors have a statistically significant effect on all four mechanical properties. However, none of the factors show a practical significant effect on the mechanical properties. As an example, the pareto charts for %El for the four populations are shown in Figures 5–8.

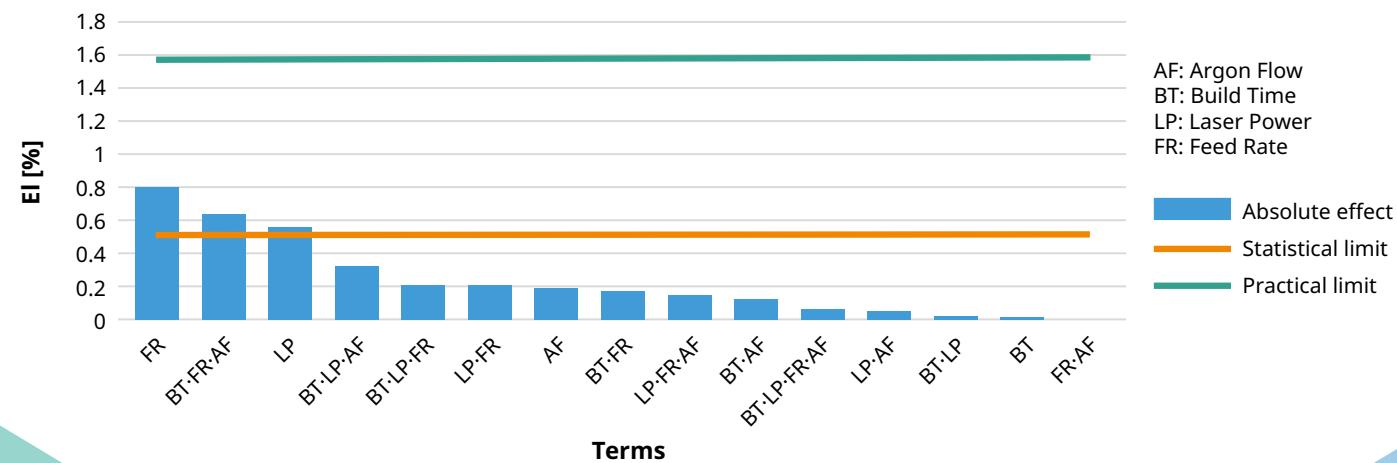


Figure 5. Mechanical — Pareto chart of Elongation with population 30XY

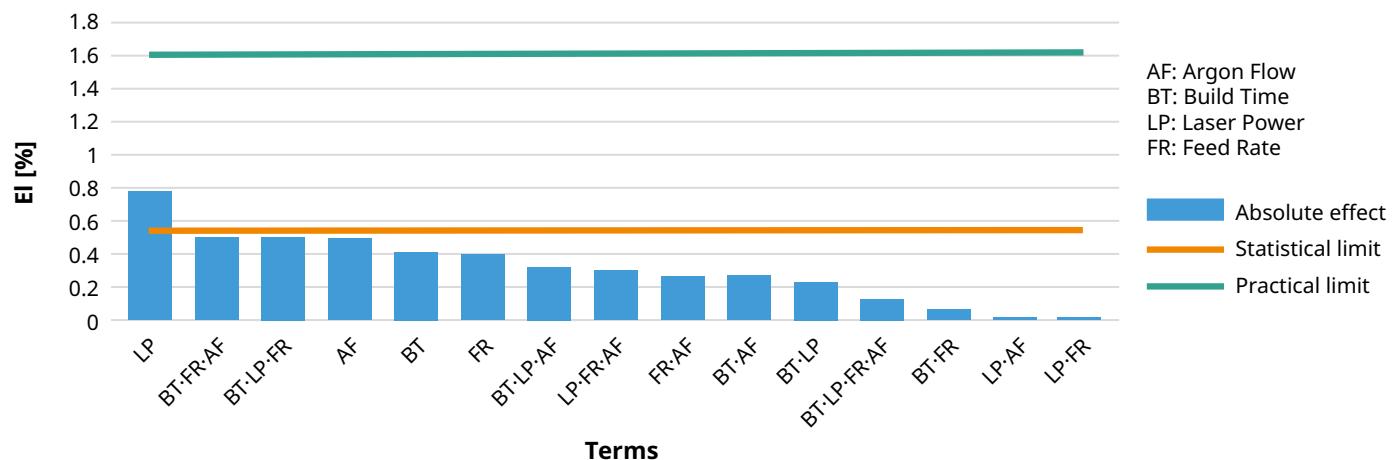


Figure 6. Mechanical — Pareto chart of Elongation with population 60XY

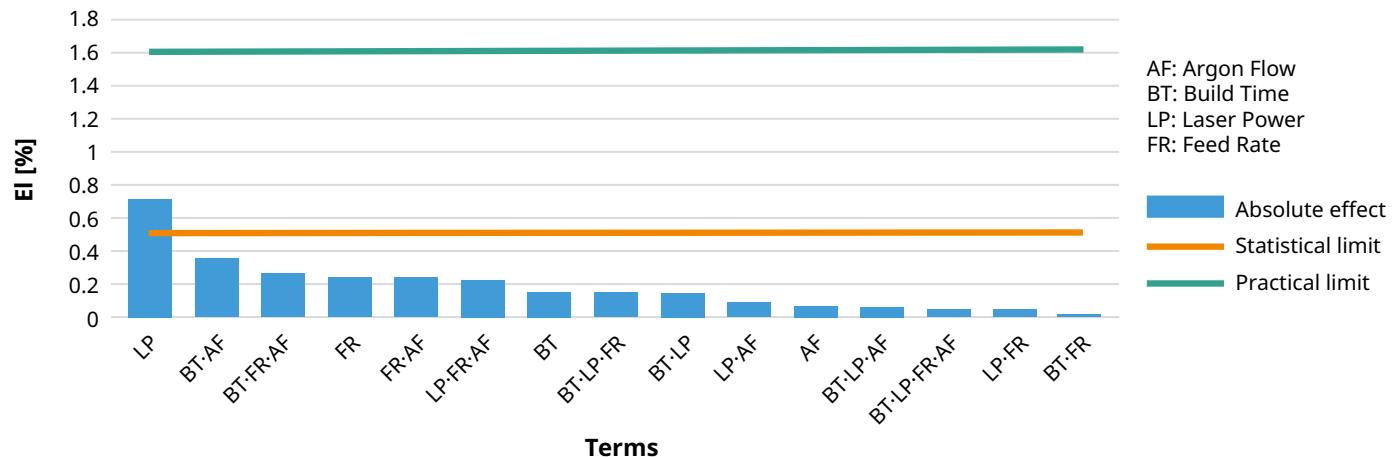


Figure 7. Mechanical — Pareto chart of Elongation with population 30Z

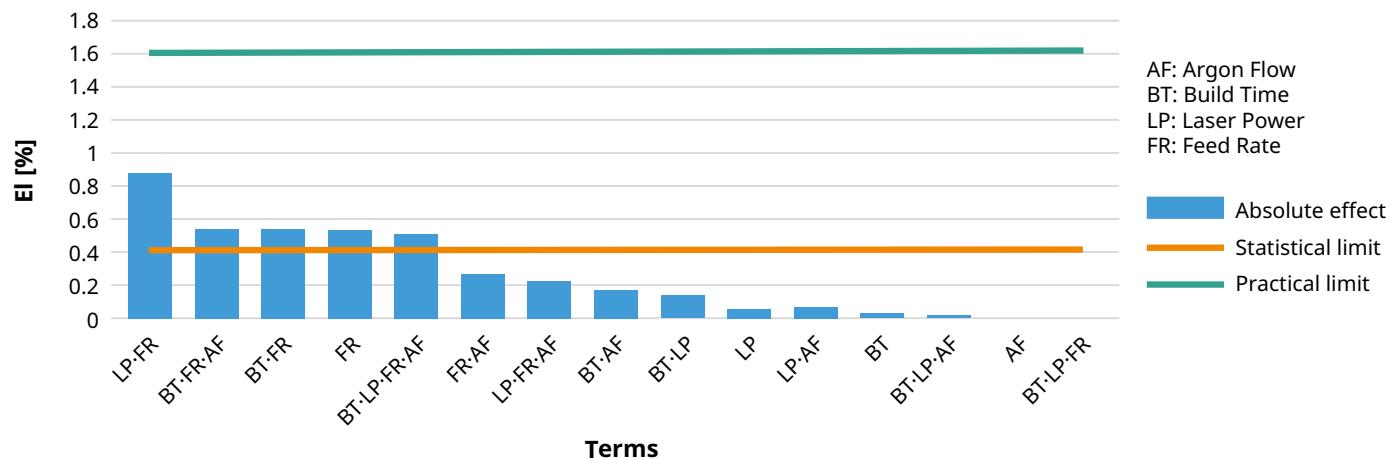


Figure 8. Mechanical — Pareto chart of Elongation with population 60Z

CHEMICAL PROPERTIES

As part of the factorial regression analysis, coupons are analyzed on all chemical elements of Ti6Al4V ELI, as defined by ASTM F3001, both for layer thickness 30 μm and 60 μm . A statistically significant difference is found for aluminum and vanadium between the 30 μm and 60 μm populations. Therefore, to normalize the data, the analysis is split into two populations: 30 μm and 60 μm . None of the differences between variable-to-variable population means for chemical properties are practically significant.

The factorial regression analysis shows that multiple factors have a statistically significant effect on the different chemical constituents. No practical significant influence of any of the factors is observed. As an example, the pareto charts for oxygen content for the two populations are shown in Figures 9-10.

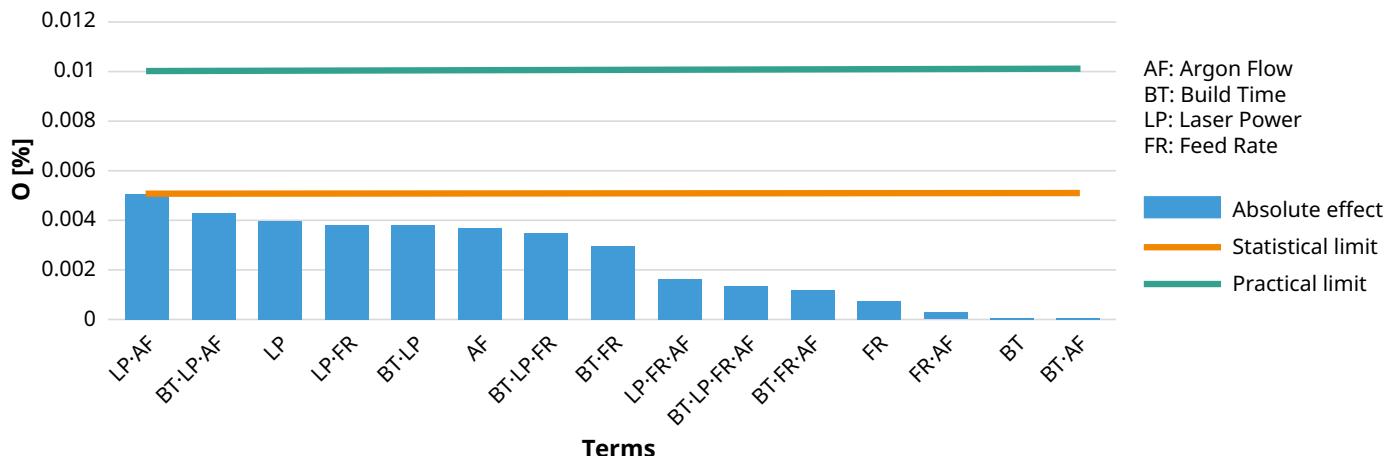


Figure 9. Chemical — Pareto chart of Oxygen with population 30 μm

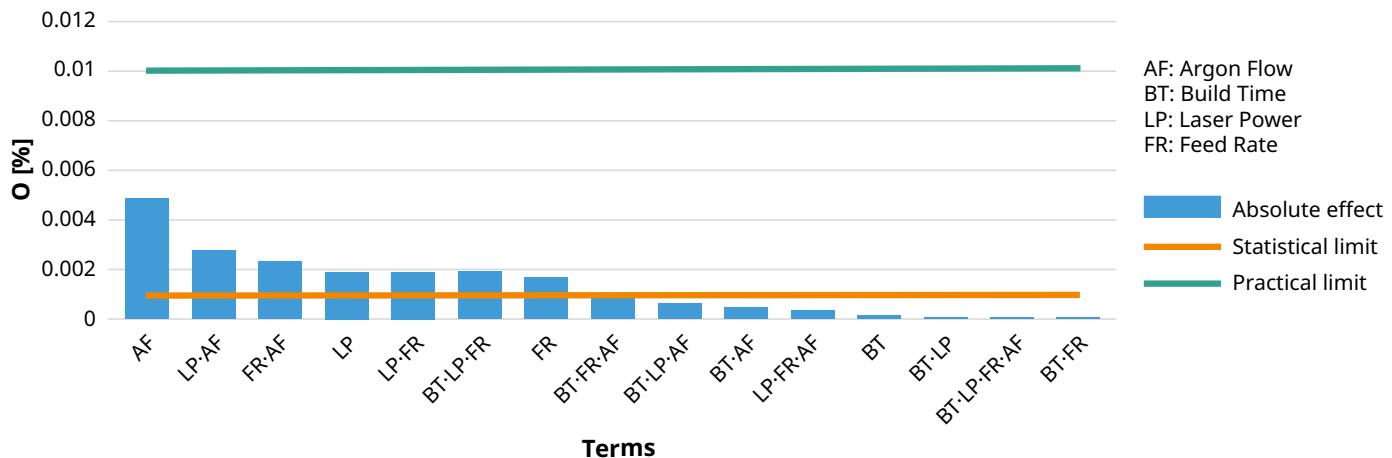


Figure 10. Chemical — Pareto chart of Oxygen with population 60 μm

ALPHA CASE

Test results from alpha case testing are judged by pass or fail criterion following ASTM F3001 where pass is given if no evidence of alpha case is observed on the test sample. None of the test samples show evidence of alpha case. Therefore, no effect of the assessed factors can be observed.

DoE CONCLUSION

In conclusion, the results of the factorial regression analysis show that none of the CPPs have a practically significant influence on the CTQs, which means none of the variables are considered “main effects” as long as they remain within the tested process window. This result shows a highly robust and stable process window. Nonetheless, an OQ is developed for the process validation based on the DoE results, using a conservative approach to determine which CPPs to challenge and their respective process windows. Calibration and maintenance cycles ensure these CPPs are maintained within the process window throughout normal production, ensuring a stable and reliable printing process. Further insight into the process capability and stability, in terms of both input material and measurable outputs, is discussed in the following sections.

Process Capability: Input Material (Powder)

BACKGROUND: POWDER BATCH/LOT HANDLING

CONTINUOUS REUSE

3D Systems' powder handling process uses a continuous reuse powder management strategy. For each new powder lot, virgin powder is loaded into the machine in order to process the first build. The second, and all subsequent builds are processed with a blend of virgin and used powder until the entire lot has been consumed. Powder feeds are topped off with the same virgin lot only as needed by volume. This powder management cycle is depicted in Figure 11 below.

Actions shown inside the blue box count as one reuse/recycle, starting with the second cycle for a particular lot. When mixing powders, the traceability to the virgin lot is maintained for each printer. Typically no different lots are mixed.

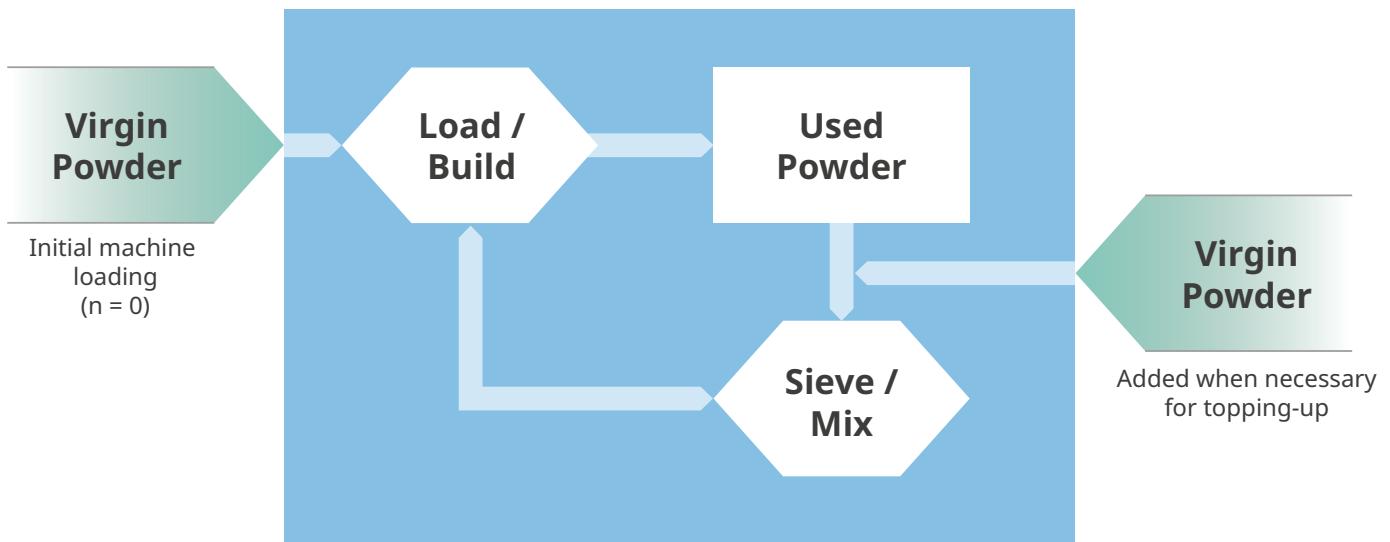


Figure 11. Metal powder management strategy used by 3D Systems

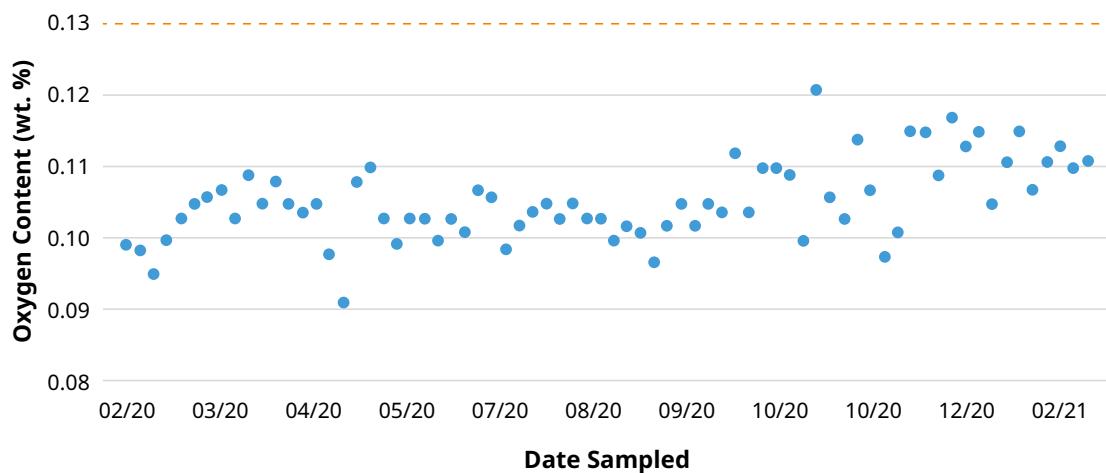


Figure 12. Time Series Plot of Powder Oxygen Content

A slight upward trend in powder oxygen wt.% is observed in these results, which is to be expected given the reactive properties inherent to titanium alloys. Despite this trend, the graph shows that by the time the powder lot had been fully consumed, powder oxygen content still had not exceeded the specification limit of 0.13%.

Additionally, a 95/95 confidence/reliability stability study was performed on the powder oxygen content data to get an indication of how many builds it would take to exceed the specification limit. The result for “shelf life” was 126 builds. In this context, shelf life is the time period in which, with 95% confidence, at least 95% of the data points are below the upper spec limit.

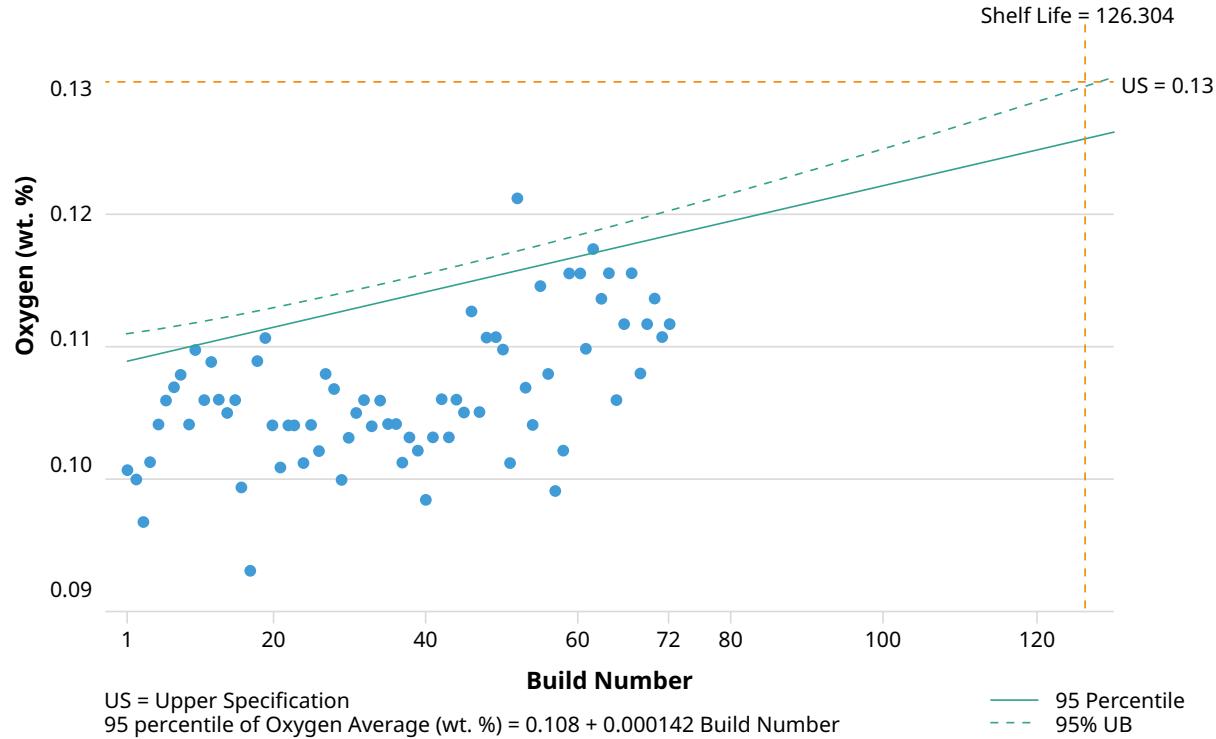


Figure 13. Shelf Life Plot

Figure 14 gives a time series plot of particle size distribution results from 20 builds throughout the powder lot. The standard deviations for each distribution are given in Table 1. Again, highly stable results are observed throughout the lifecycle of the powder lot.

Particle Size Distribution	Standard Deviation (μm)
D _v 10	0.393
D _v 50	0.404
D _v 90	0.616

Table 1. Variation in particle size distribution

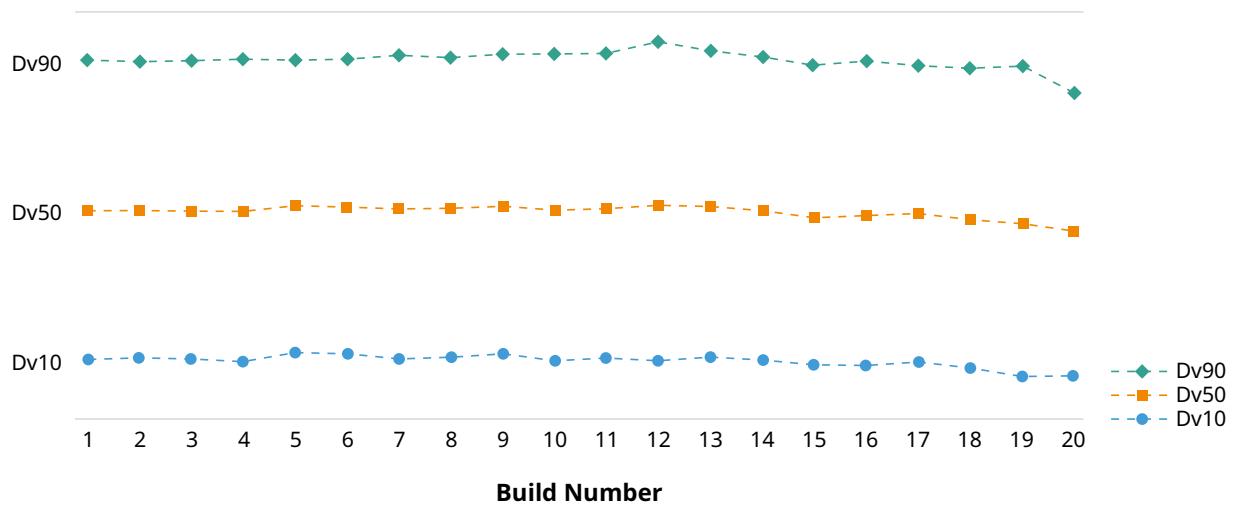


Figure 14. Time Series Plot of Particle Size Distribution

The stability observed within the powder monitoring data is a strong indication of overall process stability, especially when related to mechanical property CTQs. The graphs in Figure 15 and Figure 16 show powder oxygen content as it relates to mechanical property CTQ data for 24 DoE/validation builds.

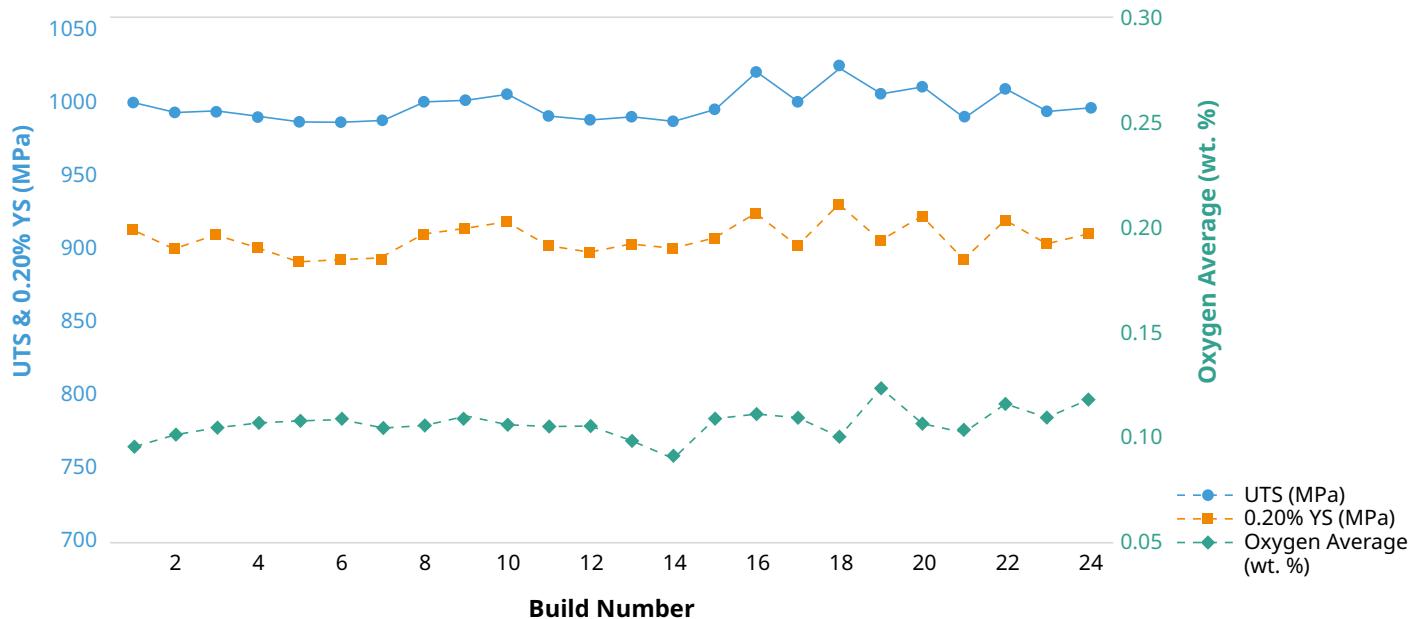


Figure 15. Time Series Plot of UTS, 0.20% YS, Oxygen Average (wt.%)

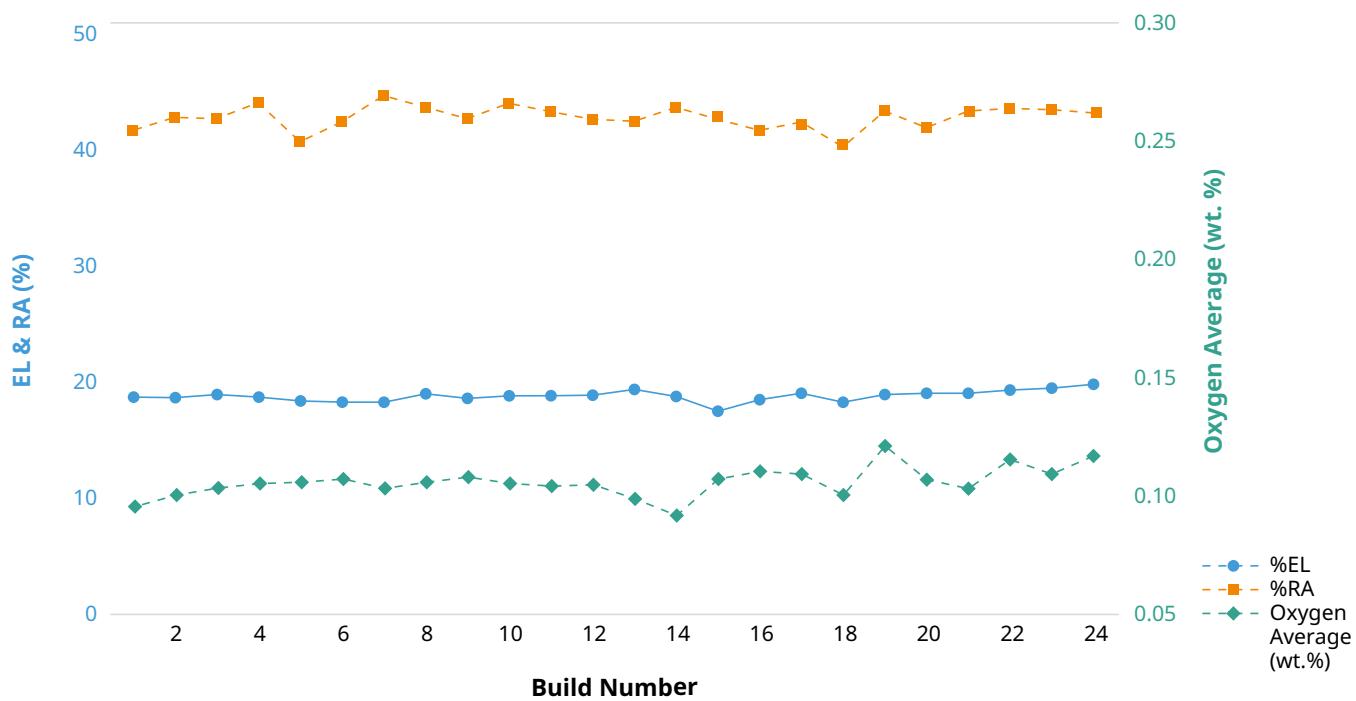


Figure 16. Time Series Plot of %EL, %RA, Oxygen Average (wt.%)

The stability data presented for powder oxygen content and particle size distribution shows that the process environment stability of DMP technology allows for reuse/recycleability that far exceeds practical requirements or limitations. There is no specific number of reuse cycles the user is limited to as long as the input material has been characterized and adequate monitoring plans have been implemented.

While this section discussed input material quality and stability as an indication of overall process performance, the next section provides a deeper dive into capability and performance of the outputs measured in terms of process capability metrics.

Process Capability: Outputs/CTQs

For further insight into how capable the DMP process is, process capability studies were performed on the CTQ data from the DoE. Though use of DoE data is atypical for capability analysis, these studies are presented with the implication that capability metrics from DoE data are worst-case due to the variation deliberately introduced to the process throughout the study.

Ppk and process sigma level capability indices are presented for all CTQs, as well as Pp for CTQs with bilateral tolerances. These metrics provide insight into both the stability, skew, and capability of the actual (overall) process.

MECHANICAL PROPERTIES

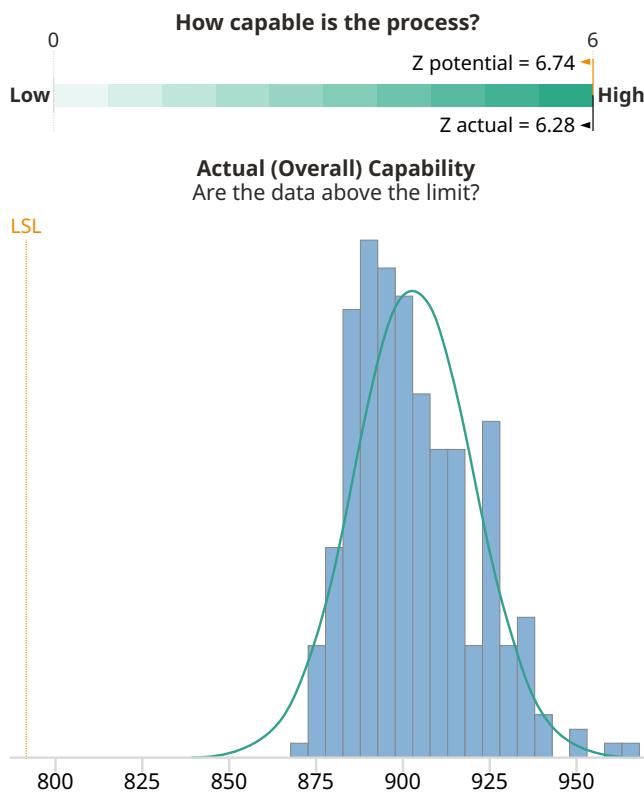
The overall process capability metrics given in Table 2 indicate the actual process (as observed within the DoE) is both stable and capable, above 6-sigma, for all mechanical properties. The high Ppk values and capability histograms given in Figures 17–20 show that the one-sided process spread is far less than the distance from the process mean to the specification limit.

Mechanical Property	Ppk	σ Level
0.2% Yield Strength	2.09	6.28
Ultimate Tensile Strength	5.01	> 10
Elongation	2.48	7.44
Reduction of Area	2.07	6.21

Table 2. Process capability metrics for static mechanical properties.

CAPABILITY ANALYSIS FOR 0.20% YS [MPa]

Summary Report



Customer Requirements	
Upper spec	*
Target	*
Lower spec	795

Process Characterization	
Mean	902.79
Standard deviation (overall)	17.160
Actual (overall) capability	
Pp	*
Ppk	2.09
Z.Bench	6.28
% Out of spec	0.00
PPM (DPMO)	0

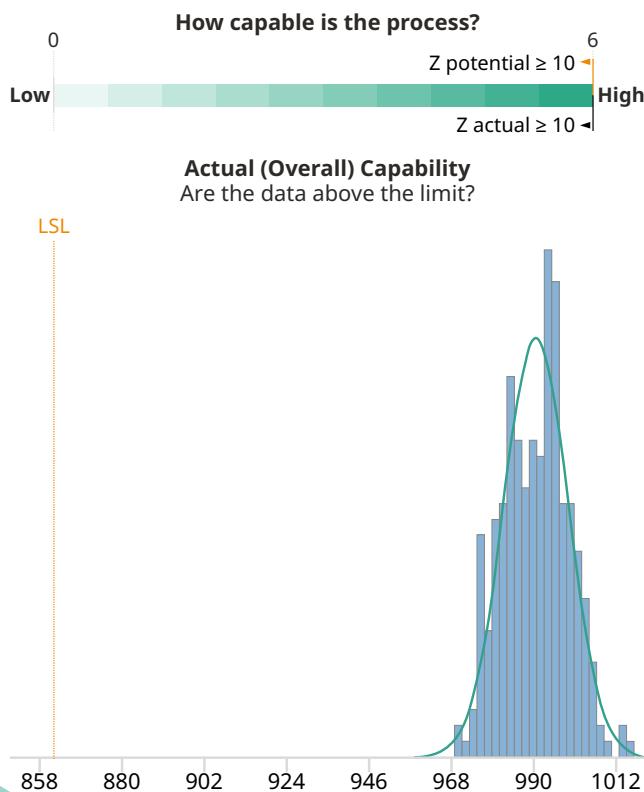
Comments

- The defect rate is 0.00%, which estimates the percentage of parts from the process that are outside the spec limits.
- Actual (overall) capability is what the customer experiences.
- Potential (within) capability is what could be achieved if process shifts and drifts were eliminated.

Figure 17. Mechanical Summary Report — Capability Analysis for 0.20% YS [MPa]

CAPABILITY ANALYSIS FOR UTS [MPa]

Summary Report



Customer Requirements	
Upper spec	*
Target	*
Lower spec	860

Process Characterization	
Mean	990.76
Standard deviation (overall)	8.7065
Actual (overall) capability	
Pp	*
Ppk	5.01
Z.Bench	15.02
% Out of spec	0.00
PPM (DPMO)	0

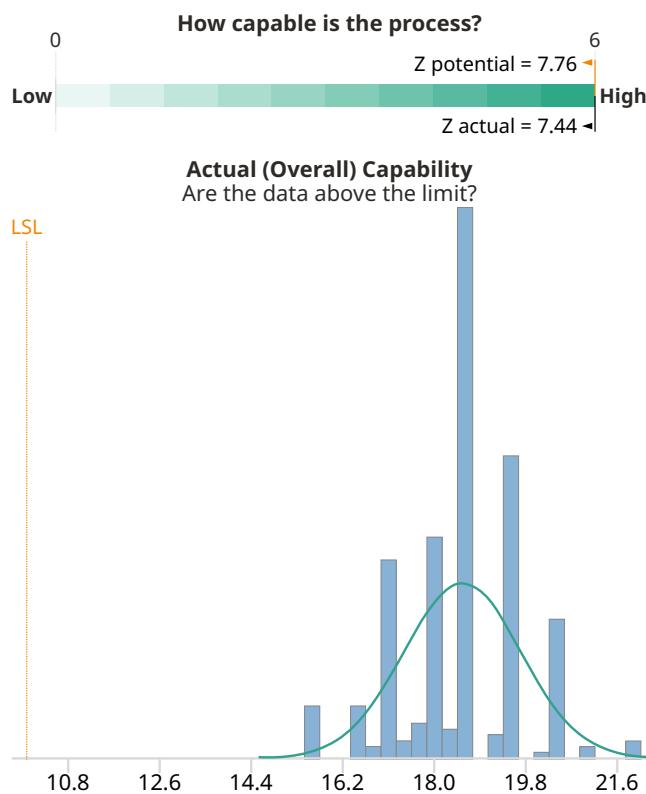
Comments

- The defect rate is 0.00%, which estimates the percentage of parts from the process that are outside the spec limits.
- Actual (overall) capability is what the customer experiences.
- Potential (within) capability is what could be achieved if process shifts and drifts were eliminated.

Figure 18. Mechanical Summary Report — Capability Analysis for UTS [MPa]

CAPABILITY ANALYSIS FOR %EL

Summary Report



Customer Requirements

Upper spec	*
Target	*
Lower spec	10

Process Characterization

Mean	18.551
Standard deviation (overall)	1.1498
Actual (overall) capability	
Pp	*
Ppk	2.48
Z.Bench	7.44
% Out of spec	0.00
PPM (DPMO)	0

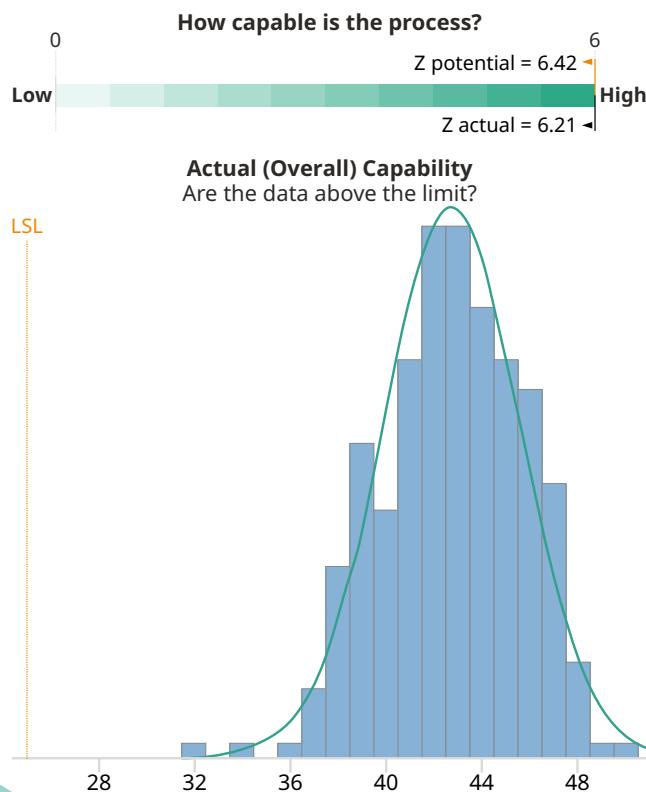
Comments

- The defect rate is 0.00%, which estimates the percentage of parts from the process that are outside the spec limits.
- Actual (overall) capability is what the customer experiences.
- Potential (within) capability is what could be achieved if process shifts and drifts were eliminated.

Figure 19. Mechanical Summary Report — Capability Analysis for %EL

CAPABILITY ANALYSIS FOR %RA

Summary Report



Customer Requirements

Upper spec	*
Target	*
Lower spec	25

Process Characterization

Mean	42.720
Standard deviation (overall)	2.8544
Actual (overall) capability	
Pp	*
Ppk	2.07
Z.Bench	6.21
% Out of spec	0.00
PPM (DPMO)	0

Comments

- The defect rate is 0.00%, which estimates the percentage of parts from the process that are outside the spec limits.
- Actual (overall) capability is what the customer experiences.
- Potential (within) capability is what could be achieved if process shifts and drifts were eliminated.

Figure 20. Mechanical Summary Report — Capability Analysis for %RA

CHEMICAL COMPOSITION

The overall process capability metrics given in Table 3 indicate the actual process (as observed within the DoE) is both stable and capable for all chemical constituents within solid test coupons, except for aluminum. When looking at the histogram and comparing the P_p to P_{pk} values for aluminum, the observation is that the process is skewed towards the high end of the specification limit. Aluminum is added as an alloying element to the material to add strength. The alloy is typically sourced at the upper end of the specification (5.5–6.5 wt%) to allow for optimal strength while also allowing for usage in higher energy processes like EB-LPF or DED (directed energy deposition) where due to the higher energy, aluminum can be vaporized yielding a final material that does not meet specification. There is no empirical evidence that aluminum can be picked up during the L-PBF process, thus even with feedstock at the high end of the specification the process is highly capable in a practical sense. Similarly, for oxygen wt.% within the coupons, the population is skewed towards the specification limit, which leads to lower capability in relation to the rest of the chemicals. This is because oxygen content directly correlates with static mechanical strength and thus a practical lower limit is used in the production material specification. However, for the purposes of this study the ASTM F3001, 3302 limits are used which is just an upper specification. Still, capability for oxygen is over 4 σ , which 3D Systems has found to be more than sufficient for the industry. The full capability analysis can be found in the appendix.

Chemical Constituent	P _p	P _{pk}	σ Level
Aluminum	2.63	0.47	1.41
Vanadium	4.11	3.86	> 10
Iron	N/A	4.38	> 10
Oxygen	N/A	1.38	4.13
Carbon	N/A	10.94	> 10
Nitrogen	N/A	5.83	> 10
Hydrogen	N/A	4.73	> 10

Note: Data for Yttrium, Other Elements (each), and Other Elements (total) were below the detection limit and therefore not included in the capability analyses.

Table 3. Process capability metrics for chemical composition.

CAPABILITY ANALYSIS FOR ALUMINUM

Summary Report

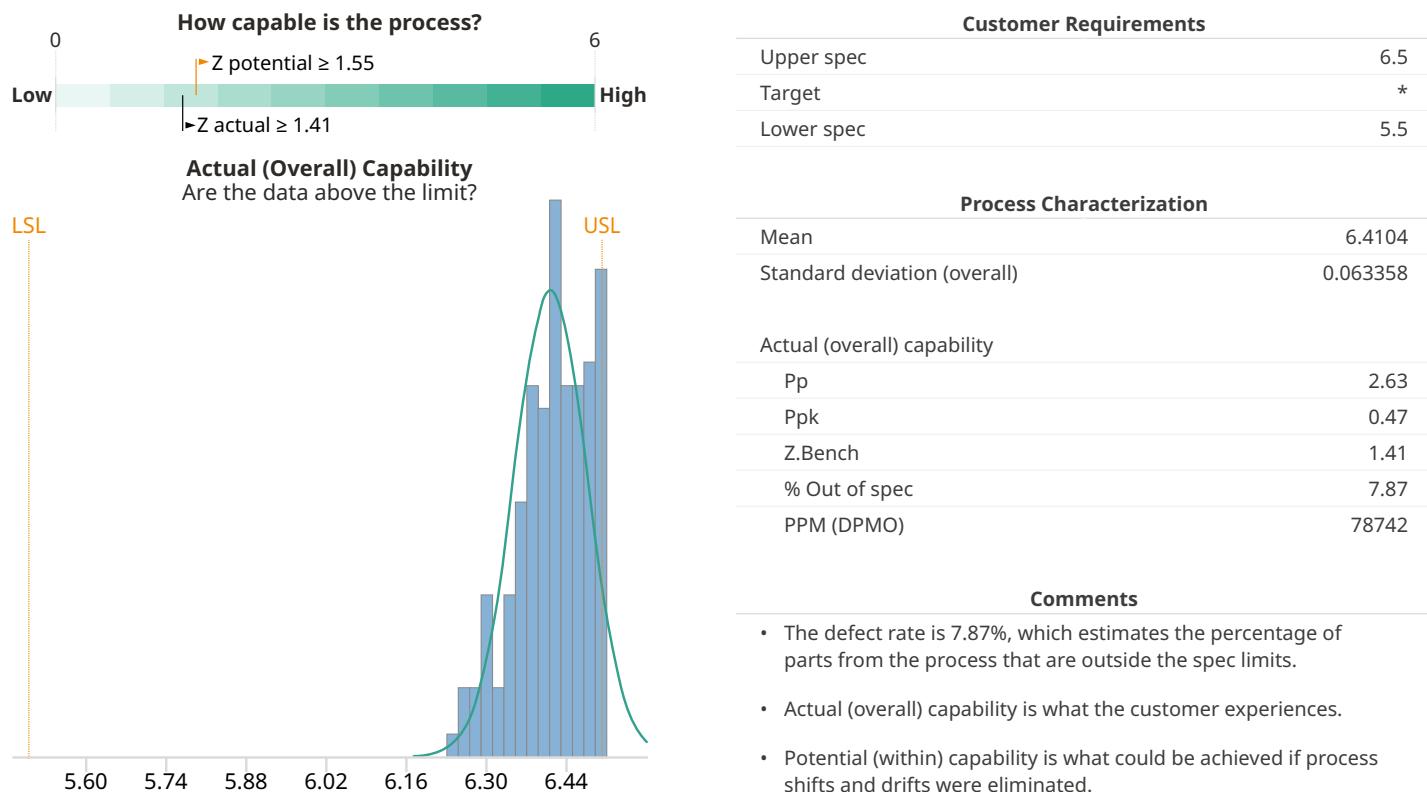


Figure 21. Chemical Summary Report — Capability Analysis for Aluminum

Conclusion

The process capability of both the input feedstock and resultant materials properties shows a highly robust process that meets or exceeds conventional manufacturing methods. The capability study performed within an interval around nominal process settings, along with the overall process characterization and more than a decade of critical application manufacturing allows for improved speed to market as new applications are developed using DMP technology. By removing as much risk from the process as possible, the nexus of application innovation and process reliability becomes clear, and with this confidence, innovation can move more freely and swiftly.

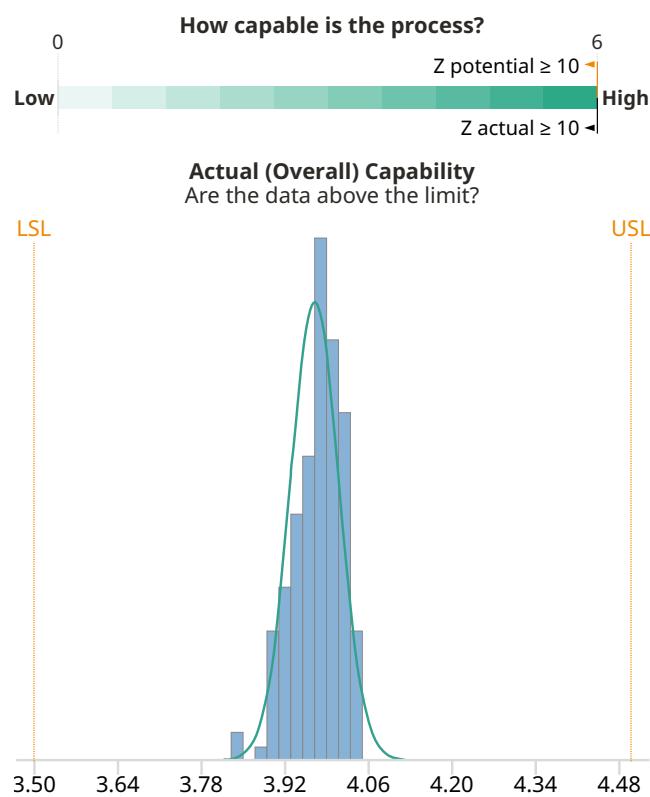
References

1. Beckers, A. *et al.* On the advances to obtain excellent and repeatable mechanical properties and build quality of LaserForm® Ti gr23 (A) across whole build platform. in 321, 03015 (14th World Conference on Titanium, 2019).
2. Cutolo, A., Elangswaran, C., Formanoir, C. De, Muralidharan, G. K. & Hooreweder, B. Van. Effect of Heat Treatments on Fatigue Properties of Ti – 6Al – 4V and 316L Produced by Laser Powder Bed Fusion in As-Built Surface Condition. 395–405 doi:10.1007/978-3-030-05861-6
3. Yan, M. *et al.* Review of effect of oxygen on room temperature ductility of titanium and titanium alloys. *Powder Metall.* 57, 251–257 (2014).
4. Oh, J. M. *et al.* Oxygen effects on the mechanical properties and lattice strain of Ti and Ti-6Al-4V. *Met. Mater. Int.* 17, 733–736 (2011).
5. Westphalen, D., Roth, K. W. & Brodrick, J. Guidance for industry - Process validation: General principles and practices. *Food and Drugs Administration (FDA)* 45, (2011).

Appendix

CAPABILITY ANALYSIS FOR VANADIUM

Summary Report



Customer Requirements	
Upper spec	4.5
Target	*
Lower spec	3.5

Process Characterization	
Mean	3.9698
Standard deviation (overall)	0.040546
Actual (overall) capability	
Pp	4.11
Ppk	3.86
Z.Bench	*
% Out of spec	0.00
PPM (DPMO)	0

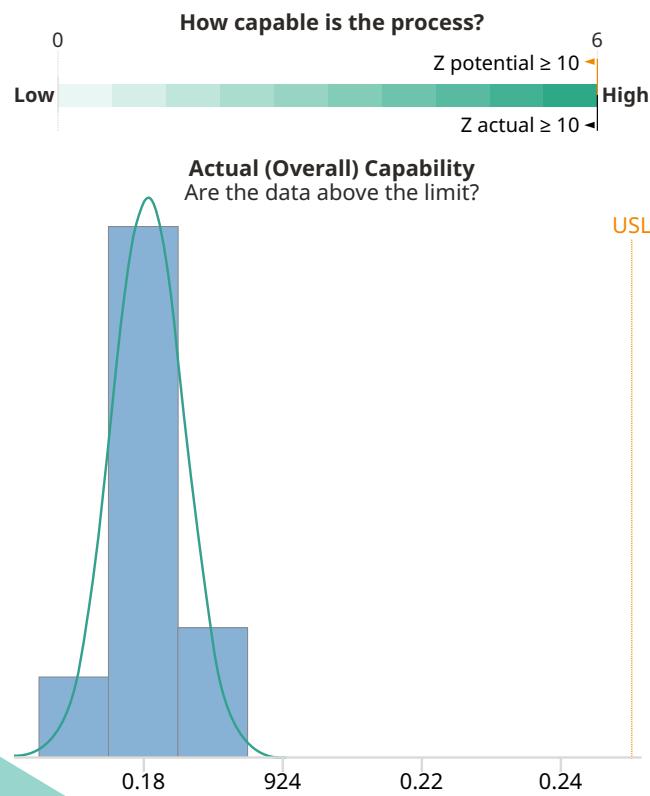
Comments

- The defect rate is 0.00%, which estimates the percentage of parts from the process that are outside the spec limits.
- Actual (overall) capability is what the customer experiences.
- Potential (within) capability is what could be achieved if process shifts and drifts were eliminated.

Figure 22. Chemical Summary Report — Capability Analysis for Vanadium

CAPABILITY ANALYSIS FOR IRON

Summary Report



Customer Requirements	
Upper spec	0.25
Target	*
Lower spec	*

Process Characterization	
Mean	0.18069
Standard deviation (overall)	0.0052751
Actual (overall) capability	
Pp	*
Ppk	4.38
Z.Bench	13.14
% Out of spec	0.00
PPM (DPMO)	0

Comments

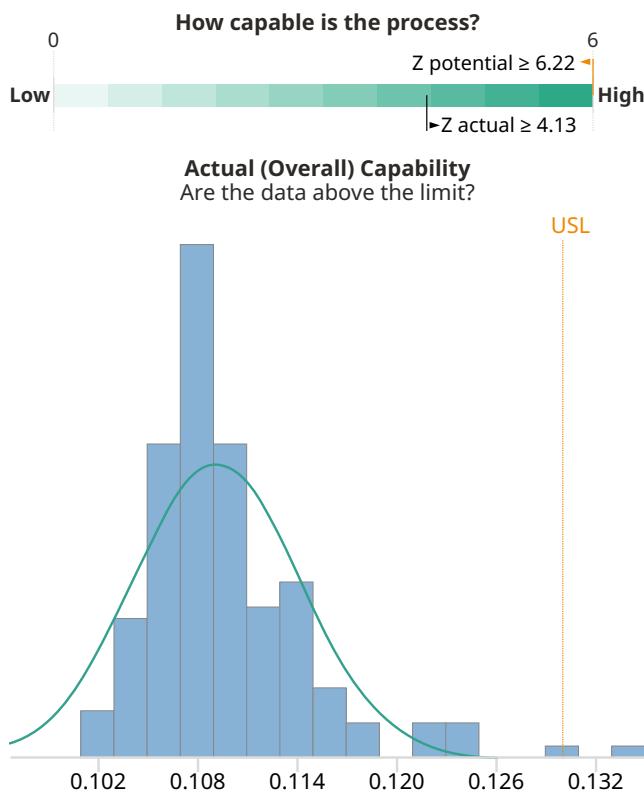
- The defect rate is 0.00%, which estimates the percentage of parts from the process that are outside the spec limits.
- Actual (overall) capability is what the customer experiences.
- Potential (within) capability is what could be achieved if process shifts and drifts were eliminated.

Figure 23. Chemical Summary Report — Capability Analysis for Iron

Appendix

CAPABILITY ANALYSIS FOR OXYGEN

Summary Report



Customer Requirements	
Upper spec	0.13
Target	*
Lower spec	*
Process Characterization	
Mean	0.10922
Standard deviation (overall)	0.0050336
Actual (overall) capability	*
Pp	*
Ppk	1.38
Z.Bench	4.13
% Out of spec	0.00
PPM (DPMO)	18

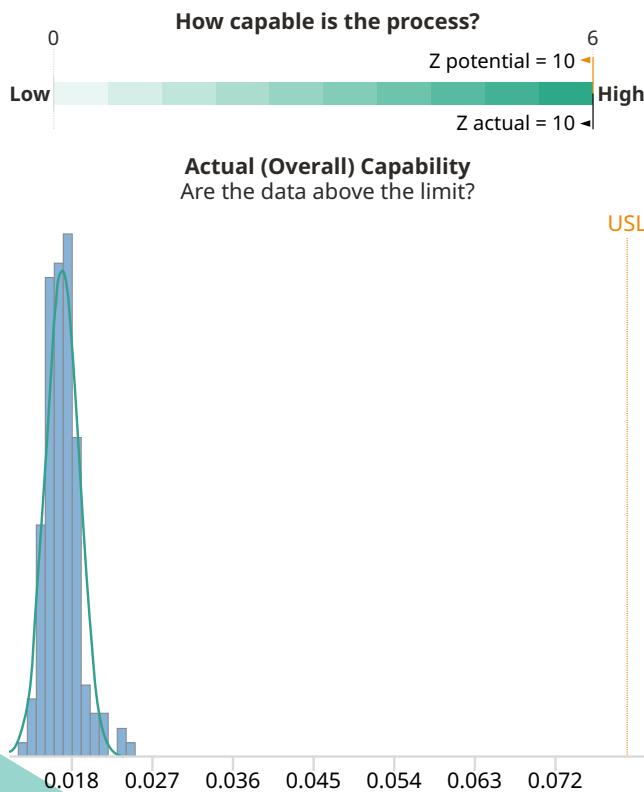
Comments

- The defect rate is 0.00%, which estimates the percentage of parts from the process that are outside the spec limits.
- Actual (overall) capability is what the customer experiences.
- Potential (within) capability is what could be achieved if process shifts and drifts were eliminated.

Figure 24. Chemical Summary Report — Capability Analysis for Oxygen

CAPABILITY ANALYSIS FOR CARBON

Summary Report



Customer Requirements		
Upper spec		0.08
Target		*
Lower spec		*
Process Characterization		
Mean		0.017394
Standard deviation (overall)		0.0019069
Actual (overall) capability		
Pp		*
Ppk		10.94
Z.Bench		32.83
% Out of spec		0.00
PPM (DPMO)		0

Comments

- The defect rate is 0.00%, which estimates the percentage of parts from the process that are outside the spec limits.
- Actual (overall) capability is what the customer experiences.
- Potential (within) capability is what could be achieved if process shifts and drifts were eliminated.

Figure 25. Chemical Summary Report — Capability Analysis for Carbon

Appendix

CAPABILITY ANALYSIS FOR NITROGEN

Summary Report

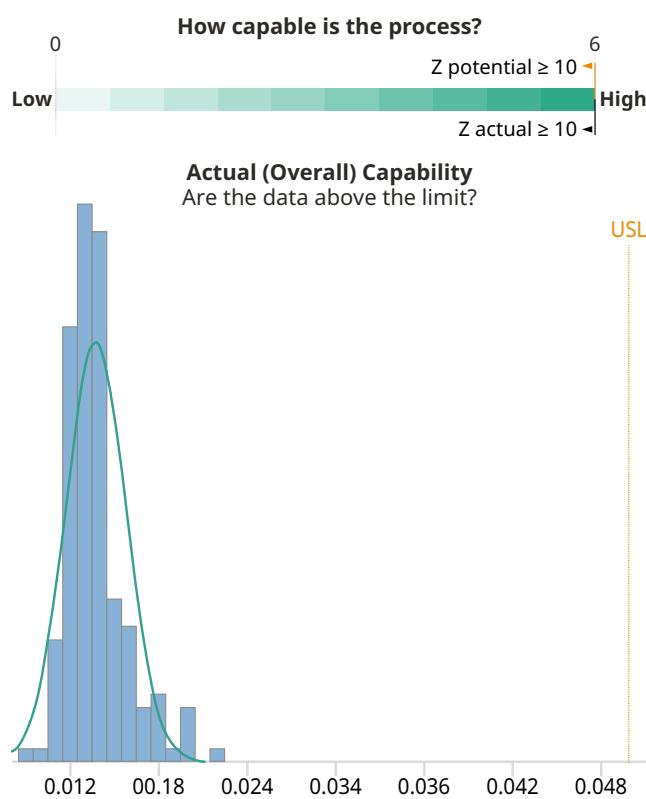


Figure 26. Chemical Summary Report — Capability Analysis for Nitrogen

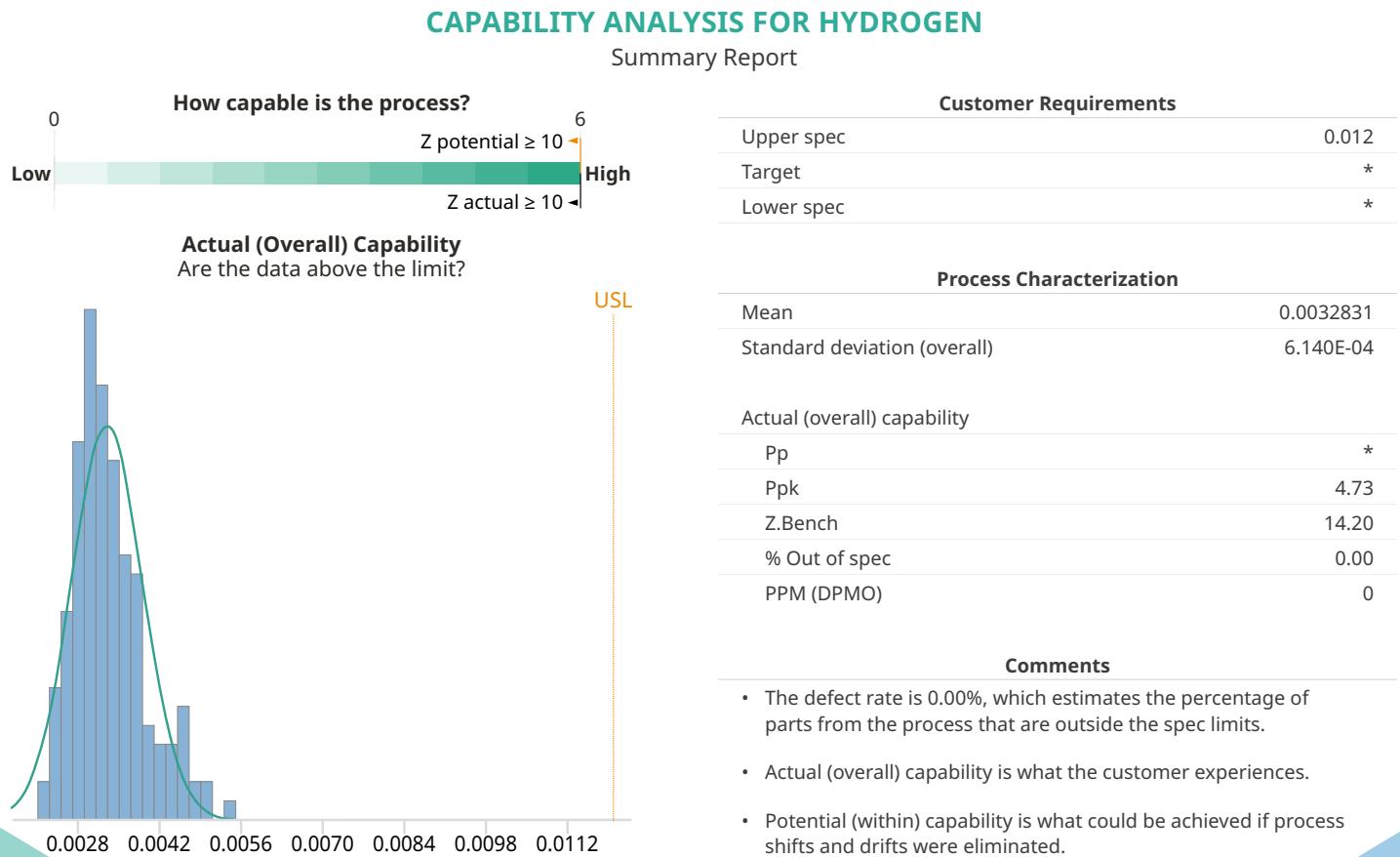


Figure 27. Chemical Summary Report — Capability Analysis for Hydrogen

What's Next? Interested in learning more about Titanium Process Capability?

Talk to an expert about which materials
and printers would work for you

GET IN TOUCH

3D Systems Corporation
333 Three D Systems Circle
Rock Hill, SC 29730
www.3dsystems.com

Warranty/Disclaimer: The performance characteristics of these products may vary according to product application, operating conditions, or with end use. 3D Systems makes no warranties of any type, express or implied, including, but not limited to, the warranties of merchantability or fitness for a particular use. Note: Not all products and materials are available in all countries – please consult your local sales representative for availability.

© 2021 by 3D Systems, Inc. All rights reserved. Specifications subject to change without notice.
3D Systems, the 3D Systems logo, ProX, and 3DXpert are registered trademarks of 3D Systems, Inc.