

# Hands-on quality



## Setting standards for process and material development and qualification in AM

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### Summary

Industrial 3D printing is becoming an accepted manufacturing method for an increasing number of parts in aerospace applications. The freedom of design offered by additive manufacturing enables the aerospace industry to solve the engineering challenges posed by future aircraft. There are strict requirements when it comes to qualifying and certifying parts, materials and processes. However, the requirements have not yet been fully standardized for additive manufacturing. We must distinguish between two certification processes: the Quality Management certification process followed by the company, and the reliability of the underlying additive manufacturing technology itself. This article will focus on technological reliability, an aspect which is important for establishing trust in the technology. We will illustrate how material and process development is performed at EOS, setting standards for quality assurance in additive manufacturing.

### This whitepaper gives answers to:

- What is required for quality in AM?
- How does EOS conduct quality assurance in product and process development?
- What could be useful standards in quality assurance?

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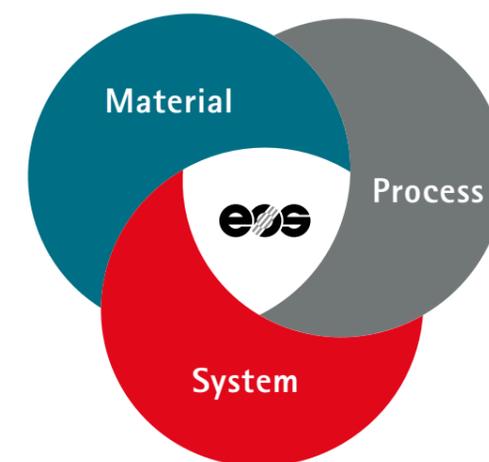
#### Customization

## The EOS Quality Triangle Determining the properties and quality of parts

The comprehensive quality assurance concept implemented by EOS ensures that products are manufactured at reproducibly high levels of quality. EOS uses an approach that is unique in the AM industry, taking each of the three central technical elements of the production process into account: the system, the material and the process – together simply described as the Quality Triangle. At EOS, properly balancing this triangle is the primary objective of every action undertaken during material and process development. The three technical components of the triangle are always interlinked; the relationships between them are the key to achieving reliable and repeatable quality and determine the properties of each part.

The first essential pillar of consistently high quality in manufactured products is the 3D printing system on which they are manufactured. A wide range of quality-assurance measures are implemented to ensure reliable compliance with production standards. An acceptance procedure (FAT – Factory Acceptance Test) is performed on every system before shipping to customers. As part of this FAT, a set of predefined reference objects are produced and tested against every relevant criterion (e.g. mechanical properties, porosity, surface finish). A second, complementary acceptance procedure (IQ – Installation Qualification) is performed on-site with the customer after installation.

Another pillar of quality is provided by certification in accordance with ISO 9001 (held by EOS since 1998) for the development & production and sale & service of laser sintering systems and metal materials and processes. Furthermore, the design, manufacture and sales of any metal materials and processes associated with EOS metal systems intended for medical device applications is conducted in accordance with ISO 13485.



# Standard Operating Procedures for Material and Process Development

Product development processes at EOS are performed in a systematic and standardized manner.

Good development and quality assurance provides consistent (statistical) data about new material and processes, from the development phase onward throughout the product life cycle.

EOS has established two internal standard procedures for Material and Process Development which are designed to produce validated and high quality products. Once the material products are released to the market, an extensive array of quality control procedures ensures that the composition of every batch of material is uniform. The processes are tested in series to secure consistency in production quality and component characteristics.

The technical development process of a material or a process begins with a feasibility study, followed by the concept and development phases. At the end of technical development, verification and validation are conducted to ensure that the design and development outputs meet the initial objectives. The scope of this verification is planned separately for each material and process product to ensure that it remains relevant for each individual alloy. The validation also unfolds according to a predefined plan to guarantee that the specified requirements are consistently fulfilled.

## Technical feasibility and development

At EOS, a metal product development project might require the development of new hardware or software and/or new processes and materials. The careful development and qualification (process verification and validation) procedure creates a robust process that, together with the qualification processes for powders and systems, guarantees that each part is endowed with the desired properties. Development requires collaboration between process and material development experts, using state-of-the-art analytical tools and test equipment. The four tools included in the EOSTATE Monitoring Suite are also elements in developing new materials and/or processes (Figure 1).

Development begins with the creation of the Specifications, followed by the concept and development phases, qualifications (known as the verification and validation phases) and finally product release. Early user feedback collected from the pilot phase is another key element that helps to ensure that the requirements are fulfilled (Figure 2).

Materials are developed by collaboration between the R&D department, the internal EOS laboratory and powder manufacturers. The market requirements documented in the User Requirement Specification (URS) are reviewed in order to identify the most important properties that this powder should possess, which naturally depend on the target application/market, part

properties and materials requirements. Any applicable regulatory requirements are also taken into account at this point, and any material-related risks are identified and mitigated. One of the tasks of the project is to establish a Failure Mode and Effect Analysis (FMEA) for the product, which is then updated throughout the material and process development project.

Process development is also tailored to the market needs established by the User Requirement Specification. These requirements and specifications are the key inputs of the process concept. The key process input variables and process outputs are determined during this phase. The process variables, for example those which affect the bulk properties, surface quality and accuracy, are identified, developed and tested as part of feasibility testing. The specifications are then documented in the Functional Specification (FS).

Once feasibility has been confirmed, the actual development is initiated. During this phase, multiple development iterations are conducted in order to ensure that specifications in the Functional Specification are met while implementing any applicable international standards. The user requirements and other factors, such as standardization, are taken into account. The development phase/design experiments are continued until the detailed Functional Specification requirements have been fulfilled.

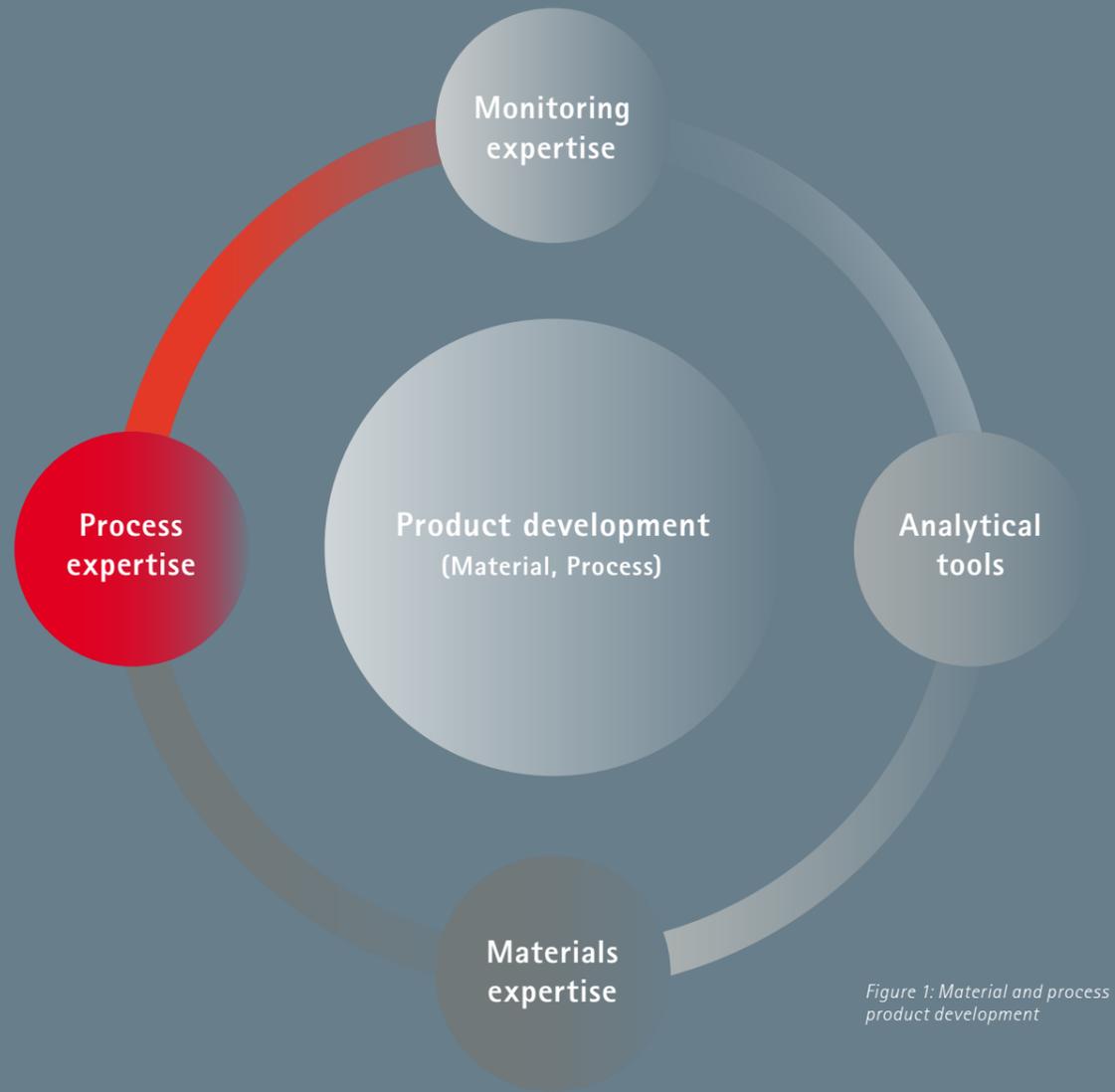


Figure 1: Material and process product development

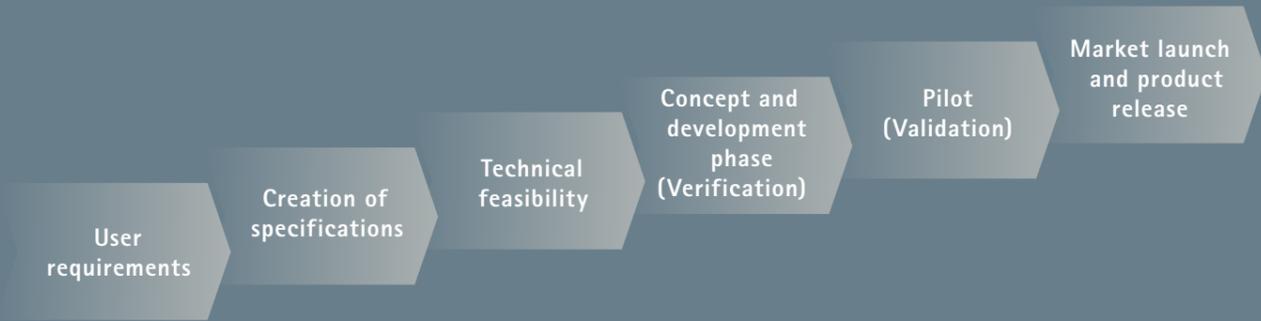


Figure 2: Visualization of EOS Product Development (MEP) standard procedures

## Verification

Verification is conducted at the end of the development phase. The aim is to generate documented proof that the requirements have been fulfilled. The output of the verification phase provides objective evidence that the requirements specified in the Functional Specification are satisfied. Usually, the verification phase also includes characterization testing, which depends on the intended uses of the final material and process products. For example, fatigue, creep, corrosion resistance or biocompatibility can be characterized.

Process and material verification apply a specific test matrix defined in the verification plan to evaluate the requirements specified by the URS. The aim is to test a large subset of the critical material-specific properties. Verification is conducted individually for each type of powder, and features predefined measurements (such as particle size distribution and chemistry). Limits are based on international material standards and/or internal specifications that were carefully developed during the concept phase.

Process verification is conducted for several of the properties defined in the Functional Specification. These properties can differ from material to material. However, in every case, the microstructure, defect densities and bulk density of the solid samples are verified, and the static tensile properties are measured. Other verified and characterized properties can be defined as a function of the application/intended use of the product.

The figure 3 below shows how the surface quality of the processed parts can be illustrated, measured and even compared to the original 3D model.

## Validation

The verification phase is followed by the validation phase, whose objective is to demonstrate that the process and material consistently fulfill the requirements defined by the specifications. Evidence is collected from multiple batches of powder and multiple

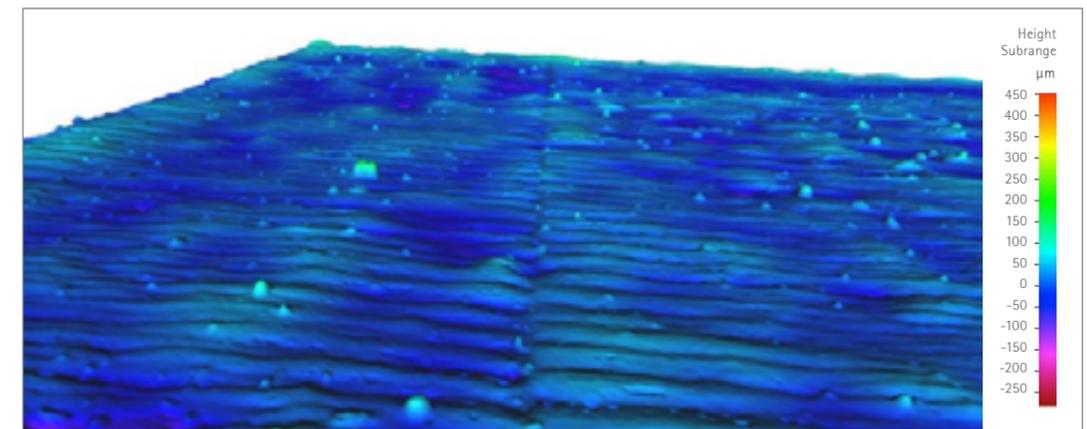


Figure 3: 3D Optical image from a sample surface

machines. The test matrix used for validation depends on whether the powder and the metal machine are released. For example, in the case of a known system platform, e.g. EOS M 290, and a new material, the test matrix would consist of two machines and three or four powder batches. The data are collected, and statistical analysis is performed. Examples of the properties considered for validation can include:

- Chemical composition (powder and processed parts) with the relevant test method for each element (combustion, fusion, ICP/OES)
- Static tensile properties (Yield and Tensile Strength, Elongation at Break) with the relevant standards (testing standards and material standards)
- Macroscopic density (measured)
- Porosity (optical microscopy with image analysis software)

If the project scope stipulates additional properties, they are also included in the validation phase. This allows both the potential capability and the actual capability of the process to be evaluated, anticipating the quality assurance phase after product development is complete. The potential capability describes the capability that could theoretically be achieved by the process if shifts were successfully eliminated; the actual capability describes the current results. The process capability report of EOS Titanium Ti64 is presented below as an example (Figure 4).

Naturally, the potential capability of the process is always higher than the actual capability. The histogram shown below presents an example case; different manufacturing conditions (machines, powders) and testing conditions (methods, test laboratory, sample size) will influence the appearance of the histogram. The capability analysis is based on the assumption that the data are normally distributed and the process is stable.

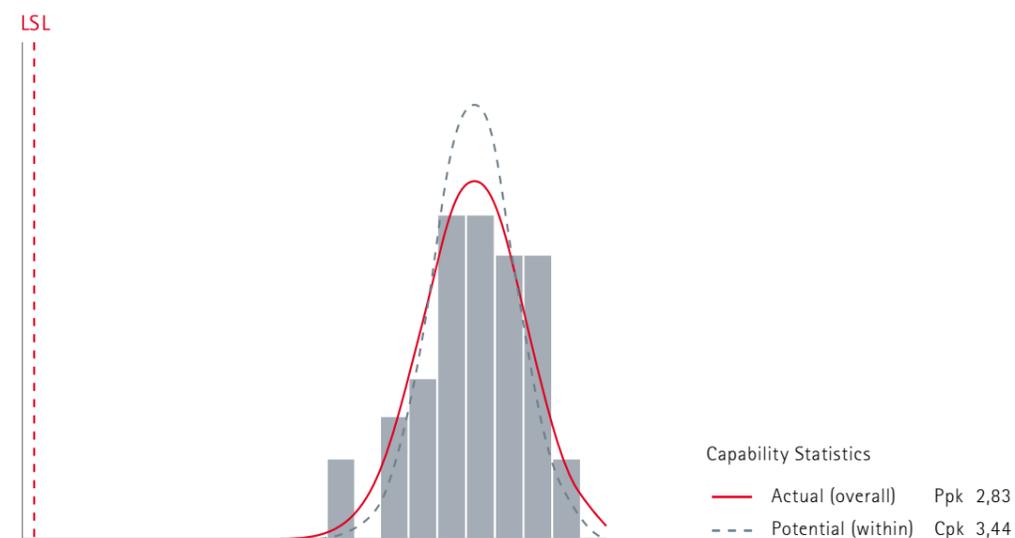


Figure 4: Process capability (based on elongation values) of EOS Titanium Ti64

## What does quality require?

### Process development

The first statistical proof of material and process quality is established during the product development phase. Process and material development includes a rigorous qualification phase (verification and validation), during which key information about the processes and materials is gathered. The effects of different machines and powder lots on the properties of the processed parts are validated. The test matrix depends on the release level of the process and the material products. The properties of built parts can be tested under both as-manufactured and heat-treated conditions (static tensile properties for example).

The process aims to achieve constant quality across different machines and powder batches. The data collected during the verification and validation phases are analyzed and documented. These data are then used to create the material data sheets (MDS).

### Quality assurance for EOS powders

The quality assurance procedure for released powders is part of the acceptance procedure of each new raw material batch received for production. Quality assurance includes a control of the powder chemistry and particle size distribution, but the properties of built parts are also investigated. Test samples are built using dedicated and qualified machines using a qualified process. Data are collected from these properties, e.g. the static tensile properties and density of built parts and their chemistry. These quality assurance data are collected and archived together with samples. This carefully conducted quality documentation process ensures that EOS procedures are fully traceable, even back to the powder suppliers.

The acceptance procedure for releasing new powder batches into production is performed by the quality department by comparing against predefined specifications. If the results are within these limits, then the batch is released, and the results are documented in the Mill Test Certificate (MTC).

Since quality control is performed for every batch of every powder, extensive archives of historical data and statistics are compiled over time. For example, specific parameters of the powder chemistry results can be tracked over time, as shown in Figure 5.

Figure 5: I-MR chart of Oxygen in EOS Titanium Ti64 powder. Summary report

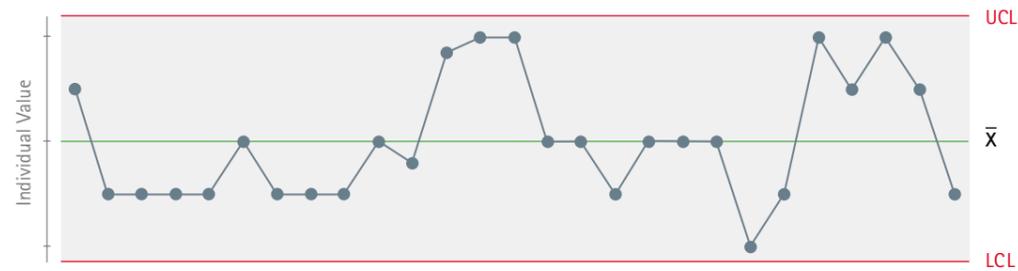


Figure 5 shows the long-term data on the oxygen content of EOS Titanium Ti64 powder. The chart shows the overall trend of the results, and makes it easy to investigate the stability of the process. The upper and lower control limits (UCL, LCL) are calculated by using the average-moving-range method to estimate the standard deviation. The UCL and LCL seen in the graph are three times the within standard deviation. The within standard deviation seen in the

graph changes when the sample size changes. The overall standard deviation for the entire population would be slightly higher than the value shown in the I-MR chart. (Individuals chart and Moving Range chart)

Figure 6 presents quality assurance data for EOS NickelAlloy IN718 (yield strength) as an example from built part properties that are collected during the powder batch quality assurance.

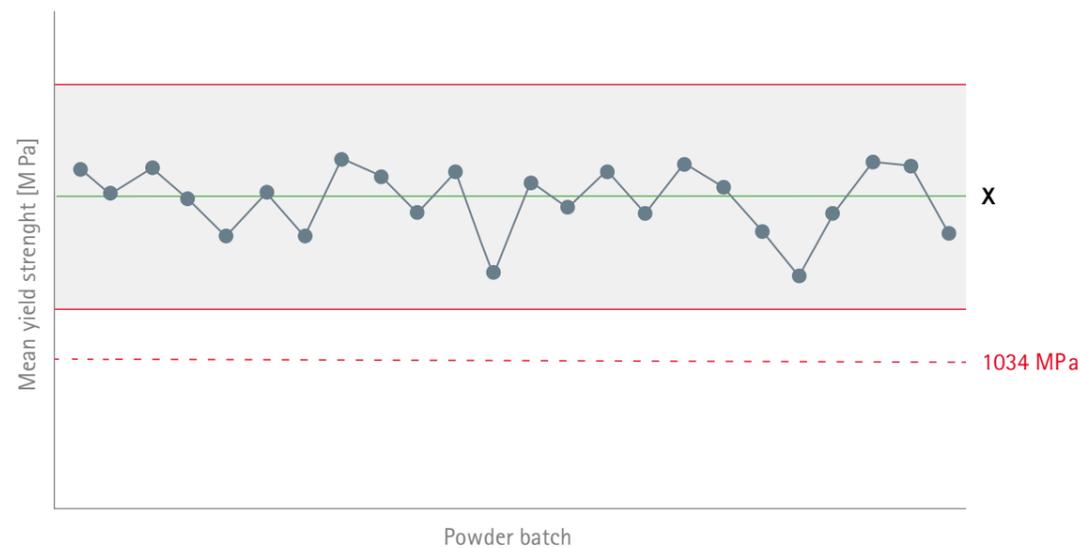


Figure 6: IN718 QA yield strength data with interval plot

Accredited and independent external laboratories are used to analyze test parts during the quality assurance and verification & validation phases. EOS also follows Good Manufacturing Practices (GMP) in the EOS laboratory. Measurement system analysis (MSA) is conducted for every piece of equipment used for quality assurance. MSA is also conducted for each measurement process to identify the extent and root causes of variation, as an integral part of the quality management system. Every system and piece of equipment is qualified, and good laboratory practices are implemented. The relevant standards for measurement systems and analyses are enforced, and full traceability is ensured by generating thorough documentation in accordance with the Quality Management System (QMS) procedures defined by ISO 9001.

### Powder supplier controls

Before process development can begin, the quality of the powders used by EOS must be guaranteed. The foundations of this guarantee are laid by establishing long-term contracts and building strong relationships with powder suppliers. Furthermore, suppliers are qualified, and their quality assurance procedures are monitored/audited. Documentation on the manufacturing process of powders is compulsory. Other obligations, such as regulatory responsibilities, liability insurance for the products, are also determined by contracts.

The purpose of the quality assurance agreement is to ensure and maintain the quality of powder products. The suppliers' responsibility is to manufacture and supply products that conform to all EOS specifications. In addition, the key processes are characterized in order to establish a common understanding of the key elements required for high quality. The manufacturing process is described in the Method of Manufacture document. This document also ensures that any changes that might affect the product are communicated to EOS. This improves the traceability and continuity of product properties. Additionally, regular feedback is shared with suppliers. This feedback might for example relate to product costs, quality, technology and supply. Supplier audits are performed regularly.

### Mill Test Certificates

The inspection certificates for powder batches (Mill Test Certificates) are created according to "Metallic products - Types of inspection documents" EN 10204, Type 3.1. Their purpose is to guarantee that all the specified requirements, applicable standards and regulations are fulfilled after the completion of testing. The Mill Test Certificate states the trade name and lot number of the powder, as well as various other information depending on the type of powder. The main powder quality characteristics, particle size analysis and chemistry, are reported for every lot, regardless of powder type. Additional information about the properties of the processed material can also be provided (e.g. bulk density, tensile properties).

# What capabilities and knowledge do we offer?

Other than product development projects that lead to commercial products, the R&D department conducts specific studies and pre-development projects. For example, a study was conducted to investigate the aging of EOS NickelAlloy HX powder.

The aging of metal powders over reuse cycles is of great interest as one of the factors which affect the costs of additive manufacturing. The results of the EOS NickelAlloy HX aging study, conducted in collaboration with University of Duisburg-Essen, indicate that the EOS NickelAlloy HX powder can be used many times without refreshing. The main factor that influences powder aging is the oxygen content, which rises steadily from cycle to cycle. If the powder is refreshed regularly, the material can be reused

for a longer period, but the effect of refreshing was beyond the scope of this particular study. Other than the oxygen content, the chemistry and particle size distribution (PSD) did not significantly differ from the virgin powder used at the beginning of the study. The PSD tended to become slightly narrower as the fine and large particles are lost during reuse cycles. Despite the changes in PSD, morphology and oxygen content, other properties such as the static mechanical properties and impact strength remained constant for the first 25 reuse cycles (more than 900 machine hours), and the hardness values remained at similar levels. The surface quality remained stable, and the defect rate, measured from the cross-cuts of solid samples, did not change.

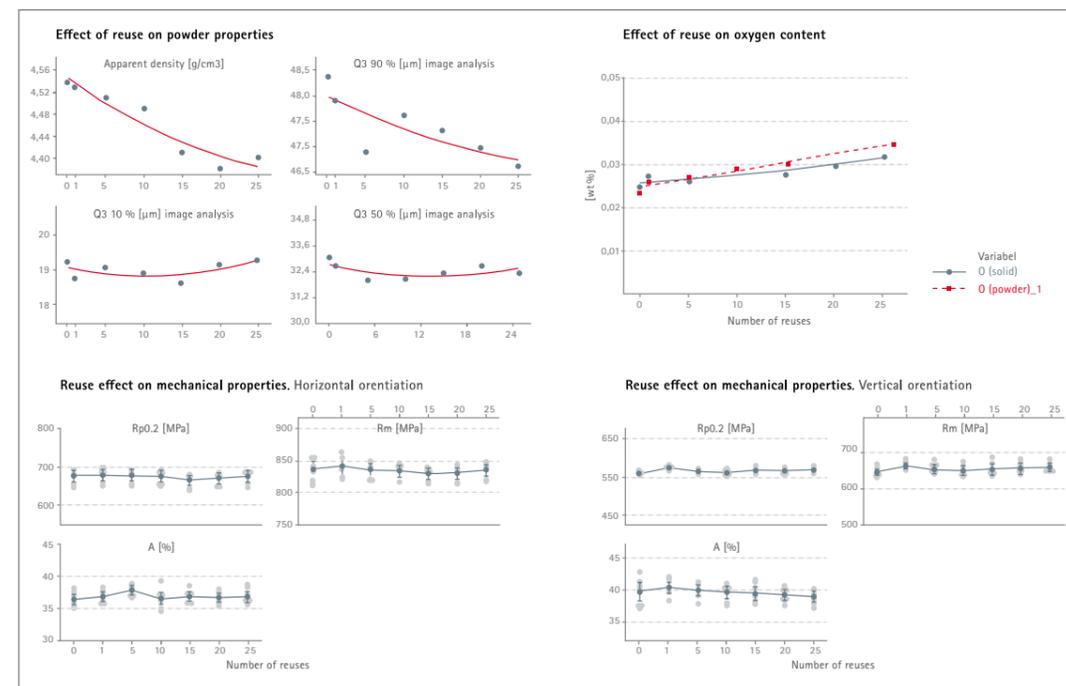


Figure 7: Top left: changes in powder properties. Top right: effect of reuse cycles on oxygen content. Down: effect of reuse cycles on static mechanical properties.

The internal range of capabilities of EOS includes systems for both powder and solid analysis. These tools can be used for both research and development and quality assurance purposes.

The quality expertise and knowledge offered by EOS draws from more than 25 years of experience with AM metallurgy. EOS has the longest-standing and best-quality experience in the field, and has played a pioneering role in the Aerospace field. The EOS development teams feature a number of dedicated material and process experts.

## Internal range of capabilities of EOS

- Particle Size Distribution (PSD)
- Particle morphology
- Powder rheological properties
- Powder densities
- Powder moisture/water content
- Bulk/solid density
- Thermal analysis (dilatometry)
- Optical microscopy
- 3D optical analysis (shape, roughness)
- Hardness

## Customization

Customized development of materials and/or processes is intended for customers with specific needs or applications. The objective is to develop new products on demand for customers who require solutions from outside of EOS's current portfolio. This might for example involve new materials, new parameter sets or new post-processes, e.g. heat treatments. The focus is always on the specific application requirements of each customer: the material and/or process is optimized according to the customer's requirements. For example, a customer who wishes to prioritize productivity could ask for a process to be designed specifically to optimize this target parameter. Alternatively, if surface quality is the main process output, the process can be adapted accordingly.

The maturity of each solution is fitted to the customer's requirements and can vary from a proven concept all the way to a fully validated product. The customer is completely free to decide the depth of development work and the final maturity level of the deliverables.

Customized products can also help to close gaps between customer requirements and existing EOS products. For example, additional data on products currently featured in the EOS portfolio can be provided, or customer-specific documentation or post-processing can be performed.

Customer-specific Material Mill Test Certificates or test reports can also be requested.

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