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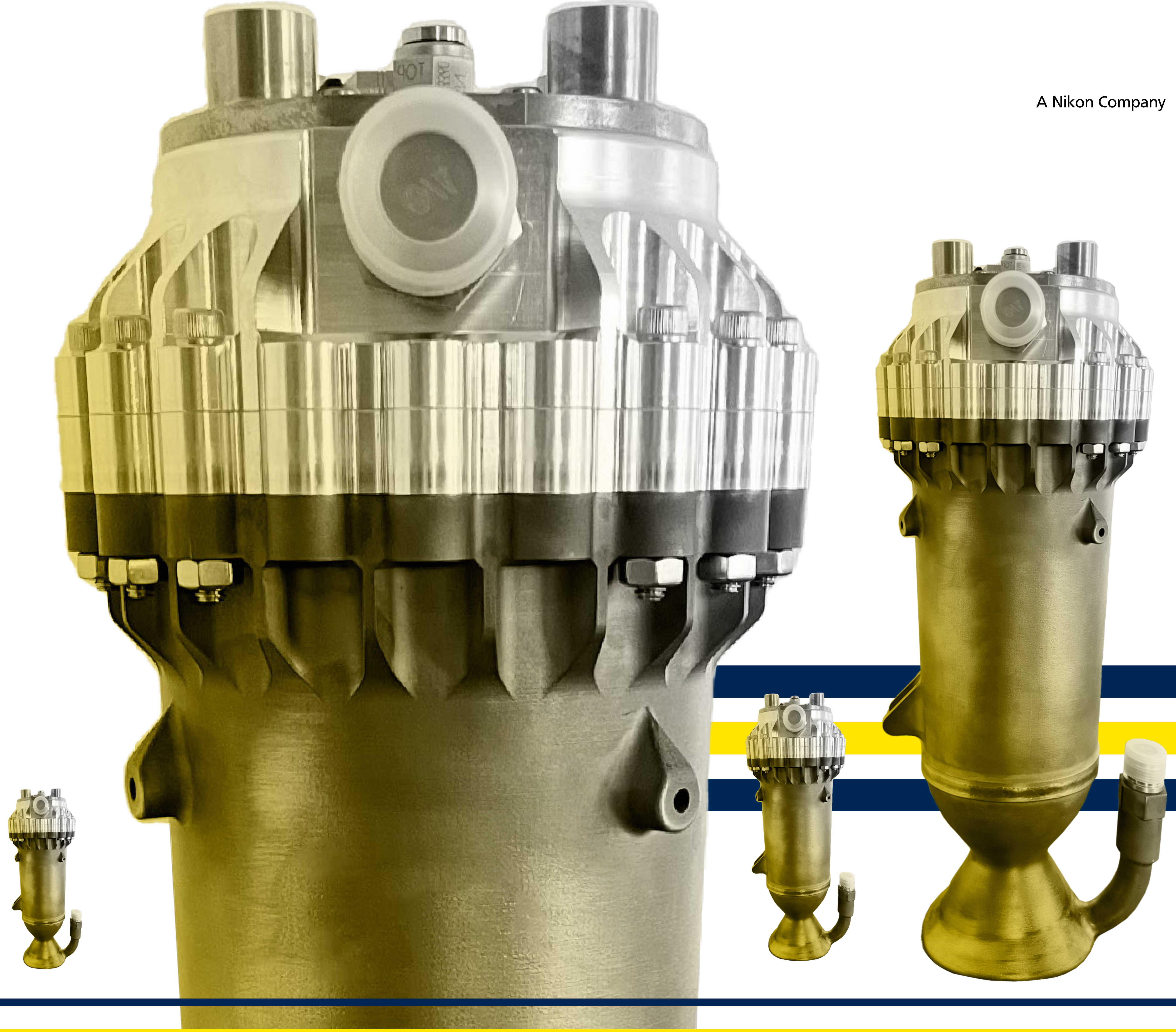
ADDITIVE MANUFACTURING, HOT ISOSTATIC PRESSING, HEAT TREATMENT, AND INSPECTION OF AN

INCONEL 718 ROCKET ENGINE



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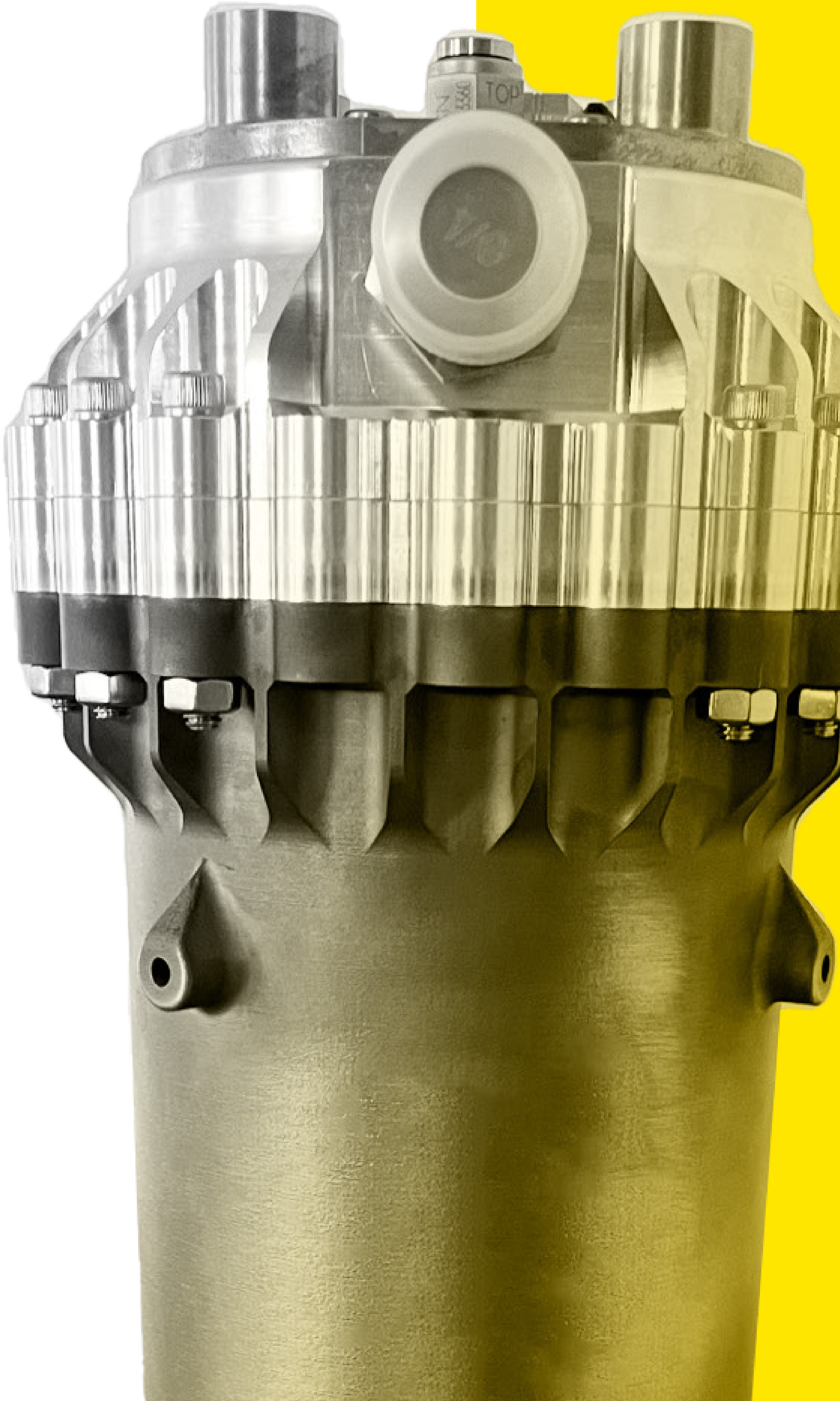
EXECUTIVE SUMMARY

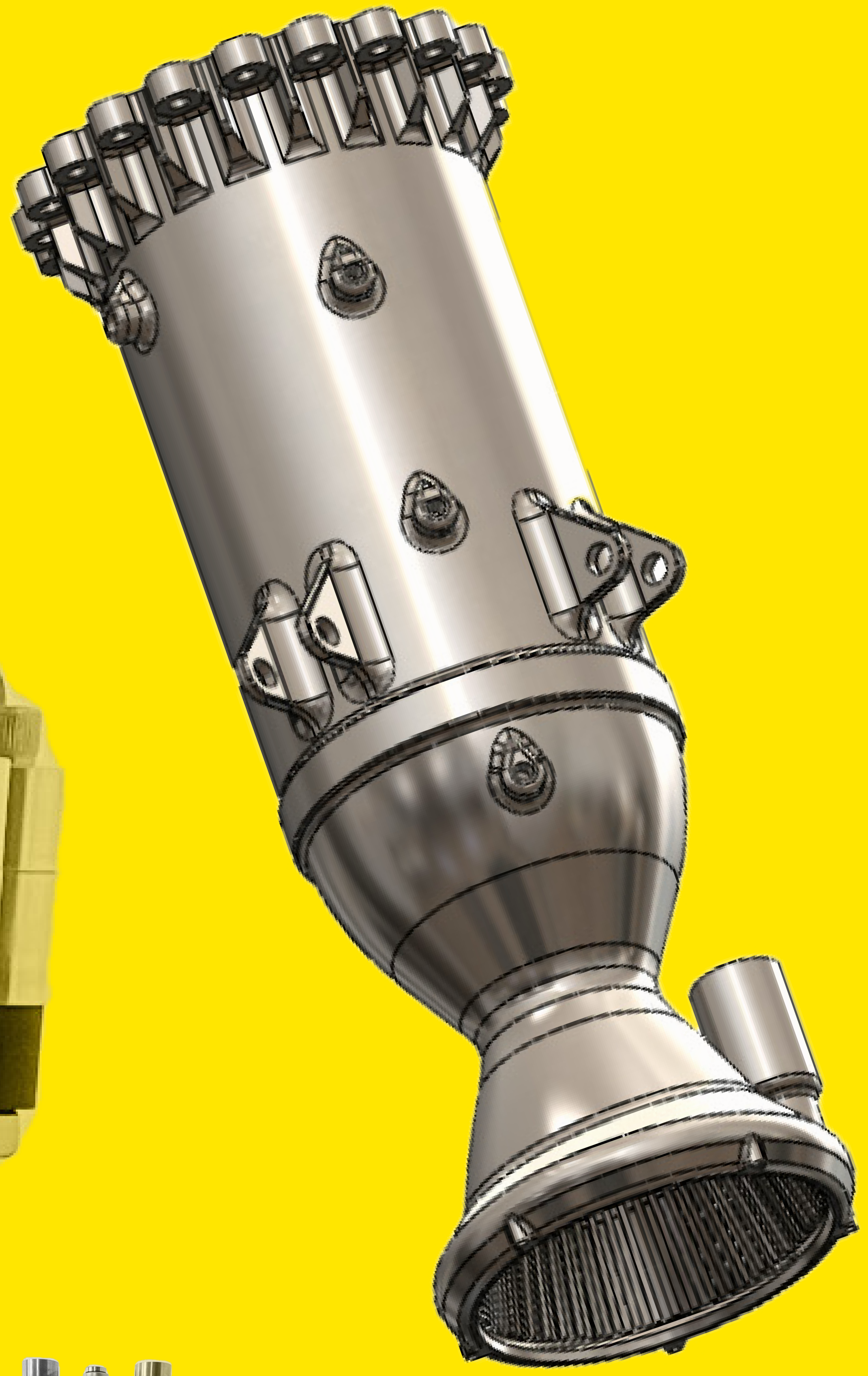
A group of San Diego State University (SDSU) aerospace engineering students designed a liquid rocket engine to be used in flight. The engine was designed to be built in Inconel 718, most commonly used for hot section components, such as booster rocket nozzles, in the space industry.

Regarding the design process, SLM Solutions guided the design for additive manufacturing for the thrust chamber components, reducing the parts from over a hundred to just five. The parts were then printed on an SLM® 280 2.0 system in the SLM North American applications center in Wixom, Michigan.

Although the additive manufacturing process provided a significant leap in the design and manufacturing process, potential imperfections in the components could be considered problematic for space applications. This was solved by utilizing Hot Isostatic Pressing (HIP) as a post-processing heat treatment at the Quintus Technologies Application Center in Lewis Center, Ohio to eliminate any potential porosity and further homogenize the material.

For quality testing, this project involved two independent sources that validate the quality of printed components produced on SLM machines. One source was the testing and recording of tensile properties from a NADCAP-approved testing facility, and the second inspection was a computed tomography (CT) scan performed by Avonix Imaging.





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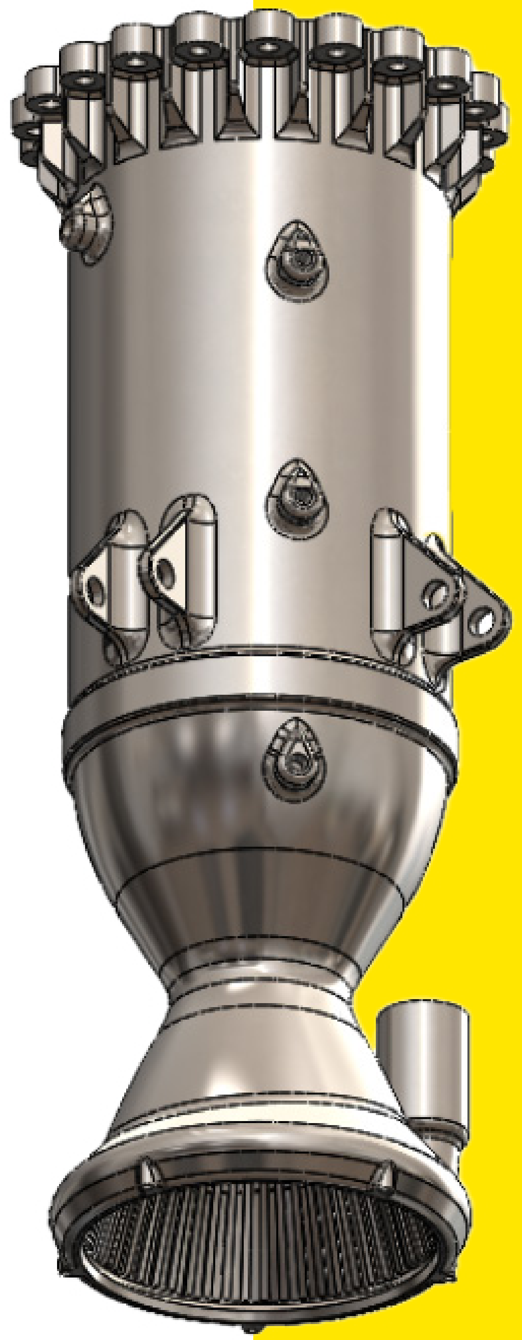
INTRODUCTION

This project aimed to develop a small-scale rocket engine capable of reaching the Kármán line, a commonly recognized boundary line for space at 100 km or about 62 miles altitude. Rockets cannot traditionally reach the Kármán line without using complex liquid-fueled engine designs. Metal additive manufacturing (also known as 3D printing) using the Selective Laser Melting (SLM) process from SLM Solutions, enables the production of complex components needed to cross the Kármán line. Additive manufacturing uses high-strength alloys, such as Inconel 718, a superalloy used in demanding applications, at high temperatures, such as gas turbine blades and rocket motors.

Over the course of a year, the students designed and tested components and held several design reviews with industry professionals in order to optimize their design. Once the design was finalized, the focus shifted from design to manufacturability. The team contacted SLM Solutions, who provided guidance on how multiple parts could be consolidated into one printed part and offered its services to bring this engine to life.

This was a truly remarkable opportunity, and with the added benefit of metal 3D printing, the team could redesign and combine components, drastically reducing the complexity of production. In the end, the engine design was reduced from around one hundred components to just five: a two-part outer shroud, a two-part inner liner, and an injector.

When designing an application for use in space, the team encountered a problem due to the nature of the powder bed fusion printing processes, the grains in additive manufactured material are not equiaxed, but columnar in the build direction. This results in anisotropic mechanical properties, which in many cases is problematic. This technology needs to be combined with another process to reduce the imperfections in the metal and increase fatigue resistance.



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THE CHALLENGES

OF BUILDING A SPACE ROCKET ENGINE

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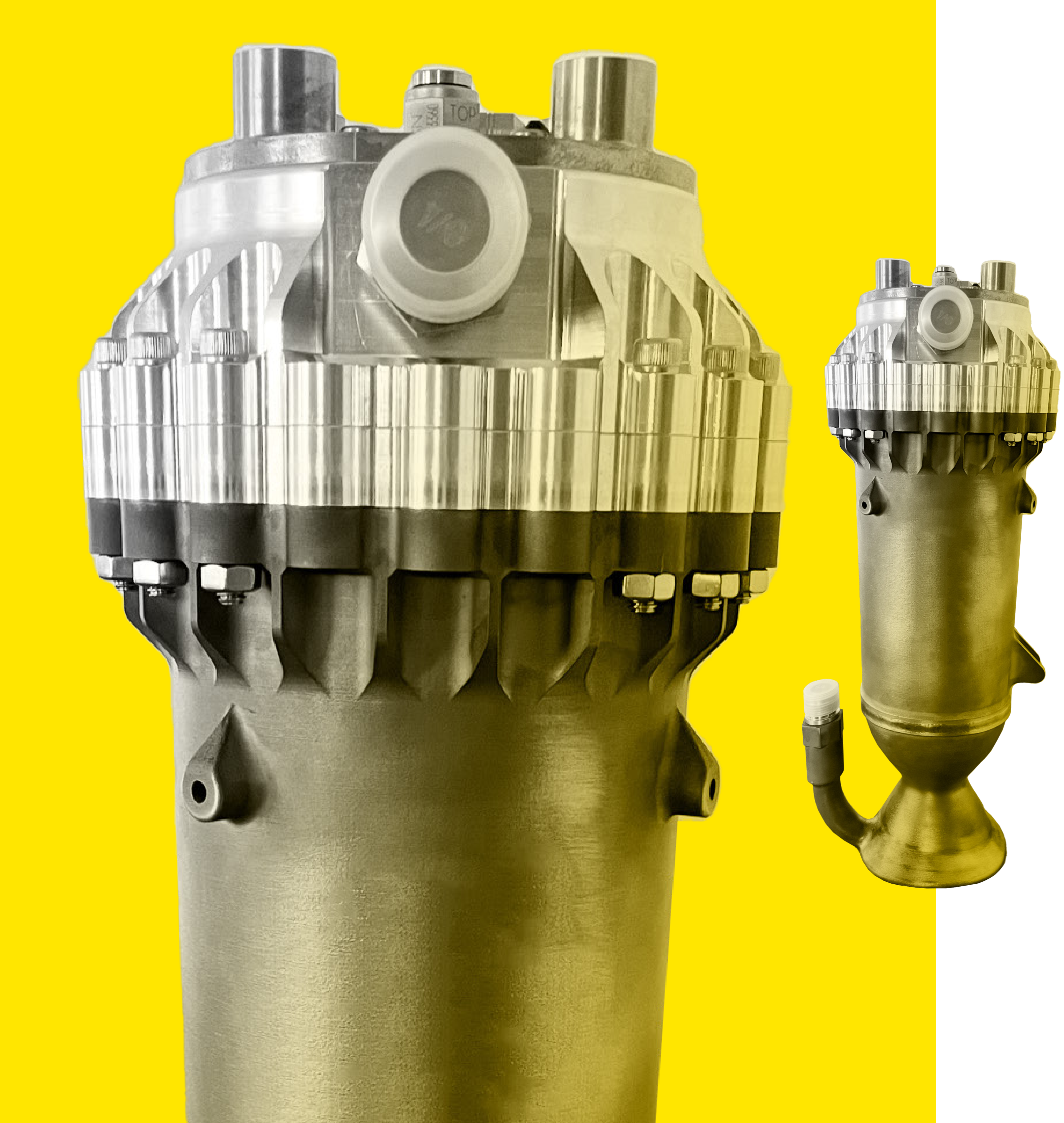

Building rocket engines for outer space has always been a challenging exercise. Rocket Engines require a high degree of complexity, in addition to being able to withstand high temperatures and stress. In addition, anything traveling above the Kármán line needs a propulsion system that doesn't rely on the lift generated by Earth's atmosphere, as the atmosphere is simply too thin. From this point on, orbital dynamic forces become more important than aerodynamic forces.

Traditionally, these engines are manufactured from several parts using casting and CNC milling techniques, which require a great deal of time and cost. This is one of the main reasons until now, crossing the Kármán line has been solely reserved for the aerospace industry.

Additive manufacturing provides a solution to this, ushering in a new manufacturing era. Complex parts that were once difficult to manufacture and assemble can now be printed on demand, creating a faster, streamlined, and more cost-effective manufacturing process.

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ADDITIVE MANUFACTURING

PARTS FOR SPACE

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Specifically, in selective laser melting, a layer of metal powder is spread onto a substrate plate; then, lasers selectively melt the powder to create the first layer of the build. A fresh layer of metal powder is evenly distributed over the build surface, and the lasers melt each successive layer to the layers underneath until the desired component is produced.

Unlike laser sintering, selective laser melting completely melts each layer into the previous for completely dense metal parts. Compared to traditional manufacturing methods, additive manufacturing enables parts with complex geometric shapes and hollow structures to be produced.

Metal AM also reduces the number of components, significantly reducing overall manufacturing time and costs. Single parts with high complexity can be printed, such as regeneratively cooled rocket engines, in materials such as Inconel 718.

The SLM® 280 proved to be the system of choice for various factors. First, the multi-laser system's agility allows you quickly develop prototypes and modify them to move rapidly through the design process and on to manufacturing. When it comes to manufacturing, the system has a high percentage of uptime and has been proven to be a reliable production machine.

The SLM® machine is currently used by multiple aerospace companies in North America, EMEA, and APAC for aerospace production because of its reliability, especially with materials such as Inconel 718, which are required for the demanding conditions of space.

SLM Solutions stitching or overlap software is an industry-leading development. Other machine OEMs struggle to fuse base materials using multiple lasers, causing companies to create in-house programs to override OEM software. This is something that doesn't need to be done with SLM® technology, as it is designed from the ground up for this purpose.

SLM®280 2.0

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THE SLM PRINTING PROCESS

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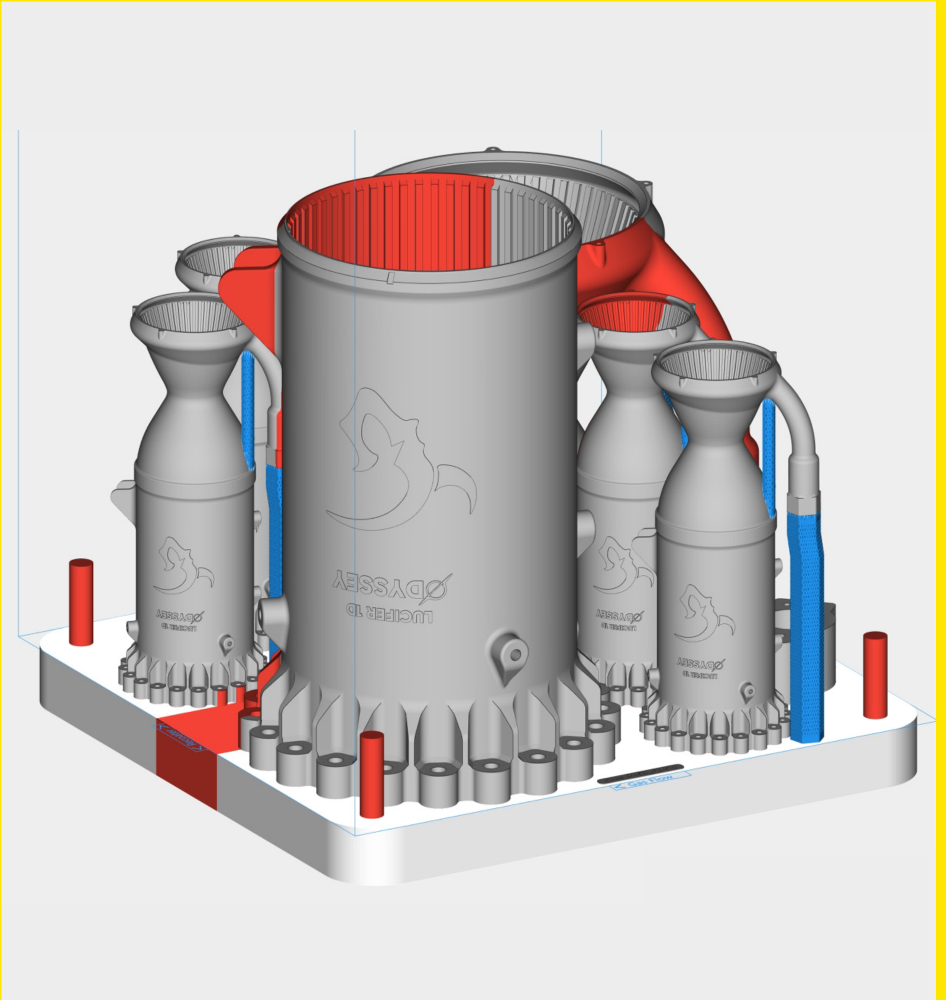
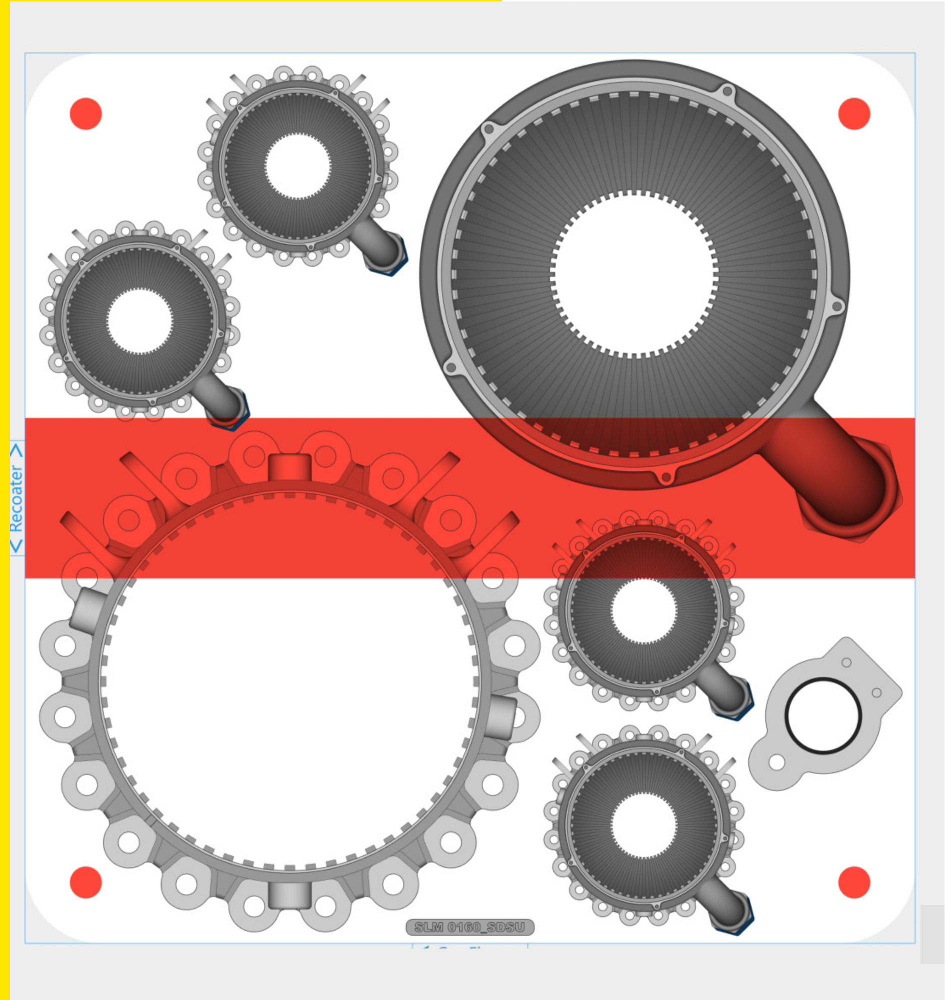
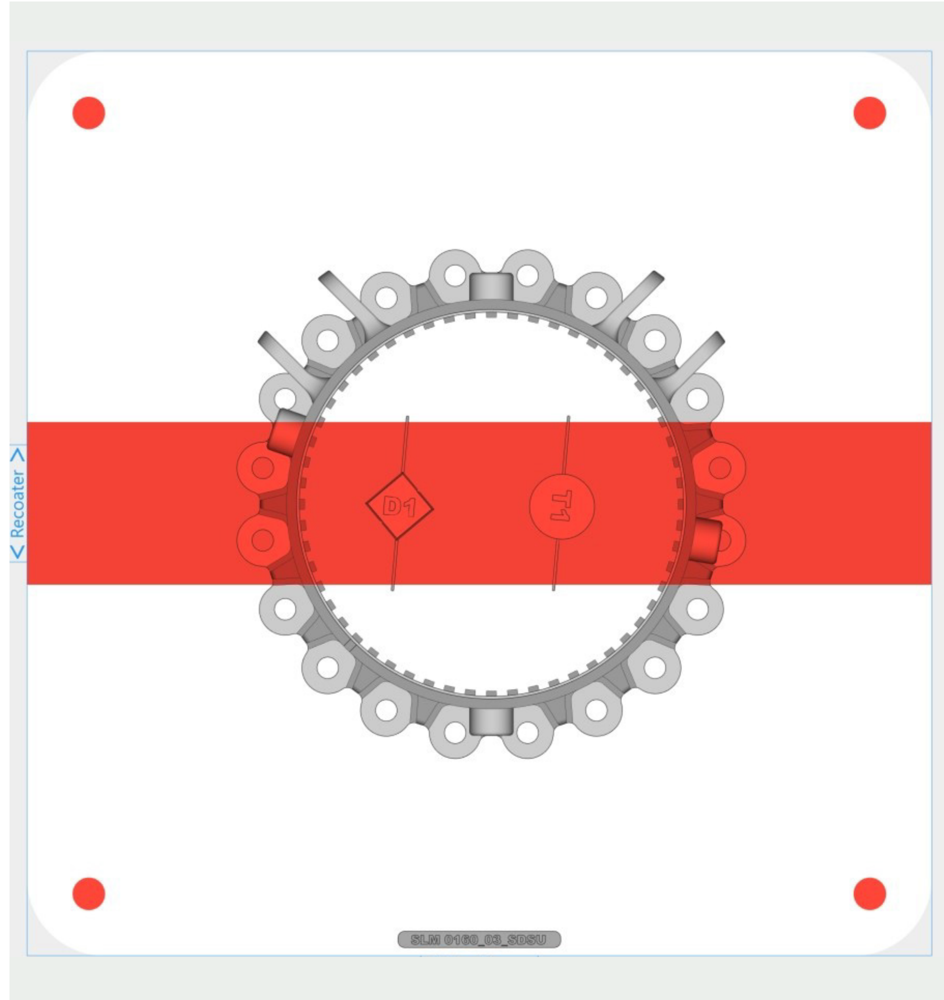


Built
Layout

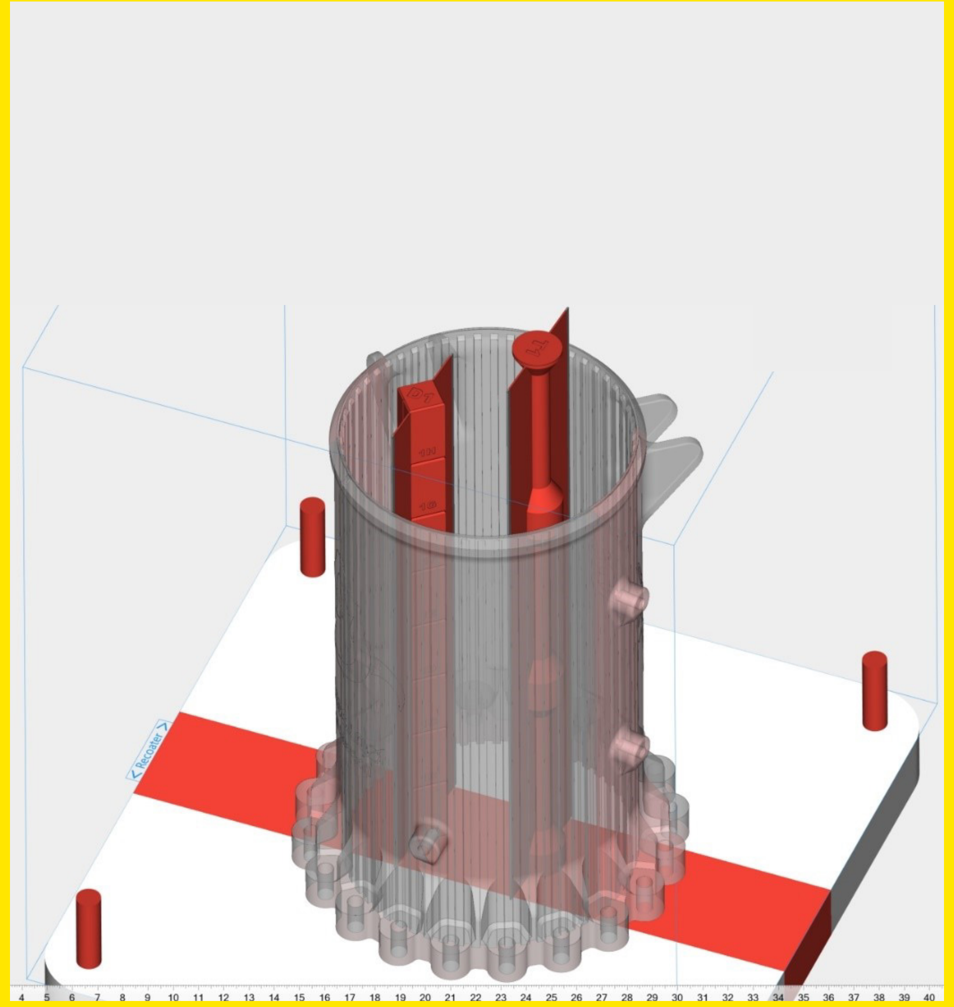


Due to the components' size and placement on the build plate, areas of the parts were printed using SLM®-patented dual laser technology.

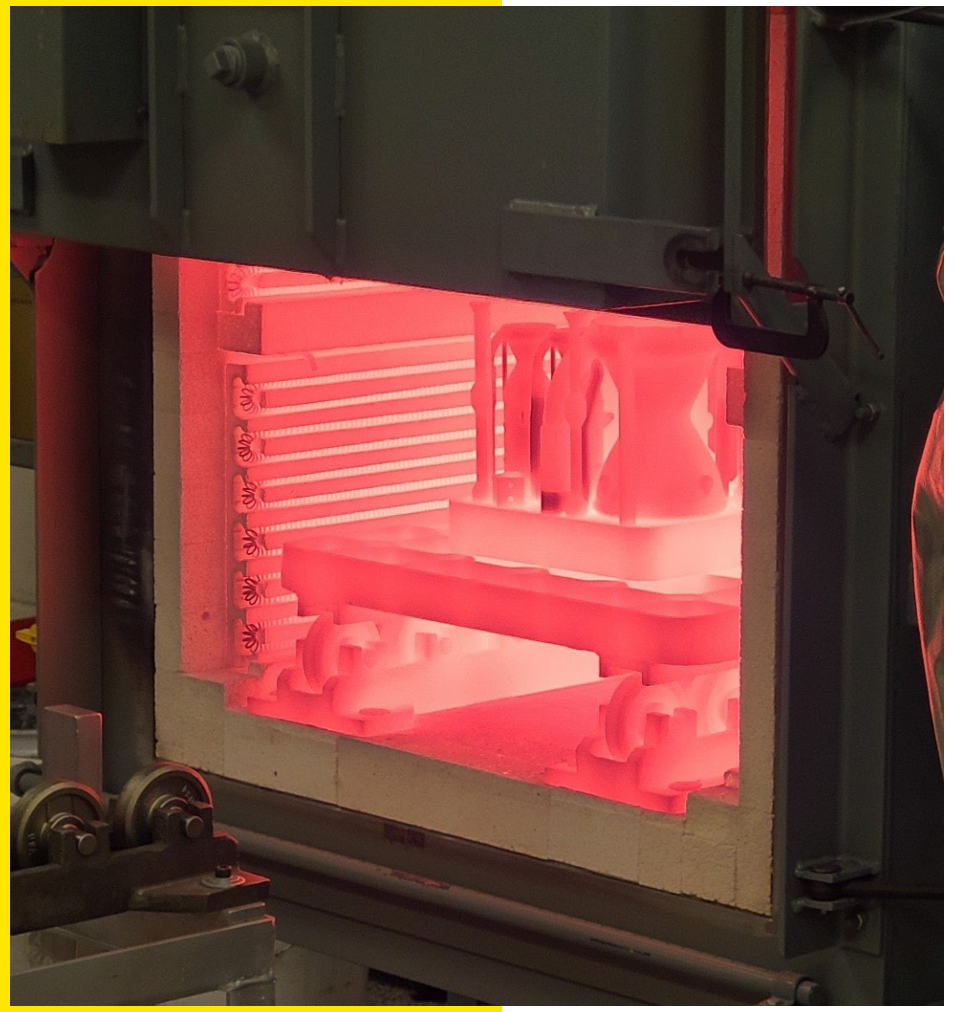


The above figure shows a print job where the top half of the rocket motor was printed along with three tensile bars taken from a tall vertical test specimen identified as T1.

The parts were printed from Inconel 718 powder with 30-micron layer thicknesses in an argon atmosphere on an SLM® 280 system. After printing was completed, the builds were de-powdered and stress-relieved at 1950 °F for 1.5 hours in an argon environment. The components were then separated from the build plate using wire EDM cutting technology.



Top half of rocket motor along with test specimens (T1)



Components being removed from oven after stress relieving process

Photo of top and bottom of rocket engine along with smaller rocket engine model

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POST PROCESSING AND HIP

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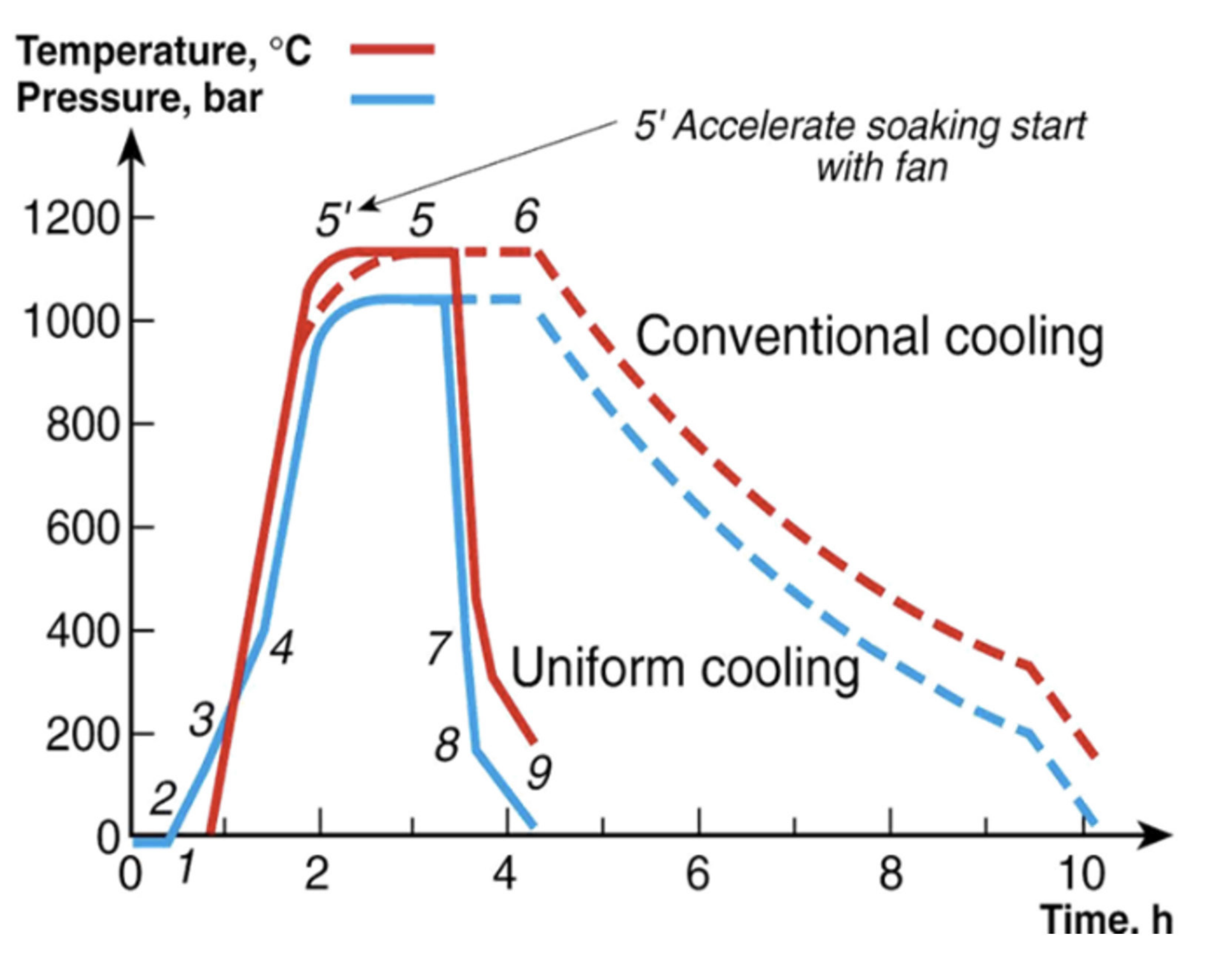
After printing, depowdering, and stress relieving at 1950 °F, SLM partnered with heat-treating supplier Stack Metallurgical Group to have these parts hot isostatic pressed (HIP) and solution and age (STA) heat treated prior to testing and use. The smaller printed assemblies of the rocket motor were HIPed only at Quintus Technologies.

HIP is a well-proven technology for reducing the size and/or eliminating defects such as pores and cracks in conventionally produced material, which is now being actively applied to additively manufactured components. These defects are prone to causing stress concentration and act as crack initiation points, having a detrimental effect on fatigue resistance and ductility. Properties can be improved using heat treatment, however defects cannot be removed by heat treatment alone. During the HIP process, components are subjected to temperatures up to 2,000°C (3,632°F) and pressures up to 207 MPa (30,000p psi) in a controlled atmosphere, usually argon gas. This combination of high temperature and pressure heals pores and cracks, homogenizes the material, eliminates residual stresses and ensures there are no remaining areas where the metal has not fused properly. This results in higher fatigue resistance and ductility compared to as-built components. A. Kaletsch et al. [1] studied the effect of HIP treatment on 3D printed Inconel 718 specimens and found that the fatigue strength increased by 28% if the material had undergone a HIP process prior to heat treatment.

Predictable material properties are crucial for the operating safety of the finished components and enables engineers to optimize their designs in terms of material thickness. A Kaletch et al. also found that the variability of the fatigue strength was substantially smaller for the specimens that had undergone a HIP treatment. This was explained by a more homogenous microstructure after the HIP treatment. Not all L-PBF machines are the same and some produce parts with questionable quality. This is why many space agencies and organizations require HIP Technology. The porosity associated with some of these machines can vary considerably with the position on the build plate during printing [2]. Parts being printed on a specific location of the build plate may have a different porosity size and size distribution than parts being printed on other locations of the plate. This variation in porosity will cause the mechanical properties, e.g. the fatigue strength, to vary. This variation can however be minimized by removing the pores with a HIP treatment.

HIP treatment is often combined with traditional heat treatments such as solution treatment and aging to further enhance the mechanical properties. Recent developments in Quintus® HIP equipment facilitate heat treatment in the HIP vessel following the densification process, so-called High Pressure Heat Treatment (HPHT™), leading to a single process to achieve fully dense parts with the desired mechanical properties. High cooling speeds and steered cooling regimes allow tailored cycles. These strategies are available by commercial heat treaters today and can be evaluated in the Quintus Technologies Application Center.

A QIH15L compact HIP system is showcased which is available for R&D support located in the Quintus Technologies Application Center.



A schematic comparing conventional and Uniform Rapid Cooling (URC©) functionality enabling High Pressure Heat Treatment (HPHT™).

THE IMPORTANCE OF HIGH PRESSURE

While many companies across the globe make an engineering decision to not use HIP technology, many companies and aerospace organizations use HIP technology as a form of added assurance and risk mitigation. The pressure is the key to the risk mitigation. In [3] a comparison is made between 3D printed test specimens of Inconel 718 that have either undergone a heat treatment at 1,200°C and 120 MPa for 4 hours, or a heat treatment at the same temperature for the same amount of time but without any applied pressure. The results clearly show that pressure is essential for reducing the number of potential defects. The heat treatment without applied pressure showed no significant defect reduction, whereas the HIP treatment lowered the defect concentration from 0.17% to 0.03%.

GRAIN SIZE AND COARSENING

The size of the crystalline grains making up a piece of metal is of great importance to its mechanical properties. The Hall-Petch equation dictates that the yield stress at room temperature is inversely proportional to the square root of the average diameter of the grains. In other words, the smaller the grains, the stronger the material. [4]

When a metal is exposed to temperatures above the recrystallization temperature, its grains will increase in size. The higher the temperature and the longer the exposure time, the more the grains grow. Traditionally, temperatures of 1,163-1,200°C (2,125-2,192°F) and an exposure time of four hours have been used for HIP treatment of Inconel 718. Several studies have shown that these settings cause substantial grain growth [3], [5], [6], [7]. In [7] Sean Gribbin et al. concludes that larger grains and content of annealing twins resulting from HIP treatment at 1163°C (2,125°F) have a detrimental effect on fatigue strength. The solution to this problem is to reduce the temperature and/or the exposure time.

LOWER HIP TEMPERATURE MINIMIZES GRAIN GROWTH

A study performed by Quintus Technologies together with University West in Sweden, shows that all the positive effects in terms of defects elimination can be achieved at lower temperatures than traditionally used, while minimizing grain coarsening [8].

The ASTM standard for HIP treatment of Inconel 718 (ASTM F3055-14a) states the temperature should be between 1,120°C (2,048°F) and 1,185°C (2,165°F). In the study the porosity and microstructure of electron beam (EBM-PBF) and laser powder bed fusion (L-PBF) test samples of Inconel 718 that had been HIP treated at 1,120°C (2,048°F) and 1,185°C (2,156°F) respectively were compared. The pressure (100 MPa) and duration (4 hours) was kept constant for all test series.

The results show that the elimination of pores was just as effective at 1,120°C as at 1,185°C. In both cases the porosity was reduced from 0.15 vol.% to 0.01-0.02 vol.%. In other words, 1,120°C is a high enough temperature to achieve full densification. However the grain size and texture were significantly different. For the EBM samples the grain growth at 1,185°C was substantial in the contour regions, but no grain growth was observed in the middle (hatch) region. At 1,120°C there was no evident grain growth at all. For the L-PBF samples there was clear grain growth at 1,185°C, and the grains in the samples HIPed at 1,120°C were significantly finer.

The results indicate that grain coarsening takes place at a higher temperature than 1,120°C and that grain growth can be mitigated if this temperature is used for HIP treatments. Further tests have shown that combining HIP treatment at 1,120°C with solution treatment and a shortened, two-step ageing process can even reduce the grain size compared to the as-built material [9].

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GRAIN STRUCTURE

Due to the nature of the powder bed fusion printing processes, the grains in additively manufactured material are not equiaxed but columnar in the build direction. This results in anisotropic mechanical properties, which in many cases is problematic. Since HIP is performed at a temperature above the recrystallization temperature, a HIP treatment can help homogenize the microstructure. This has been investigated in [8] where the grain structure for 3D printed Inconel 718 before and after HIP treatment at 1,120°C (2,048°F) was compared. For L-PBF material, the material recrystallized completely, resulting in an equiaxed microstructure. For electron beam EBM-PBF printed material, the columnar structure remained even after the HIP treatment. A. Kaletsch et al. came to similar results in [1] where they concluded that a HIP treatment fully homogenizes the grain structure for L-PBF printed Inconel 718.

HIP OF THE ROCKET ENGINE

Taking the knowledge gained on HIP of additively manufactured Inconel 718, Quintus Technologies applied an optimized HIP cycle to homogenize the microstructure and minimize grain growth while targeting a fully dense structure. The smaller printed assemblies of the rocket motor were HIPed in the Quintus Application Center applying a soak with a temperature of 1,120°C (2,048°F), pressure at 100MPa (14,500psi), while being held for 4 hours. The cycle also leveraged the URC® functionality to rapidly cool the HIP unit to minimize process time for improved productivity.

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QUALITY ASSURANCE

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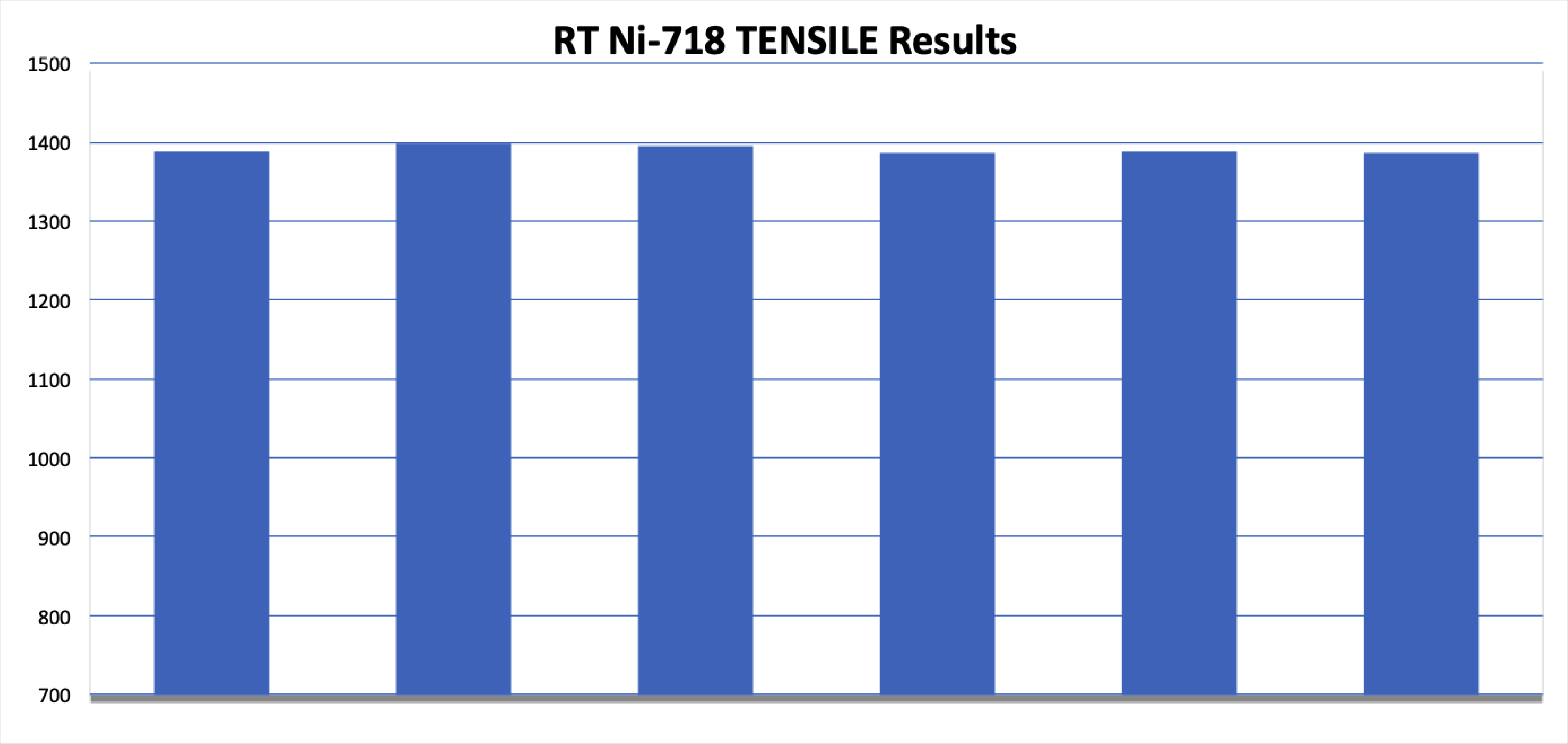
TENSILE PROPERTIES

The test specimens were tested at room temperature (RT) at Element Hamburg, Germany, for tensile properties. The results are posted below.

Ni-718 RT Tensile Results				
	YIELD	YIELD	UTS	UTS
SPECIMEN	RpO2 (MPA)	KSI	Rm (MPA)	KSI
1	1197	173.6105	1389	201.4578
2	1195	173.3204	1399	202.9082
3	1196	173.4654	1396	202.4730
4	1186	172.0151	1387	201.1677
5	1190	172.5952	1389	201.4578
6	1180	171.1448	1386	201.0227
High	1197	173.610	1399.000	202.908
Low	1180	171.145	1386.000	201.023
Average	1190.67	172.692	1391.000	201.748
SD	6.68	0.969	5.254	0.762



Repeatable tensile test results along with repeatable yield strength results of the six specimens, show that SLM® machine parameters and superior quality were consistent across the build plate. Three of the test specimens were printed with two lasers.

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X-Ray Computed Tomography (CT) Scanning

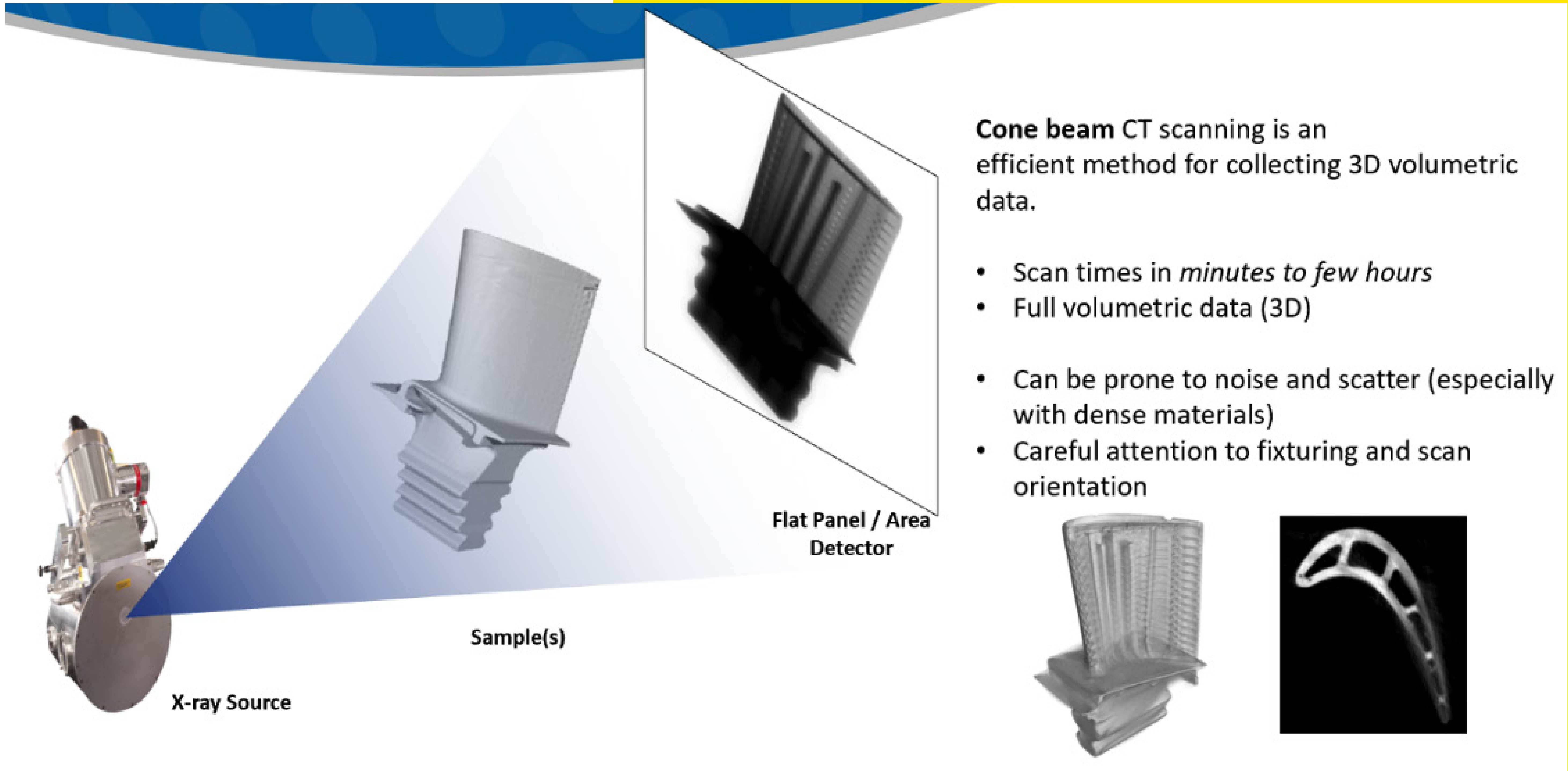
CT Scanning is a non-destructive imaging method which results in cross-sectional and or volumetric datasets revealing a specimen's internal geometry, structure and features. It is commonly used for inspection of flight hardware and other mission-critical components. The advent and rise of metal additive manufacturing has further fed the need for non-destructive evaluation technologies, such as CT scanning. The ability of metal additive manufacturing to realize complex internal features and geometries renders many traditional surface inspection techniques useless for part qualification.

Industrial x-ray CT scanning has been around in practice for many decades and has evolved into a necessary inspection technique in engineering and qualification of manufactured components. Industrial CT scanning is analogous to medical CAT scanning that many are familiar with. Key differences in approach, mainly the utilization of higher x-ray energies and longer imaging acquisition times, enable high-resolution inspection of larger and more dense materials and structures. Industrial x-ray CT also makes use of micro-focus x-ray technologies, which helps enable spatial resolutions in excess of 100-micrometers, sometimes down to single microns.

The basic process of CT scanning involves the acquisition of hundreds or thousands of x-ray images, through all angles around a specimen. These x-ray images, or radiographs, are then reconstructed into a three-dimensional volume, which can be virtually cross sectioned or processed into a point cloud or CAD geometry. There are a wide variety of tools and techniques to acquire, reconstruct, and process CT scan data. The ultimate approach will depend heavily on the application, specifically the part material, size, complexity, as well as factors such as throughput and resolution requirements.

This project enabled two primary scanning approaches; a 3D cone beam scanning approach and 2D linear array canning approach. Examples of both approaches are shown in Figures X and Y. Three-dimensional, or cone beam CT scanning leverages high resolution flat panel imaging devices. This scan method captures several thousand radiographic images, each several megapixels in resolution. This process produces a large three-dimensional volumetric dataset in a single scan. While cone beam CT scanning can be tailored to be fast and efficient, it may be prone to noise and unwanted imaging artifact, which are seen especially in denser materials such as steel and nickel-based alloys. The degradation of image quality can prevent the detection of defects and the ability to extract internal surface data for measurement.

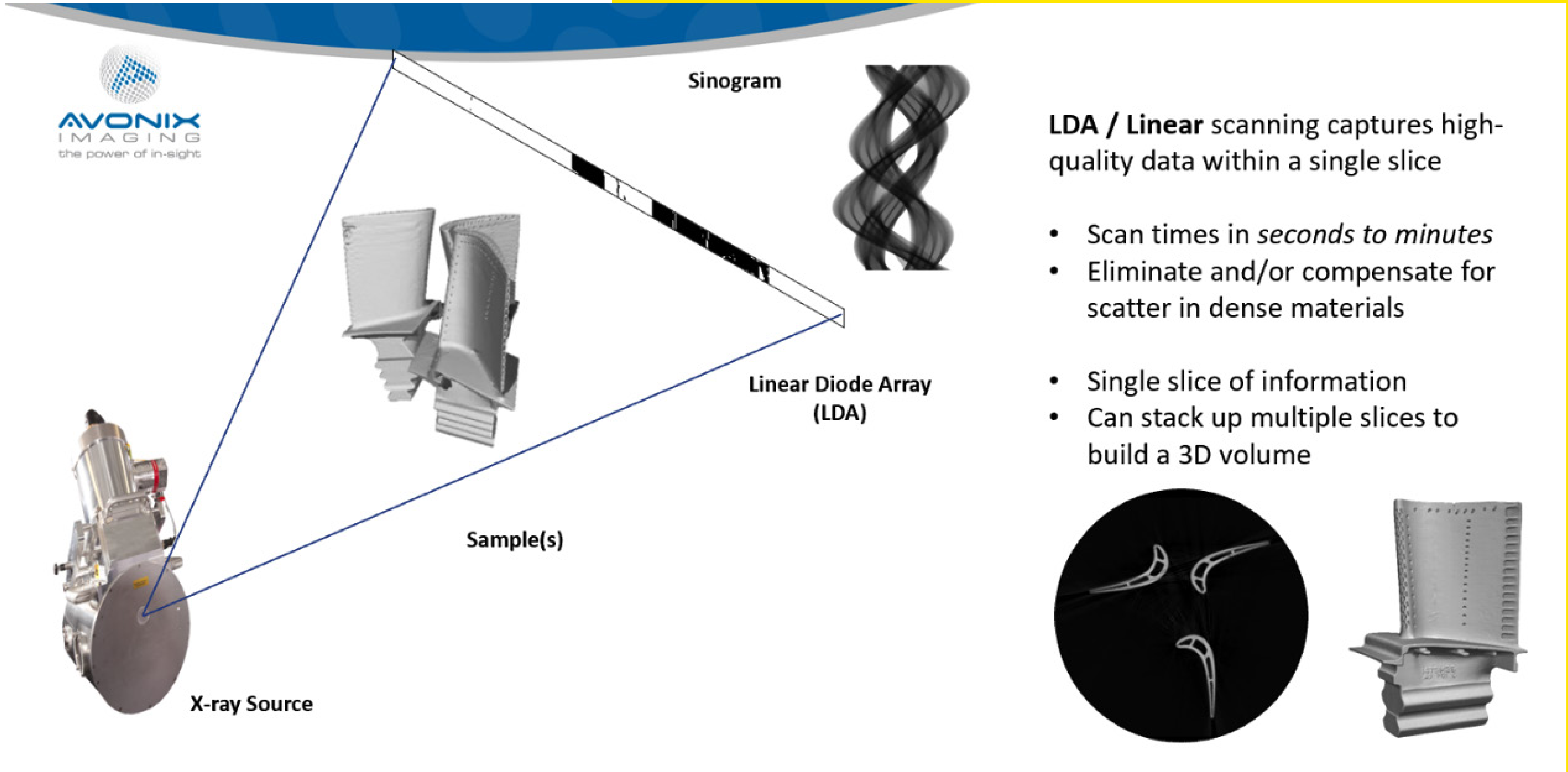
To overcome image degradation a two-dimensional line scan can be used. In contrast to cone beam CT, in this technique, the x-ray beam is collimated into a fan beam, and an image is projected on to a one-dimensional line detector, typically one pixel tall, by several thousand pixels wide. The main advantage of this methodology is the reduction in unwanted x-ray scatter, which further improves the overall image quality. The main tradeoff is imaging time. Only one plane at a time is captured in a single rotation, therefore, additional imaging time is needed to build a full three-dimensional volume. 2D CT scanning can be very efficient in production application, where quality checks are performed only on certain datums, however, scans may take hours to a day in order to render a full three dimensional dataset.



Some people in industry believe that in-situ monitoring and Non-Destructive Testing such as CT Scanning are competing technologies. This is not a correct assumption. The truth is these technologies are complementary and should be used alongside with one another as well as other quality tools.

In-situ process monitoring is a technology area that occurs during the build, and involves the measurement of thermal or chemical processes during the material processing. Like CT scanning, in-situ monitoring has rapidly evolved into a deployable solution for many AM processes. There are those that see in-situ monitoring and non-destructive inspection as competing technologies. They are in-fact complimentary, with process monitoring providing critical data during the AM processing, and CT scanning serving as a method of validation of those data. Today, validation of in-situ data must occur.

X-Ray CT Scanning is a critical tool to qualify parts, particularly large parts, more economically than having to cut X-Ray CT Scanning can measure wall thicknesses and show the level of material density, in-situ technology cannot do this.



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The rocket motor was first scanned using a cone beam scanning approach. The Avonix Team used the following technique:

450KV MICROFOCUS SOURCE

440KV, 255UA X-RAY CONDITIONS

61.5-MICRON VOXEL SIZE

3,000 PROJECTION IMAGES

4MM COPPER FILTRATION

45-MINUTE SCAN TIME

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The Avonix Team found no indication of porosity in the printed part, within the resolution limits of the scan.

Oftentimes, a reference quality indicator, or RQI, may be used to validate the results of a CT scan. Because image quality and detectability of defects in CT are often dictated by a part's material and geometry, it's important for the RQI to represent the part as closely as possible. This technique is very helpful in practice and consists of a representative part, with known defects or features. The RQI can be scanned alongside, or before/after the part under inspection. Characterization of the known defects is recorded and the technique may be validated. The use of additive manufacturing to embed known defects is a key advantage in the development of RQIs.

To further improve the image quality, the part was scanned a second time using a Linear Approach.

The Avonix Team used the following technique:

450KV MICROFOCUS SOURCE

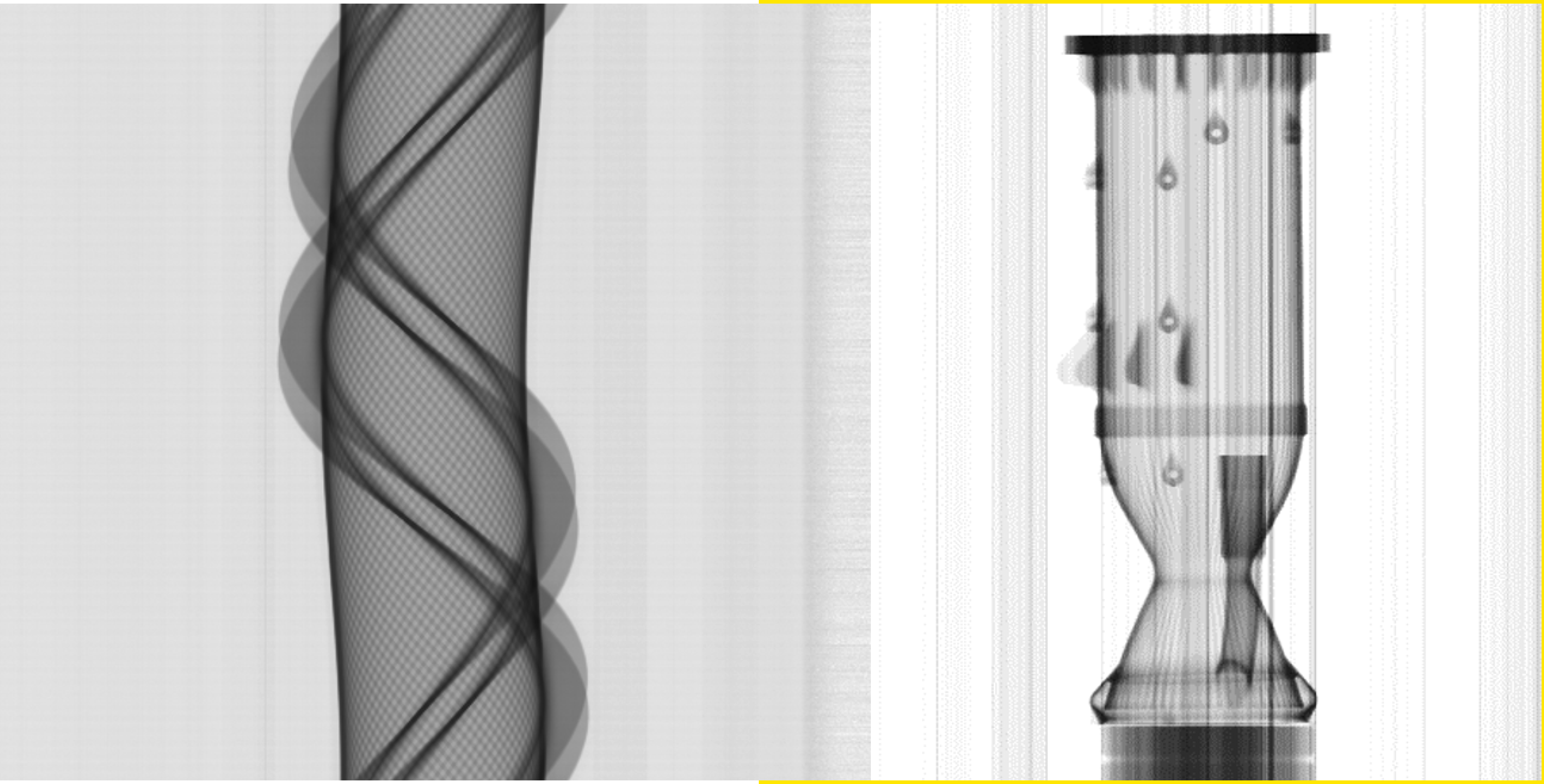
440KV, 455UA X-RAY CONDITIONS

100-MICRON VOXEL SIZE

1,608 Z-SLICES

2MM COPPER FILTRATION

45-SECONDS PER SLICE (19HR TOTAL)



Again, the Avonix Team found no evidence of cracking or porosity within the resolution limits of the scan.

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CONCLUSION

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In this whitepaper, we have documented the manufacturing process for the liquid rocket engine, which combines metal additive manufacturing with Hot Isostatic Pressing and heat treatment to produce applications in Inconel 718 for the engine.

Metal additive manufacturing considerably reduces the build parts and complexity, while HIP treatments minimize the potential defect content and adds a level of risk mitigation if the user believes it is necessary, leading to higher fatigue strength and less variability in mechanical properties.

We have highlighted the problem with current HIP standards for 3D printed Inconel 718 and that they originally have been developed for castings. Additively manufactured material has a different microstructure than castings and require different settings for optimum results.

Tests with lower HIP temperature, higher pressure, and the use of integrated HIP and heat treatment show promising results in minimizing grain size and improving mechanical properties. In addition, HIP can break up the columnar grain structure in L-PBF printed components, thereby homogenizing mechanical properties.

In addition, we have run quality assurance tests with two companies. Element Hamburg was responsible for testing the tensile structures in a NADCAP-approved testing facility. At the same time, the Avonix team was responsible for CT scanning the application using both a cone beam and a linear scanning approach.

All tests concluded there was no porosity in the printed part, which reinforced that metal additive manufacturing in conjunction with HIP can be used to make high-grade applications for space and aviation without sacrificing quality.

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2. Jim Shipley, Heat Treatment of AM Parts by Hot Isostatic Pressing, presentation, Quintus Technologies (2019).

3. Snea Goel, Tejas Gundgire, Johny Varghese, et al., Role of HIPing and heat treatment on properties of Alloy 718 fabricated by electron beam melting, Euro PM2019– HIP Post Processing of AM Parts, Maastricht, Netherlands.

4. Wikipedia, Grain growth, https://en.wikipedia.org/wiki/Grain_growth

5. Snea Goel, Anumat Sittiho, Indrajit Charit, et al., Effect of post-treatments under hot isostatic pressure on microstructural characteristics of EBM-built Alloy 718, Additive Manufacturing 28 (2019) 727-737.

6. Arun Ramanathan Balachandramurti, Johan Moverare, et al., Microstructural influence on fatigue crack propagation during high cycle fatigue testing of additively manufactured Alloy 718, Materials Characterization 149 (2019) 82-94.

7. Sean Gribbin, Saeede Ghorbanpour, et al., Role of grain structure, grain boundaries, crystallographic texture, precipitates, and porosity on fatigue behaviour of Inconel 718 at room and elevated temperatures, Materials Characterization 149 (2019) 184-197.

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THANK YOU

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