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LATEST DEVELOPMENTS IN HIGH PRESSURE HEAT TREATMENT OF AEROSPACE COMPONENTS

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Abstract

Hot Isostatic Pressing (HIP) has been used to remove shrinkage porosity and internal defects in cast products for many years, predominantly to improve mechanical properties and fatigue resistance. Of late, focus has moved to further improve thermal processing routes through innovative use of the heat and pressure developed during densification. Studies have demonstrated significant benefits for production processes such as Additive Manufacturing (AM), casting and metal injection moulding (MIM) where demands on quality are significant and where internal defects and porosity are of concern.

Recent developments in the use of in-process heat treatment directly after the densification of materials (High Pressure Heat Treatment) have shown further improvements in material properties such as strength and fatigue resistance, facilitating weight savings and optimal performance. Combined post processing using HIP including Solution Heat Treatment (SHT) and Aging leads to the opportunities to automate production and improve digitalised quality improvement methodologies.

In this presentation, solutions for various aerospace alloys as well as recent results from trials and studies will be discussed.

Keywords: Additive Manufacturing, Hot Isostatic Pressing, Mechanical Properties, Combined HIP and Heat Treatment

1. HIP, a well-known go-to technology for aerospace components

Hot Isostatic Pressing (HIP) has been used to remove shrinkage porosity and internal defects in cast products for many years, to improve mechanical properties and fatigue resistance. Of late, focus has moved to further improve thermal processing routes through innovative use of the heat and the inert gas pressure from the densification process. Studies have demonstrated significant benefits for production processes such as Additive Manufacturing (AM), casting and metal injection molding (MIM) where demands on quality are significant and where internal defects are an area of concern.

Recent developments in the use of in-process heat treatment directly after the densification of materials (High Pressure Heat Treatment) have shown improvements in material properties such as strength and fatigue resistance, facilitating weight savings and optimal performance. Combined post processing using HIP including Solution Heat Treatment (SHT) and Aging leads to the opportunity to automate production and improve digitalized quality improvement methodologies.



Figure 1 – Saving weight is necessary to reduce carbon emissions for commercial aircraft

2. Challenges

The corner stones of the aerospace industry are:

- Passenger safety and risk mitigation
- New fuels and propulsion solutions
- Lighter structures and the need for new material solutions

2.1 Passenger Safety and Risk Mitigation

Structural integrity is in focus for every single component involved in the aerospace industry, with exacting demands on quality and function as material failure is not an option. Component and process qualification are focus areas leading to predictable properties that can be used in definition of design limits to guarantee safe operation.

Qualification of products is a tightly controlled process for each component including all steps in the production chain as well as inspection and quality control. For the aerospace industry HIP helps to guarantee performance.

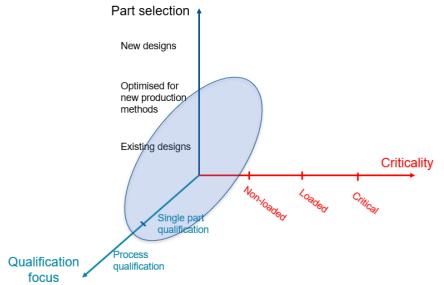


Figure 2 – Illustration of how major OEMs (Original Equipment Manufacturers) classify qualification of components and processes. The central ring shows the status for additively manufactured parts

Once processes are qualified for a specific production route, these remain locked for the duration of the program. It is therefore extremely important to ensure optimization steps are taken prior to finalization of new production route.

Hot isostatic pressing is a critical process in the post processing chain for aerospace components which is seen as essential in combination with part and process qualification as well as non-destructive testing.

2.2 New fuels and propulsion solutions

The aerospace industry is rapidly moving towards greener solutions, with a combination of improved fuel efficiency, new engine designs and the development of new Sustainable Aviation Fuels (SAVs). Below is a summary of some of the current focus areas.

Fuel	Sources [1][2][3]	Challenges [1]
SAV additives for blending with aviation kerosene	Hydro-processing of fatty acids, alcohols, synthetic gas or biocrude	Carbon based additive Significant processing chain
Hydrogen	Gas separation, thermochemical cycles, water electrolysis, photocatalysis	Storage vessels 4x compared to aviation kerosene [2]
Battery solutions	Li-ion batteries Solid State batteries	Safety for liquid electrolytes [4] Battery weight Efficiency

Table 1: Future propulsion solutions

The new fuels give rise to a variety of problems which need to be solved. These include fuel storage, containment in the airframe structure and safety aspects tied to the different media.

2.3 Lighter structures and the need for new material solutions

Weight plays a key role in the amount of fuel burn, especially during take-off. Up to 10% of the fuel burn (% is dependent on flight distance and aircraft model) is seen during climb to cruising altitude. Fuel containment solutions and the resulting airframe design can play a greater role for future propulsion technologies.[2]

An aircraft weight reduction of 1 kg results in a fuel saving 20-30 grams of fuel per 1000 Km which is significant for larger aircraft. The cost per available seat kilometer (CASK) varies typically between 0.04 to 0.10 Euros for commercial aviation, and a 1 ‰ reduction of a typical CASK of 0.06 Euro would require reductions in the region of 300 kg to 1000 Kg for typical airliners. [1]

Reduction of weight requires new and innovative material solutions, and here HIP plays a vital role.

There are many challenges in an aerospace environment, such as vibration and fatigue loading, thermal expansion/contraction, and corrosion. Performance of the components is critical, and saving weight therefore requires increased strength and/or further enhanced properties to negate the challenges. Traditional use of HIP ensures removal of porosity and improved fatigue life, and now further technology advancements allow improvement of design limits facilitating lighter weight structures.

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3. Critical technology to ensure safety

3.1 Background to Hot Isostatic Pressing (HIP)

Hot isostatic pressing is a process that has been used since the 1960s to remove porosity in materials in order to improve their integrity. The HIP equipment itself consists of a pressure vessel, which can contain incredible pressure, usually up to 207MPa (30000 psi) in which the payload is heated in an inert atmosphere. Argon gas is normally used as the inert pressure medium, but

nitrogen is also common for HIP of some materials. Whilst under pressure, heaters are used to soften material at temperatures typically 80-90% of their melting point, utilizing a molybdenum furnace. Graphite furnaces are used for materials requiring temperatures in excess of 1400°C (2552°F), such as ceramics. Temperatures, pressures, and dwell times depend on the payload material. In this extreme environment, mechanical deformation and creep of the material closes porosity and micro cracks allowing diffusion to heal the surfaces resulting in a homogenous microstructure. The resultant material shows dramatically reduced scatter in mechanical properties including fatigue resistance, ductility, and fracture toughness. Predictable properties then allow improved designs and increased levels of safety.

Removal of porosity and defects is key to improving fatigue resistance of materials to prevent premature failure in critical environments.

3.2 Combined HIP and Heat Treatment Cycles, High Pressure Heat Treatment

There have been two significant trends in recent years. Larger and larger HIP units are being introduced, to cope with larger volumes of product, and also larger sizes of component. A more significant development has been development of controllable high-speed cooling (Uniform Rapid Cooling, URC®) and in-HIP quenching (Uniform Rapid Quenching, URQ®) have been developed by Quintus Technologies. These breakthrough technologies enable full heat treatment of parts following the densification step of the HIP cycle. This is known in the industry as High Pressure Heat Treatment, HPHT. Tailored heating and cooling steps can be used to repetitively achieve improved material properties, often combined with adjusted temperatures, pressures and hold times to optimize the microstructure, adding industrial robustness to production processes. Figure 3 below, illustrates typical thermal processes used for a laser powder bed fusion additively manufactured product.

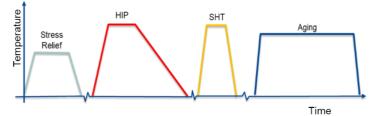


Figure 3 - Typical thermal processes for additively manufactured parts. ©Quintus Technologies

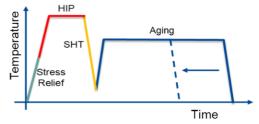


Figure 4 - High Pressure Heat Treatment (HPHT) process for the same parts, using an integrated heat treatment approach with possibilities to shorten aging time and overall cycle time. ©Quintus Technologies

Figure 4 shows how heat treatment processes can be combined using HPHT[™] to not only shorten processing time, but also to provide improved and repeatable material properties, whilst reducing the need to reheat material several times This approach has been proven in many studies in recent years [5][6][7][8][9][10].

Recent work carried out on both titanium and aluminium alloys, including Ti-6AI-4V, F357 and AISi10Mg shows the opportunity to utilize in-HIP quenching of parts using the Uniform Rapid Quenching, URQ® technology specifically. Publication of these studies is expected during 2022 although some studies have already been published. [8],[9], [21], [22]

3.3 Modern HIP equipment from Quintus Technologies

Key technology drivers identified by Quintus, include several essential heat treatment parameters:

- Heating rate
- Temperature uniformity
- Cooling rate
- Pressure control
- HIP atmosphere
- Payload condition

The pressure used during the HIP process has a direct correlation with the heat transfer between the load and the furnace atmosphere. Quintus knowledge in this area enables simulation of heating and cooling during the HPHT cycle, which can then be verified through trials. This gives performance enhancing control over the microstructure expectations and quality in the production cycles.

The example below shows modelling of Ni-base turbine blades at two different pressures (Figure 5), clearly showing the difference of cooling rate depending on the HIP pressure after 200 seconds during the Uniform Rapid Cooling, URC® cooling cycle.[12]

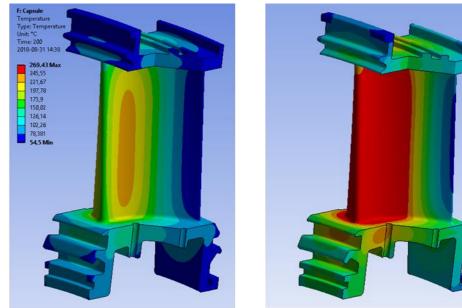


Figure 5: Finite element modelling of Uniform Rapid Cooling, URC® of a nickel base super alloy material at different pressures.

Control of temperature and pressure over time are key performance areas for Quintus hot isostatic presses. They enable steering of tailor-made cycles which ensure repeatable, robust, and prequalified microstructures.

This is made possible through technical developments in the equipment over recent years as can be seen in the temperature log below (Figure 6), from an installation verification cycle for a fully loaded QIH 60 HIP, equipped with HPHT capability. This is a typical HIP used for investment castings and additively manufactured parts.

Modern, fan driven systems give rise to extremely good temperature uniformity on par with class 1 and class 2 furnaces in the pyrometry standard, AMS2750[11] (Figure 7). Temperature uniformity during cooling is also critical to achieve a uniform microstructure and performance for all parts within the hot zone.

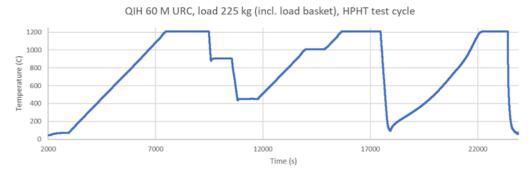
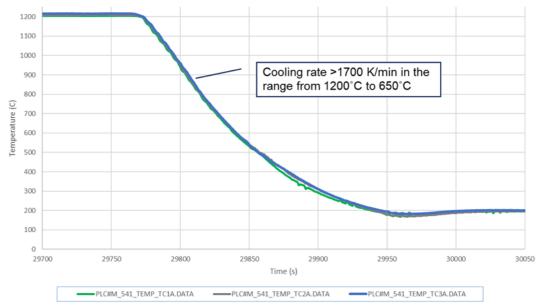


Figure 6: Installation verification cycle for a Quintus QIH 60 M URC with full payload



QIH 60 M URC, load 225 kg (incl. load basket), HPHT test cycle

Figure 7: The final cooling segment from the verification cycle shown above in Figure 6, with cooling rate >1700 K/min in temperature range 1200-650 °C. Three thermocouples show extremely good temperature uniformity during cooling

Modern HIP machinery from Quintus Technologies offers excellent control of thermal cycles and pressure adjustments, where latest developments have also led to the reduction of high-temperature oxides on the surface of HIPed parts. This is of course interesting with respect to improved fatigue and corrosion resistance as well as savings in finishing operations and the potential environmental impact resulting from them.

A common phenomenon with titanium alloys, is the ingress of oxygen below the material surface, to form alpha-case. This is a brittle phase in the material this is most often removed electrochemically or mechanically prior to use of the produced aerospace component to ensure mechanical performance and reduce risk or fatigue cracks initiation.

Both surface and sub-surface oxides can be addressed in the clean HIP processing in modern Quintus HIP units. This is achieved without the need to wrap or bag parts to shield surfaces from the HIP atmosphere. This is a more sustainable and more efficient process requiring fewer resources and with lower environmental impact. Below are some Alloy 718 images after HIP demonstrating typical HIP processing and recent advances in state-of-the-art Quintus technology (Figure 8).



Figure 8: Alloy 718 bars treated with traditional hot isostatic pressing (left) and modern Quintus processing (right)

3.4 Essential Alloys and how they are fabricated

Flight critical components are processed using HIP to ensure integrity with significantly increased fatigue life, in the range of 10 to 100 times [20]. Increased ductility and fracture toughness are also a result of defect healing. This technique has been utilized for many years on cast and powder metallurgy near-net-shape (PM NNS) materials and is becoming increasingly important for more recent production methods such as metal additive manufacturing. Turbine blades are typical such components, as shown in Figure 9, Figure 10.



Figure 9: An aero engine is a complex piece of equipment with high fatigue loads and temperature variations

There are many materials used today in the aerospace industry, from titanium and aluminium alloys to nickel base super alloys and ceramics. Some examples of material groups are,

- Light-weight alloys (e.g., titanium alloys, aluminium alloys, magnesium alloy
- Heat resistant alloys (e.g., nickel base super alloys, cobalt alloys)
- Ceramics (e.g., silicon nitride, alumina, zirconia)
- Composite materials (e.g., metal matrix composites)



Figure 10: An aero engine must be able to cope with extreme environments and give maximum thrust when called upon

The list of materials is of course extensive, and the application of innovative technologies is growing. Operating environments and consequently, demands on the material properties are specific for each of area of use, product, and processing technology. Table 2 shows how applications, environments and desired properties are connected.

Application	Environments	Critical (predictable) properties
Airframe,	Fatigue loading	High fatigue resistance
landing gear	Cryogenic service	Ductility
		Weldability
		Low density / mass (Weight reduction)
		Formability
Engine	Fatigue loading	Corrosion resistance
-	Elevated temperatures	Wear resistance
	Thermal cycling	Weldability
	Exposure to radiation	Creep strength
		Impact toughness / ductility
		Weight

Table 2: Applications, environments, and desired material properties

4. At the forefront of innovation

Quintus Technologies has been actively involved in supporting research and development into new products, processes, and materials for the aerospace industry for many years. Today there are many ongoing programs where development of new and innovative HIP solutions play an active role. Some examples of these are listed in the reference section. We encourage increased cooperation regardless of technology readiness level (TRL).

5. Summary

HIP is an essential technology for the aerospace industry in light of high demands in the reduction of environmental impact of aviation, requiring the highest performance possible from materials, including repeatability and reliability of performance. The use of in-HIP heat treatment, High

Pressure Heat Treatment, HPHT[™] is a proven technology to achieve improved material performance and strength, and this modern HIP equipment equipped with HPHT[™] capability is now widely available in the market. This technology is being specified for production applications based on extensive research programs and trials.

About Quintus Technologies AB

Quintus Technologies is the global leader in high pressure technology. The company designs, manufactures, installs, and supports high pressure systems for sheet metal forming and densification of advanced materials. Quintus has delivered over 1,900 systems to customers within industries such as aerospace, automotive, energy, and medical implants. The company is headquartered in Västerås, Sweden, with a presence in 45 countries worldwide. For more information, visit https://quintustechnologies.com/

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References

- [1] Fuel economy as function of weight and distance, Rolf Steinegger Dipl. Bau.-Ing ETH SIA SVI EMBE. DOI 10.21256/zhaw-3466
- [2] Sustainable aviation fuels, European Union Aviation Safety Agency environmental report. https://www.easa.europa.eu/eaer/
- [3] Sustainable aviation fuel Review of technical pathways, U.S (United States) Department of Energy. www.energy.gov/eere/bioenergy, September 2020
- [4] A review of safety considerations for batteries in aircraft with electric propulsion, Sripad et.al. MRS Bulletin 46, 435-442 (2021)
- [5] IATA Fact Sheet 7: Liquid Hydrogen as a potential low carbon fuel for aviation, August 2019. www.iata.org/contentassets/d13875e9ed784f75bac90f000760e998/fact_sheet7-hydrogen-factsheet_072020.pdf
- [6] Effect of γ' precipitate size on hardness and creep properties of Ni-base single crystal superalloys: Experiment and simulation SFB/Transregio 103 'From Atoms to Turbine Blades', Ali et.al. Materialia. Vol 12 Aug 2020 https://doi.org/10.1016/j.mtla.2020.100692
- [7] An integrated HIP Heat-Treatment of a Single Crystal Ni-Base Superalloy Ruttert et.al. The Minerals, Metals & Materials Society 2020 S. Tin et al. (eds.), Superalloys 2020, The Minerals, Metals & Materials Series https://doi.org/10-1007/978-3-030-51834-9_38
- [8] Effect of different heat-treatment routes on the impact properties of an additively manufactured AlSi10Mg alloy, M Tocci et.al. University of Brescia, The Minerals, Metals & Materials Society and ASM International 2020 https://doi.org/10.1007/s11661-020-05905-y
- [9] Parameter and process optimization for the additive manufacturing process in the powder bed using the

example of the alloy Ti6Al4V, D.Ahlers Paderborn Univ. PhD Thesis. ISBN 978-3-8440-7424-6

- [10] Microstructure evolution-based design of thermal post treatments for EBM-built Alloy 718, Goel et.al. University West, December 2020 Metals & corrosion J Mater Sci (2021) 56:5250–5268
- [11] Hot isostatic pressing in metal additive manufacturing: X-ray tomography reveals details of pore closure Du Plessis, MacDonald https://doi.org/10.1016/j.addma.2020.101191
- [12] Optimized cooling to achieve reduced lead time and optimal mechanical properties of HIPed and heattreated parts, J Gårdstam, Quintus Technologies EPMA (European Powder Metallurgy Association) HIP Seminar 2019 Sint Niklaas
- [13] Aerospace Material Specification Pyrometry Standard AMS2750F
- [14]Effect of a New High-Pressure Heat Treatment on Additively Manufactured AlSi10Mg Alloy, Tocci et.al. https://doi.org/10.1007/s11661-020-05905-y
- [15]Optimization of heat treatment parameters for additive manufacturing and gravity casting AlSi10Mg alloy, Girelli et.al. doi:10.1088/1757-899X/264/1/012016
- [16] Titanium aluminides processing by additive manufacturing a review, Soliman et.al. Doi: doi.org/10.1007/s00170-022-08728-w
- [17] Metal additive manufacturing in aerospace: A review. Blakey-Milner et.al https://doi.org/10.1016/j.matdes.2021.110008
- [18] Total Energy U.S. Energy Information Administration (EIA)Total Energy U.S. Energy Information Administration (EIA) www.eia.gov/totalenergy/data/monthly/index.php#petroleum
- [19] Hot isostatic pressing (HIP) to achieve isotropic microstructure and retain as-built strength in an additive manufacturing titanium alloy (Ti-6AI-4V). Benzing et.al. Elsevier Materials Letters Volume 257
- [20] J. T. Staley Jr et al., The effect of hot isostatic pressing (HIP) on the fatigue life of A206-T71 aluminum castings, Materials Science and Engineering A 465 (2007)
- [21] E. Wycisk, AMPOWER Ti-6AI-4V HIP study (2020), Unpublished
- [22] A. Wessman, Quintus Tech Talk regarding unpublished results, April 2022