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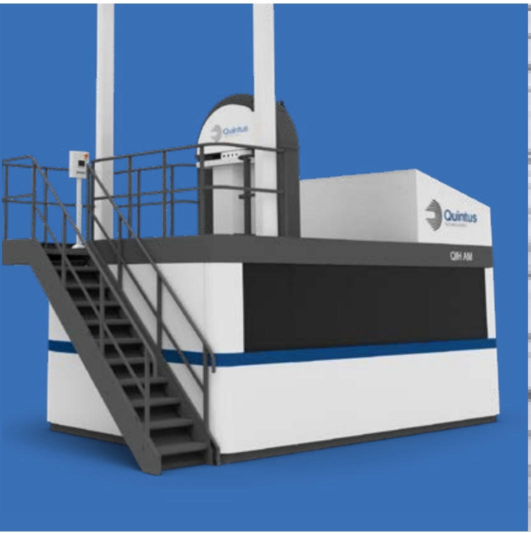
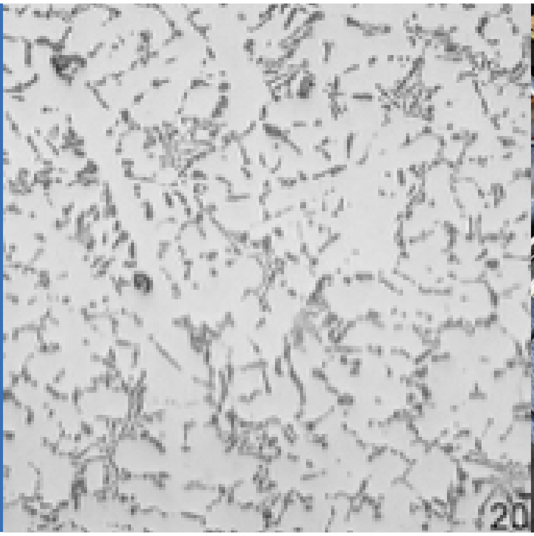
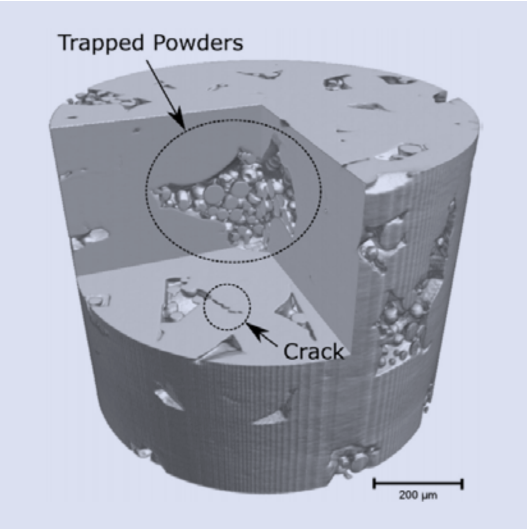
ebook series

High Pressure Heat Treatment:

Leading the Renaissance of Hot Isostatic Pressing





Prepared in cooperation with
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INTRODUCTION

In an ever-changing world where production efficiency, reduced environmental impact, and improved process reliability are in focus, some well-known technologies are seeing a renaissance. Hot isostatic pressing (HIP), is one such technology, which until now has been an essential process to densify porosity and improve the mechanical properties of materials for components. Subsequent heat treatment has been used to achieve final product properties. HIP has been around many years in its traditional form, and recent advances in technology and a changing business climate have shown just how bright the future is.

An increased interest from heat treatment service providers is driving industry growth because of the functionality of the equipment. Companies have installed HIP capability as a natural complement to their other heat treatment equipment, while expanding their localized service offering.

This has broadened the availability of the technology and increased the number of technical discussions and trials to verify the benefits of HIP. The recent introduction to the market of new and modern range of HIP equipment with robust built-in heat treatment capability has made this possible. Modern HIP machinery is an extremely good fit with the traditional heat treatment market, offering the opportunity to further adjust material properties through tailored HIP cycles. Many OEMs have also decided to insource the HIP process to gain flexibility in their production and to gain control over previously sub-contracted processes. Of course, with in-house equipment, the opportunities to develop processes and own intellectual property increase.

The outlook for HIP technology has never been brighter, helped by technology shifts that are accelerated by recent global events. The additive manufacturing market is growing rapidly, helping drive the development of the market for smaller units.

Figure 1. A modern hot isostatic press, equipped with High Pressure Heat Treatment technology.

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BACKGROUND OF HOT ISOSTATIC PRESSING

A hot isostatic press (HIP), is a furnace inside a pressure vessel designed to withstand extreme pressure. HIP technology was originally developed in the 1950s in the quest to make man-made diamonds from graphite, where very high pressures were needed. Basically, two alternative solutions were developed: one with a monolithic forging and end closures, and the other with a wire-wound vessel and frame. Each of these solutions is designed to handle the design pressure and temperature in the hot zone with various solutions for adjusting pressure and maintaining temperature uniformity.

The Swedish company ASEA, later the "A" of ABB, developed the wire-wound technology through a strong innovation team and first patented their solution on March 18, 1955, following the development of presses to produce synthetic diamonds in 1953. At the same time, gas-pressure bonding was developed at Battelle Memorial Institute, Columbus, Ohio, in 1955, where it was used in an isostatic diffusion bonding process for cladding of nuclear fuel elements. This would eventually be called hot isostatic pressing. For many years, Battelle conducted comprehensive equipment development which initially operated at a maximum temperature of 1526°F (830°C) and a pressure of 69MPa (10,000 psi) in a hot-walled monolithic forged structure.

Wire-wound technology continued to be developed by the high-pressure technology division of ABB, which is now known as Quintus Technologies company where technology is still based on wire winding of pressure-bearing components. Today, hot isostatic pressing equipment is designed for, and operated at, working temperatures up to 3632°F (2000°C) and pressures up to 207MPa (30,000 psi) as standard, with a choice of several different furnace options for the same pressure vessel. Even higher pressure equipment is available on the made-to-order basis.

What industries use HIP today?

Most industries benefit from the use of modern HIP equipment today, and there is significant growth in demand.

Some examples include:

- Civil aviation
- Space
- Medical
- Automotive
- Machining
- Mining
- Oil and Gas
- Power generation
- Electrical goods
- Defense

The diversity of components which are processed using hot isostatic pressing is significant, with component sizes ranging from millimeters to meters in diameter.

Heating components softens the material, and combined with the immense isostatic pressure of the inert process gas in the HIP vessel, creep leads to the mechanical closure of pores and defects. Diffusion acts to heal the surfaces of the pores and defects, leading to a homogenous material. As the pressure on the workpiece is from all directions, uniform closure of defects is obtained. Deformation is minimal for most wrought materials and can be calculated from the expected volume of the porosity removed. More significant deformation is seen when densifying very porous materials such as powder in cans, but this can also be calculated.

The high pressure gas atmosphere acts on all surfaces simultaneously, and internal porosity is eliminated as a result. Surface-connecting defects such as cracks and fissures cannot be closed using HIP, as the gas enters the defect also (Figure 2).

The elimination of internal defects has major effects on the properties of the material as stress concentrations and potential crack initiation points are removed. This results in dramatically improved fatigue life, ductility, and fracture toughness, leading to reduced scatter and more predictive design limits. In turn, weight-saving and cost reduction is facilitated, which makes this increasingly relevant for applications where weight is important, e.g., aerospace, automotive and offshore platforms.

The absence of internal defects has other benefits also, allowing machining and polishing of surfaces without revealing unwanted blemishes. This makes HIP a key process in the production chain for aesthetically important applications such as jewelry, as well as in production of equipment for gas-tight applications.

Hot isostatic pressing is a batch process, and parts are loaded into the chamber using load baskets or standing on a load base, depending on size and customer requirements. Quite often operators prefer two load bases to be able to unload/load, while another cycle is running for productivity reasons. Each load, as with other batch type furnaces, is subjected to a specific environment which is affected by the gas used, the cleanliness of the load, furnace furniture, and operational procedures. Once the load is sealed and pressurized, impurities in the gas cannot be removed, so thorough purging and vacuum cycles are needed prior to pressurization to ensure a clean environment. Typically, 4.8 argon gas is used, although nitrogen gas is commonly used for some materials.

Typically, HIP cycles run at eighty to ninety percent of the melting point of the material, to induce creep, diffusion, and homogenization of the microstructure. There are some exceptions to this rule of thumb, including ceramics and some metals such as titanium which are processed at temperatures well below melting point for other reasons. Molybdenum furnaces designed for temperatures up to 2552°F (1400°C) are used for most metals, and higher temperature materials are often processed in graphite furnaces.

Figure 2. *Schematic of a component before and after HIP, showing closed internal porosity remaining surface connecting defects.*

Before HIP

After HIP

The largest traditional application area for HIP has been the densification of castings for industries with very high demands on fatigue resistance, such as the aerospace and gas turbine industries. The advent of additive manufacturing is leading to an ever-increasing demand for HIP to ensure material performance in all manner of demanding application areas from orthopedic implants to racing cars and rocket engines.

Figure 3. Additively manufactured rocket engine thermally processed using HIP and heat treatment in the same cycle – High Pressure Heat Treatment

(Courtesy of Accurate Brazing and Launcher/John Kraus Photography)

Common alloys that are processed using HIP include:

- Aluminum
- Titanium
- Ni-base super alloys
- Cobalt Chrome
- Stainless steels
- Ceramics
- TiAl
- Copper alloys
- Duplex alloys
- Tool steel
- Ausferrite Ductile Iron (ADI)
- High Entropy (HE) alloys
- Tungsten carbide

Densification of Products

All modern-day metallurgical processes have well-established practices for using HIP. Cast material has been processed using HIP since the introduction of industrial processes in the mid-1960s with the aim of removing porosity and improving fatigue resistance. (Figures 4 and 5).

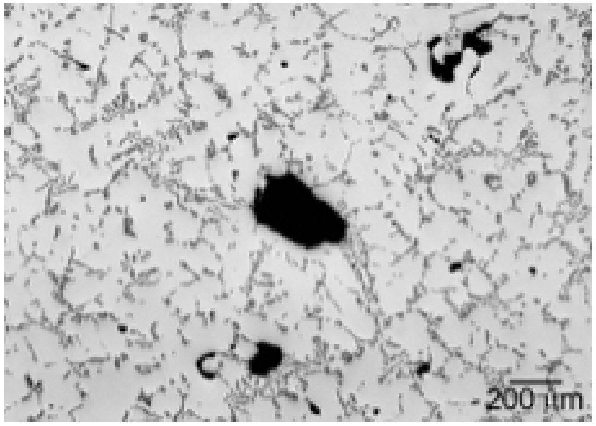

Figure 4. *Porosity in cast material.* [3]

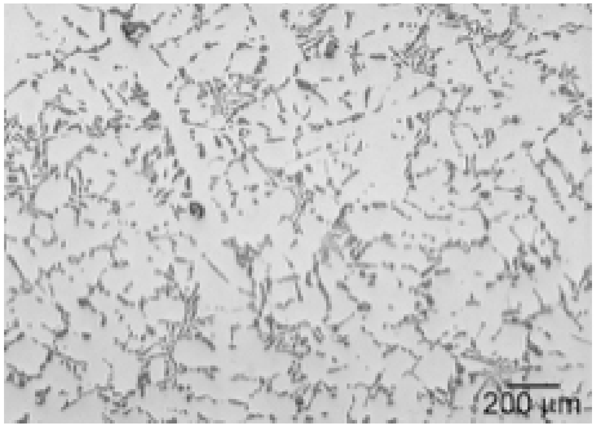

Figure 5. *Post HIP Micrograph of the casting.* [3]

Additive manufacturing (AM) is fast becoming a viable contender with many traditional industrial production processes. As for the high-end castings market, HIP is a process chosen for thermal post-processing of additively manufactured components for critical applications. There is often a need to improve material properties by removing residual porosity and other defects in the material, and here HIP is an essential process in the production chain. Micro-porosity, micro-cracking, stress induced cracking, and other detrimental defects are some of the issues with AM processes. Many of these can be resolved using HIP. Even many "non-weldable" alloys are possible to print using less energy density in combination with HIP. Some examples of defects typically seen in components produced using AM can be seen in Figure 6.

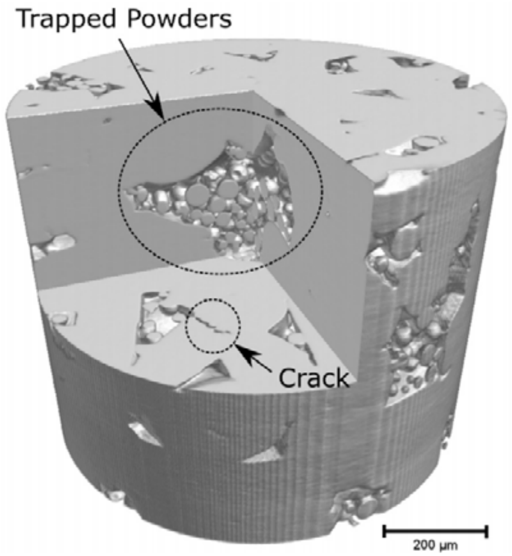

Figure 6. *Typical build defects arising from additive manufacturing processes.*[5]

Printing defects such as lack-of-fusion porosity, residual porosity, trapped powders, and microcracking give rise to stress concentrations and initiation points for cracking and corrosion.

These have negative influences on fatigue properties, ductility, and fracture toughness.

Consolidation of Powder

Although cast products account for a very large portion of the material processed using HIP today, there are many other processes where it is used. A good example is the use of powder metallurgy to produce extremely complex shapes, which has been extensively used in the oil and gas and nuclear industries. Here metal powder is placed in a complex shape can which is then evacuated and sealed prior to HIPing. A solid metal near-net-shape part with virtually no post-processing needs (Figure 7) is produced. The same technique is made for production of more simple shapes such as billets in high speed steel and tool steel.

Figure 7. *Powder metallurgical near net shape manifold structure with integrated hubs and tees produced using HIP.*

©MTC Powder Solutions

Diffusion Bonding

Combinations of materials are often bonded together using HIP to achieve a metallurgical bond. This is especially true for wear materials, which are clad onto substrates to improve erosion resistance in specific areas using the HIP diffusion bonding process. With a combination of materials in a single HIP cycle, the most appropriate HIP cycle parameters need to be carefully based on the melting points and heat treatment parameters of the materials involved.

Performing thermal processing treatments including stress relief, solution heat treatment, quenching and aging are all possible in HIP equipment today (see below) in conjunction with the traditional HIP cycle. This is an interesting and growing market for modern HIP equipment. HIP is no longer only densification.

The HIP Cycle

The HIP cycle consists of several steps, which are programmed into one sequence comprised of loading, vacuum cycle (to remove contaminants such as oxygen), heating, including pressurization, holding, cooling, de-pressurization, and release.

It is in the cooling phase of the HIP cycle that the difference in HIP design becomes clear, with wire-wound HIP equipment proving much more efficient in removing heat effectively from the vessel. A wire wound system has coolant channels placed close to the vessel to maintain temperature control of the wire package. These can also be used to remove heat to assist cooling. By forcing the process gas over the cylinder wall using fans or ejectors while simultaneously cooling the cylinder wall, high cooling speeds can be achieved. This process, known as Uniform Rapid Cooling, URC®, was initially developed to increase productivity by shortening the HIP cycle time by speeding up the cooling step. Dramatic cycle time reductions compared to conventional cooling (or turning off heaters and allowing the vessel to cool naturally) are seen. Cycle times vary depending on alloy and starting temperature and size of the vessel but are based on the same principles. This can be seen in Figure 8, where Uniform Rapid Cooling, URC® is illustrated.

The degrees of freedom for a HIP cycle have increased dramatically with the introduction of modern wire-wound HIP systems. With the possibility to control high cooling rates very accurately with a fan, everything from stress relief to quenching under pressure is possible.

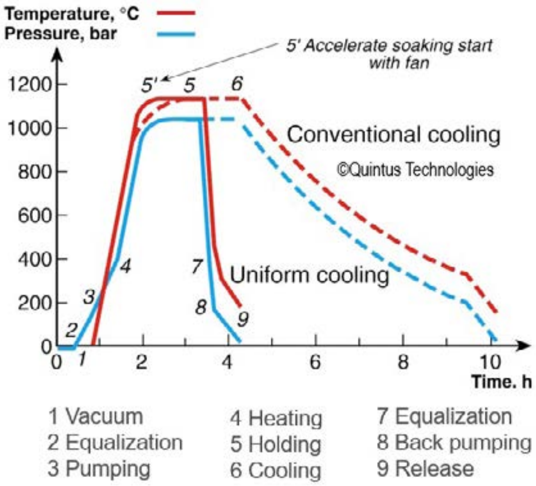

Figure 8. *Uniform cooling vs conventional cooling.*
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High Pressure Heat Treatment™, HPHT™

The ability to control the cooling rate after the soaking time is a significant and key development for the HIP industry. This leads to the possibility to combine several thermal processing steps into one cycle, all conducted at the same time under inert gas pressure. This technology is especially of interest for additive manufacturing (AM), but also for the tailoring of microstructures for nickel base super alloys and other metal injection molded (MIM) or investment cast materials.

Stress relief occurs during the heating segment of the HIP cycle. Many companies HIP material while still on the build plate to avoid damage and warping of parts prior to HIP. This is especially of interest for crack sensitive alloys.

The HIP soak temperature is usually above the solution heat treatment (SHT) temperature, and as such, the load can either be cooled to the desired SHT temperature prior to starting cooling or can be cooled directly from the HIP soak temperature. The choice route depends on the alloy as well as the desired properties of the material. Cooling the material from the SHT temperature at a chosen cooling rate is fully possibly in modern HIP equipment using fan control, and cooling can also be stopped at a chosen temperature prior to subsequent aging if needed. The combined, or integrated heat treatment approach inside the HIP vessel, is known as High Pressure Heat Treatment (HPHT™) (Figures 9 and 10).

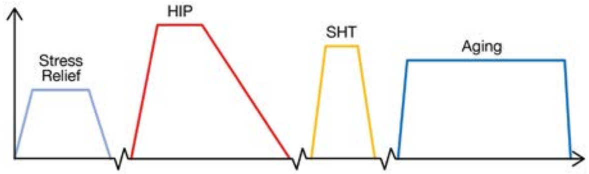

Figure 9. *Typical Thermal Processes for Additive Manufactured Parts*

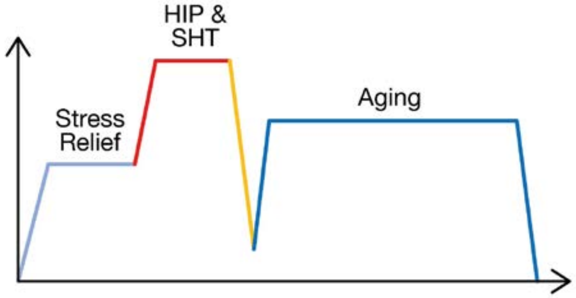

Figure 10. *High Pressure Heat Treatment process for the same parts, using an integrated heat treatment approach.*

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Steered Cooling

There are several developments in terms of the controllability of the HIP process that have now become central to many HPHT™ discussions. Especially important is the applicability of HPHT™ as a process to adjust and tailor microstructure. The cooling rate of the gas in the HIP can be steered using load thermocouples (LTC) to set the cooling rate of the component by closely controlling the gas cooling rate. This is an important quality aspect to ensure material properties in the center of thicker component sections.

The machine can therefore autonomously steer the temperature gradient based on the thickest component (Figure 11).

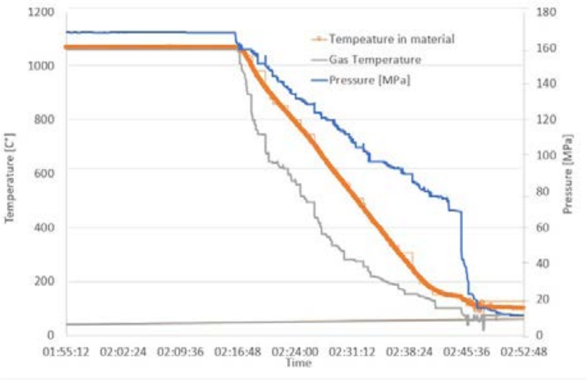

Figure 11. *Example of steered cooling for a nickel base super alloy. Cooling rate set at 86°F/min (30°C/min) controlled by a load thermocouple, LTC.*

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Higher Pressure Yields Higher Cooling Rates

The effect of the HIP pressure on the heat transfer coefficient, HTCα, has been demonstrated in recent studies.[1] By using this knowledge, it is possible to model the cooling rate for a specific machine, specific part geometry, material and HIP parameter set (pressure, temperature, hold time, cooling rate). The model can then be verified using steered cooling in actual cycles. The heat transfer is improved with the use of pressure, and as a result the cooling rate achieved increases with the HIP pressure, so various scenarios can be simulated.

This was demonstrated in the finite element simulations (FEM) shown in Figure 12, where the cooling rate of the same turbine blade was modelled at 150 MPa and 50 MPa. The results were demonstrated through metallurgical investigation. In short, parts can be cooled more quickly by using higher pressures.

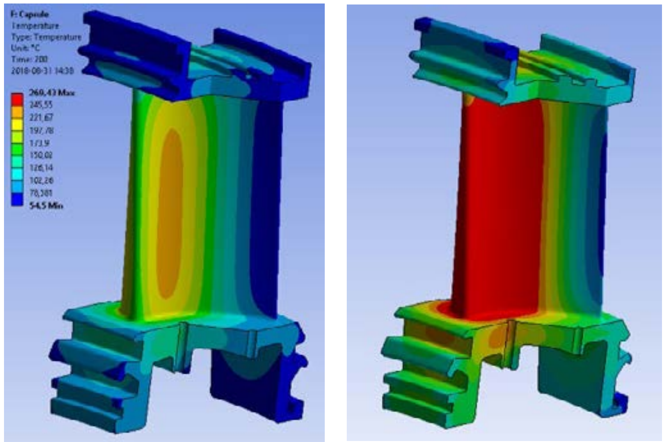

Figure 12. *Finite element modeling at 150MPa (left) and 50 MPa (right) showing the effect of pressure on the heat transfer coefficient, HTCα* [1]

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Cycle Flexibility in Modern HIP/HPHT™ Systems

The cooling rate can be steered and predicted, and specific segments can also be added to the HPHT™ recipe to change cooling gradients at given points. Cooling can even be halted at an intermediate temperature to add a dwell time before further cooling. Temperature and pressure can be controlled independently, adding to possibilities for tailored cycles.

So how fast can a modern HIP cool the payload? Smaller laboratory monolithic pressure vessels, even when they are equipped with cooling mechanisms, are limited to fairly low cooling rates of up to approximately 180°F/min (100°C/min) in the gas. As the pressure vessel volume increases, the ability to remove heat drops rapidly due to the increasing wall thickness of the pressure containment forging. Consequently, for larger production vessels, cooling rates are well below this. Wire-wound production HIPs are able to cool at rates in excess of 360°F/min (200°C/min) in the payload material itself, and smaller equipment focused on additive manufacturing can reach cooling rates in excess of 1440k°F/min (8000°C/min). So, production HIP equipment on par or faster with oil quenching is available in the market. This gives opportunities to quench material while under pressure in the HIP.

Hot isostatic presses come in all manner of sizes and configurations, from small laboratory machinery up to extremely large vessels. Currently the largest press in the world can be found at Metal Technology Co. Ltd. in Japan, with a hot zone of 6ft, 8in diameter (2.05m) and 13ft, 9in (4.2m height), (Figure 13). Larger presses are, of course, always of interest for even larger components, and the possibilities exist to produce much larger machinery should the need arise.

Figure 13. *The largest hot isostatic press in the world*

©Metal Technology Co. Ltd.

Reducing Discoloration

Due to the very high pressures used in the HIP process, an understanding of the partial pressure of gases is important. As an example, active gases even at low concentrations in the HIP vessel prior to the cycle, can give rise to a very high partial pressure when pressurized. This can lead to discoloration and surface oxides on parts in the payload if not handled correctly by the company performing the HIP operation.

Quintus has been working actively to reduce this issue by improving equipment and best practices in terms of HIP operation to reduce contamination. As a result, it is now possible to produce titanium parts with virtually alpha case free surfaces. The same is true for nickel base alloys where oxides are sometimes an issue due to high alloy content of elements with high affinity for oxygen, such as chromium, nickel, aluminium, titanium, and cobalt. Surface contamination can lead to negative effects on fatigue and corrosion resistance.

Therefore, modern-day practices provide great opportunities to reduce the amount of post processing after the hip cycle, thus saving significant sums of money.

Tailored HIP Cycles

The tailoring of HIP cycles to improve properties is a new area with great interest for many companies. Due to the excellent controllability of modern equipment, tailored heating, sustain and cooling cycles can be programmed for specific material properties. This is an area of extensive research and is especially applicable for materials needing high cooling rates or having an extremely tight cooling corridor.

An excellent example of tailored HIP cycles can be seen in recent work by Goel et.al at University West in Sweden which showed the possibility to reduce the standard twenty-one hour thermal treatment cycle for EBM produced IN718 by eleven hours with improved properties (Figure 14).

Process Route Optimization

Components that are under extreme stress in their applications, such as aero-engine turbine blades or industrial gas turbine blades, use HIP to increase fatigue life to ensure equipment safety and longevity. By combining HIP and heat treatment steps as described above, significant process route improvements can be made. Figure 15 shows an example of a CMSX-4 nickel base super alloy post treated with the traditional heat treatment route using vacuum furnaces (casting + homogenization heat treatment + solution heat treatment + 2-step aging) compared with a combined HPHT™ cycle. Re-growth of porosity after HIP is avoided by conducting heat treatment under pressure in the HIP. An advantageous fine γ /γ' microstructure was demonstrated, with excellent creep properties.

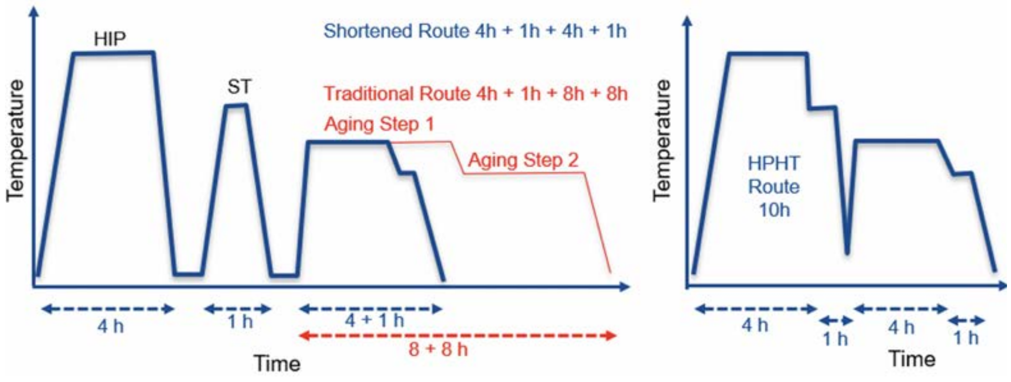

Figure 14. *Illustration of a traditional production route (red solid line) and shortened (blue solid line) post-treatments for EBM Alloy 718 compared with a full HPHT™ cycle, reducing cycle time by 50%.* [2]

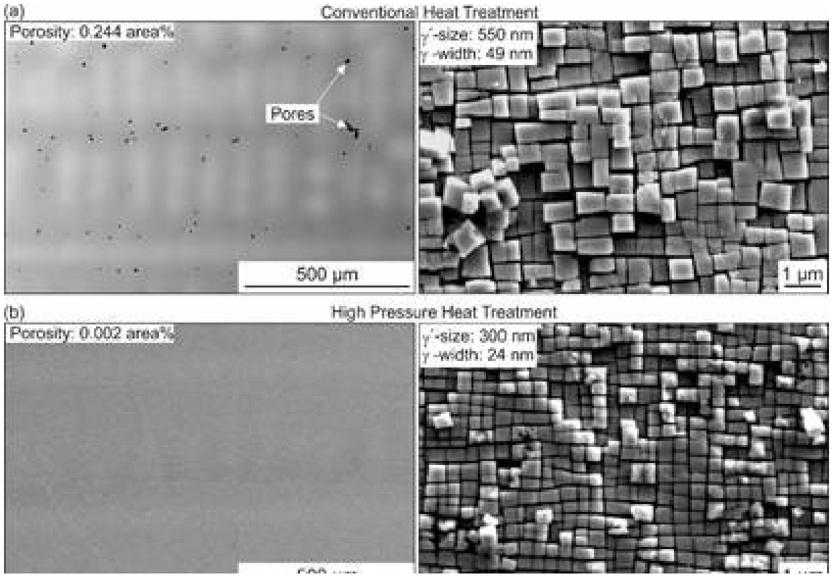

Figure 15. *Comparison of production routes using conventional heat treatment vs High Pressure Heat Treatment™ .* [4]

A Sustainable Future is Digital

Reduction of waste in the supply chain is an area of great interest for many companies. This can involve reducing material and electricity consumption, while increasing productivity and the automation of production lines. Hot isostatic pressing has an important role in all these areas with the introduction of machine digitalization of high-pressure heat treatment equipment. Integration of the equipment into digitalized production lines enables product and process improvements, including rationalization of flows and removal of some steps as well as improved monitoring and production planning. The data extracted from the production equipment, including the HIP, can be used to analyze and improve the production of the entire system from an overall perspective, facilitating lean manufacturing and machine learning.

By healing material rather than scrapping it, significant cost can be saved, while reducing the environmental impact due to yield losses and reproduction. The consolidation of thermal processing into one machine reduces the time spent at elevated temperatures, and as a result the overall electricity consumption can be reduced. This also has benefits for the microstructure of the material, avoiding unnecessary grain growth to improve mechanical properties.

Some companies have seen an opportunity to HIP their entire production demand to avoid yield losses. This approach maximizes the use of the HIP vessel and reduces the overall cost per component while ensuring the quality of the material. As a result, weld repair and inspection costs are reduced significantly.

Only surface-connected porosity becomes an issue as it can unfortunately not be healed using HIP (Figure 2).

Economic Benefits Can Drive Insourcing of HIP Technology

The heat treatment capability of modern HIP systems leads to a significant reduction in the production cycle time and thus, improved productivity of the supply chain. By insourcing the technology, savings can be made in handling and overall cost.

Figure 16. *Acetabular cup with typical dimensions 50 x 50 x 50 mm in Titanium Grade 5.*

Figure 16 shows a typical acetabular cup orthopedic implant produced in titanium grade 5 (Ti6Al4V) using additive manufacturing. Assuming that the components are not allowed to be in contact with each other during the HIP cycle, the number of load levels and parts per level can be calculated based on the design envelope. In this way, the number of parts and number of load baskets per cycle can be calculated. The HIP cycle time is affected by the heating rate, sustain time, and cooling rate, and the overall cycle time dictates the total productivity for the process.

For this study, the production volumes are based on annual production from a typical orthopedic implant producer, and two sets of HIP parameters have been compared. Time and costs involved in off-site HIP as well as goods-receipt inspection have been included in the service supply alternative.

Figure 17 shows potential cost savings in the range of 13 percent, corresponding well in excess of one million USD per year for the orthopedic implant producer.

Part	Acetabular cup
Material	Titanium Grade 5 (Ti6Al4V)
Service provider HIP	920°C, 100 MPa, 2 h (1688°F, 15,000 psi), Various HIPs
Inhouse HIP	820°C, 200 MPa, 2 h (1508°F, 30,000 psi), Quintus QIH48 MURC

Table 1. *Input to case study.*

Flexibility and Availability Can Drive Outsourcing

Many companies prefer to purchase HIP services from renowned service providers. There are a wide range of companies offering HIP and HPHT™ services today, making it possible for OEMs to utilize an ever-growing sub-supplier base to ensure flexibility, especially when expanding production.

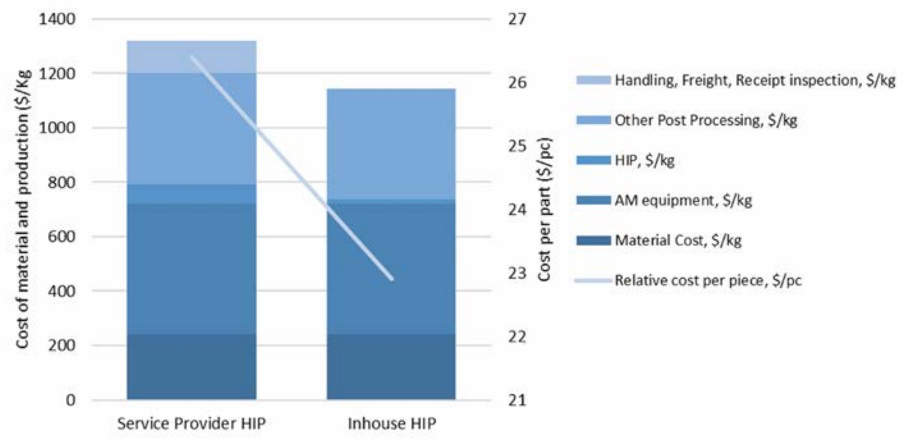

Figure 17. *Cost comparison for Titanium Grade 5 acetabular cup production using a typical HIP service provider vs in-house high-pressure heat treatment.*

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A ROBUST FUTURE

Additive manufacturing is now posing questions to the HIP community with respect to standardization. The majority of industrial standards used today were developed many years for wrought material. AM-produced material is well known for its fine microstructure, and from a metallurgical point of view, optimized standards for fine grained material would be beneficial for the implementation of improved designs. There are several international standards currently being worked on for additive manufacturing, although post processing is somewhat of an afterthought in some cases.

As production processes become more stable, improvements in the thermal processing routes will be needed to maximize the potential for different materials.

The development of AM has focused on full-density production prior to post processing and the porosity levels seen in castings are not tolerated. Using porous production with subsequent hot isostatic pressing has been around for many years for cast material; however, this has not yet been fully explored for processes such as AM or even MIM.

As for all production processes, lean manufacturing is key to improving product quality, minimizing costs, and maximizing productivity. Reducing waste and increasing throughput should always be a focus, but simply adding production capacity may not be the way forward. The addition of HIP with heat treatment capability as part of the production chain can facilitate robust and lean processes through reduction of yield losses, logistics, and quality related costs. Lean Additive Manufacturing™ is now becoming more of a discussion point.

Hot isostatic pressing is here to stay, and with many successful years ensuring safe service in critical applications, the doors to the future are wide open.

For more information, please contact
Jim Shipley
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