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Use of High-Pressure Heat Treatment (HPHT™) for L-PBF F357

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Abstract. Recent advancements in hot isostatic pressing (HIP) equipment now offer the ability to integrate HIP and heat treatment in the HIP furnace with the aid of controllable high-speed cooling and in-HIP quenching. This approach not only offers improvement in productivity but provides a path to prevent anomalies during heat treatment including thermally induced porosity (TIP) and part quench cracking or distortion. This manuscript will cover the approach of High Pressure Heat Treatment (HPHTTM) applied to SLM Solutions laser powder bed fusion (L-PBF) printed high strength aluminum alloy F357. Microstructure, tensile properties, and part distortion are evaluated. The results capture a post process method making it possible to prevent hydrogen blistering, mitigate defects present in the L-PBF material, offer strength properties exceeding that of MMPDS cast properties, and minimize geometric distortion of complex part geometries.

Introduction

High strength aluminum alloys are of great value to the aerospace industry and their market share reflects this with a revenue share of ~31% reported in 2019 [1]. The demand is due to the advantages offered such as excellent strength-to-weight and stiffness-to-weight ratios, good machinability and formability, corrosion resistance, and low-cost compared to more exotic materials. As a result, these alloys are of wide use for non-critical and structural applications offering improvements in fuel efficiency and reduction in weight of the aircraft [2].

F357, also commonly referred to as AlSi7Mg, is a high-strength aluminum alloy supporting this demand. Traditionally it's a cast precipitation hardenable Al-Si-Mg alloy that has been extensively studied. Its increase in Mg over AlSi10Mg offers improved balance in mechanical properties even at elevated temperatures. However recent development interest has been placed for its use in additive manufacturing (AM) techniques including L-PBF.

AM technology is expanding the design window providing the possibility to manufacture more innovative shapes and complex geometries not possible by conventional fabrication techniques. New sets of design freedoms are enabling lower production cost and part failure risk, improved performance including lightweighting, and lower material usage as part complexity increases. This is creating additional interest and use for high-strength aluminum alloys including F357 in the aerospace industry. In fact, AM as it has been marked with significant growth rates in recent decades, doubling the global AM market size every three years through 2023. And it's the aerospace industry that has been leading the charge in AM development for metallic non-critical and mission-critical components including aluminum alloys [3].

Despite these advantages and opportunities that exist there has been a slow adoption of AM high-strength aluminum alloys in the aerospace industry. This is largely due to the print defects inherent to the L-PBF process impacting part performance and reliability and lack of quality standards for AM components [3, 4].

Lack-of-fusion and keyhole defects are one of the most common print related anomalies present in AM processes. These defects can negatively impact microstructure and mechanical properties limiting structural integrity and use [4]. A method commonly used to minimize or eliminate such defects is hot isostatic pressing (HIP). However it has been reported in literature that hydrogen porosity and blistering is a common issue in aluminum castings and L-PBF components [5, 6, 7]. The hydrogen porosity and blistering present in castings can be managed with appropriate melt strategies. Due to the rapid solidification of L-PBF such strategies can't be applied, and it has been reported that the hydrogen supersaturated state in the solidified alloy is further enhanced. So despite the HIP process providing a fully dense structure, the need to perform conventional post-processing thermal treatments, such as the T6 solution anneal treatment at elevated temperatures, hydrogen pores may nucleate and grow [5]. These anomalies deteriorate the high density achieved by the HIP process and can negatively impact the microstructure and mechanical properties of the material.

An additional challenge associated with the heat treatment of AM high-strength aluminum alloys is often the requirement to quench from the solution anneal temperature in order to achieve the full age-hardening potential of the alloy. Ideally, the quench is faster than the critical cooling rate, but slow enough to avoid part distortion and even cracking. Often in practice this is not the case. Historically a water quench strategy has been applied to F357 castings resulting in optimum mechanical properties despite the propensity to distortion often requiring rework or scrap [8]. AM F357 is faced with the same challenge if there is a desire to meet or exceed mechanical property requirements of cast variants.

Due to the challenges highlighted above there has been a lack of consistency with post process heat treatment for high-strength aluminum alloys including F357. The benefits offered by HIP can be eliminated by hydrogen blistering. Also, water quench applied from the T6 heat treatment can lead to distortion. Performing a stress relief only heat treatment or direct age offers an approach to avoid both hydrogen blistering and distortion but doesn't take advantage of the age-hardening potential of the alloy, thus mitigating lightweighting potential.

Therefore, there exists a need in the industry to define a standardized and robust post-processing approach. Tocci et. al. [9] investigated the use of a novel heat treat strategy applied to AM and AlSi10Mg to achieve densification and hardening effects typical of T6 heat treatment in one step. The following work captures a case study carried out by University of Arizona, SLM Solutions, and Quintus Technologies expanding on this strategy, evaluating the application of HPHT in a Quintus Technologies' Uniform Rapid Quenching (URQ®) furnace for L-PBF F357. Density, microstructure, mechanical properties, and distortion of this novel approach are evaluated. Results capture a post processing method offering excellent tensile properties while avoiding thermally induced porosity and distortion.

High Pressure Heat Treatment – An Introduction

Quintus Technologies is a leader in high pressure technology and has introduced the next generation of HIP systems providing capabilities beyond conventional densification. Decade's worth of advancements in equipment design, system functionality, and control now offers a path to perform HIP and heat treatment in a combined cycle, referred to as HPHT^{TM}. This novel methodology provides a more sustainable processing route with improved productivity and energy efficiency. It also offers opportunities to further optimize microstructures for improvement in material properties coupled with ease of manufacturability.

Typical thermal processes performed on AM materials include HIP, homogenization, solution heat treatment, rapid air cooling or quenching, aging, and tempering. With Quintus' high strength wire-wound pressure vessels it is possible to perform operations all in the HIP. A thinner vessel forging is enabled by high strength wire winding. The vessel also incorporates cooling channels adjacent to the forging and wire-winding package for a more effective heat extraction from the system. The novel design continues internally to the pressure vessel with the ability to perform forced convection cooling, or the mixing of hot and cold process gases, with the aid of fans or ejectors. Such a system offers the ability to perform controllable Uniform Rapid Cooling (URC®) or URQ®. URC increases the natural cooling speed by up to a factor of 10-15, while stirring the gas so the cooling is more uniform in the hot zone. URQ is very fast cooling (>2000ºC/min gas rate with cycle load down to 600ºC), allowing quenching of most materials in the same or higher rate as commercial oil quench equipment. Quintus delivers both techniques with the option of controlled cooling. An additional benefit of the highly pressurized gas involved in the cooling segment produces an entire production load that will receive more or less the same cooling rates. Originally introduced for productivity improvement, these technologies now offer the cooling rates, accuracy, and uniformity to perform many of these post processing steps all in a HIP vessel.

There are many reported benefits by applying the combined HPHT route such as reduced number of process steps, reduced cycle time and lead time, and improved process and quality control. Other advantages include spending less time at elevated temperatures helping to preserve the fine grain AM microstructure by minimizing grain growth. It has also been shown that by carrying out all elevated temperature process steps under pressure thermally induced porosity can be prevented or mitigated. Finally, manufacturability can be improved through HPHT as this approach reduces the cooling or quench severity during cooling segments which can often lead to part distortion or cracking. The lower temperature differential between the components and gas temperature compared to conventional approaches and application of high pressure both aid in lowering thermal gradients and stresses with a component.

Materials and Methods

F357 cylindrical samples were printed by SLM Solutions. The machine used for this project was an SLM 280 2.0 with a build envelop of 280 mm X 280 mm X 365 mm (11.0 in X 11.0 in x 14.4in) using standard print parameters including 30-micron layer thickness. The machine was equipped with a twin laser system of 400 watts each. In this study, as-built, stress relief only, and five HIP variants were investigated, see Table 1 for a summary of stress relief and HIP variant conditions. All heat treated samples were stress-relieved at 285ºC (545ºF) for 2 hours and cooled at a rate equal to air cooling or faster. For the baseline variant, HIP was performed at 515ºC (959ºF) at 100 MPa (14.5 ksi) for 3 hours with a slow cool (10ºC/min(18ºF/min)) followed by a standard T6. The standard T6 consisted of a solution heat treatment at 530ºC (986ºF) for 6 hours in air followed by a water quench and an age cycle at 160ºC (320ºF) for 6 hours. For variants 4.1-4.4 all HIP cycles represented a HPHT strategy by combining the HIP, solution heat treatment, and quench all in the HIP vessel under high pressure with the use of the URQ furnace. Temperature and pressure were modified for these variants targeting 500ºC (932ºF)/530ºC (986ºF) and 100 MPa (14.5 ksi)/170 MPa (24.7 ksi). The lower temperature HPHT variants performed at 500ºC (932ºF) was evaluated to assess the impact HIPing at lower temperatures had on maintaining a finer AM microstructure, whereas the high-temperature variants performed at 530ºC (986ºF) was evaluated to assess the impact on achieving an optimum supersaturated solid solution state of hardeners. The high-pressure variants performed at 170 MPa (24.7 ksi) were assessed to evaluate the influence increased cooling power offered with the higher pressure and density gas medium.

Table 1. HIP variants, all samples were stress-relieved at 285°C (545°F) for 2 hours and cooled at a rate equal to air cooling or faster.

Variant	HIP Parameters	T6/Age Specifics
Baseline	515°C/100MPa/3 Hrs + Slow Cool	Standard T6, solution heat treat in air furnace, 530°C/6hrs + Age cycle
4.1	530°C/100MPa/3 Hrs + URQ	Age cycle only, 160°C/6hrs
4.2	500°C/100MPa/3 Hrs + URQ	Age cycle only, 160°C/6hrs
4.3	530°C/170MPa/3 Hrs + URQ	Age cycle only, 160°C/6hrs
4.4	500°C/170MPa/3 Hrs + URQ	Age cycle only, 160°C/6hrs

Results

The microstructure for each variant was evaluated. In general variants 4.1-4.4 showed similar microstructures with respect to shape and size distribution of the eutectic silicon. However, variants 4.1-4.4 showed a finer distribution of eutectic silicon relative to the Baseline variant. Micro photos are provided in Fig. 1 for the Baseline and 4.3 variant.

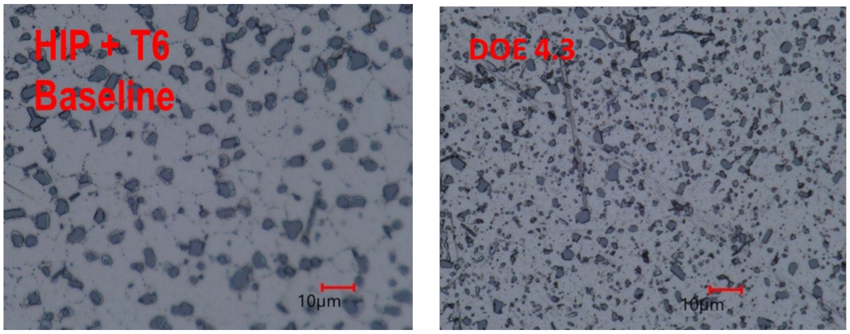

Figure 1. Micro photos of Baseline and 4.3 variants.

ASTM E8 standard tensile coupons were machined and room temperature tensile properties were evaluated for the as-built, stress relief only, as well as the five HIP variants. Results for the 0.2% yield strength (YS) and ultimate tensile strength (UTS) were compared to Metallic Materials Properties Development and Standardization (MMPDS) Cast A357/F357 property data [10] as a benchmark with a summary provided in Fig. 2. Similar trends are observed for YS and UTS. HIP variants 4.1-4.4 display a significant advantage over the as-built and stress relief only conditions, with Baseline outperforming all variants. HIP variants 4.1-4.4 exceeded the benchmark value, with HIP variants 4.1 and 4.3 showing improved strength over the other HPHT variants due to the higher temperature soak segment in the HIP vessel. Elongation for Variants 4.1-4.4 was greater than 15%, equivalent to or better than the Baseline variant. Overall HIP Variants 4.1-4.4 specimen showed no evidence of blistering and negligible distortion relative to water quench samples.

Distortion for HIP Variant 4.1 HPHT cycle was evaluated on a L-PBF F357 trial part shown to be distortion sensitive to the standard T6 water quench. Normalized distortion results are summarized in Fig. 3 for two locations prone to distortion. Overall, the stress-relieved and HIP conditions show little to no increase in dimensional deviation. The as-HT material, comparable to the Baseline variant in this study that utilized a water quench after the solution treatment, shows a significant increase in dimensional deviation. However, the HIP Variant 4.1 mitigated distortion with results similar to stress relieved or As-HIP conditions.

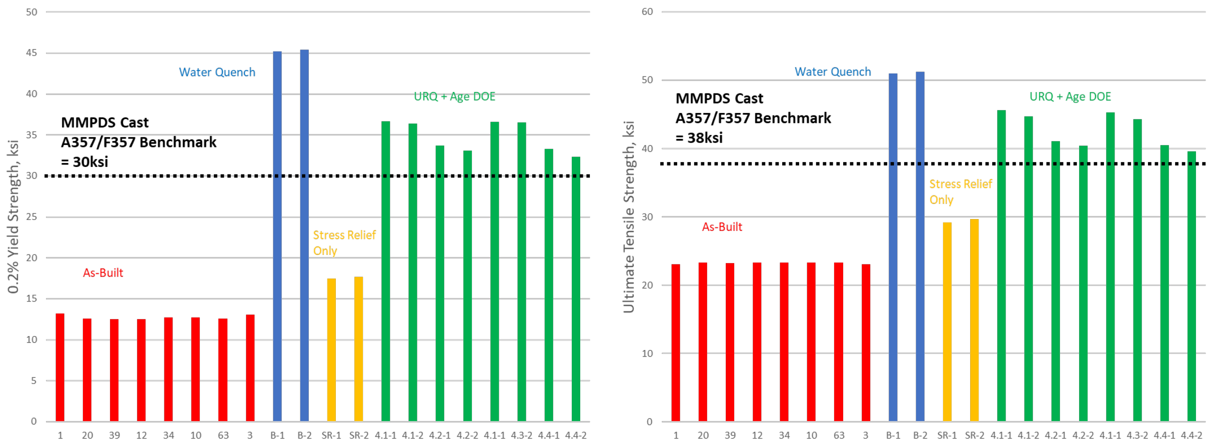

Figure 2. 0.2% YS and UTS results for "As-Built" (sample ID 1, 20, 39, 12, 34, 10, 63, 3), "Water Quench" (sample ID B-1, B-2), "Stress Relief Only" (sample ID SR-1, SR-2), and "URQ + Age DoE" consisting of the URQ HIP variant results compared to MMPDS Cast A357/F357.

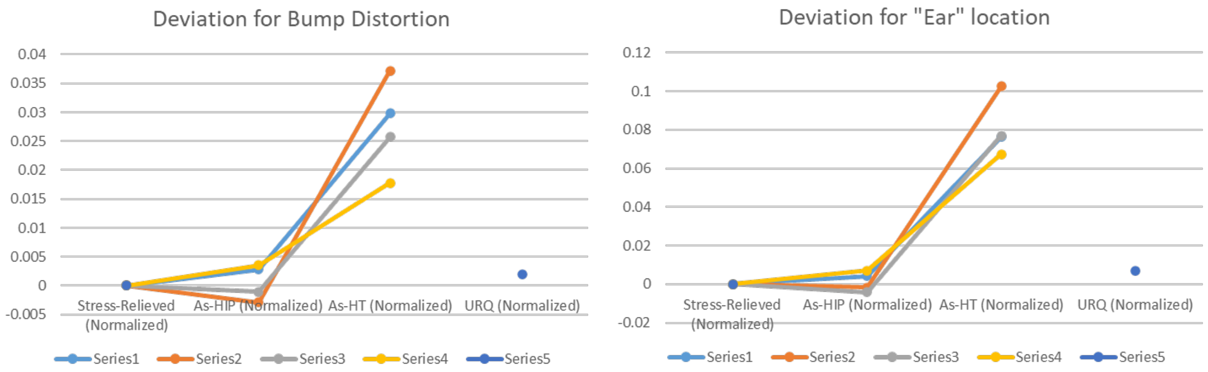

Figure 3. Distortion data at two locations for a distortion-sensitive L-PBF F357 trial part for multiple post-processing conditions.

Conclusions and Future Work

The recent advancements in modern HIP design and equipment are now providing the opportunity to perform HPHT with aid of high-pressure rapid cooling and quenching. The added variable of pressure creates novel approaches to combine HIP and heat treat all in the HIP vessel. This method not only eliminates a process step(s) but also has the potential to remove defects and optimize the microstructure for improved mechanical properties. In this work HPHT was applied to high-strength precipitation-hardenable aluminum alloy F357 produced by L-PBF. These results capture a HPHT processing route applied to L-PBF F357 offering material with no observable defects, strength values exceeding MMPDS cast F357/A357 benchmark properties with excellent ductility, and negligible distortion of a quench sensitive part.

Future work will include post-process optimization studies with a focus on the HIP/solution segment, material characterization of the silicon particle size and size distribution, and ultrasonic low cycle fatigue and high cycle fatigue screening.

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