Novel heat treatment methodology for residual stress relieve of AlSi10Mg without compromise of mechanical strength

Lim, Chong Heng

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FOR RESIDUAL STRESS RELIEVE OF AlSi10Mg
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SCHOOL OF MECHANICAL AND AEROSPACE ENGINEERING

**A thesis submitted to the Nanyang Technological University in partial
fulfilments of the requirements for the degree of Doctor of Philosophy**

2020

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research, is free of plagiarised materials, and has not been submitted for a higher degree to any other University or Institution.

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Lim Chong Heng

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I have reviewed the content and presentation style of this thesis and declare it is free of plagiarism and of sufficient grammatical clarity to be examined. To the best of my knowledge, the research and writing are those of the candidate except as acknowledged in the Author Attribution Statement. I confirm that the investigations were conducted in accord with the ethics policies and integrity standards of Nanyang Technological University and that the research data are presented honestly and without prejudice.

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Assoc. Prof. Li Hua

Author Attribution Statement

This thesis contains materials from one paper accepted at a conference in which I am listed as an author. The thesis also contains materials for two papers under preparation.

Chapter 2 is published partially as Lim, C. H., Krishnan, M., & Li, H. (2018). Methods of residual stress reduction for metal parts manufactured using selective laser melting (a review). Proceedings of the 3rd International Conference on Progress in Additive Manufacturing (Pro-AM 2018), 315-320. doi:10.25341/D4901Z

The contributions of the co-authors are as follows:

- Lim, C. H. conducted the literature review under the guidance of Ph.D. supervisor Li, H., and co-supervisor Krishnan, M.
- Li, H. and Krishnan, M. provided the comments on the manuscript and edited the manuscript drafts.

Materials in Chapter 3 and Chapter 4 are currently in preparation for publishing as Lim, C. H., Li, H., Krishnan, M., Novel method for residual stress reduction in AlSi10Mg parts manufactured by selective laser melting without compromising mechanical properties.

The contributions of the co-authors are as follows:

- Lim, C. H. designed and conducted the experiments under the guidance of Ph.D. supervisor Li, H., and co-supervisor Krishnan, M.
- Lim, C. H. analyzed the data and proposed the model for residual stress reduction
- Li, H. and Krishnan, M. provided the comments on the manuscript and edited the manuscript drafts.

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The contributions of the co-authors are as follows:

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- Lim, C. H. analyzed the data and made the observations.
- Krishnan, M. and Li, H. provided the comments on the manuscript and
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Lim Chong Heng

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Abstract

Additive manufacturing (AM) has made the fabrication of complex parts possible which otherwise, would not have been possible with conventional subtractive manufacturing. Laser Beam Powder Bed (LBPB) Fusion, commonly known as Selective Laser Melting (SLM), is one of the methodologies in AM for metal fabrication. Many types of research have been done pertaining to fabricating functional parts via LBPB methodology, and one of the challenges faced is the residual stresses that reside in the metal parts due to repeated uneven heating and cooling process. Residual stresses can cause the LBPB process to fail by dimensional warping of the parts, and also compromise the functionality of the final product especially if there is tensional stress on the surface, introducing the risk of premature failure below its operational load. AlSi10Mg is a great strength-to-weight ratio alloy that is commonly manufactured by the LBPB process. It is an excellent material suitable for fabrication via the LBPB process, as it has proven to have superior mechanical properties as compared to that made by the conventional cast method. The current conventional method of relieving the residual stresses of LBPB manufactured AlSi10Mg involves heat treatment to 300°C. However, AlSi10Mg manufactured by the LBPB process is much more sensitive to heat treatment as compared to conventional cast AlSi10Mg, resulting in a drastic reduction in mechanical strength after heat treatment. Performing heat treatment at a lower temperature will result in less reduction in the mechanical strength of the material, but the residual stress will not be completely relieved. Hence, the current industrial method of residual stress relief is achieved at a compromise of its mechanical property, which poses an engineering dilemma.

This thesis presents a novel method of residual stress relief of LBPB manufactured AlSi10Mg without any compromise of its compressive strength, discussed in section 3.1.2. and the results verified in Chapter 4. Also, two novel phenomena were

observed in the study of microstructural characterization of the material in sections 5.4 and 5.5 respectively.

The novel residual stress relief methodology involves uneven air cooling after heat treatment at a relatively lower temperature, therefore inducing thermal residual stress during the cooling process, such that it cancels out the existing residual stress that remains after the heat treatment. This method successfully relieved the residual stress without compromising the compressive strength of the part. The effect of heat treatment temperature on the compressive strength of the part is justified via scanning electron microscopy (SEM) images.

A novel phenomenon was observed on the trend that low angle grain boundaries (LAGB) are found to be concentrated in grain orientated towards $\langle 001 \rangle$ direction. Semi-transparent grain orientation map superimposed on grain boundaries map reveals this trend via visual observation.

Another novel phenomenon was observed of the relationship between LAGB density and the magnitude of residual stress relief was made in which both parameters show a similar trend at different treatment temperatures. This further substantiates the nature of the new methodology of stress relief where a larger induced residual stress results in an increase in dislocation, and therefore an increase in LAGB.

General observation of the grain orientation map, and precipitation distribution of intermetallic compounds are also discussed.

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Abbreviations

3D	3-Dimensional
AM	Additive Manufacturing
ASTM	American Society for Testing and Materials
CHD	Central Hole Drilling
CAD	Computer-Aided Design
DFAM	Design for Additive Manufacturing
EBM	Electron Beam Melting
EDS	Energy Dispersive X-ray Spectroscopy
EBSD	Electron Backscatter Diffraction
EDM	Electrical Discharge Machining
FGM	Functionally Graded Materials
FEMM	Finite Element Method Magnetics
HIP	Hot Isostatic Pressing
IN718	Inconel 718
IPF	Inverse Pole Figure
LBPB	Laser Beam Powder Bed
LFMT	Low Frequency Alternating Magnetic Treatment
MS1	Maraging Steel Grade 300

MIG	Metal Inert Gas
PMQ	Permanent Magnet Quadrupoles
STA	Solution Treatment and Aging
SLM	Selective Laser Melting
SEM	Scanning Electron Microscope
XRD	X-Ray Diffraction
LAGB	Low Angle Grain Boundaries
BH-UAC	Bottom Heated-Uneven Air Cooling
FH-SC	Furnace Heated-Slow Cooling
FH-UAC	Furnace Heated-Uneven Air Cooling

Chapter 1. Introduction

1.1. Background and Objective

Residual stress exists in materials that underwent processes involving heat followed by uncontrolled cooling, such as the welding process. The existence of residual stress in manufactured parts can result in premature failure even when subjected to loads within its design limits. With the increase in popularity of additive manufacturing in the recent years, the problem of residual stress becomes an even more serious issue as it can cause dimensional warping if not addressed properly. Many different methods have been developed to address the problems associated with residual stress in additive manufacturing process, some of which are unique to a certain process or material, while some methods are universal. This study aims to investigate a method currently used in the industry to relieve residual stress associated with AlSi10Mg alloy manufactured by additive manufacturing, particularly laser beam powder bed process, identify the problems associated with the method, and to develop a new process to address the problems. The stress relief capability of the new method and its effect on mechanical property will be investigated experimentally, and verified with microstructural characterization.

1.2. Laser Beam Powder Bed Additive Manufacturing Process

Additive Manufacturing (AM), previously known as rapid prototyping, is defined by the American Society for Testing and Materials (ASTM) as "a process of joining materials to make objects from 3-dimensional (3D) model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies" [1]. As illustrated in Figure 1, traditional methods of manufacturing such as milling, drilling, laser cutting, and grinding, are subtractive in nature and involve machining a larger block of material into the desired shape and size. Additive manufacturing creates a solid shape by adding a new layer of material on top of the preceding block. Common terms used

to describe this process include 3D printing, rapid prototyping, direct computer-aided design (CAD) manufacturing, desktop manufacturing, instant manufacturing, CAD-oriented manufacturing, layer manufacturing, material deposit manufacturing, material addition manufacturing, material increment manufacturing, and solid freeform manufacturing [2].

Figure 1: Illustration of subtractive and additive manufacturing [2]

Benefits of AM over traditional manufacturing processes include resource savings with high “buy to fly” ratios, allows design optimization as complex parts can now be manufactured, and reduced use of toxic chemical uses [3]. AM processes can be classified according to the starting material used, as liquid-based, solid-based, and powder-based system [2]. Laser beam powder bed (LBPB) additive manufacturing, also commonly known as selective laser melting (SLM), is one of the techniques under powder based systems that are capable of producing end-usable metal parts with complex geometries. By the series of melting of metallic powder followed by solidification of the melted pool, metal components build up layer by layer forming the complete part [4]. The use of metal powders in LBPB technique is of great interest in many industries, such as aluminum [3] and titanium alloys [5] in aerospace, alpha-beta titanium alloy (Ti6Al4V) [6], and stainless steel 17-4 [7] in biomedical, and cobalt-chromium (Co-Cr) in dental [8]. However, not all types of materials are suitable to produce end-usable metal parts by LBPB technique as different material suffer from different amount of residual stress [9].

1.3. AlSi10Mg

AlSi10Mg has been manufactured traditionally by casting for various applications, such as heat sink, manifolds, and engine parts [10]. Also commonly known as A360 alloy and well known for its high strength to weight ratio. The first successful application of AlSi10Mg in LBPB was done in 2010 [11], spinning off further research of this methodology for the fabrication of this material. Many types of research focused on optimizing parameters for the LBPB process, and problems encountered include high reflectivity of aluminium alloys causing heat loss [3, 12] and low flowability of powders that affects the consistency of powder layer during the LBPB process [11]. To date, manufacturers of LBPB machines have managed to develop their own set of parameters capable of manufacturing full density and functional parts with superior mechanical properties. The advantages of adopting additive manufacturing for the production of this material is not just limited to the general advantages of additive manufacturing over conventional methodologies such as casting, but the material manufactured by additive manufacturing has shown superior mechanical properties over its conventional counterparts [13]. The introduction of additive manufacturing and the benefits of this technology have made the production of AlSi10Mg via this methodology an attractive option.

1.4. Residual Stress

Residual stress is defined as stresses that remain in a structure or part even after the removal of externally applied loads [14]. Residual stress is not necessary to maintain equilibrium between the body and the environment [15], as they are in equilibrium balanced by a combination of tensile and compressive stress within the body. The Heyn spring model shown in Figure 2 below illustrates the balance of compressive and tensile residual stresses within a system [16].

Figure 2: Heyn spring model of a balanced residual stress system [16]

Residual stress can be classified under Type I, Type II, and Type III [17-19]. Type I represents the macro residual stress on the continuum level without consideration of the micro-level. Type II represents the micro residual stress on the grain scale level, while type III refers to residual stresses on the atomic scale. Type I residual stress is of more significant importance in consideration of material's strength, as compared to Type II and III [20]. Also from a macro fatigue point of view, the effects of Type II and III residual stresses tend to be washed out by the plastic deformation in the crack tip area, making Type I residual stress the most important factor of consideration [15]. Residual stress readings measured from a common method such as X-ray diffraction (XRD), is a combination of both Type I and II residual stress, while Type III residual stress is relatively much lower than Type I and II [18].

While residual stress in a structure exists as a combination of tensional and compressional stresses at different regions [21], tensional stress at the surface or subsurface would pose a higher risk of fatigue failure as compared to if it was compressional stress. This is because the surfaces of structures are usually subjected to the largest amount of stress when subjected to bending load and cyclic loading. Tensional residual stress on the surface is one of the main causes of initiation and propagation of cracks in weldment [22] and thus, many studies on residual stresses are targeted in the surface or sub-surface region.

While many types of research have been carried out to eliminate or reduce residual stresses in manufactured parts, residual stress itself can be used to improve the mechanical properties of materials. The fatigue life of materials can be enhanced by

inducing compressive residual stress on critical regions on the surface and subsurface [23-26]. Inducing compressive stress on the surface of glass and ceramics help to increase the strength of the material [15]. Although the induced compressive stress on the surface will also result in tension stress in the middle core region, these regions are relatively defect-free as compared to the top surface, and initiation of crack growth is much less likely to occur from the center, as compared to from the surface.

The formation of residual stresses was classified under 3 types of deformation in Griffith's book: thermal deformation, mechanical deformation, and combined thermal and plastic deformation [27]. The residual stresses that develop on the surface of a part due to thermal load such as welding tend to be tensile, while compressive residual stresses generally develop for mechanical loadings, such as milling or cutting [28]. However, if mechanical loading causes microstructural changes due to an increase in temperature, the residual stress on the surface could be either compressive or tensile depending on the material used and the machining conditions [29]. Studies have also shown that thermal-induced stresses on the surface of aluminum alloys due to rapid cooling after heat treatment tend to be compressive [30, 31].

1.5. Thesis Overview

Despite the advantages of the LBPB process, residual stress is a by-product of the additive manufacturing process which can cause the final part to fail if not controlled. While existing heat treatment methods are able to relieve residual stresses in LBPB manufactured parts, it also results in a deterioration in the mechanical properties in AlSi10Mg [32]. A technological gap exists where a new type of treatment methodology is required to relieve the residual stress without compromising the mechanical properties.

This thesis focuses on the development of a novel method of relieving undesired surface residual stress of AlSi10Mg manufactured by the LBPB process. Chapter 2 covers the literature review of residual stress development in LBPB, the hindrance of residual stress on LBPB manufactured parts in terms of functionality, and methods of relieving residual stresses. The relation between microstructure and mechanical properties of AlSi10Mg is also explored and different methods of residual stress characterization. 0 presents a novel methodology of heat treating LBPB manufactured AlSi10Mg that requires not just a lower temperature than the conventional method, but also preserving the mechanical strength that would have been compromised by conventional methods. Some other discontinued experimental work performed by the author that produced small residual stress relief will also be presented. Chapter 4 presents the experimental results that validate the concept proposed in 0. Microstructural characterization, including novel phenomena observations, are presented in Chapter 5.

Chapter 2. Literature Review

2.1. Residual Stress in Laser Beam Powder Bed Process

In the LBPB process, dimensional distortion is a common problem whereby printed parts tend to warp upon cooling if the print parameters were not set well, support structures are not designed well or not positioned at critical area [33], or the design of a component to be printed is not suitable for the LBPB technique.

Figure 3: Delamination of LBPB printed part due to residual stress induced distortion [34]

Experimental results from the LBPB process show a reduction in residual stress by more than 50% when a completed SS316L part is removed from its base plate [20], suggesting the possibility of a large dimensional distortion upon stress relief. During the LBPB process, rapid heating, melting, followed by solidification results in thermal gradients across the profile of the part, inducing uneven expansion and contraction at different regions and warping to occur [20, 35, 36]. As described by Mercelis [20], the mechanism of residual stress induction involves compressive stress induced on the top surface during heating and tensile stress during cooling as illustrated in Figure 4.

Figure 4: Compressive residual stress induced at heated region when exposed to heat source, and tensile residual stress when cooled [20]

The author Liu [37] explained the mechanism of residual stress development during the LBPB process as illustrated in Figure 5 [37], explaining in terms of the interaction of bulk material between 3 regions - Region I (melted zone), Region II (heat affected zone), and Region III (non-heat affected zone).

Figure 5: Residual stress development on surface and sub-surface region of parts during the LBPB process [37]

Stage 1 denotes the stage when the laser irradiates a point of the part, causing melting of the powder in the region and hence zero residual stress in Region I. Region II sees a rise in temperature due to the heat from the laser and expands while restrained by Region III, resulting in compressive stress in Region II and tensile stress in Region III. Stage 2 denotes the stage when the laser moves away from the targeted point, causing immediate rapid cooling in the material at the specific region. Region I solidifies and undergoes rapid cooling and contraction while being restrained by Region II. This induces tensile stress on Region I, and even more compressive stress on Region II. The increase in compressive stress in Region II also induces compressive stress in Region III, causing the tensile stress in Region III to reduce. At stage 3, the top surface Region I continues to cool even further and the tensile residual stress increases. Likewise, the contracting effect due to Region I induces more compressive stress in Region II, and the residual stress in Region III eventually becomes compressive as well. However, this residual stress distribution in stage 3 does not reflect the actual residual stress nature of parts that have been removed from the base plate as the residual stress. Depending on the material property and residual stress magnitude and distribution, removal of support structures and base plates can

result in minor or major dimensional changes in the parts, causing residual stress to redistribute throughout the whole body.

The basis of the mechanism of residual stress development by Liu involves restriction in expansion and contraction and has been modelled in several studies. A mathematical model by Mercelis successfully predicted the residual stress profile verified by experimental measurement, with residual stress induction due to shrinkage of the top upper layer [20].

The issue of residual stress in the manufacturing industry is often associated with the welding process. The cause of residual stress development during the LBPB is similar to that developed during welding, whereby a steep temperature gradient develops due to rapid heating from the energy source and slow heat conduction [20]. When a concentrated energy source melts the solid materials, a liquefied melt pool forms which have a higher heat transfer rate as compared to the surrounding solid-state parts due to convective heat transfer [38-40]. When the heat source leaves the melt pool, the high heat transfer from the melt pool allows it to solidify rapidly. The steep temperature gradient due to uneven heating causes plastic straining at different regions due to uneven expansion or contraction rates. The similarity in the formation of residual stress during welding as compared to the LBPB technique meant that researches based on welding could be used as a foundation to our understanding of residual stress in the LBPB processes.

Figure 6: Similarity in concentrated heat scan between (a) Welding [41], (b) LBPB process [42]

Studies have been done on using AM on welding [43], whereby the stress distribution is found to be similar to that produced in welded metal parts [44] with the deposited region in the heat-affected zone experience tensional residual stress, while surrounded by regions experiencing compressional residual stress. However, for the LBPB process, melting and solidification take place line by line over the entire surface of the part or structure being manufactured, while the melting and solidification in the weld medium take place linearly along a line on the parent material. The relatively complicated process of the LBPB thus results in a more complicated mechanism that develops a different distribution of residual stress as compared to welding.

2.1.1. Limitation of Laser Beam Powder Bed Applications due to Residual Stress

Although to date, AM supports the manufacture of a wide range of materials such as aluminum alloys [45], tool steels [46], cobalt alloys [47], copper [48], and Inconel alloys [49], the coverage of the printing capability is still limited. Excessive residual stress is one of the limiting factors for the limited adoption of existing metal alloys in the LBPB process [50]. As different alloys have different mechanical and thermal properties, the unique heat distribution and thermal cooling rate mean their relative susceptibility to thermal distortion is different as well. The cooling rate of the melted powder after being exposed to the energy source during AM affects the microstructure that forms during solidification, and a cooling rate of 7×10^7 K/s is often considered to be achieved in the LBPB process [51]. Zhang described the restriction of development and application of the LBPB processed parts to "residual stress, defects, and columnar grains with anisotropy" [52]. Studies have been done to investigate the "printability" of existing alloys based on common LBPB parameters [9], and the residual stress that develops in each alloy is an important factor. Research has been done to reduce residual stress in the LBPB produced parts such as

alpha-beta titanium alloy (Ti6Al4V) and Inconel 718 [53] to address the dimensional distortion and poor mechanical properties that can render the resulting parts unsuitable for operation [54]. When the residual stress exceeds the yield limit of the material, plastic deformation can occur [55], resulting in a poorer mechanical property causing premature failure below its operating load [56].

2.1.2. Reduction of Residual Stress

The methods to reduce welding residual stresses are classified by Berezhnyts'ka [57] into 2 categories: methods to prevent the formation of residual stress, and methods to relieve residual stresses in finished parts. Preventing the formation of residual stress involves the regulation of temperature field during or before fabrication by intense removal of heat, concentrated heating, or preliminary heat. The residual stress relief approach involves the regulation of temperature and application of external force on the finished parts. The category of methods of preventing the formation of residual stress can be further branched out into another 2 groups - pre-processing and in-situ prevention. The categorization of residual stress reduction methods is illustrated in Figure 7.

Figure 7: Categorization of residual stress reduction methods

Pre-processing planning involves the pre-fabrication stage of the LBPB process, involving the design of built parts and settings parameters before the start of the LBPB process. In-situ prevention involves using optimum parameters and

methodologies via the design of experiment approach [58]. during the melting process itself. Relieving residual stress in finished parts or post-treatment methods are used to improve the quality of the final parts, by reducing the residual stress that has been induced during the LBPB process.

2.1.2.1. Pre-Processing Planning - Design of dimensions and Orientation of Built Parts and Plate

Due to the complexity of each type of AM process, the design for additive manufacturing (DFAM) approach helps to the optimized geometry of the component. DFAM is used in the creation of shapes, sizes, and geometric mesostructures, with material compositions and microstructures to best utilize AM technologies, so as to achieve desired material performance [59]. DFAM tools using finite element simulation such as NX by Siemens [60], have the capabilities of predicting residual stress for specific LBPB conditions and parameters, thus providing potential for reducing residual stress by optimizing the parameters. The accuracy of some models developed by researchers was verified by experimental results, such as in SS316L [61], Inconel 718 [62], and alpha-beta titanium alloy (Ti6Al4V) [63] in terms of residual stress profile with depth and stress-induced deformation. However, some hypothetical AM conditions such as localized preheating [64] were modeled but not yet verified experimentally. The dimension of the part being manufactured affects the residual stress that will develop. In the welding of two plates, the induced residual stress in thin plates is lower than that of thick plates [65]. In the LBPB process, fabrication of a taller part will result in larger residual stress as compared to a shorter part for SS316L [20] and Maraging Steel Grade 300 [66]. The heat transfer rate from the scanned surface of a fabricated part affects the thermal gradient and residual stress formation. Thicker build plates are more efficient in heat transfer and lower residual stress in built parts [20, 66]. Hence, materials that are more prone to residual stress require the use of a thicker build plate, such as Inconel 718 and Inconel 625.

2.1.2.2. Pre-Processing Planning - Design of support structures

Support structures are required for some alloys in the LBPB process to prevent dimensional distortion due to residual stress concentration, and thus keep the parts firmly attached to the build platform throughout the LBPB process [67]. Metallic support structures improve heat transfer between the metal part and base plates [33], reducing the thermal gradient across the profile and reduces residual stress. However, if the residual stress is large enough to cause dimensional distortion, the presence of support structures helps to prevent such distortions but will result in higher residual stress in the final build part [20, 40, 65, 68-70]. Although there is commercial software available for designing support structures, such as Magics by Materialise [71], they are mostly developed based on supports for the critical angle of overhang features and not designed to cater to warping due to stress [33]. Hence, further research is required to integrate the consideration of residual stress with the use of commercial software.

Figure 8: Dimensional distortion occurs when overhang angle is lower than the critical angle [72]

Overhang features parallel to the powder bed surface require the most support [73] where the overhang angle is zero. Additional post-processing machining required to remove the supports from the manufactured part might induce further residual stress into the part [29]. The use of low melting point or soluble materials as support simplifies the post-processing work required; however, it is currently not applicable to the metallic LBPB process. To reduce the time required for support structure removal, pulsed laser instead of continuous scan could be used for printing support

structures [74], which reduces the density support structure produced [36]. Benchmarking was done to relate residual stress resulted warping with different types of overhang features [75], and attempts to optimize the number of support structures were carried out [33]. The stress distribution of a printed part due to the dimensional restraints of support structures or based plate was found to be tensional at both the top and bottom surfaces where the restraints from support were acting [20], which can affect the fatigue lifespan of the part.

2.1.2.3. Pre-Processing Planning - Preheating of powder-bed

Preheating is the most common residual stress relief method in welding processes. Standards for preheating of different material and welding types have been adopted by industries [76]. One explanation is the diffusional relaxation of the materials due to preheating allows the relief of residual stresses [77]. Also, preheated steel materials with elevated surrounding temperature reduce the thermal gradient between the weld seam and its surrounding, thus reduces the cooling rate during the welding process [65, 78, 79]. Similarly, preheating the powder-bed in AM before printing helps to lower the thermal gradient between the laser exposed region and the rest of the powder bed. An experimental result from the LBPB process shows a reduction of residual stress by approximately 8 to 17% for the SS316L part with a preheat temperature of 200°C [20].

Commercially available LBPB machines can offer a limited preheat temperature up to 200°C [80, 81], while electron beam melting (EBM) can achieve as high as 800°C by defocused electron exposure between each scan [82]. Also, for large build parts on LBPB, it is not possible to maintain uniform heating throughout the build plate. Simulation of localized preheating of the LBPB process shows a reduction in residual stress by 18% [64].

2.1.2.4. Pre-Processing Planning - Maintaining a Semi-Solid State during Additive Manufacturing Process

A possible method applicable to certain metallic alloys was inspired by a process developed for polymers. Given that rapid solidification is one of the causes of residual stress development in the AM process, a novel method to reduce the rate of solidification was explored by making use of the different melting point and freezing point in supercooling polymers [83]. Nylon 12 and 13 have a unique property whereby the melting point is higher than the freezing temperature [84, 85]. The powder bed is preheated and maintained at a temperature between its solidification and melting point, thus any regions exposed to the laser get melted but do not solidify completely. Holding the printed part in a semi-solid state throughout the process results in reduced residual stress.

However, this method requires the material to exhibit two different temperatures required for melting and solidification, and the ability of the AM machine to preheat the powder bed to a temperature high enough to maintain the powder in a semi-solid state. EBM will be more suitable as compared to LBPB in terms of powder bed preheat capability. To reduce residual stress using this concept on metallic alloy, a lower solidification and melting temperature are preferred which corresponds to the eutectic composition [86] saving the energy required [87]. Eutectic Al-Si alloy without support structures was originally prone to warpage at overhung region due to residual stress build-up, but was successfully manufactured by LBPB without the use of support structures using this methodology as shown in Figure 9 [88, 89].

Figure 9: Successful fabrication of AlSi12 parts with Al and Si12 powder using LBPB without the use of support structures [88]

However, alloys with eutectic composition have relatively low melting point [90] and might not be suitable for high temperature applications.

2.1.2.5. Pre-Processing Planning - Material Property used

The choice of materials to be used in the LBPB process affects the residual stress. To minimize residual stress, the material of the part should have a low volumetric coefficient of thermal expansion, minimal temperature gradient developed during the manufacturing process, low heat input per unit length (equates to lower input power and higher scanning speed), high flexural rigidity, high elastic modulus, high moment of inertia, and high density of alloy powder [9].

Functionally Graded Materials (FGM) with varying properties were patented in the 1980s [91] and the finite element model showed that FGM with varying composition has reduced residual stress as compared to parts with a uniform property when exposed to high fabrication temperature [92]. While the formulation of FGM by varying chemical composition in the LBPB process is difficult, FGM in the LBPB process could be produced by varying porosity [93]. FGM has been successfully attempted in the LBPB process [93, 94]. There is a possibility to design a part by incorporating DFAM and FGM to fulfill the functional requirements of the part and reduce residual stress.

In the study of machining induced residual stress [95], compressional surface residual stress predominates on harder materials due to an increased shear angle effect, a factor that is unique to machining induced stress and not applicable for AM.

The residual stress development in LBPB materials is similar to that of a laminate composite made of different layers of materials with different elastic modulus. In the LBPB process, the addition of each layer of molten material introduces a mismatch in material property with the preceding solidified layer. In laminate composites, residual stress can be reduced by optimizing the ratio of elastic modulus between each

material layer [96]. Regions expected to experience tensile residual stress should have a higher elastic modulus, while regions expected to experience compressive stress should have a lower elastic modulus [96]. FGM with varying microstructures has been printed using the LBPB process by varying the power input of the laser [94]. A potential application of this finding can be useful to LBPB with the ability to print functionally graded parts with varying properties in different areas. Methods to produce complex FGM had been proposed by classifying typologies of bi-material gradients using mathematical descriptions [97, 98]. By predicting the residual stress distribution of a part manufactured using the LBPB process using computational simulations, FGM with a varied distribution of elastic modulus across the entire body can be designed such that region expected to be subjected to tensile stress to have higher elastic modulus, while region expected to be subjected to compression to have lower elastic modulus.

2.1.2.6. In-Situ Parameters - In-Situ Heat Treatment of Laser Beam Powder Bed processed parts

Heat treatment of each melted and solidified layer during the LBPB process was performed experimentally on SS316L. Rescanning each layer with 50% of the laser power used to fuse the SS316L powders (100W at 100%) helps to reduce the tensional residual stress by approximately 30%. However, using 100% of the laser power used in the heat treatment of SS316L would cause re-melting of the layer, and the residual stress will not be reduced [20]. For some cases, experimental results show that re-scanning of chrome-molybdenum steel (JIS SCM440) with 100% laser power (50W on average) produced a 55% reduction in residual stress [99]. These two results suggest that the optimum laser power could be used for the in-situ heat treatment of each layer varies for different types of material.

2.1.2.7. In-Situ Parameters - Energy source power and scanning speed

The combination of energy source power and scanning speed determines the amount of energy concentrated per unit length onto the powder material. The ratio of energy source power to scanning speed equates to the amount of energy input per unit length and was used as a parameter of study in several works of literature, for welding [100] and also for AM [101]. A high energy input per unit length is crucial for highly reactive materials such as aluminum which tends to develop a layer of oxide and requires a higher laser power and slower scanning speed to break down the oxide [3]. However, using higher laser power and slower scanning speed to achieve high energy input per unit length causes an increase in residual stress [24, 102]. Keeping the power input constant, a higher scanning speed results in lower residual stress in welding [65, 102] as well as in AM for SS304L material [103].

After absorbing the energy from the energy source, the dynamics of heat transfer in the part plays a determining role in the residual stress distribution. Non-dimensional parameters can be used to represent the heat transfer – the Fourier and Marangoni number in a computational model [104]. Fourier number is the ratio of heat dissipation rate to heat storage rate, a higher Fourier number corresponds to a higher heat dissipation rate and lower residual stress. Marangoni number indicates the strength of the convective transport of heat in the melt pool, which is driven by spatial variation in interfacial tension known as Marangoni stress [105] and is directly related to the input power density of the energy source. A lower input power density obtained by lower laser power and higher scanning speed results in a lower Marangoni number and consequentially lower residual stress.

2.1.2.8. In-Situ Parameters - Dwell time between each layer

Dwell time in the LBPB process refers to the intermittent time between each layer of scan and also equates to the duration allowed for the solidified part to cool down. The

effect of dwell time on the residual stress in the solidified part was found to be dependent on the material itself [106]. An increase in dwell time between the deposition of each layer during the LBPB process was proven to reduce residual stress for Inconel 625, while the trend is opposite for the case of alpha-beta titanium alloy (Ti6Al4V).

2.1.2.9. In-Situ Parameters - Scan Strategy

While the welding sequence in the welding process affects the residual stress in the welded part [107, 108], scan strategy in AM affects the residual stress distribution in the AM part. Residual stress perpendicular to the scan direction was found to be higher than the residual stress parallel to the scan direction in stainless steel SS316L manufactured by the LBPB process [20]. However, another experimental result shows that the residual stress parallel to scan direction was larger than the stress perpendicular to it for the same material [37]. A possible reason attributing to this contradicting result could be due to the scan direction relative to the dimension of the print. It was found that continuous scanning in a long path produces higher residual stress in the same direction as compared to that produced by a shorter scan path [20]. As such, instead of attributing residual stress magnitude to the direction of the scan, it would be more appropriate to relate the stress magnitude to the combined effect of scan distance, direction, and pattern instead. This combination of scan parameters is known as the scan strategy. Studies have been done to develop and test new scanning strategies to reduce residual stress [109, 110], and many LBPB manufacturers have developed their proprietary scan strategies.

2.1.2.10. In-Situ Parameters - In-Situ Parameters - Using larger layer thickness

Layer thickness affects not just the resolution of the print product, but also the ability of the selected laser power to completely melt the layer of powder materials due to

changes in the laser power heat intensities, and the heat distribution along with the depth of the part. Using a thicker layer thickness results in lower residual stress in tool steel 1.2709 (X3NiCoMoTi 18-9-5) manufactured by LBPB as per simulation and experimental results [111]. Although using a thicker layer thickness also helps to reduce printing time, it reduces the laser power intensity and increases porosity, and reduce the density of the print product [35, 112]. Issue of delamination might occur prematurely between each layer if the external load in addition to the remaining residual stress exceeds the bonding strength. A non-dimensional parameter – lack of fusion index, was used to represent the extent of fusion of the molten pool of a newly melted layer into the previously deposited layer [9]. The lack of fusion index is the ratio of penetration depth of the molten pool to the layer thickness and requires a value of more than 1.15 for successful inter-layer bonding. If a thicker layer is to be used for a quicker process and reduced residual stress, sufficiently high power and slower scanning speed is required to ensure through melting but will result in higher residual stress induced on the product which might offset the original intention of reduced residual stress. The application of using a larger layer thickness for lower residual stress in the print part also comes with a compromise of print resolution.

2.1.2.11. In-Situ Parameters - Ensuring Sufficient Hatch Overlap

During the LBPB process, the tracks of the laser beam will be overlapped by a distance known as the hatch overlap, to increase the strength of the part [113, 114]. Insufficient hatch overlap can result in the development of microcracks along the overlap zone between two adjacent passes. A sharp step surface profile that develops when there is insufficient overlap causes residual stresses to concentrate along with it, resulting in higher stress in that region, promoting crack growth. The importance of the overlap of each deposition pass was demonstrated on nickel-based superalloy Rene88DT [115]. Parts manufactured with insufficient overlap (30%) resulted in relatively long residual stress-induced cracks (3-10 mm) as compared to micro-cracks

(100-300 μm) if sufficient overlap (40%) was available. While the author found it difficult to eliminate the micro-cracks caused by residual stress concentration simply by adjusting the laser parameters, the long cracks could be easily eliminated with sufficient overlap. Although these results were based on the laser solid forming process instead of the LBPB process, the similarity in the resulting sharp step profile due to insufficient overlap in both processes, suggests a possible similarity in the trend for the LBPB process as well.

Cracks were found to develop in the interdendritic regions of the overlap zone. Due to the formation of low melting point eutectics (γ and γ') along columnar grain boundaries and interdendritic region during solidification, repeated exposure to high temperature due to the second deposition pass results in crack initiation in those regions. The presence of γ and γ' phases in the crack regions from the Energy Dispersive X-ray Spectroscopy (EDS) results shows that cracking resulted from liquation cracking. Cracks develop mainly at the top of the final deposit as cracks that develop near the substrate are healed by the re-melted γ and γ' phases above them.

EDS showed that low melting point eutectics (γ and γ') are formed along columnar grain boundaries and interdendritic regions.

Although overlap is required for crack prevention, an increase in overlap reduces the density of the part being printed. This is attributed to the increased number of melt pool exposed to high temperature incorporating powder material into it, leaving insufficient powder for other regions [36]. Hence, an optimum hatch overlap amount is important to ensure sufficient crack prevention while not affecting the density.

2.1.2.12. In-Situ Parameters - In-Situ Magnetic Treatment with Pulsed Current

A static magnetic field of 0.024 Tesla was applied perpendicular to the base plate surface during the laser-metal inert gas (MIG) hybrid welding process of SS304

material, as illustrated in Figure 10, and the residual stress was reduced as shown in Figure 11 [116]. The material with in-situ magnetic field treatment also has an improvement in tensile strength and micro-hardness. The improvement was attributed to the interaction between the magnetic field and electric current in the melt pool.

Figure 10: Schematic and photo of the application of static magnetic field in laser MIG process [116]

Figure 11: Comparison of residual stress of weld seam with magnetic field treatment of 24 mT versus without magnetic treatment. Left: Residual stress distribution in longitudinal direction, Right: Residual stress distribution in transverse direction. WZ: Weld-zone, HAZ: Heat affected zone [116].

The installation of a magnet below the base plate, as illustrated in Figure 10, can be applied to the LBPB process in EOS M290. However, the setup uses a laser MIG process which includes the application of pulsed electric current as high as 160A through the specimen, a feature not available in LBPB machines currently. Thus, the interaction of the magnetic field and pulsed current through the specimen to relieve residual stress is not directly applicable to the LBPB process.

Similar work was done for laser beam welding of aluminum part, whereby the magnetic field was applied parallel to the build plate but perpendicular to the weld

direction as illustrated in Figure 13 [117]. Laser beam welding does not involve the application of an electrical current through the weld material and is similar to the LBPB process. However, although this literature does not focus on residual stress reduction, the results obtained suggest a possibility of stress reduction in the welded part. The paper reports that current is induced in the melt pool when a magnetic field was applied perpendicular to the weld direction, which further interacts with the magnetic field and result in damping of the melt pool flow. Heat transfer in the melt pool by convection reduces and conduction dominates, causing changes in the temperature distribution. The final obtained weld cross-sectional area changes from a "wineglass shape" to that of a V-shape as shown in Figure 12, in which the author suggests a possible reduction in peak stress due to a more even residual stress distribution. A similar test was done [118] and it was found that 0.5 Tesla of a static magnetic field was sufficient to suppress the melt pool velocity and achieve a similar V-shape weld cross-sectional area.

Figure 12: Cross sectional view of weld beads. Left: Wineglass shaped weld beads observed without exposure to magnetic field during welding. Right: Regular shaped weld bead produced by exposure to magnetic field during welding [117].

Figure 13: Left: Schematic of the application of static magnetic field in laser beam welding. Right: Illustration of the direction of induced electric current and electromagnetic force due to interaction between magnetic field and fluid flow direction [117].

2.1.2.13. Post Treatment - Induced Reversal Stresses

Having compressive residual stress on the surface of a part helps with a better fatigue lifespan as compared to having tensile stress on the surface [23-26]. Residual stress can be reduced by deliberately inducing a reversed residual stress.

Shot peening [119], hot isostatic pressing (HIP) [115], autofrettaging [120] and cold expansion [53] are used to induce compressive residual stress onto high tensile stress regions to slow down the crack growth [121]. Imposing compressive residual stress on critical regions not only helps to delay the onset of crack growth but also slows down the rate of crack growth as well [121]. Shot peening is performed generally on physically reachable surfaces, HIP is capable of treating hard to reach surfaces, autofrettaging for the inner surface of enclosed pressure vessels, and cold expansion for stress-prone holes in structures. HIP treatment was found to be effective for healing long cracks (3-10 mm) in nickel-based superalloy Rene88DT that resulted from residual stress formation due to insufficient overlap of deposition pass, and also for micro-cracks (100-300 μm) [115]. However, the results suggest that mechanical properties might be compromised if HIP is used to treat long cracks as compared to micro cracks. No traces were observed for the healed micro-cracks, while traces of second phase particles were observed in the healed long cracks. The second phase particles are MC-type carbides formed due to the re-solidification of γ and γ' phases

in the interdendritic regions, which limits the effectiveness of the HIP treatment to improve the mechanical property. However, in general, HIP treatment was able to improve the tensile strength, ductility of the material by relieving the residual stress. It was found that improvement in the fatigue lifespan does not depend only on the maximum compressive stress induced in the sub-surface region. The improvement in fatigue lifespan also depends on the depth of the plastically deformed region, which corresponds to the area under the graph of the residual stress - depth distribution within the compressive region [122].

In welding of steel, phase changes to martensite using a low-temperature transformation consumable in the weld material induces compressional stress on the weld seam due to phase expansion, relieving the tensile residual stress [123]. As the material used in LBPB is generally homogenous, this method does not apply to LBPB as it makes use of the different properties of two different materials. However, the ability to induce a phase expansion of the newly deposited layer in future work inspired by this methodology would be useful.

However, given that residual stress exists as both compressive and tensile in a structure, induced compressive stress on the surface will also result in an increase in tensile stress in other parts of the structure. Depending on the shape and nature of the structure itself, excessive induced compressive stress on some parts of the surfaces can result in increased tensile stress on other surfaces, nulling the effectiveness of the treatment [124]. Temperature also plays an important role in the effectiveness of induced compression treatment [125].

2.1.2.14. Post Treatment - Post Heat Treatment of part

Heat treatment of parts after the LBPB process or welding is one of the most common methods used to relieve the residual stress. Standards of post heat treatment after welding has been adopted by industries [76]. Residual stress reduced by 70% in

chrome-molybdenum steel fabricated by LBPB after going through post-heat treatment [99]. Heat treatment also prevents deformation due to residual stress during build plate removal in SS316L [126]. Solution treatment and aging (STA) was able to improve the properties of Inconel 718 additively manufactured by laser powder deposition process, whereby the improved tensile strength became comparable to traditional wrought material due to the precipitation of γ'' and γ' strengthening phases [127].

The temperature was shown to have a larger impact on tensile residual stress on the surface and subsurface than the duration of treatment [128]. Tensile residual stress was found to be redistributed and some regions reduced to compressive residual stress instead. Although the general increase in compressive residual stress can be detrimental to dimensional stability, the change from tensile to compressive residual stress is beneficial to the fatigue properties of the material.

However, heat treatment was also found to decrease the tensile strength of aluminum-zirconium alloy [129], titanium alloy [130] and maraging steel grade 300 [131]. Corrosion susceptibility of certain materials such as 7N01 (Al-Zn-Mg) [132] and Fe-30Mn alloy [133] increase after repeated heat treatments. The toughness of maraging steel decreased after undergoing the heat treatment process [134]. Also, materials that are susceptible to re-heat cracking such as steel alloyed with Cr, Mo, V, and B are not suitable to undergo heat treatment [125].

2.1.2.15. Post Treatment - Cryogenic Treatment of Part

Other than using heat treatment, exposing the part to cryogenic temperature also helps in relieving residual stress. Unlike heat treatment, cryogenic treatment does not compromise on the material properties. Post cryogenic treated steel may have an improvement in its operational life and mechanical properties [135, 136]. Not only

was residual stress reduced, but Inconel 718 subjected to cryogenic treatment also have an improvement in strength, ductility, and nano-hardness at room temperature.

A test conducted on a welded aluminum alloy showed a reduction of 12 ksi (82.73 MPa) in the heat-affected zone and 9 ksi (62.05 MPa) in the parent metal when exposed to liquid nitrogen (-184°C) gradually, held for 24 hrs, and reheated to ambient temperature without thermal shock [137]. Two cycles of exposure to 12 hours of liquid nitrogen followed by reheating to room temperature reduced the residual stress of welded SS316 stainless steel from 260MPa to 58MPa [138]. Repeated cryogenic treatment on the same specimen was found to reduce the surface tensile residual stress even further on Inconel 718. The surface residual stress was reduced from a 53 MPa to -72 MPa after a round of cryogenic treatment (submerged in liquid nitrogen of -196°C for 2 hours, reheated back to room temperature), and a further reduction to -95 MPa after the second round of treatment [139].

A combination of cryogenic and heat treatment was performed as well on carburized steel. Cryogenic treatment followed by tempering of carburized steel was found to reduce compressive residual stress on the surface, while specimens that did not undergo cryogenic treatment first had an increase in compressive residual stress after annealing [140]. If heat treatment results in excessive dimensional distortion due to excessive compressive residual stress on the surface, a balance between beneficial compressive residual stress on the surface versus dimensional distortion can be made through cryogenic treatment as possible future work. The balance between having beneficial compressive residual stress on the surface (better fatigue lifespan) versus considerable dimensional distortion due to excessive compressive stress can be made with the application of cryogenic treatment.

2.1.2.16. Post Treatment - Magnetic field treatment

Patents have been filed regarding the use of a magnetic field to reduce residual stress in metals as a post-processing method [141] as well as specifically for welding induced stress [142]. The use of magnetic field treatment to reduce residual stress is not only faster and simpler than traditional treatment methods such as heat treatment, but it also avoids the harmful effects and drawbacks of heat treatment [143]. The essential difference between heat and magnetic treatment lies in the way plastic deformation is induced for stress relaxation [144]. The key feature of the magnetic treatment is the selective plastic deformation along dislocations in the material, whereas stress relaxation during heat treatment involves the flow of materials continually and relatively uniform plastic deformation across an area. However, both heat and magnetic field treatment induce a “self-organization process” [145] for the specimen to recover back to a certain organized condition, and thus provides a residual stress relief solution.

Low frequency alternating magnetic treatment (LFMT) with the frequency between 1 to 30Hz was found to have residual stress relaxation effects on welded specimens subjected to welding induced residual stress. However, the effect of frequency of the magnetic field is not proportional to the stress relaxation magnitude at different locations of the welded specimen. To improve the efficiency of magnetic field treatment, the direction of magnetic field imposed should be perpendicular to the direction of heat source motion during welding instead of parallel to maximize residual stress relief as suggested in the experimental results done by Song, Y.L., L. Hua, and B. Wang [146]. It was found that residual stress of low alloy steel could be reduced by 26-28% when a magnetic field of 2 Tesla at 2Hz was applied perpendicular to weld direction, as compared to 20-21% reduction for parallel. Other modes of magnetic treatment such as pulses were also effective for residual stress relief. A pulsed magnetic field was able to reduce residual stress by 12-24% in mid-

carbon steel [144] and 10% reduction in 16Mn steel plates welded with H08Mn2Si [147]. Other than dynamic and quasistatic magnetic treatment, a static magnetic field was found to improve the fatigue lifespan of ferromagnetic mild steel [148], which might be attributed to the reduction of surface tensional residual stress which has always been closely related to fatigue lifespan. Like an alternating magnetic field, static field treatments are also found to have a “healing effect” on low carbon steel specimens, whereby specimens exposed to microstructural damage under cyclic loading can recover from magnetic field treatment and have an improved fatigue lifespan [145]. Given the close relationship between fatigue durability and residual surface tensile stress, the improvement in fatigue property suggests the possibility of residual stress relief using a static magnetic field.

Although the hardness of steel specimens was found to decrease after magnetic field treatment, it was shown that the relief of residual stress after the treatment is not due to the hardness change [149]. Suggested mechanisms of stress relaxation compiled by Song and Hua [150] include dynamic magnetostriction [143, 149], the magneto-plastic effect [150], micro-plastic deformation due to changes in dislocation density [151], and alteration of energy absorbed by domains [146]. However, the effects of residual stress relief due to magnetostriction were found to be limited [152] and the magneto-plastic effect was suggested to have a larger role.

When combined with electrical current treatment, magnetic field treatment has been proven to be even more effective in residual stress relief [147]. The pulsed magnetic field of 2.1 Tesla at 0.5 Hz using 25 pulses over 50 seconds combined with pulsed current treatment of peak 3000A using 15 pulses at 0.02 seconds each for 5 times were able to relieve 60% of stress, as compared to relief in 10% using a pulsed magnetic field and an increase in 20% using pulsed current individually. Cai, Z.P., and X.Q. Huang attributed this to the combined effect of magneto-plastic and electro-plastic effect, where conduction electrons help to drive dislocations further and faster.

Some authors hypothesized there is a correlation between electric current and magnetic field [145], as a changing magnetic field can produce eddy current, while electric current can produce magnetic field as well.

Some of the parameters that indicate the magnetic properties of the material includes relative permeability (non-dimensional), susceptibility (non-dimensional), and magnetization (kA/m) [90]. The type of material applied to magnetic field treatment is limited to those that exhibit sufficient magnetic properties. While most of the works of literature involving magnetic treatment are based on iron alloys that are ferromagnetic, paramagnetic materials with relatively weaker magnetic properties have been proven to respond positively to magnetic treatment as well. Tungsten Carbide-Cobalt (WC-Co) detonation spray coating was relieved from its residual stress by 50% to 60% through magnetic field treatment [153]. Aluminum 7055 alloy [154] and aluminum matrix composites [155] with much poorer magnetic properties were also proven to undergo residual stress relief when exposed to magnetic field treatment. In the aluminum 7055 specimen, it was found the stress starts to increase when the imposed magnetic field strength exceeds a threshold value of 3T. The existence of a threshold point of magnetic field application and duration was also discovered, whereby the fatigue lifespan of low carbon steel starts to deteriorate when the applied field and duration is too large, resulting in an excessive eddy current generation and heat, causing heavy oxidation [145].

2.2. Conventional Post-Heat Treatment Methods

This section focuses on common conventional post-heat treatment methods for aluminum alloys and AlSi10Mg. Conventional post heat treatment for AlSi10Mg includes annealing at 300°C for 2 hours [156] and T6 heat treatment. Many modified heat treatment methodologies have been done pertaining to T6 heat treatment [157, 158] as it has been shown to improve mechanical properties of cast AlSi10Mg.

Unanimous result finding shows that higher heat treatment temperature results in poorer mechanical properties in terms of compressive and tensile strength.

2.2.1. Annealing with Furnace Controlled Cooling

Annealing of AlSi10Mg at 300°C for 2 hours has been the stress relieve methodology proposed by EOS for the alloy manufactured using EOS LBPB machines and powder [156]. Other than observing the duration of treatment and temperature, the cooling must be slow and controlled [159], usually performed in the furnace itself with the cooling duration equivalent or longer than the heat treatment itself. Many studies have been done on this heat treatment methodology [158, 160, 161].

2.2.1.1. Effect on Residual Stress

Annealing of AlSi10Mg at 300°C for 2 hours was proven to relieve residual stress to less than 20 MPa from compressive stress of approximately -80 MPa induced due to rough machining [12]. The same set of the experiment also shows that heat treatment at 200°C for 1 hour is insufficient to relieve the stress to near 0 value.

2.2.1.2. Effect on Mechanical Properties

The duration of 2 hours of annealing at 300°C has been proven to be the ideal duration for maximum tensile strength and yield strength and elongation [162], as seen in Figure 14. The author has also established that the tensile residual stress that resides in AlSi10Mg specimens fabricated by the LBPB process was successfully relieved by annealing at 300°C for 2 hours, followed by slow controlled cooling.

Figure 14: Effect of different annealing time on AlSi10Mg manufactured by the LBPB process, with annealing temperature of 300°C [162]

Table 1 below shows the tensile strength of AlSi10Mg in as-built condition and after annealing at 300°C for 2 hours. The experiment by Takada shows that the yield strength, ultimate tensile strength, and hardness undergo a significant drop after the annealing process [32]. Yield stress reduced by 20% to 35%, tensile strength reduced by 40%, while total elongation increased by 8.7% to 11.1%.

Table 1: Tensile properties of AlSi10Mg as fabricated by LBPB and after annealing at 300°C for 2 hours [32]

Tensile direction	0.2% proof stress/MPa		Tensile strength/MPa		Total elongation/%	
	X/Y direction	Z direction	X/Y direction	Z direction	X/Y direction	Z direction
As-fabricated	279	220	475	476	7.5	5.5
Annealed at 300 °C for 2 h	180	175	285	290	18.6	14.2

While annealing at 300°C exhibited a significant drop in mechanical strength, another study found that treatment at a lower temperature of 250°C caused a lower reduction of yield and tensile stress by 14% and 2.8%, and elongation increase by a lower amount of 3.9% [163].

2.2.1.3. Effect on Microstructure

Figure 15: Schematic showing the change in microstructure of AlSi10Mg after annealing at 300 °C for 2 hrs [32]

The illustration shown in Figure 15 shows the schematic of the change in the microstructure of AlSi10Mg after annealing at 300 °C for 2 hours [32]. The original eutectic microstructure of α -Al / Si which forms the cellular network that contributes to the strength of the material starts to dissolve and new Si precipitates form in the α -Al phase.

SEM images in Figure 16(a) and (c) below show the dissolution of the Si fibrous network after heat treatment at 300 °C for 2 hours. Holding the treatment temperature for a longer duration of 8 hours at a lower temperature of 160 °C causes precipitation of Si in primary α -Al phase in between the fibrous network structure as shown in Figure 16(b). The fibrous network structure of Si is also still intact in Figure 16(b).

Figure 16: SEM image of (a): As built condition after the LBPB process (b): After subjected to 160 °C for 8 hours, air cooled, (c): After subjected to 300 °C for 2 hours, air cooled [158]

While having proven that the eutectic fibrous network of Si starts to get broken up significantly at 300°C with the duration of 2 hours treatment time, Rafieazad found that annealing at a lower temperature of 200°C leaves the fibrous network mostly intact as shown in Figure 17(a), and the author suggested adopting a stress-relieving annealing temperature at 200°C or below [164].

Figure 17: 3D representation of SEM micrographs of AlSi10Mg after undergoing annealing treatment for 2 hours at temperature a) 200 °C. b) 300 °C. c) 350 °C [164]

Although in Rafieazad's experiments, the AlSi10Mg was manufactured by direct metal laser sintering process, the eutectic microstructure of Si network as shown in Figure 17 is the same as that obtained from the LBPB process in Figure 16, as both methodologies involve rapid cooling via layering process, which was attributed to the reason for the formation of such unique structures which are not found in conventional cast AlSi10Mg.

Studies have also shown that after the heat treatment, brittle intermetallic phases of $\text{Al}_9\text{Fe}_2\text{Si}_2$ and $\text{Al}_{4.5}\text{FeSi}$ were found to have increased as shown in Figure 18, in which the author mentioned this phenomenon being detrimental to the mechanical property of the material [160].

Figure 18: Phase distribution of intermetallic phases after heat treatment. Specimen A1 refers to heat-treated AlSi10Mg at 300 °C for 2 hours and slow cooling. Specimen A2 refers to heat treatment at 535 °C for 1 hour, followed by water quenching, 190 °C aging for 10 hours, followed by furnace quench [160].

2.2.2. Solution Heat Treatment with Quenching and Artificial Aging

T6 heat treatment is a conventional heat treatment used for casted AlSi10Mg, to improve its mechanical strength. It involves treatment at approximately 520°C to 550°C for 2 hours, water quenching, and artificial aging between 160°C to 180°C for a range of 30 minutes to 18 hours [161]. Many modified versions of such treatment have been studied with varying solution treatment temperature and duration, quenching medium, aging temperature, and duration. This treatment is relatively much longer and tedious than that of the annealing treatment discussed in section 2.2.1.

2.2.2.1. Effect on Residual Stress

A set of solution heat treatments with quenching and artificial aging has been shown to be effective in relieving residual stress of AlSi10Mg parts manufactured by the LBPB process as shown in Table 2 below [165]. Solution treatment with quenching and aging process - HT3 and HT4, has been effective in relieving the residual stress of 75 MPa to a low value of ± 20 MPa. These show a successful relieve of residual stress similar to pure annealing – HT1 and HT2, with final residual stress values of 6MPa and 22MPa. The mechanical properties in Table 2 will be discussed in the next section.

Table 2: Top: Heat treatment methodologies, Bottom: Effect of different heat treatment methodologies on mechanical properties and residual stress of AlSi10Mg manufactured by the LBPB process [165]

No.	Heat treatment parameters					
HT 1	300 °C/ 3 hours					
HT 2	250 °C/ 2 hours					
HT 3	520 °C/ 6 hours/ water quench/ 175 °C/ 4 hours					
HT 4	510 °C/ 0.1 hour/ water quench/ 170 °C/ 8 hours					
HT 5	no heat treatment (as-built)					

No.	YS $R_{p0.2}$ [MPa]	UTS R_m [MPa]	Elongation [%]	X direction [mm]	Y direction [mm]	R. S.* [MPa]
HT 1	139	225	8.8	+0.19	+0.19	+6
HT 2	204	303	4.4	+0.52	+0.51	+22
HT 3	258	288	2.8	-0.24	-0.23	-17
HT 4	246	287	3.2	-0.25	-0.23	-17
HT 5	237	400	3.5	+1.97	+1.94	+75

2.2.2.2. Effect on Mechanical Properties

As per Table 2 above [165], while the solution treatment with quenching and aging treatment - HT3 and HT4, is successful in relieving the residual stress, the treatment caused a reduction in ultimate tensile strength by approximately 28% and a slight improvement in yield strength by 8%.

Table 3 below shows the summary of the effect of T6 heat treatment on the mechanical properties of AlSi10Mg manufactured by the LBPB process. Similar to that of annealing at 300°C in Table 1, the tensile strength and hardness reduced, while elongation increased. Although T6 heat treatment is of a higher temperature than 300°C, the T6 heat treatment saw a smaller reduction in yield stress by 11% (Table 3) as compared to 20-35% (Table 1) after annealing at 300°C. This difference is due to the additional aging process in the T6 treatment which causes precipitation of Si particles and strengthening effect. This is supported by the result in Figure 19 which shows an increase in tensile strength after artificial aging for 12 hours [157]. Another study also shows the improvement in strength due to the aging process of T6 heat treatment [165]. This is the reason for the popular adoption of aging treatment

for conventional cast materials which does not have the strengthening effect of Si cellular network structure that LBPB parts have.

Table 3: Summary of effect of T6 Heat treatment on mechanical properties of AlSi10Mg [166]

Property	Effect of heat treatment (%)
Nano-hardness	$-(16 \pm 1)$ ↓
Micro-hardness	$-(20 \pm 1)$ ↓
Ultimate tensile strength	$-(12 \pm 5)$ ↓
Yield tensile strength	$-(11 \pm 1)$ ↓
Tensile elongation at failure	$+(179 \pm 57)$ ↑
Compressive strength at 25% strain	$-(56 \pm 0.003)$ ↓
Yield compressive strength	$-(47 \pm 2)$ ↓

Figure 19: Tensile stress strain curve of AlSi10Mg subjected to T6 heat treatment. Left: Modified T6 heat treatment without artificial aging. Right: Corresponding T6 heat treatment with artificial aging [157]

After T6 heat treatment, the compressive strength of the alloy decreased most significantly among other mechanical properties as shown in Table 3. Compression load up to 230 kN (700MPa) conducted by Aboulkhair shows a significant drop in the strength as shown in Figure 20 and Table 4 below.

Table 4: Compressive properties of AlSi10Mg in as built condition versus after T6 heat treatment[166]

Condition	Yield compressive strength (MPa)	Compressive strength at 25% strain (MPa)
As-built	317 ± 2	714 ± 1
Heat treated T6	169 ± 6	466 ± 6

Figure 20: Compressive stress-strain curve of AlSi10Mg in as-built condition versus after subjected to T6 heat treatment [166]

2.2.2.3. Effect on Microstructure

T6 heat treatment causes precipitation of Mg_2Si [167] which helps to improve ductility without compromising on other mechanical properties [157] and contributes to mechanical strength by pinning dislocation [168]. However, intermetallic precipitates such as $Al_9Fe_2Si_2$ can also form [167]. Modified T6 heat treatment with 1 hour of solution treatment instead of 2 hours shows the presence of $Al_{4.5}FeSi$ as shown earlier in Figure 18 [160]. As mentioned also in section 2.2.1.3, the author mentioned about the detrimental effect these intermetallic precipitates have on the mechanical properties.

2.2.3. Heat Treatment with Quenching and Mechanism of Residual Stress

Induction

After solution or aging heat treatment, the cooling process can be a slow and controlled manner to ensure no residual stress is induced on the part, or via rapid quenching into a medium. Mediums of quenching include liquid nitrogen or sub-zero

temperature liquid for rapid cooling, water at ambient temperature, or air cooling for relatively gradual cooling.

Rapid cooling has been found to induce compressive residual stress on heat-treated aluminum alloy [30, 31]. Rectangular Al-Cu alloy specimens treated at 500°C were subjected to different quenching rates in water, with the temperature of water ranging from 20°C to 100°C. It was found that specimens exposed to a higher cooling rate have the largest compressive stress on their surface as compared to those exposed to a slower cooling rate [31]. Via the slitting method, the author measured the residual stress profile of the entire depth of the specimen and concluded tensile stress existing in the center portion, while the residual stresses in the outer portion are compressive. Although the mechanism was not discussed by the author, the mechanism can be deduced from Figure 21 below.

As the outer surface undergoes rapid cooling, it contracts at a faster rate than the inner core. The restriction in the contraction of the outer surface results in tensile stress on the surface and compressive stress on the inner core as shown in Figure 21 – Stage 1. The duration of time spent in stage 1 is relatively short as compared to Stage 2, as the rapid cooling process via quenching causes the outer surface to reach ambient temperature quickly. When the outer surface approaches ambient temperature as seen in Stage 2, contraction no longer takes place. The inner core, however, continues to cool and undergoes contraction. The outer surface now restricts the contraction of the inner core and induces tensional stress in the core, while compressive stress is induced on the outer surface.

Figure 21: Mechanism of residual stress induction due to rapid cooling after heat treatment.

Due to the longer duration of time spent in Stage 2, the final residual stress that resides on the outer surface is compressive and in the inner core is tensile, as illustrated in Figure 22.

Figure 22: Illustration of residual stress distribution in specimens after rapid cooling via quenching.

This mechanism of residual stress development has been well established in the study of thermal tempering of glass and modelled results are aligned with experimental results [169]. Due to the transparent nature of glass, the entire residual stress profile across the depth of glass can be measured non-destructively and directly using

scattered light polariscope and the results proved that the mathematical model is accurate. Figure 23 below shows the temperature and residual stress profile of tempered glass across its depth, whereby the distribution follows a parabolic curve [170]. The compressive stress magnitude is approximately twice that of the tensile stress in the center, while the compressive layer depth forms 21% on the top surface and tensile stress forms 58% of the entire depth.

Figure 23: Temperature and residual stress profile of glass plate subjected to quenching [170]

The residual stress distribution obtained experimentally from Al-Cu alloy is shown in Figure 24 below, which is the same as that proposed in Figure 22.

Figure 24: Residual stress distribution of Al-Cu alloy after quenching at different rates. Sample A1 was subjected to the largest cooling rate, and A4 was subjected to the lowest cooling rate [31]

2.3. Microstructure and Mechanical Properties of AlSi10Mg

2.3.1. Microstructure of AlSi10Mg Manufactured by Laser Beam Powder Bed

In low magnification, scan tracks and melt pools from the LBPB process are visible from optical microscopy as shown in Figure 25 below.

Figure 25: Optical image of AlSi10Mg in as-built condition. Left: Cross sectional view. Right: Top view [171]

It has been established in many studies that the rapid cooling of the LBPB process results in the formation of Si in the form of a fibrous network structure in α -Al phase [161, 166] as shown in Figure 26. Not just unique to the LBPB process, AlSi10Mg manufactured by direct metal laser sintering in which also involves rapid cooling also has the same microstructure [164, 172]. The schematics of the silicon structure as illustrated in Figure 27. Padovano attributed the formation of this structure to the high cooling rate of approximately 10^6 °C/s that results in Al-rich cells surrounded by Si-rich eutectic structures in the intercellular regions [161].

Figure 26: Microstructure of AlSi10Mg built by the LBPB method [166]

Figure 27: Schematics of the formation of eutectic microstructure of Si in α -Al phase

Fibrous networks of Si phase was observed in LBPB manufactured parts, while such structures could not be found in cast parts as shown in Figure 28. The lack of supersaturated Si precipitation in cast parts and only found in LBPB manufactured parts are attributed to the unique characteristic of the LBPB process itself – rapid cooling.

Figure 28: Microstructure of AlSi10Mg. Top left and right: as cast AlSi10Mg. Bottom left and right: LBPB manufactured AlSi10Mg [173]

Table 5 below shows the comparison of the microstructure of LBPB manufactured AlSi10Mg specimens versus conventional cast material [174]. In as-built conditions, LBPB parts possess better mechanical properties due to their continuous Si segregation structure as compared to Si in the flake form in conventional cast material. Upon precipitation hardening via heat treatment, the strength of LBPB parts becomes weaker than before due to the coarsening of Si particles and dissolve of the

original Si network structure, while that of conventional cast material becomes stronger due to an increase in Si precipitates contributing to precipitate strengthening.

Table 5: Comparison of microstructure of LBPB manufactured AlSi10Mg versus conventional cast material [174]

Point of Comparison	SLM Material	Conventional Cast Material
As-fabricated microstructure	fine microstructure with continuous Si segregations on the grain boundaries of α -Al	coarse dendritic microstructure ^[18] with Si in the form of flakes ^[20]
Precipitation hardened microstructure	microstructure coarsens to become a solid solution of Si particles in a matrix of α -Al	fine Si particles form and cluster at the grain boundaries of α -Al ^[20] generally a globular microstructure.
Precipitation hardening effect on hardness	hardness decreased for the investigated range	hardness increases until peak hardening (~10 h AA) then decreases with over aging ^[21]

2.3.2. Grain Orientation of AlSi10Mg Parts

Microstructural grains of the LBPB process tend to be elongated towards the build direction [175]. Many studies have shown the elongated grains of AlSi10Mg manufactured by the LBPB process tend to form [32, 176, 177], such as illustrated in Figure 29. Due to the LBPB process, grains have a preferred orientation towards the build direction, usually denoted as $\langle 001 \rangle$ as the directional convention.

Figure 29: Cross-sectional view of EBSD scan along the Z direction of AlSi10Mg manufactured by LBPB. Left: Grain orientation map. Right: Red coloured portion denotes grains orientated within 15° from $\langle 001 \rangle$ build direction [32]

Likewise, the top surface view of the grain orientation map also shows preferred grain orientation towards the built direction as shown in Figure 30. Anisotropy is evident comparing the IPF map of the cross-sectional view in Figure 29 and the top view in Figure 30. The grain from the top view shows uniaxial grain structure instead of elongated grains in the cross-sectional.

Figure 30: Top view of EBSD scan along the Z direction of AlSi10Mg manufactured by LBPB. (a) to (d) shows specimens manufactured by different laser beam intensity (increasing order) [176]

2.3.3. Mechanical properties and anisotropy of AlSi10Mg Manufactured by Laser Beam Powder Bed

It was found in several studies that residual stress induced in AlSi10Mg parts is lower in the LBPB process as compared to casted parts [178, 179]. LBPB manufactured AlSi10Mg possesses better tensile strength than its traditional cast counterpart [173]. The experiment results also show that LBPB manufactured AlSi10Mg materials are more prone to strength deterioration after heat treatment as compared to cast counterparts. This is due to the fibrous network of Si phase in LBPB parts which constitutes the strength of the material gets broken up by the heat treatment, thus making LBPB parts more vulnerable and sensitive to heat treatment than cast counterparts. The existence of this unique fibrous network structure of Si in LBPB manufactured parts also contributed to its higher hardness as compared to that manufactured via the casting method [178]. Padovano [161] reviewed that the mismatch in the atomic size of Si (0.118 nm) and Al (0.143 nm) atoms results in lattice deformation that limits the movement of dislocations [180], thus contributes to

the strengthening effect of AlSi10Mg in as-built condition from LBPB [174]. Experimental results have also shown the anisotropic properties of AlSi10Mg made from the LBPB process [181], with different tensile strength in vertical and longitudinal direction.

Studies have shown that the grain size of AlSi10Mg does not change significantly with heat treatment temperature up to 307°C, and only increases moderately for higher temperature treatment [171]. In another study, another series of aluminum alloy - A357, which is closely associated with AlSi10Mg due to their similar composition, mechanical properties, and microstructure, has shown negligible grain size changes after being subjected to different heat treatment temperature, and the author concluded the effect of grain size changes on the mechanical properties to be negligible [158]. Li attributed the higher strength of as-built AlSi10Mg as compared to heat-treated specimens with Hall Petch relationship – as-built specimens feature a smaller distance between Si particles in as the network structure, therefore increasing Al-Si interface and reduces dislocation movement, as compared to the reduced Al-Si interface in heat-treated specimens leading to a reduction in material strength [157]. Hence, changes in mechanical properties of the material after heat treatment are mainly due to the changes in fibrous eutectic network structure instead of the negligible changes in grain size.

2.3.4. Effect of strain on Low Angle Grain Boundaries and Kernel Average Misorientation

Studies have shown that heat treatment will reduce low-angle grain boundaries [32] due to the reduction of dislocations [182]. Results from past work also show the possible effect of induced residual stress resulting in strains that cause dislocation and increase in low angle grain boundaries (LAGB). The experiment by Takada [32]

shows a higher peak in low angle boundaries after a 6 hours duration of 530°C treatment as compared to that of a 2 hours duration treatment at a lower temperature of 300°C. This increase in low angle boundaries could be induced due to the fast cooling quenching process that follows the 530°C treatment instead of the slow cooling from the annealing process of 300°C treatment.

2.4. Characterization of Residual Stress

While stress cannot be measured directly and can only be derived from force and area, residual stress can only be derived from other measurable parameters depending on the methodology. Characterization of residual stress measurements can be classified as destructive, semi-destructive, and non-destructive methods. Non-destructive testing of residual stresses, also known as physical methods, is popular as they do not cause any destruction to the specimen, and no additional residual stress will be induced in the process of testing. Ultrasonic measurement relates residual stress built up in materials to the velocity of ultrasound through the body. This technique can measure the average residual stress over the depth at different parts of a specimen [183] but can give inaccurate results when phase or microstructural changes occur during the AM process. XRD works by measuring the angles where the maximum intensity of diffracted X-rays is detected when an X-ray is targeted at a specimen. The interplanar spacing of the lattice planes of the materials can then be obtained using Bragg's law. By comparing the interplanar spacing of the stressed material with that of an unstressed one, the resulting residual stress can be obtained. XRD is suitable for small and complex specimens but is difficult to differentiate between micro and macro residual stresses, and excessive residual stress-causing plastic strains can result in inaccurate results [184]. The magnetic method involves measuring the Barkhausen "noise" generated in materials when exposed to a magnetic field. By relating the stress-sensitive noise to residual stress, the compressive or tensile nature of the residual stress can be known [44]. This method is

suitable for thin specimens but only works for ferromagnetic materials [185]. While the sign of residual stress can be obtained, this method is not able to measure the magnitude of the stress.

Mechanical methods of residual stress testing such as CHD (Central Hole Drilling) are semi-destructive and parts of the specimen get removed. The principle behind this method is the off-loading of bulk materials through the process of residual stress relief and the measurement of residual strain. The residual stress profile along the depth of a specimen can be obtained relatively quickly by the CHD method but is accurate with depth up to the size of the hole diameter [15]. To measure residual stress deeper in the specimen, development has been made for deep hole drilling [186]. However, the measurements using this method will be compromised if the residual stress in the specimen is larger than 50% of the yield stress of the material [187] and corrections need to be made [188]. Also, this method is only suitable for macro residual stress measurement and does not include micro stresses [44]. This methodology, however, assumes that the material is isotropic [189], something which is not expected to be in additive manufactured materials that have been known for their anisotropy characteristics. Some of the important mechanical properties for evaluating residual stresses are Young's modulus, thermal dilatation coefficient, Poisson ratio [40], strain, and yield strength [190].

Mathematical models involving the residual stress development of laser or electron beam powder bed process usually involve 2 models - heat transfer and heat source intensity models [191]. The heat transfer model involves the solidification process of the melted zone and the interaction with surrounding solid material. The mathematical model considers the latent heat for melting and boiling, and the heat transfer with the surrounding via conduction, convection and radiation [192]. The source intensity model involves the consideration of vaporized powder and the penetration of energy into the solid body from the laser or electron beam. Solid phase

that vaporizes into gaseous phase or melted into liquid phase are removed from the model as they no longer contribute to macro residual stress build up via restrains in compression and contraction. Navier stoke's equation are used to model the fluid flow within the melt pool before solidification. The powder material is modelled based on heat conductivity of the bulk material, as a function of the powder bed packing density.

2.5. Analysis of Literature Review – Challenges of Conventional Industrial Adopted Heat Treatment Methods

From the literature review, it can be seen that the presence of residual stress limits the ability to produce functional parts and limits the benefits of LBPB process. Hence, many studies focus on reducing residual stress by pre-process planning, selecting appropriate in-situ LBPB parameters, or as a post processing methodology. Pre-process planning such as design of build dimensions, support structures and the orientation of build parts on build plates require users to have a generic knowledge of the dependency of residual stress on these design parameters. Other pre-processing parameters such as selecting the pre-heat temperature of powder bed, selecting materials less prone to residual stress induction and maintaining semi-solid state during LBPB, are highly dependent on the material itself. In-situ LBPB parameters such as laser power, scanning speed, hatch overlap, dwell time between each layer and scan strategies are common parameters that are often provided by LBPB manufacturers and metal powder vendors based on their research results, in order to produce parts with mechanical properties comparable or superior to conventional casted parts. Tweaking parameters in order to reduce residual stress induction can result in deterioration of the mechanical properties. While pre-processing planning and in-situ methods and LBPB parameters are important to minimize the residual stress during the build and ensure a successful print, these methodologies are only able to reduce the residual stress and unable to eliminate residual stress completely.

Post treatments such as heat treatment has been adopted as the most common method to relieve all remaining residual stress that could not be prevented in pre-processing planning and in-situ methods.

The literature review in section 2.2 has shown that the vendor suggested stress relief temperature of 300°C for 2 hours is effective in reducing the residual stress of AlSi10Mg to near-zero value, and the 2 hours duration has been optimized for the strongest mechanical properties. However, stress relief is achieved at the expense of other mechanical properties such as hardness, tensile, and compressive strength. The Si network structure that provides the strength for the material is mostly intact at 200°C, suggesting minimal damage done to the material strength. However, stress relieves treatment temperature lower than 300°C have been proven to be incapable of relieving all of the residual stress of AlSi10Mg samples. Although the effect of different heat treatment temperatures and duration on the residual stress of AlSi10Mg had been extensively investigated, there were no reports that show a successful attempt to reduce the temperature required for residual stress reduction to near zero value. This poses an engineering dilemma between mechanical strength and residual stress reduction, and this problem has been highlighted by several authors before [157, 165].

The cooling process after the stress relief heat treatment should also be furnace cooled or a process that is sufficiently slow enough to maintain a residual stress-free state upon reaching room temperature. This significantly reduces the efficiency as the duration of furnace cooling can be equivalent to the duration of heating itself. The T6 heat treatment takes the longest duration which can take more than 10 hours. Although utilizing a faster cooling rate for the heat treatment will significantly reduce the time required for the process, it will also induce additional thermal residual stress on the material, reducing the effectiveness of the process altogether.

The residual stress induction mechanism from rapid cooling has been well established from past works, producing residual stress profile that is parabolic across the depth. Upon further analysis of the parabolic residual stress curve as shown in Figure 31, the area bounded by the curve and x-axis in the red to green zone has a ratio of 1:1. The ratio of the area of red to green zone signify the ratio of the amount of tensile force exerted in the middle region with respect to the compressive force exerted on the top and bottom regions. This is aligned with the balance of compressive and tensile forces within a body and supports the Heyn's spring model shown earlier in Figure 2. This also suggests that an inverse relationship exists between the residual stress magnitude and the depth of influence. As illustrated in Figure 32, the depth of influence in the center region in tension is larger as compared to that of the top and bottom layers in compression. As such, the tensional force in the center is spread over a larger area as compared to the compressional force acting on the relatively thinner surfaces.

Figure 31: A parabolic temperature gradient produces surface compression which is twice the interior tension in magnitude. Ratio of the area of green to red is found mathematically to be 1:1.

Figure 32: Illustration of residual stress distribution across the depth of a quenched plate. Green region denotes the region in compression and red region denotes the region in tensile stress.

Last but not least, although many types of research have been done to correlate microstructural evolution with different heat treatment parameters, little has been done to correlate microstructural observations with residual stress reduction from these treatments.

Chapter 3. Proposed Novel Heat Treatment Methodology

This chapter proposes a novel heat treatment methodology to address the challenges and technological gap discussed in Chapter 2. The details and process of experiments conducted are presented in this chapter.

3.1. Novel Heat Treatment Methodology for Residual Stress Reduction Without Compromise of Mechanical Property

Given that thermal residual stress can result from uneven cooling due to fast cooling after heat treatment, understanding the mechanism of residual stress formation from uneven cooling can help us to “customize” the induced residual stress by controlling the uneven cooling process. Having knowledge of the nature (compressive or tensile) of the residual stress on a different location on parts, an uneven cooling process can be customized to induce residual stresses that are equivalent but opposite as that of the initial residual stress, canceling them out. This gives rise to the possibility of using a lower temperature for heat treatment such that the mechanical properties are not compromised excessively, yet achieving sufficient residual stress relief. This new methodology requires a cooling rate that is faster than furnace cooling to induce thermal residual stress. As different surfaces of specimens require different amounts and nature of residual stress induction for stress relief, the cooling rate should not be too rapid that results in a compressive effect on all top surfaces as illustrated in Figure 21. The methodology presented in this chapter utilizes a bottom plate heat treatment instead of a traditional furnace environment. The reliability of the residual stress trend obtained from this method is validated by comparison with that obtained from a traditional furnace-treated environment, with both exposed to a similar uneven cooling process. As the new methodology relieves existing stress by superpositioning of new induced stress and does not rely solely on heat itself, the temperature required for this new methodology is lower than that of the conventional

methodology. Given that the mechanical property weakens at a higher temperature, this methodology achieves residual stress relief at a lower temperature and hence minimizes mechanical property deterioration.

3.1.1. Initial Residual Stress of AlSi10Mg from LBPB Process

To be able to customize an uneven cooling process to relieve the remaining surface residual stress in AlSi10Mg alloy after low-temperature heat treatment, the nature of the initial stress and its distribution on the part needs to be known. To simplify the effect of uneven cooling on the specimen to just the top and bottom surface, the specimens used have a relatively thin thickness with respect to their top and bottom surface dimensions as shown in Figure 33. The specimens were removed from the build plate via electrical discharge machining (EDM) without any form of heat treatment.

Figure 33: Top left: Dimensions of specimen used for this study 10 x 10 x 4mm. Top right: Specimens before removal from the build plate. Bottom: Convention of the Longitudinal and Transverse direction

Residual stress measurements were done on the top and bottom surface with the longitudinal and transverse directions standardized as shown in Figure 33. Surface residual stress characterization was performed using XRD in the longitudinal and transverse direction for both top and bottom surface respectively. The initial residual stress results are discussed in section 4.1, with a relatively much larger tensile residual stress on the bottom surface and a mixture of compressive and mildly tensile stress in the longitudinal and transverse direction on the top surface. Thus, the main concern of the effect of residual stress is on the bottom surface.

3.1.2. Novel Method of Heat Treatment (Bottom Heated-Uneven Air Cooling) – Mechanism of Residual Stress Induction

The physical basis of the residual stress induction mechanism proposed in this section was based on the past studies done by other authors. The mechanism of residual stress induction during LBPB process and during rapid cooling has been extensively studied and modelled by other authors as mentioned in section 2.1. The physical basis of the mechanism of residual stress induction is that restriction in expansion of a layer of bulk material results in compressive stress induced in that layer, and a corresponding tensile stress in the restricting layer. Likewise, restriction of contraction of a layer of material results in tensional stress induced in that layer, and a corresponding compressive stress in the restricting layer. The mechanism of residual stress induction due to rapid cooling was described in detail in section 2.2.3 and further analysis done in section 2.5. The physical basis of the mechanism of residual stress induction is that rapid cooling results in the outer surface reaching ambient temperature and restricts the inner contracting region, resulting in compressive stress induced on the outer surface and tensional stress in the inner region. The dominance of a stage over the other shows that the duration of time spent in each stage also determines the magnitude of residual stress induced in the part.

The mechanism of type I residual stress development on the macro scale due to uneven cooling was a combination of both LBPB process described in section 2.1 and rapid cooling process in section 2.2.3. It was observed that the residual stress in LBPB fusion produced AlSi10Mg samples have tensile residual stress on the bottom surface, followed by mildly tensile and compressive residual stress on the top surface after removal from the build plate, which is shown in section 4.1 later on. Inducing an uneven air cooling process immediately after the heat treatment with a bottom heat plate will induce compressive and tensile stress on the specimen, with its distribution depending on the thermal gradient between the bottom and the top surface as illustrated in Figure 34.

After Heat Treatment:

Mechanism 1:

Figure 34: Model of residual stress induction due to uneven cooling. The arrows denote the nature of residual stress induced in different regions of the specimen. After heat treatment: Illustration of the specimen on the bottom heat plate at the start of the cooling process immediately after heat treatment. Mechanism 1: Residual stress is induced during the cooling process when the entire specimen body undergoes uneven thermal contraction. Mechanism 2: Residual stress induced during the cooling process when the top surface temperature is nearing ambient temperature. Mechanism 3: Residual stress induced in the last stage when the bulk of top material is nearing ambient temperature.

The illustration in Figure 34 after heat treatment shows the side view 10 x 4 mm face of the specimen with the 10 x 10 mm face area placed on a heat plate. Each specimen undergoes heat treatment at temperatures 160°C, 200°C, 300°C, 400°C and 500°C respectively for 2 hours, immediately followed by an uneven air cooling process while still positioned on the heat plate. During the cooling process after the 2 hours of heat treatment, the heat plate is subjected to natural cooling from its exposed surface area to the ambient temperature as well as conduction by the specimen above it. The specimen undergoes cooling from its top surface area exposed to ambient temperature. Residual stresses will be induced due to the uneven cooling rate between the top and bottom surface. The heat transfer via its side surface area (10 x 4

mm) is assumed to be even on all 4 sides with negligible difference due to the symmetry of the shape of the specimens, hence they do not induce additional residual stress.

Upon the initiation of the cooling process, the top surface of the specimen is exposed to ambient air temperature while the bottom surface is in direct contact with the cooling heat plate with a higher temperature $T_1^{\circ}\text{C}$ as illustrated in Figure 34 – after heat treatment.

As the cooling commences, the rate of cooling and correspondingly the rate of contraction reduces as the depth of the material increases with respect to the top surface - the rate being fastest on the top surface and slowest at the bottom surface. During the uneven cooling process, the bulk of the material at the top surface experiences induced tensile stress due to its faster rate of contraction constrained by the bottom surface. In contrast, the bottom surface experiences compressive stress due to its slower rate of contraction as illustrated in Figure 34 – defined as “Mechanism 1”. The magnitude of internal forces induced in Mechanism 1 is expected to be relatively larger than that induced in subsequent mechanisms, as the thermal gradient between the specimen itself with respect to the surrounding is at its maximum. However, the actual amount of residual stress induced from Mechanism 1 is dependent on other factors which will be elaborated in subsequent Mechanism 2 and 3. The main driving force of Mechanism 1 is the restrained contracting top region due to the bottom region.

As the cooling progresses, the temperature of the upper surface of the specimen approaches that of the ambient temperature such that contraction in the upper layer becomes insignificantly small as illustrated in Figure 34 “Mechanism 2”. The region directly below the top “non-contracting” layer continues to cool and undergoes contraction. This contraction process is restrained by the top “non contacting” layer

and the bottom warmer region which is contracting at a relatively slower rate due to the heat plate at the bottom. This restrains in contraction induces tensile stress on the middle region, while inducing compression stress on the top non-contracting region and the bottom slower contracting region as illustrated in Figure 34 “Mechanism 2”. Using Figure 2 from the literature review, the Heyn spring model presents the balance of internal forces within a system maintaining an equilibrium. Applying the balance of internal forces within the specimen in equilibrium, the magnitude of induced compressive force on the top and bottom region should be balanced by the tensional force in the middle contracting layer, indicating that the induced compressive force on the top and bottom region is relatively smaller than that of tensional force in the middle region. Given that stress is defined as the force acting on a unit area, the magnitude of the induced residual stress in each layer is dependent on the thickness of the affected region. Having induced thermal forces acting over a larger region, the induced stress is expected to be lower than if the same magnitude of thermal force were acting over a smaller region. The driving force of Mechanism 2 is the restrained contraction of the center layer due to the top and bottom regions.

As the cooling continues, the bulk of the top region of the specimen no longer undergo significant contraction as the thermal gradient between the material and ambient surrounding gets smaller as depicted in Figure 34 “Mechanism 3. The lower portion of the specimen still exposed to the warm heat plate continues to cool down and continues to undergo contraction. The mismatch in contraction between the upper non-contracting region and the lower contracting portion of the material results in induced compressive stress on the top region and tensile stress on the bottom region. Similar to that of mechanism 2, applying the balance of thermal-induced force based on the Heyn spring model, the magnitude of induced tensional stress at the bottom region and compressive stress on the top region is dependent on the relative thickness of each of these two affected regions. Towards the end of the

cooling process, the ratio of the non-contracting region is much larger than that of the contracting region, suggesting that the induced compressive residual stress on the bottom surface is larger in magnitude than the compressive stress on the top surface. This mechanism that takes place towards the end of the cooling process is named “Mechanism 3” as illustrated in Figure 34. The driving force of Mechanism 3 is the restrained contracting layer at the bottom region due to the top region.

Thermal residual stress develops via Mechanism 1, Mechanism 2 followed by Mechanism 3 in this mode of air cooling with a bottom warm plate. The final induced residual stress on the specimen depends on the combination of 2 factors:

- a. The relative duration of time spent in each mechanism throughout the cooling process.
- b. The magnitude of residual stress induced in each affected region, which has an inverse relationship with the thickness of the affected layer region.

The 2 factors mentioned above are highly dependent on the treatment temperature itself and the ambient temperature. Higher treatment temperature will likely result in a longer duration spent in Mechanism 1, as it takes a longer time for the top surface of the specimen to reach near the ambient temperature for Mechanism 2. Similarly, a lower treatment temperature will result in a longer duration spent in Mechanism 2, as a relatively shorter time is required for the top surface to reach near the ambient temperature. Lower starting temperature also results in a lower effect from Mechanism 3 as the driving force behind this mechanism due to contraction of the bottom portion will be too small. The treatment temperature also determines the starting temperature of the bottom heat plate upon the initiation of the cooling process, affecting the thermal gradient of the heat plate with the surrounding, determining the cooling rate of the heat plate. This affects the extend of mismatch in contraction rate between the bottom region and the region above it, the thickness of

the affected region, and the magnitude of induced residual stress for all three mechanisms.

As mentioned in section 3.1.1, the initial residual stress of the specimen has a relatively larger tensile residual stress on the bottom surface in contrast to the relatively lower compressive and tensile residual stress on the top surface. By optimizing the heat treatment temperature which is also the starting temperature of the bottom plate upon the start of the cooling process, it is possible to achieve a set of induced residual stress that is capable of canceling out the remaining residual stress in the specimens after heat treatment at a lower temperature than the vendor suggested temperature of 300°C. In this thesis, this new method will be termed as the “bottom heated-uneven air cooling” process, “BH-UAC” in short.

3.1.3. Advantage of Novel Method of Heat Treatment (Bottom Heated-Uneven Air Cooling) versus Conventional Annealing (Furnace Heated-Slow Cooling)

The conventional annealing methodology proposed by EOS for AlSi10Mg specimens manufactured via the LBPB process is to perform heat treatment at 300°C for 2 hours for residual stress relief, followed by slow and even cooling to prevent new thermal-induced residual stress. The conventional annealing methodology in this thesis will be termed as “furnace heated-slow cooling”, “FH-SC” in short. A comparison of resulting residual stress of BH-UAC and FH-SC will be investigated at a different treatment temperature of 160°C, 200°C, 300°C and 400°C. The differences in the residual stress results could be attributed to the two variables between these two sets of experiments – furnace heated versus bottom heated plate, and also slow cooling versus uneven air cooling. To understand if there is any difference in residual stresses induced due to the effect of a furnace heated and bottom heated plate, a further test is needed as shown in section 3.1.4.

3.1.4. Further Discussion of Novel Method of Heat Treatment (Bottom Heated-Uneven Air Cooling)

To investigate if there is any effect of heat treatment via furnace heated versus bottom heated treatment on residual stress, a comparison of resulting residual stress of BH-UAC and FH-UAC (furnace heated-uneven air cooling) will be investigated at a different treatment temperature of 160°C, 200°C, 300°C, 400°C and 500°C. Specimens subjected to FH-UAC are exposed to air cooling while still positioned within the furnace itself, rested on ceramic insulation material with relatively low thermal conductivity and high heat capacity. The high heat capacity characteristics of an insulation material serve as a heat source to the specimen positioned on it, gradually cool down during the cooling process, serving a similar role as that of the heat plate used in the BH-UAC process. The comparison of residual stress results between these two processes can then be used to verify if the difference in results in 3.1.3 is due to the difference in cooling rate itself, or if the difference in heating mode (furnace versus bottom heat plate) contributed to the difference too.

3.1.5. Effect of Novel Method of Heat Treatment (Bottom Heated-Uneven Air Cooling) on mechanical property and anisotropy

Heat treatment has been proven to deteriorate the mechanical properties of AlSi10Mg alloys manufactured by LBPB fusion such as tensile strength, hardness, and compressive strength. This is attributed to the breaking up of the inter-cellular eutectic structure of the Si phase when AlSi10Mg is subjected to heat treatment. From the literature review, compressive strength was found to reduce the most (56% drop in compressive strength and 47% drop in compressive yield strength) as compared to tensile strength (11% to 12% reduction) after heat treatment [166]. Hence in this experiment, the resistance to the compressive stress of the AlSi10Mg specimens is used as an indication of the mechanical property response to different heat treatment

temperatures with a cooling process discussed in section 3.1.2. Compressive strength is expected to reduce as the heat treatment temperature increases. With as-built AlSi10Mg specimens as a reference, while it has been established that the material strength becomes weaker in terms of tensile strength, yield strength, and hardness after 2 hours of heat treatment at 300°C [12, 32, 158, 160], 250°C [163, 165], 450°C, 500°C, 550°C [157], there has not been previous work done to investigate the presence of transition temperature for the mechanical property deterioration. The effect on compressive strength of the specimens after heat treatment temperature between 250°C to 300°C will be investigated to investigate the transition in material strength.

Figure 35: Illustration of cylindircal specimens machined via EDM from LBPB fusion process, in vertical and horizontal direction with respect to additive manufacturing build direction

Anisotropy is characteristic of parts manufactured by the LBPB method and homogenized by heat treatment. This unique property is due to the manufacturing process of LBPB, whereby parts are built layer by layer in the build direction (z-axis), whereas the building process in the x and y directions are continuous, based on the scan path of the laser beam. As seen in Table 1, the yield strength and ductility of parts manufactured by LBPB method are different in x-y direction as compared to the z direction in the as-built condition, with the yield strength in the z direction differing from the x-y direction by approximately 27%, and elongation differing by

36%. Upon heat treatment, the anisotropy gets eliminated and the material strength becomes uniform in both different directions.

In this experiment, cylindrical shape specimens of 13mm height and 10mm diameter are machined via EDM from rectangular cuboids manufactured by the LBPB fusion process, as illustrated in Figure 35 above. The cylindrical specimens are machined in the vertical and horizontal directions with respect to the build direction in the LBPB fusion process to investigate anisotropy in mechanical property changes at different heat treatment temperatures.

3.1.6. Microstructural Characterization for Residual Stress and Mechanical Property Changes

Literature reviews have shown that the mechanical property of AlSi10Mg is expected to deteriorate upon heat treatment at 300°C and above, and past work has established the relationship between mechanical property and the cellular network structure of the Si phase. With an increase in heat treatment temperature, the density of dislocations is expected to be reduced and analysed by EBSD. Each specimen subjected to different heat treatment temperatures is characterized via SEM and EBSD for examining the relationship between residual stress and mechanical property changes to microstructural changes. Characterization of samples via EBSD was performed on the top surface of the specimens. The analysis of microstructure in this study focuses on the top surface of the specimens due to the nature of the mechanism of residual stress induced as shown in Figure 34.

3.1.7. Remark on Novel Heat Treatment Methodology

The specimens used for this experiment are manufactured from LBPB process, after removal from the build plate via EDM without prior heat treatment. The initial residual stress values on the top and bottom surfaces in 2 perpendicular directions are measured via XRD and used as the basis of the residual stress changes upon

subjected to heat treatment. The mechanism of residual stress induction in the novel heat treatment method has been proposed on the basis of established mechanisms from previous studies done by other researchers. This proposed mechanism is based on residual stress induction due to restriction in expansion and contraction between adjacent layers of material, the effect of dominance of a certain mechanism stage in rapid cooling, and the derived inverse relation between the depth of residual stress influence and the magnitude of residual stress induced. The specimens are subjected to 3 different mode of heat treatment at different temperature – the conventional treatment termed as furnace heated-slow cooling (FH-SC), the novel method termed as bottom heated-uneven air cooling (BH-UAC) and also a verification test termed as furnace heated-uneven air cooling (FH-UAC). The effect of the novel heat treatment method (FH-UAC) and the conventional method (FH-SC) on mechanical property at different treatment temperature will be investigated, and compressive test is used for this experiment. Compression test is performed on the specimens in the transverse (LBPB build direction) as well as in the longitudinal direction (perpendicular to LBPB build direction) to investigate the effect on anisotropy. To correlate the mechanical property to the microstructural changes, material characterization is performed using SEM and EBSD.

3.2. Equipment and Processes

The specimens are manufactured via the LBPB fusion process, with process parameters based on proprietary AM parameters tested to be ideal settings by the manufacturer. Specimens are removed from the build plate via EDM without prior heat treatment without visible dimensional distortion. Cylindrical specimens for compression tests are wire cut by EDM process from cuboid specimens manufactured from the LBPB fusion process. Other equipment used for heat treatment, compressive

test, residual stress measurements, and microstructural characterization will be presented in this section.

3.2.1. Specimen Fabrication - Laser Beam Powder Bed Fusion Machine

Figure 36: Dehumidification of AlSi10Mg powders at 80°C prior to LBPB process

EOS metal powder was used for the fabrication of AlSi10Mg specimens. To ensure the quality of the powder, the powder was dehumidified at 80°C for 4 hours under vacuum conditions in a dehumidifying oven as shown in Figure 36 above.

During loading of the powder into the dispenser chamber, the powder was sieved through a sieve mesh using a shovel as shown in Figure 37 below. The sieve mesh ensures that powder particles larger than 90 μm size are removed and only smaller size powder particles are loaded onto the dispenser chamber.

Figure 37: Left: Sieving of powder through sieve mesh using shovel. Right: Remaining AlSi10Mg powder particles larger than 90 μm caught by sieve mesh.

The LBPB process was performed using EOS M290, with its infill parameters based on EOS default settings for EOS AlSi10Mg powder as shown in Table 6. Figure 38

below shows the alternating scan angle of 67° between each layer. The alternating scan angle randomizes any directional property caused by the unidirectional scan in each layer. This eliminates anisotropy of the manufactured part along with the directions perpendicular to the build direction. The combination settings used for laser power, scan speed, layer thickness, and hatch spacing equates to 73J/mm^2 of energy density used.

Table 6: Processing parameters for LBPB fusion process of AlSi10Mg samples – infill region

Scan Strategy	Stripe
Single Exposure Laser Power (W)	370
Scan Speed (mm/s)	1300
Layer Thickness (μm)	30
Hatch Spacing (mm)	0.13
Layer Orientation Angle ($^\circ$)	67
Stripe width (mm)	7
Overlap Stripes (mm)	0.02
Oxygen Level (%)	<0.1
Recoater Speed (mm/s)	150
Preheat Temperature ($^\circ\text{C}$)	35

Figure 38: Illustration of the alternating scan angle for each layers in LBPB process [193]

Figure 39: Left: Laser beam powder bed fusion in process chamber. Right: Specimens on build plate after completion. Build plate is made of aluminium alloy with dimension 252 x 252 x 30mm.

3.2.2. Residual Stress Characterisation – X-Ray Diffraction

The X-Ray diffraction methodology was used to measure the surface residual stress of the specimens. To ensure accuracy of the effect of treatment methodology on the residual stress relief, the measurement was performed on the same specimens before and after treatment for a direct comparison. 4 readings are obtained from each specimen, namely top surface longitudinal direction, top surface transverse direction, bottom surface longitudinal direction, and bottom surface transverse direction, as illustrated in Figure 33 (right).

The equipment used is Xstress 3000 G3 X-ray machine by StressTech in compliance with BS EN15305:2008 non-destructive testing standard. X-ray generation was via Cr $K\alpha$ radiation at 30kV and 9.0mA at 15 seconds exposure, deployed on the 311 planes. Measurement was done in 13 oscillations using the $\text{Sin}^2\psi$ method the ψ angle was varied between the tilt angles -38° and $+38^\circ$. The software used in the data analysis is Xtronic 1.12.0 by StressTech. The 2θ peak shift was determined using the cross-correlation method. The error bars were based on ISO/IEC Guide 98-3:2008 where the quality of the signals determines the uncertainty related to each measurement.

3.2.3. Surface Preparation for Microstructure Characterisation

Cross-section polisher SM-09010 by JOEL was used to polish the AlSi10Mg samples for microstructural characterization. The process involves milling via argon-ion accelerated at 6kV and an average beam current of 0.16mA in a vacuum environment.

Figure 40: Left: JOEL SM-09010 Cross Section Polisher. Middle: Zoomed in view of polished surface. Right: Specimen holder

To prepare the specimen surfaces for optical microscopy and SEM, Keller's reagent was used to etch the polished surface for 10 seconds.

3.2.4. Heat Treatment via Heat furnace

Furnace BRF 14/5-2416 by Elite Thermal Systems Limited was used for the furnace heat treatment of AlSi10Mg specimens, namely FH-SC and FH-UAC discussed in 3.1.3 and 3.1.4 respectively. Two different heat furnace heat treatment processes were done as follow:

- a. FH-SC treatment involves subjecting different specimens to 160°C, 200°C, 300°C and 400°C for 2 hours, followed by controlled furnace cooling at 2°C/min.
- b. FH-UAC involves subjecting different specimens to 160°C, 200°C, 300°C, 400°C and 500°C for 2 hours, after which the furnace door was opened for air cooling. The specimens were positioned on insulation ceramics during the

heating process, as well as throughout the cooling process. During cooling, the bottom ceramic act as a hot bottom surface to provide an uneven cooling condition between the top surface and the bottom surface of the specimen. Setup is as shown in Figure 41.

Figure 41: Left: Heat furnace BRF 14/5-2416 by Elite Thermal Systems Limited. Right: Interior view of heat furnace. Specimen positioned on top of ceramic insulation. Heating rod is located above the specimen.

3.2.5. Heat treatment via Bottom Heated Plate

Heat treatment via bottom heat plate was performed using XRD Bruker D8 Discover for the novel method of BH-UAC. The heat treatment temperature sets the surface plate temperature for the treatment itself. The treatment is performed under vacuum conditions for 2 hours at temperatures 160°C, 294°C, 200°C, 300°C, 400°C and 500°C for the specimens in Figure 33 for comparison with FH-SC and FH-UAC process in section 3.2.4. Cylindrical specimens as shown in Figure 35 for compressive stress test are also heat-treated at 164°C, 200°C, 300°C, 400°C and 500°C with this the same setup. The cooling process takes place immediately upon the completion of the heat treatment via natural air cooling. Only one specimen undergoes heat treatment at every one time to ensure consistency in heat transfer.

Figure 42: Setup of XRD Bruker D8 Discover for heat treatment with bottom heat plate. Left: Side view of specimen placed on the heat plate. Middle: Top view of specimen placed on the heat plate. Right: Graphite dome to provide a vacuum environment during heat treatment.

3.2.6. Compressive Test

Instron 5569 electromechanical test machine was used to perform compressive tests on the specimens with dimensions shown in Figure 35 conducted at room temperature. As mentioned in section 3.1.5, specimens were wire cut from their parent cuboid specimens in the longitudinal direction (perpendicular to build direction) and transverse direction (along the build direction in the LBPB process). The compressive force was applied on each specimen after subjected to heat treatment via bottom heat plate at 200°C, 294°C, 300°C, 400°C and 500°C described in section 3.2.5. The procedure and equipment used are in compliance with ASTM standard E9-19 [194]. Short specimens with a length to diameter ratio of 1.3 according to the standard, satisfies the requirement for obtaining compressive yield strength from the experiment. The compressive force is applied up to 45kN and stopped before failure occurs, with strain values sufficient to obtain yield strength. Yield stress values are obtained from the 0.2% offset method from the standard.

Figure 43: Left: Instron compressive test setup. Right: Specimens after the compressive loading till 45kN. Specimen exposed to 300°C experiences a larger compression displacement as compared to that of 294°C

3.2.7. Microstructural Characterisation

Microstructural characterization was performed using JOEL 7800F FESEM and EBSD using a backscattered electron detector by Oxford Instruments. Data acquisition and analysis were performed using the software Aztec and AztecCrystal. The top surface of the specimens which is furthest away from the bottom heated surface is selected for microstructural analysis. Specimens are polished using the cross-section polisher mentioned in section 3.2.3.

Figure 44: JOEL FESEM 7800F equipped with backscatter electron detector.

3.3. Minor Effects of other Methodologies on Residual Stress

This section presents some of the past work done to relieve residual stress of Maraging steel grade 300 (MS1), Inconel 718 (IN718) and AlSi10Mg by applying static magnetic field or cryogenic treatment. Figure 45 shows the specimens of MS1 and IN718 of dimensions 20 x 20 x 15mm, and AlSi10Mg of 8 x 3 x 3mm fabricated by LBPB process.

Figure 45: Specimens manufactured by LBPB process. Left: MS1 specimen with a strain gauge applied on its top surface. Middle: IN718 specimen with a strain gauge on its top surface. Right: AlSi10Mg specimen.

3.3.1. Application of Static Magnetic Field on MS1 and IN718

To obtain a uniform magnetic field and to minimize the effect of diminishing field strength with respect to distance from the poles, Permanent Magnet Quadrupoles (PMQ) derived from Halbach Array [195] was used. Permanent Neodymium magnetic cubes of N52 grade with 25mm dimension are arranged into 8 segments with configuration shown in Figure 46 below.

Figure 46: Left: Top view of 8 segments of 25mm neodymium magnets arranged into PMQ, specimen positioned at the center with a transverse direction parallel to the concentrated magnetic field. The direction of the arrow points towards away from the north pole. Right: Isometric view of PMQ setup.

The PMQ setup concentrates magnetic field into the center portion as illustrated in Figure 47 below. The simulation of magnetic flux density obtained in Figure 47 is based on an open source software Finite Element Method Magnetics (FEMM) [196] on 2 dimensional planar analysis. The accuracy of the software was described to be fast and accurate by Baltzis [197]. However, the quantitative results obtained assumes 2 dimensions only which might not be true in reality. The magnetic flux density in the center of the PMQ was measured using Magna Tesla Meter MG-701 to be approximately 0.72 Tesla as opposed to 0.91 Tesla obtained from the simulation. The discrepancy is due to the leakage of magnetic field in the 3rd dimension which was not accounted for in the 2-dimensional based software, which was verified by magnetic field reading in the Z direction using the Tesla meter. The magnetic field concentrated towards the center of the PMQ setup was predicted to be uniform in the Y direction as seen in Figure 47 and was verified to be true using the Tesla Meter.

Specimens are positioned at the center of the PMQ setup where magnetic flux is concentrated and uniform, and the duration of each treatment is 24 hours.

Figure 47: Magnetic flux distribution of PMQ setup simulated

3.3.2. Residual Stress Characterization by Hole Drilling: Central Hold Drilling and Electronic Speckle Pattern Interferometry

CHD was used for sub-surface residual stress measurement of MS1 and IN718 samples. Restan MTS3000 by SINT Technology was used for the hole drilling operation, based on ASTM E837 [198]. Drill bit with endmill diameter of 1.8mm is used to drill incremental steps in the center of a three-grid strain gauge rosette. K-RY61-1.5/120R rosette strain gauge was used. 200 grain sandpaper was used to grind the surface of specimens, followed by cleaning with alcohol with lint-free paper.

Figure 48: Numbering sequence of K-RY61-1.5/120R rosette strain gauge

EVAL 7 software developed by the same company performs back-calculation on the residual stress from the strain values obtained from Restan MTS3000. Strain released for each of the 40 drill steps is acquired until the maximum drill depth of 1mm. EVAL 7 software contain calculations compliant with ASTM E837 and also other algorithms, extending the scope of the standard. The ASTM standard does not factor

in the effect of hole eccentricity in the calculation, while the algorithm of HDM method [199] in EVAL 7 software performs eccentricity correction. The reliability of the eccentricity correction of the HDM method was compared with another software - H-DRILL [200], where the HDM method was proven to produce more accurate results. Plasticity occurs if the residual stress that develops near the hole during CHD is more than 80% of the yield stress of a thick specimen, the results can then only be used as "indicative" and not "quantitative", with an overestimation of the quantitative values [198]. An evaluation on the different calculation methods in EVAL 7 was done to see the effect of plasticity near the drilled hole on the deviation from the mean stress value [201]. When no plasticity occurs, the Integral method was found to have the least relative error, followed by HDM method, while the E837 Non-uniform method having the most. HDM method was used in these experiments as eccentricity exists in the holes drilled during CHD.

Instead of using strain gauges, electronic speckle pattern interferometry (ESPI) was used to measure the residual stresses of AlSi10Mg samples via the hole drilling method. The process also follows the ASTM E837 hole drilling standard, with the exception that it measures the surface distortion via ESPI and correlates the distortion to residual stresses based on the material properties. The equipment used is PRISM by Stresstech.

Figure 49: Hole drilling via ESPI using PRISM by Stresstech

3.3.3. Effect of Static Magnetic Field Treatment on Residual Stress

Figure 50 shows the average residual stress profile of 4 different conditions - as built condition, after magnetic field treatment, after heat treatment, and a combination of heat followed by magnetic field treatment. The residual stress is compressive near to the surface, transits from compressive to tensile between 0.3 to 0.4mm from the top surface, and remain tensile in deeper depth for all 4 conditions. Magnetic field appears to have reduced the compressive residual stress at the top surface between 0 to approximately 0.4mm, but no significant effects were observed for depths deeper than 0.4mm in the tensile stressed region. The peak compressive residual stress was reduced by a maximum of about 22% from -489 MPa to -379 MPa at 0.125mm depth. Heat treatment (490°C for 6 hours) reduced the residual stress significantly by a maximum of 72% from -489 MPa to -134 MPa at 0.125mm depth, and a relatively consistent reduction in residual stress by about 50% in the tensile region beyond the 0.4mm depth. Beyond the 0.4mm depth, the residual stress profile of the specimens for all 4 test conditions increases at a decreasing rate and stabilizes near to the end of the drill depth. Magnetic field treatment after heat treatment has no effect on the residual stress distribution.

Figure 50: Effect on residual stress after heat treatment, magnetic field treated, and heat treated followed by magnetic field treatment.

Figure 51 shows the average residual stress of Inconel 718 with respect to depth in as built condition and after magnetic field treatment. Magnetic field treatment has negligible effect on the residual stress profile of Inconel 718, from the surface to 0.6mm depth. Beyond 0.6mm depth, the stress profiles of the two conditions follow different trends.

Figure 51: Effect of static magnetic field treatment on IN718

It can be observed that while static magnetic field has a mild stress relieving effect in the subsurface region of maraging steel, there is negligible effect on Inconel 718. The results suggest the dependence of this methodology on the magnetic properties of the material itself.

3.3.4. Effect of Cryogenic Treatment on AlSi10Mg

AlSi10Mg specimens were subjected to cryogenic treatment at -196°C for 24 hours before controlled warming back to room temperature. The specimens were positioned upright as per Figure 45(right), directly below the cooling outlet where nitrogen gas at -196 °C was injected into the chamber cryogenic chamber.

Residual stress was characterized by the ESPI method using PRISM by Stresstech. 5 points were drilled on the specimens shown in Figure 52, each hole drilled on a new sample. Two measurements were taken for each point, and the average of the residual stresses was taken and shown in Figure 53.

Figure 52: Residual stress measurement on 5 positions on AlSi10Mg samples

Figure 53: Effect of cryogenic treatment on residual stress of AlSi10Mg

It can be seen that the method of cryogenic treatment had reduced the residual stress of P1 out of the 5 other points. This reason could be due to the relative positions of the 5 points with respect to the source of cryogenic fluid injected from the top nozzle in the chamber. As this treatment was outsourced to an industrial thermal treatment company, in-situ temperature taking and further analysis of the cause could not be done. However, the residual stress reduction in P1 could be due to uneven expansion and contraction due to uneven cooling and warming process. Position P1 was nearer to the cooling fluid outlet, therefore having a lower temperature than the other positions P2 to P5. During the warming process after the treatment, the larger thermal gradient between P1 and the surrounding resulted in a faster rate of expansion as compared to that of P2 to P5. This might have induced compressive stress on P1, resulting in the results obtained in Figure 53.

3.3.5. Remarks on Results

The effect of static magnetic field on residual stress of MS1 was shown to be less effective than that of conventional heat treatment, and negligible effect on Inconel 718. While the literature review suggests that alternating magnetic fields have a larger effect on residual stress especially in cryogenic conditions, the limitation of equipment and resources available makes it not possible to conduct further tests in this direction. Also, IN718 is known to have an improvement in mechanical properties after heat treatment, reducing the benefits of developing an alternative method for stress relief for this material. The direction of research is focused on AlSi10Mg instead which is known to have a larger technological gap whereby the current methodology of conventional heat treatment results in a reduction in mechanical strength. The cryogenic treatment of AlSi10Mg came to a halt as further experiments to investigate the treatment chamber could not be done, as the treatment was outsourced to an industrial thermal treatment company.

3.4. Remarks on Methodologies

The concept of the novel methodology for residual stress reduction without compromise of mechanical properties of AlSi10Mg specimens is presented in section 3.1. Several experiments were conducted to verify the concept behind the methodology and properties characterized using the equipment and process shown in section 3.2. Sets of experiments conducted are as follow:

- a. The effect of the vendor suggested heat treatment process with FH-SC was compared with that of BH-UAC on the residual stress reduction at different temperatures. The optimum temperature required for the most efficient residual stress relief for both methods was compared.
- b. The effect of FH-UAC versus the new proposed method BH-UAC at different temperatures.

- c. The effect of different heat treatment temperatures using the new BH-UAC method on anisotropy and compression strength of the AlSi10Mg specimens.
- d. Analysis of the microstructure of AlSi10Mg specimens after subjected to BH-UAC at different temperatures via optical microscope, SEM and EBSD.

Chapter 4. Discussion of Experiment Results for Validation of Novel Heat Treatment Methodology

This chapter presents the results obtained from the experiments conducted in 0 and analysis of the results. The effect of residual stress of the top and bottom surface in longitudinal and transverse direction are as follow.

4.1. Comparison of Residual Stress Reduction after Novel Heat Treatment Method (Bottom Heated-Uneven Air Cooling) versus Conventional Annealing (Furnace Heated-Slow Cooling) at Different Temperature

The purpose of this experiment is to find out the optimum temperature required for residual stress relief using the annealing methodology proposed by EOS – heat treatment in a furnace for 2 hours followed by slow and controlled cooling (FH-SC). This experiment also strives to investigate the effect on residual stress by the new proposed method of heat treatment by BH-UAC discussed in 3.1.2, and also obtain the optimum temperature required for residual stress relief. The two variables used in this experiment are namely the nature of heat source – furnace environment versus heat source from a bottom plate, and the nature of cooling – slow and controlled cooling versus uneven air cooling.

Figure 54 shows the resulting residual stress after 2 hours of heat treatment via FH-SC, BH-UAC, and the average as-built residual stress without treatment. The residual stress of the as-built specimen is obtained by taking the average of the specimens used before FH-SC and BH-UAC. The as-built condition denoted by the green colour line shows the as-built residual stress condition of the specimen after EDM cutting from the LBPB build plate. The top surface of the specimen is compressive (-35MPa to -42MPa) in the longitudinal direction and slightly tensile (14MPa to 18MPa) in the transverse direction. The residual stress is much higher on the bottom surface (100MPa to 128MPa) in both directions. The maximum residual stress value of

128MPa is less than 50% that of the average yield strength of the material shown in the later part under section 4.3, hence there is insignificant plastic deformation due to the residual stresses.

As observed in Figure 54, the vendor EOS suggested heat treatment of 300°C via the traditional method of slow furnace cooling is the minimum temperature required for the residual stress relief. At a temperature higher than 300°C, the specimen remained residual stress relieved with remaining stresses within ± 10 MPa, suggesting that 300°C is sufficient to relieve the specimen from residual stress.

Figure 54: Effect of different heat treatment temperatures on residual stress: a novel method of BH-UAC vs convention FH-SC with respect to the original residual stress before heat treatment. (a): Top surface longitudinal direction, (b): Top surface transverse direction, (c): Bottom surface longitudinal direction, (d): Bottom surface transverse direction

The resulting residual stress from heat treatment via the new BH-UAC method is represented by the orange curve in Figure 54. As opposed to the results from the FH-SC method, the BH-UAC method achieved residual stress relief to ± 10 MPa at 200°C instead of 300°C for all 3 directions and surfaces except for the top longitudinal direction, having a mild compressive residual stress value of -28MPa from its original average stress of -42MPa. The final residual stress after 200°C treatment on the top surface transverse direction, bottom surface longitudinal, and

transverse direction remain within ± 10 MPa. Treatment at 300°C via the new method of BH-UAC however, induced larger tensile residual stress on all 4 readings as compared to that of 200°C.

The residual stress results shown in Figure 54 is due to the residual stress induced on the part due to the rapid and uneven cooling process, and different heat treatment temperature results in a different dominating mechanism. The dominating mechanism determines the magnitude and nature of residual stress induced due to the rapid cooling in this novel heat treatment method. As mentioned in section 3.1.2, the heat treatment temperature which is equivalent to the initial temperature of the specimen and the bottom heat plate will determine whether Mechanism 1 or Mechanism 2 is the dominating stage. The effects of Mechanism 3 will become significant only if the initial temperature of the heat plate is high enough to continue to experience sufficient thermal gradient across the depth to induce significant stresses in each layer. In Mechanism 3, The tensile effect of the bottom contacting layer will however be larger than the compressive effect on the top due to the relatively thinner layer at the bottom layer as illustrated in Figure 34.

The new method of BH-UAC induced a slight compressive residual stress on the top surface of the as-built specimens at a lower heat treatment temperature of 160°C while inducing tensile stress at a higher temperature at 300°C and 400°C. 200°C appears to be the transition temperature where mild tensional and compression stress is induced on the top surface in the longitudinal and transverse direction respectively. On the bottom surface, the heat treatment process induced compressive stress for all treatment temperatures for both methods. Based on the mechanism discussed in section 3.1.2, Mechanism 2 is dominant in the BH-UAC method at a low temperature of 160°C, as the specimen takes a relatively shorter time for the top surface to cool down to near ambient temperature. The influence of Mechanism 3 is the weakest as

the driving force of Mechanism 3 being weaker due to the low thermal gradient between the low-temperature bottom surface and the surrounding. This accounts for the compressive stress induced on the top and bottom surface at 160°C. The compressive stress at the bottom surface is relatively larger than the top due to the combined effect of Mechanisms 1 and 2, while the compressive effect on the top surface due to Mechanism 2 is reduced due to the tensile effect from Mechanism 1.

At a higher temperature of 300°C and 400°C, the tensile effect on the top surface increases significantly as Mechanism 1 is the dominating process. The stress-induced on the bottom surface is still compressive due to the combined effect of mechanisms 1 and 2. Although the influence of Mechanism 3 increases at higher temperature, the compressive force induced in the non-contacting region as shown in Figure 34- Mechanism 3 is spread over a relatively larger area than the bottom thinner region, hence reducing the compressive stress induced on the top region. Thus, tensile residual stress is induced in the top surface at higher temperature than at lower temperatures due to the dominating Mechanism 1. Compressive stress is still induced on the bottom surface but smaller in magnitude as compared to that at a lower temperature of 200°C due to the tensile effect from Mechanism 3.

To summarize the results discussed, the AlSi10Mg specimens manufactured from the LBPB process after removal from the build plate via EDM suffer the most tensile residual stress on the bottom surface. The top surfaces of the specimens are mildly tensile and compressive. The vendor suggested annealing furnace temperature of 300°C has been proven to be the minimum temperature required for complete stress relief for this material which is aligned with the results from literature reviews. A new heat treatment method BH-UAC was found to be successful in relieving residual stress at a lower temperature of 200°C. 200°C also appears to be the transition temperature where there are a slight compression and tensile induced stress on the top

surface based on the mechanism discussed in section 3.1.2. The residual stress values corresponding to different treatment temperatures for both methodologies are found to be different. However, up till this point, it is inconclusive to attribute this difference due to the different cooling process - uneven cooling process versus controlled furnace cooling, or it is due to the nature of the heat source being a furnace environment or from the bottom surface of the specimen. This will be addressed in section 4.2.

4.2. Comparison of Residual Stress after Novel Heat Treatment Method (Bottom Heated-Uneven Air Cooling) versus Furnace Heated-Uneven Air Cooling at Different Temperature

The experiment in this section is to validate if there is any effect on the residual stress relief due to the nature of heat source direction - heat treatment via furnace environment versus heat treatment from the bottom surface of the specimen. This experiment is required to verify if the difference in residual stress result shown in section 4.1 is attributed to the nature of cooling itself or attributed to the nature of the heat source during the treatment. For both sets of experiments, the specimens undergo uneven air cooling with a higher temperature on the bottom surface as per the process explained in section 3.2.4 and 3.2.5 respectively. The variable of these two sets of experiments is the source of heat treatment – furnace heated environment versus bottom heating. The residual stress results are presented in Figure 55 below:

Figure 55: Effect of different heat treatment temperatures on residual stress: a novel method of BH-UAC vs FH-UAC heat treatment. (a): Top surface longitudinal direction, (b): Top surface transverse direction, (c): Bottom surface longitudinal direction, (d): Bottom surface transverse direction

It can be observed from Figure 55 that the residual stress from the two processes follows a similar trend, therefore suggesting the same mechanism of residual stress induction took place for both processes as shown earlier in Figure 34. While the mechanism of residual stress induction is the same, the duration of the cooling process undergoing mechanisms 1, 2, and 3 is different, the ambient and bottom surface of the specimens are also different. The ambient temperature in the furnace is higher than that of the bottom heat plate setup during the cooling process due to the nature of the equipment and setup. The heating source of the furnace is located above the specimen which will continue to transmit heat into the ambient environment during the cooling process. In contrast, the heat source of the bottom heat treatment process is located at the bottom of the specimen on the heat plate, therefore having relatively less effect on the ambient temperature but more of the bottom surface of the specimen itself. Comparing the relative size of the heating rod in the furnace and that of the bottom heat plate, the time required for cooling is relatively longer for the furnace as compared to that of the bottom heated plate.

At a low temperature of 160°C and 200°C, the FH-UAC process induced less tensile stress on the top surface as compared to the BH-UAC process. This is because the

duration of time spent in Mechanism 2 is longer in the FH-UAC method as compared to that of the BH-UAC method, with the top portion of the specimen in the furnace reaching the ambient temperature faster due to the relatively higher ambient temperature in the furnace environment. Mechanism 2 induces compressive stress on the top surface that cancels off a portion of the tensile stress induced on the top surface from Mechanism 1, resulting in an overall less tensile effect. The bottom surface shows no obvious difference with BH-UAC achieving a more compressive effect in the longitudinal direction at 200°C, while FH-UAC achieving a more compressive effect in transverse at 200° and both directions at 160°C.

At a higher temperature of 300°C, 400°C, and 500°C, there is no clear increase or reduction in tensile stress-induced on the top surface, with a lower tensile stress impact from the FH-UAC method than from the BH-UAC method in the longitudinal direction and the reverse trend for transverse direction. At the bottom surface, the induced tensile residual stress from the BH-UAC treatment is larger than that of the FH-UAC treatment. This can be attributed to a poorer heat transfer between the bottom insulation ceramic to the specimen above in the FH-UAC treatment, due to the highly textured surface of the ceramic surface, therefore reducing surface contact with the specimen and thus, the less tensile effect on the bottom surface from Mechanism 3.

A similar trend in residual stress profile can be observed for FH-UAC process and BH-UAC. Although there are variances in the magnitude of residual stress, the similarity in trend suggests that specimens in both sets of experiments had undergone the same mechanism as illustrated in Figure 34. The difference in magnitude is due to the different setup that results in a different temperature gradient, and thus affecting the magnitude of residual stress-induced. The results in this section proved that the difference in residual stress trend observed in Figure 54 was due to the cooling process itself and not due to the mode of heat treatment – furnace heated versus

bottom heated. With the results from sections 4.1 and 4.2, we can conclude on the effect of uneven cooling on the trend of residual stress induced at different temperatures.

4.3. Effect of Novel Heat Treatment Method on Compressive Strength and Anisotropy at Different Temperature

The experiments in this section are to examine the effect of different temperatures using the bottom heat treatment methodology on the mechanical property of AlSi10Mg. As compressive strength deteriorated the most as compared to hardness and tensile after heat treatment as shown in Table 3, compression test is used for this study. While it is well established from many previous works that higher temperature will result in a reduction in the material strength, this experiment is to confirm that the bottom heat treatment methodology will produce the same effect as furnace treatment condition used in previous works from the literature review. This experiment also aims to investigate the presence of any transition temperature that results in a sudden reduction in material strength, and intermittent temperatures between 200°C to 300°C were tested. This experiment also aims to investigate the treatment temperature of which the anisotropy of the material gets eliminated.

4.3.1. Compressive Stress-Strain Curve at Different Temperature

The effect of the treatment temperature of BH-UAC on the compressive strength of the specimens is shown in Figure 56 and Figure 57 for longitudinal and transverse direction respectively.

Figure 56: Compressive stress vs strain of AlSi10Mg specimens subjected to different heat treatment temperatures via BH-UAC in the longitudinal direction. Top: Stress-strain curves until a maximum load of 45kN. Middle: Enlarged view of L1. Bottom: Enlarged view of L2.

Figure 57: Compressive stress vs strain of AlSi10Mg specimens subjected to different heat treatment temperatures via BH-UAC in the transverse direction. Top: Stress-strain curves until a maximum load of 45kN. Middle: Enlarged view of T1. Bottom: Enlarged view of T2.

As seen in Figure 56 and Figure 57 (top), the compressive stress-strain curves of the AlSi10Mg specimens are similar for as-built and treatment temperatures between 200°C to 294°C for both longitudinal and transverse direction, with respect to the LBPB build direction as described in section 3.2.6. Upon transition from 294°C to 300°C heat treatment, the compressive strength of the specimen undergoes a drastic and sudden change with a sudden reduction in elastic modulus, and an increase in ductility. The stress-strain curves of the specimens subjected to 300°C, 400°C, and 500°C follow a similar trend.

From the enlarged view of L1 in Figure 56 (middle) and T2 in Figure 57 (middle), it can be seen that the compressive stress curve for as-built and 200°C treated temperature are identical with negligible difference. With an increase in heat treatment temperature to 250°C, 275°C, and 288°C, there is a gradual systematic shift in the stress-strain curve upwards by approximately 10 to 15MPa for each increment, suggesting a gradual and slight increase in compressive strength. Upon heat treatment at 294°C, there is a slight drop in the stress-strain curve from that of 288°C. As seen in the enlarged view of L1 in Figure 56 (middle), the stress-strain curve of 294°C starts to deviate from the common trend of the other curves from 0.075 strain onwards, portraying a slight increase in ductility.

4.3.2. Effect of Heat Treatment Temperature on Yield Strength and Anisotropy

The compressive yield stress also shows a sudden reduction when the heat treatment temperature changes from 294°C to 300°C as shown in Table 7. As presented in Table 7, the yield stress of as-built specimen in the transverse direction is 308MPa, similar to the value of 317MPa obtained by Aboulkhair [166] in Table 4. The yield stress of as-built condition and that of 200°C are identical in both longitudinal and transverse directions differing by just 1 to 2%. As the temperature increases from

200°C to 250°C, 275°C and 288°C, the yield strength increases as well to its maximum value at 288°C.

Table 7: Compressive yield stress via 0.2% offset of AlSi10Mg specimens subjected to heat treatment from bottom surface at different temperatures

<i>Yield Stress via 0.2% offset (MPa)</i>	<i>Longitudinal</i>	<i>Transverse</i>	<i>% Difference between Longitudinal & Transverse</i>
<i>As-Built</i>	237	308	30%
<i>200°C</i>	240	301	25%
<i>250°C</i>	256	315	23%
<i>275°C</i>	279	340	22%
<i>288°C</i>	298	355	20%
<i>294°C</i>	268	315	18%
<i>300°C</i>	135	143	6%
<i>400°C</i>	129	127	-2%
<i>500°C</i>	121	126	4%

However at 294°C, the specimen exhibits a slight reduction in yield stress by 10 to 11% in the longitudinal and transverse direction as compared to the specimen treated at 288°C. The region between 294°C and 300°C marks a transition point, where addition of 6°C from 294°C to 300°C will result in a significant reduction of yield stress by approximately 50% and 55% in the longitudinal and transverse direction respectively. The transition from 300°C to 400°C saw a slight reduction in yield stress by 6% and 11% in the longitudinal and transverse direction. From 400°C to 500°C, the yield stress changed insignificantly by 6% and 0.7% in the longitudinal and transverse direction. Comparing the yield stress reduction after treatment at 500°C from the as-built condition, the transverse yield stress reduced by 59%, while the compressive yield stress dropped by 47% after T6 heat treatment at 520°C [166].

This is attributed to the precipitation strengthening effect due to the aging process in the T6 treatment.

It can be seen that anisotropy in terms of yield strength reduces with increasing heat treatment temperature, with an abrupt reduction between 294°C and 300°C. The yield stress in transverse direction differs from that of the longitudinal direction by approximately 30% under as-built conditions, by 25% after 200°C treatment, and reduces gradually until 18% after treatment at 294°C. After the transition temperature of 294°C, the yield stress in the transverse direction differs from that of the longitudinal direction by approximately 6% after 300°C treatment, 2% after 400°C treatment, and 4% after 500°C treatment.

4.3.3. Effect of Heat Treatment Temperature on Final Height after Compression and Anisotropy

Table 8: The final height of AlSi10Mg specimens subjected to heat treatment from bottom surface at different temperature after the application of 45kN of compressive force

<i>Final Height after Compression (mm)</i>	<i>Longitudinal</i>	<i>Transverse</i>	<i>% Difference between Longitudinal & Transverse</i>
<i>As-Built</i>	<i>10.43</i>	<i>10.84</i>	<i>3.93%</i>
<i>200°C</i>	<i>10.43</i>	<i>10.83</i>	<i>3.84%</i>
<i>250°C</i>	<i>10.51</i>	<i>10.90</i>	<i>3.76%</i>
<i>275°C</i>	<i>10.55</i>	<i>10.99</i>	<i>4.17%</i>
<i>288°C</i>	<i>10.43</i>	<i>10.96</i>	<i>5.11%</i>
<i>294°C</i>	<i>10.19</i>	<i>10.88</i>	<i>6.77%</i>
<i>300°C</i>	<i>6.44</i>	<i>6.39</i>	<i>-0.78%</i>
<i>400°C</i>	<i>6.36</i>	<i>6.33</i>	<i>-0.47%</i>
<i>500°C</i>	<i>6.36</i>	<i>6.33</i>	<i>-0.47%</i>

Table 8 shows the final height of each of the specimens after subjected to the compressive stress of 45kN. The final height of each specimen is an indicative measure of the ductility of the specimens in response to compressive stress. The percentage difference in the final height of the specimens between longitudinal and transverse directions is an indicative measure of the change in anisotropy properties of the specimens after being subjected to different heat treatment temperatures. Similar to the yield stress results in Table 7, the anisotropy characteristics of AlSi10Mg specimens reduce significantly after heat treatment of 300°C and above with less than 1% difference. There is a sharp peak by almost 2 times in anisotropy after heat treatment at 294°C compared with treatment at 200°C and as-built condition. However, the anisotropy in terms of compressive deformation increases as temperature increase from as-built condition to 294°C, reaching a maximum after treatment at 294°C.

4.3.4. Remarks on Compressive Test Results

The yield stress results suggest that heat treatment at 300°C and above is undesirable to the compressive property of AlSi10Mg if strength is an important consideration for its functional applications. Heat treatment at 200°C preserves the original compressive properties of the as-built condition. Treatment at 288°C yields the largest yield strength of the material. Although anisotropy still exists relatively significantly in the parts after low-temperature heat treatment (294°C and below), the larger yield stress after low-temperature heat treatment as compared to high-temperature treatment compensates for it. However, although heat treatment of 300°C and above is detrimental to the compressive strength and also tensile strength based on literature reviews, the ductility of the material increases which is beneficial to its functional applications.

4.4. Remarks on Results

The new method of BH-UAV has been proven to be capable of relieving residual stresses at a lower temperature than conventional annealing, and also at a faster rate. The new method of BH-UAV utilizes uneven air cooling to induce residual stress such that it counters and relieves any residual stress that remains in the part after heat treatment. This allows a lower temperature of 200°C required for heat treatment as compared to that of the convention method of 300°C.

The combination of experimental results in 4.1 and 4.2 has proven that the main driving force behind this new methodology for residual stress relief is the uneven air-cooling rate after the heat treatment process. Residual stress results from the new method BH-UAC and FH-UAC both show a similar trend in resulting residual stress at different treatment temperatures, which proves that the fundamental mechanism of residual stress induction in Figure 34 is not affected by furnace heating or bottom plate heating.

Mechanical property test via compressive loading has shown that the lower temperature of 200°C used in the new BH-UAC method caused a negligible effect on the compressive yield strength of AlSi10Mg parts. A sharp change in the mechanical property was observed between the treatment temperature of 294°C and 300°C. Anisotropy was still observed in the specimen subjected to 200°C of treatment and the ductility of the specimens was lower than specimens that were exposed to a higher temperature.

Chapter 5. Correlation of Mechanical Properties and Residual Stress with Microstructure

This section presents the microstructural characterization of the AlSi10Mg specimens at different heat treatment temperatures conducted with bottom heat plate followed by an uneven air-cooling process (BH-UAC) described in section 3.2.5.

Optical Microscopy reviews the cross-sectional macro view and the top surface of the AlSi10Mg specimens shown in Figure 58. The top surface microscope image shows the scan tracks with hatch spacing approximately $100\mu\text{m}$, with a layer orientation angle of 67° with respect to previous layers. The cross-sectional image shows the melt pool layer thickness to be approximately $30\mu\text{m}$. These observations are aligned with the parameters used for the LBPB process as shown in Table 6.

Figure 58: Optical microscope images of AlSi10Mg specimens in as-built conditions. Left: Top surface view. Right: Cross-sectional view.

5.1. Effect of Si Microstructure on Mechanical property via Scanning Electron Microscopy

The SEM images obtained for as-built condition, 200°C, 294°C, 300°C, 400°C and 500°C are shown in Figure 59. The images are in the center of scan track region where the density of the Si phase is the highest.

Figure 59: Secondary electron images of AlSi10Mg samples subjected to different heat treatment temperature. a) as built condition, b) 200 °C, c) 294 °C, d) 300 °C, e) 400 °C, f) 500 °C

In line with literature reviews, AlSi10Mg specimens as built from the LBPB process is made up of cellular and nano-scale fibrous interconnected network of Si as shown

in Figure 59(a). The light grey phase shows the Si eutectic phase and the darker phase shows the supersaturated α -Al matrix. Upon heat treatment to 200°C for 2 hours, the network structure of Si phase is intact and lumps of Si structures start to form as shown in Figure 59(b). While some parts of the Si network structure become thinner, some portion starts to thicken and coarsens. Upon heat treatment to 294°C, coarsening continues on localized parts of the Si network structure, and new formation of precipitates start to form randomly along with the network structure as shown in the bright spots seen in Figure 59(c). Figure 60 below shows a high magnification view of the structure of the specimen after treatment at 294°C, showing an increase in precipitates in between the cellular fibrous network structure which were not observed in specimens subjected to treatment at 200°C. This increase in precipitates in between of the fibrous structure contributes to the increase in yield strength of the specimens obtained in Table 7.

Figure 60: Secondary electron images of AlSi10Mg subjected to 294°C, showing formation of precipitates between cellular network structure of Si.

The fibrous network structure at this point, however, is still visible and interconnected, contributing to the mechanical strength of the material. Upon transition of heat treatment temperature from 294°C to 300°C, there is a sharp change in the Si fibrous network structure where the interconnectivity gets broken up. While the tracks of fibrous structure could still be traced, the long chains are no longer continuous. The lighter shades of grey shown in Figure 59(d) is made up of a

combination of broken Si spheroid shape remaining from the previous fibrous structure and newly formed ones. At a higher heat treatment temperature of 400°C, the Si phase becomes more refined and smaller in size as seen in Figure 59(e). After 500°C of heat treatment, the silicon particles coarsened in size as compared to that of 400°C.

5.2. Observations of Grain Orientation Map from EBSD

The EBSD technique was used to analyze the microstructural changes that arise due to different temperatures used for the residual stress relief. The crystallographic orientation of the AlSi10Mg specimens subjected to BH-UAC treatment at different temperatures is represented by IPF maps in Figure 61 to Figure 66.

Figure 61: Inverse pole figure grain orientation and boundary map of the top surface of AlSi10Mg specimen in as-built condition.

Figure 62: Inverse pole figure grain orientation and boundary map of top surface of AlSi10Mg specimen after heat treatment at 200 °C.

Figure 63: Inverse pole figure grain orientation and boundary map of top surface of AlSi10Mg specimen after heat treatment at 294 °C.

Figure 64: Inverse pole figure grain orientation and boundary map of top surface of AlSi10Mg specimen after heat treatment at 300 °C.

Figure 65: Inverse pole figure grain orientation and boundary map of top surface of AlSi10Mg specimen after heat treatment at 400 °C.

Figure 66: Inverse pole figure grain orientation and boundary map of top surface of AlSi10Mg specimen after heat treatment at 500 °C.

The step size used for the EBSD scans from Figure 61 to Figure 66 was 0.35µm which limits the minimum grain size detectable to 0.35µm. LAGB are defined to be between 2° to 10° denoted by the grey boundary division lines. The colours of each grain follow the inverse pole figure shown in Figure 61, with grains oriented towards <001> direction to be red, green colour for <101> direction, and blue for <111> direction. While the cross-section of the specimen is made up of elongated grains from literature review, the grains from the top surface show the projection of the cross-section of the elongated cells which are equiaxed. The melt tracks can be observed with smaller grains concentrated along-track boundaries, larger grains form up the center of scan tracks similar to literature review [176]. Similar to the images obtained from the optical microscope in Figure 58, the overlay orientation angle of 67° can be observed between each melt track. Smaller grain sizes found along the intersection between each scan path were observed similar to the literature review

[158, 176]. This is due to the repeated exposure to heat from the laser, causing repeated melting and solidification. In the orientation map for all specimens, there is a common trend with preferred grain orientation in the $\langle 001 \rangle$ direction in the non-melt pool boundary as indicated by the red-coloured grains. Grains are orientated randomly with no preferred orientation along the melt pool regions for all specimens as well. The specimens used in these experiments were manufactured with 73J/mm^2 laser energy intensity as mentioned in section 3.2.1, and the grain orientation results are similar to that of specimens manufactured with 77.8J/mm^2 and 100J/mm^2 energy intensity from the literature review [176] as shown in Figure 30(a) and (b). For higher laser power of 122.2J/mm^2 and 144.4J/mm^2 , grains along the melt pool regions tend to also adopt a preferential orientation towards $\langle 001 \rangle$ LBPB build direction. Also, similar to the literature review mentioned in section 2.3.3, heat treatment has no significant effect on the grain size of AlSi10Mg [158].

5.3. Observations of Precipitates Distribution at Different Treatment Temperature

Figure 67 to Figure 72 below shows the high angle grain boundaries (reddish brown lines) and distribution of precipitates of Mg_2Si and intermetallic compounds $\text{Al}_{4.5}\text{FeSi}$ and $\text{Al}_9\text{Fe}_2\text{Si}_2$ after different heat treatment temperature via BH-UAC.

In as-built conditions as shown in Figure 67, specks of precipitates are found distributed mainly along high angle grain boundaries, an observation that is expected due to higher energy state along high angle grain boundaries. The precipitates are found mainly as individual specks instead of concentrated lumps. Due to the higher proximity of high angle grain boundaries along the intersection of scan tracks during LBPB, specks of precipitates are more densely populated along the scan track intersections.

Figure 67: EBSD map showing high angle grain boundaries and distribution of precipitates of Mg_2Si and intermetallic compounds $Al_{4.5}FeSi$ and $Al_9Fe_2Si_2$ in as build condition

Figure 68: EBSD map showing high angle grain boundaries and distribution of precipitates of Mg_2Si and intermetallic compounds $Al_{4.5}FeSi$ and $Al_9Fe_2Si_2$ after BH-UAC treatment at 200 °C

Figure 69: EBSD map showing high angle grain boundaries and distribution of precipitates of Mg_2Si and intermetallic compounds $Al_{4.5}FeSi$ and $Al_9Fe_2Si_2$ after BH-UAC treatment at 294 °C

Figure 70: EBSD map showing high angle grain boundaries and distribution of precipitates of Mg_2Si and intermetallic compounds $Al_{4.5}FeSi$ and $Al_9Fe_2Si_2$ after BH-UAC treatment at 300 °C

Figure 71: EBSD map showing high angle grain boundaries and distribution of precipitates of Mg_2Si and intermetallic compounds $Al_{4.5}FeSi$ and $Al_9Fe_2Si_2$ after BH-UAC treatment at 400 °C

Figure 72: EBSD map showing high angle grain boundaries and distribution of precipitates of Mg_2Si and intermetallic compounds $Al_{4.5}FeSi$ and $Al_9Fe_2Si_2$ after BH-UAC treatment at 500 °C

Upon heat treatment, as shown in Figure 68 to Figure 72, there is an obvious increase in density of Mg_2Si along grain boundaries, particularly in the center of scan tracks where the grain size is larger. Instead of existing as individual specks, Mg_2Si are now spread linearly along the grain boundaries. There is a general slight increase in the

percentage of intermetallic compounds $\text{Al}_{4.5}\text{FeSi}$ and $\text{Al}_9\text{Fe}_2\text{Si}_2$, but a more significant increase in the percentage of Mg_2Si from 0.3% in as built condition to 0.7% after 500°C of treatment. The results obtained are aligned with literature review which also reported increase in precipitation at higher temperature of heat treatment.

5.4. Novel Phenomena Observed - Relationship between Low Angle Grain Boundaries Density and Grain Orientation of AlSi10Mg

Figure 73 to Figure 78 below shows the semi-transparent view of the inverse pole diagram, band contrast, and boundary map of the top surface of specimens exposed to different heat treatment temperatures via BH-UAC. Grain boundaries are denoted by low angle and high angle grain boundaries. LAGB are defined by grains with misorientation angles between 2° and 10°, while high angle grain boundaries are defined by grains with misorientation larger than 10°. Low angle grains are shown in Figure 73 to Figure 78 via light grey colour as shown in the legend in Figure 73. The inverse pole triangle diagram showing the colour coding of each grain is also shown in Figure 73.

Figure 73: Semi-transparent inverse pole figure grain orientation, band contrast, and boundary map of the top surface of AlSi10Mg specimen in as-built condition.

Figure 74: Semi-translucent inverse pole figure grain orientation, band contrast, and boundary map of the top surface of AlSi10Mg specimen after subjected to 200 °C heat treatment.

Figure 75: Semi-translucent inverse pole figure grain orientation, band contrast, and boundary map of the top surface of AlSi10Mg specimen after subjected to 294 °C heat treatment.

Figure 76: Semi-translucent inverse pole figure grain orientation, band contrast, and boundary map of the top surface of AlSi10Mg specimen after subjected to 300 °C heat treatment.

Figure 77: Semi-translucent inverse pole figure grain orientation, band contrast, and boundary map of the top surface of AlSi10Mg specimen after subjected to 400 °C heat treatment.

Figure 78: Semi-translucent inverse pole figure grain orientation, band contrast, and boundary map of the top surface of AlSi10Mg specimen after subjected to 500 °C heat treatment.

For all specimens from Figure 73 to Figure 78, it is observed that the majority of the LAGB are concentrated within the red colored regions, indicating a majority of low angle boundaries being concentrated in grains with an orientation towards build direction of $\langle 001 \rangle$.

Figure 79 to Figure 84 below shows the grain orientation and boundaries map for specimens highlighting only grains orientated towards LBPB build direction of $\langle 001 \rangle$ within 0° to 10° , and 0° to 15° of tolerance. The inverse pole figure is shown in Figure 79. As seen in Figure 79 to Figure 84 (left), within the grains orientated in 10° variance from build direction, 52% to 61.3% of grain boundaries within these grains are low angle boundaries with misorientation angles between 2° to 10° , indicating a high concentration of dislocations within these grains. The density of LAGB reduces if the variance of grain orientation from $\langle 001 \rangle$ is increased to 15° as shown in Figure 79 to Figure 84 (right).

Figure 79: Grain orientation map showing grains with orientation within Left: 10° deviation from LBPB build direction of $\langle 001 \rangle$, Right: 15° deviation from LBPB build direction of $\langle 001 \rangle$ for specimens in as-built condition

Figure 80: Grain orientation map showing grains with orientation within Left: 10° deviation from LBPB build direction of $\langle 001 \rangle$, Right: 15° deviation from LBPB build direction of $\langle 001 \rangle$ for specimens after heat treatment at 200°C

Figure 81: Grain orientation map showing grains with orientation within Left: 10° deviation from LBPB build direction of $\langle 001 \rangle$, Right: 15° deviation from LBPB build direction of $\langle 001 \rangle$ for specimens after heat treatment at 294°C

Figure 82: Grain orientation map showing grains with orientation within Left: 10° deviation from LBPB build direction of <001>, Right: 15° deviation from LBPB build direction of <001> for specimens after heat treatment at 300 °C

Figure 83: Grain orientation map showing grains with orientation within Left: 10° deviation from LBPB build direction of <001>, Right: 15° deviation from LBPB build direction of <001> for specimens after heat treatment at 400 °C

Figure 84: Grain orientation map showing grains with orientation within Left: 10° deviation from LBPB build direction of <001>, Right: 15° deviation from LBPB build direction of <001> for specimens after heat treatment at 500 °C

Table 9 below shows the summary of density change of LAGB in grains, and density of grains with 10° and 15° variance from the build direction. It can be seen that the area of grains orientated within 10° from building direction forms up between 10.5%

to 17.83% of the entire area scanned via EBSD. Increasing the variance from build direction to 15° sees to a corresponding increase in the percentage of grains area to a range of 22.68% to 35.21%, which is approximately double the area that of 10° variance. Although the amount of <001> orientated grains area doubled from 10° to 15° variance, the corresponding percentage of low angle grain within the <001> grains reduced by approximately 21.1% to 26.9%.

The quantitative results indicate that the largest amount of low-angle grain boundaries in the specimen are concentrated in grains orientated within 10° from build direction of <001> which is aligned with visual observation of Figure 73 to Figure 78. The density of LAGB reduces as the orientation of the grains starts to deviate from the <001> direction.

Table 9: Percentage of LAGB in grains orientated towards build direction of LBPB, and percentage of grains with a variance of 10° and 15°

Treatment temperature of specimen	Percentage of LAGB in grains between 0° to 10° from build direction (%)	Percentage of LAGB in grains between 0° to 15° from build direction (%)	Percentage reduction in LAGB (%)	Percentage of grains within 10° from build direction (%)	Percentage of grains within 15° from build direction (%)	Percentage increase in grains area (%)
As Built	60.9	44.5	26.9	15.36	29.63	92.9
After 200°C	61.0	48.1	21.1	14.43	27.73	92.2
After 294°C	52.0	40.9	21.3	15.81	28.3	79.0
After 300°C	57.2	44.9	21.5	17.83	35.21	97.5
After 400°C	61.3	44.9	26.8	10.5	22.68	116
After 500°C	58.4	46.5	20.4	13.85	27.85	101

This phenomenon of having a high concentration of LAGB within the grains orientated towards LBPB build direction suggests the generation of residual stress during the grain growth stage of LBPB. Strains from the stress induced within each grain during grain growth cause dislocations within the grains.

5.5. Novel Phenomena Observed - Relationship between Low Angle Grain Boundary Density and Magnitude of Residual Stress Relieve

The results in Table 9 shows that the percentage of LAGB within 10° from build direction does not reduce with an increase in heat treatment temperature. Peak value is found at 200°C and 400°C while having the lowest value after subjected to treatment at 294°C as shown in Figure 85.

While the literature review shows that heat treatment will reduce LAGB after heat treatment [32] due to reduction of dislocations [182], results from past work also shows the possible effect of induced residual stress resulting in dislocation and increase in LAGB. This is further substantiated by the results shown in Figure 85, which shows the similarity in the trend of the magnitude of stress relief induced on the specimens with the trend of percentage of low angle boundaries in <001> orientated grains. The stress relieves magnitude curves of the bottom surface of the specimens shown in Figure 85 is obtained by the magnitude of the difference in residual stress of as-built condition and post-heat treatment, which represents the combined effect of the amount of thermal residual stress induced during the cooling process and stress relieved during the heat treatment. The largest effect on residual stress was after heat treatment at 200°C, which corresponds to a peak value of low angle grain boundary density. Upon heat treatment of 294°C, the effect on residual stress dropped to a minimum, which corresponds to a minimum trough of low angle grain boundary percentage as well. A similar trend is observed with 300°C and 400°C where the effect on residual stress increases with a corresponding increase in low angle grain boundary density, with a similar drop at 500°C. This similarity in trend between low angle grain boundary density in <001> orientated grains and the residual stress effect due to heat treatment for the bottom surface, suggests that strains from the stress induction during the uneven cooling process had induced

dislocations in grains orientated towards $\langle 001 \rangle$ direction. This is further substantiated with the similar trend of the KAM (kernel average misorientation) values represented by the yellow curve in Figure 85, which suggests a corresponding increase in misorientation due to dislocations generated from induced stresses.

As the magnitude of stress relief curves was the difference in residual stress between the as-built condition and the post-treatment, the values for the as-built condition are not included in Figure 85. However, the original value of KAM was 0.39, and the percentage of low angle boundaries was 60.9% for the as-built condition.

Figure 85: The similarity in the trend of KAM, percentage of LAB within 10deg from build direction, and compressive residual stress effect due to heat treatment.

5.6. Remarks on Results

Several novel phenomenon were observed by microstructural characterization in section 5.4 and 5.5. Firstly, LAGB was observed to concentrate in grains orientated towards the build direction of the LBPB process. The observation was made visually from the semi-transparent overlap of grain orientation and grain boundaries map. The trend was also quantified by the percentage of LAGB found in grains orientated within 10° tolerance from <001> direction, and comparing with that of 15° tolerance. Secondly, the magnitude of residual stress reduction and density of LAGB in <001> orientated grains at different treatment temperature were observed to follow the same trend. This shows that the new method of residual stress relief via inducing counter stresses has resulted in an increase in dislocations, which corresponds to the trend of KAM mean value as well. The increase in dislocation density resulted from the concentration of stress along existing dislocations when thermal stress was induced due to rapid cooling, resulting in an increase and branching of dislocations from existing ones. Larger magnitude of residual stress induced resulted in an increase in stress concentration along dislocations, resulting in a larger density of dislocations and LAGB. In contrast, conventional heat treatment methods for residual stress relieve are based on stress relaxation and a relatively higher treatment temperature is used, allowing recovery of the material by relieving stored internal energy through dislocation motion and subsequently a corresponding reduction in dislocation density.

Microstructural observation via SEM reveals the reason for the abrupt mechanical property deterioration between 294°C and 300 °C, which was due to the destruction of Si network structure that provided the mechanical strength of AlSi10Mg manufactured by LBPB. Grain orientation shows consistency with that of literature reviews, with unidirectional grains concentrated in the middle of LBPB scan tracks, and randomly orientated grains along the intersections of scan tracks. Smaller grains were found along the intersections of scan tracks and larger grains form up the

middle portion of the scan tracks. Precipitation of intermetallic compounds $\text{Al}_{4.5}\text{FeSi}$ and $\text{Al}_9\text{Fe}_2\text{Si}_2$ were found to increase slightly after exposure to heat treatment, while Mg_2Si was found to increase more significantly. While the literature review suggests the detrimental effect of the intermetallic compounds and the improvement in mechanical properties due to Mg_2Si precipitates, the experimental results in section 4.3 show that the effect of Mg_2Si on the compressive strength is less significant as compared to the change in the Si network structure after heat treatment.

Chapter 6. Conclusions and Future Work

6.1. Conclusions

The results in Chapter 4 show that the novel method of BH-UAC can relieve the residual stress to $\pm 10\text{MPa}$ at a lower temperature of 200°C , as compared to the conventional annealing temperature of 300°C via FH-SC. Although the top surface residual stress in the longitudinal direction is still at -28MPa , this mild and compressive residual stress on the surface is much less detrimental as compared to tensile stress in terms of bending load or fatigue.

This new method not only reduces the energy required for heat treatment but also reduces the duration required for the entire treatment process. The convention methodology of FH-SC requires slow and controlled cooling from 300°C to room temperature at approximately $2^\circ\text{C}/\text{min}$, which equates to more than 2 hours of cooling time, which is longer than the 2 hours heat exposure duration itself. The new methodology utilizes fast cooling which takes approximately 10 minutes to cool from 200°C to room temperature, which is 12 times faster than the slow cooling method. Although this new methodology of the BH-UAC process utilizes a bottom heat plate for heat treatment, it was proven that treatment using a furnace with uneven air cooling (FH-UAC) also produces the same residual stress trend but with slightly different magnitude due to the different cooling rate due to the environment.

The mechanism of the residual stress induction through this novel method of heat treatment was established based on past literature reviews, with a combination of concepts derived from LBPB and rapid cooling process. Based on established mechanisms derived from LBPB process, restriction in contraction of a layer results in an induction of tensile stress in that layer, while the adjacent restricting layer experiences compressive stress. The established mechanism of residual stress induction of rapid cooling of metals and tempered glass illustrates the concept of dominance of mechanism stages and the effect it has on the final residual stress induced in the part. With the application of Heyn's spring model on thermal tempering of glass, the concept of the inverse relationship between the magnitude of residual stress with the depth of the layer of influence was proven in the parabolic stress profile generated in glass, and hence incorporated into the proposed mechanism of residual stress induction from this novel method of heat treatment. The nature of the resulting residual stress and magnitude obtained from experimental results are aligned with the proposed mechanism.

The compressive stress test shows that the new method of BH-UAC at 200°C does not cause any reduction in compressive yield strength in specimens. There is a sudden reduction in yield stress from 294°C to 300°C, indicating there is an abrupt transition point between 294°C and 300°C. This is supported by the SEM images that show the fibrous network of Si phase still intact after 294°C of treatment, and the breaking up of the chain after 300°C of treatment. Compressive yield strength was found to be at its peak after treatment at 288°C, which was contributed to the increased precipitation in between the fibrous network of Si phase shown in the SEM image obtained at 294°C. Anisotropy was found to reduce with increasing temperature, and also undergoes an abrupt reduction in the transition from 294°C to 300°C. However, although there is still anisotropy existing in the specimen after being subjected to the new BH-UAC process at 200°C, the smaller yield stress value

of 240MPa in the longitudinal direction is still larger than that obtained for treatment temperature of 300°C and above. Hence, even with the existence of anisotropy in parts after the new method of the BH-UAC process, the compressive yield strength is still larger than a homogenous part that has undergone the conventional treatment at 300°C. The elongation, represented by the final height of the specimen after compression, also shows no changes after heat treatment via the BH-UAC method.

In section 5.4, a novel observation was made from the grain orientation maps obtained from the EBSD method, showing a larger amount of low-angle grain boundaries being found in grains orientated towards build direction. The observation was done visually via semi-translucent images of grain orientation, band contrast and boundaries maps show a higher concentration of LAGB in red-colored grains (orientated towards $\langle 001 \rangle$ LBPB build direction). The observation was also concluded with quantification by the percentage of LAGB within grains orientated towards $\langle 001 \rangle$ LBPB build direction.

A novel relationship was found with EBSD results showing a similar trend in LAGB with the residual stress reduction at different temperatures. A larger magnitude of residual stress reduction using the new method of BH-UAC means larger residual stress was induced on the specimen that counters the original stress. The induction of larger residual stress correspondingly leads to a larger strain and subsequently causing dislocations, which corresponds to an increase in low angle grain boundary density and also a higher KAM value. While brittle intermetallic phases such as $\text{Al}_9\text{Fe}_2\text{Si}_2$ and $\text{Al}_{4.5}\text{FeSi}$ were found to increase as the temperature of the treatment increase, the brittle effect due to these are insignificant as compared to the breaking up of eutectic Si structures, which is consistent with previous works in literature review stating the dominance of Si structure on the mechanical strength. The increase in the Mg_2Si phase along grain boundaries contributed to the increase in ductility after heat treatment.

Comparing with the conventional method of heat treatment at using FH-SC, while this new method of BH-UAC is successful in relieving residual stress at a lower temperature, require less cooling time, and retains the original compressive strength in as-built condition, it also comes with a compromise of anisotropy, and no improvement in ductility and elongation. Depending on the requirements and applications of the functional part this material is to be used for, the novel method of BH-UAC will be useful if ductility plays a less significant consideration where compressive loading is more important.

6.2. Future Work

Future work includes the following:

- Developing a modified version of BH-UAC for parts with complex geometry. This work uses specimens of a simple shape as a test of concept. However, in real life, functional parts are usually of a more complex shape and this methodology of BH-UAC cannot be directly duplicated and applied on such specimens. As proven in this thesis, the main factor that allows this method to work is the uneven air cooling process that is slower than quenching but faster than furnace-controlled cooling. Complex shape parts with different residual stress distribution will require customization of heating surfaces angled to provide surface contact and to result in uneven air cooling. While it might not be possible to perform a bottom heated methodology for the 2 hours duration for complex shape parts or relatively big specimens, the heating can be performed in a furnace as it was proven that furnace will produce similar trends on residual stress at different temperature as compared to bottom plate heating. The uneven air cooling process which is the key to this methodology can be achieved by customized heat plates to be placed in contact with targeted surfaces on the specimen after furnace heating.

Processes need to be developed such as controlling the heat plate temperature and duration, and jigs for quick installation of the heated plates onto the parts immediately after the furnace heating.

- Performing in-situ controlled heating of the bottom heat plate during the cooling process of the BH-UAC method. One of the main factors that affect the magnitude of induced residual stress on the treated part is the duration of time spent in each mechanism as discussed in section 3.1.2. Other specimens with different magnitudes of initial residual stress require a different magnitude of reversed residual stress induction for complete stress relief. Adding in-situ heating to the bottom plate enables the cooling rate of the bottom plate to be controlled, therefore improving the flexibility of this methodology.
- Performing in-situ controlled cooling on the top surface of the specimen during the cooling process of the BH-UAC method. While ambient air cooling is sufficient for the specimens used in this thesis with relatively low residual stress on the top surface, it might not be sufficient for other specimens with more significant residual stress on the top surface. Similar to the proposal of performing in-situ heating of the bottom surface, adding in-situ controlled cooling on the top surface of the specimen introduces flexibility of the methodology, enabling the cooling rate of the top surface to be controlled, directly enhancing the effect of induced residual stress on the top surface.
- Performing design of experiment (DOE) approach to three parameters – top and bottom surface cooling rate, and the heat treatment temperature itself. By combining in-situ cooling of the top surface and in-situ heating of the bottom surface during the cooling process of BH-UAC, the temperature of the heat treatment required to achieve the same residual stress relief changes as well.

The compressive test results show that the maximum compressive yield strength is achieved at a heat treatment temperature of 288°C due to increased Si precipitates. For a heat treatment temperature of 288°C, further tests can be conducted to obtain the required values of cooling and heating rate that will eliminate the residual stresses. This way, the methodology not only achieves a stress relief effect, but also achieves the ideal yield strength of the material.

- Performing tensile tests on specimens subjected to BH-UAC at different treatment temperatures. While compressive load was investigated in this study, tensile loading can be investigated in future work. While there is a transition point between 294°C to 300°C that leads to a sudden change in the compressive strength, future work can involve investigating the existence of transition temperature for tensile loading.
- Performing sub-surface residual stress measurement instead of just top surface measurement for a holistic understanding of residual stress distribution after heat treatment via BH-UAC.
- Performing microstructural characterization for specimens manufactured with higher energy intensity during the LBPB process. The literature review has shown that the number of grains orientated towards <001> build direction is affected by the energy intensity in terms of energy per unit area shown in Figure 30. Currently, the specimens used in these experiments were manufactured using 73J/mm² that resulted in scan track interactions having randomly orientated grains. Further experiments can be done to increase the energy intensity of the laser during LBPB to manufacture specimens with more grains orientated towards <001> direction by increasing energy intensity to 122.2J/mm² and above, and to investigate the distribution of LAGB in such specimens.

- In order to validate the mechanism of the residual stress induction due to the novel heat treatment method, the residual stress profile of the specimen can be obtained from computational modelling. By varying the bottom heat plate and specimen initial temperature and the residual stress profile obtained, the experimental results of the surface residual stress can be used to gauge the accuracy of the model results and the mechanism of stress induction can be verified via the residual stress profile throughout the specimen.

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Publications

Journal Papers:

- Lim, C. H., Li, H., Krishnan, M., Novel method for residual stress reduction in AlSi10Mg parts manufactured by LBPB without compromising mechanical properties. (In preparation)
- Lim, C. H., Krishnan, M., Li, H., Effect of heat treatment on compressive strength of AlSi10Mg manufactured by selective laser melting (In preparation)

Conference Paper:

- Lim, C. H., Krishnan, M., & Li, H. (2018). Methods of residual stress reduction for metal parts manufactured using selective laser melting (a review). Proceedings of the 3rd International Conference on Progress in Additive Manufacturing (Pro-AM 2018), 315-320. doi:10.25341/D4901Z