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LUT School of Energy Systems
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HEAT TREATMENT OF INCONEL 718 MANUFACTURED BY LASER POWDER BED FUSION

LASERPOHJAISELLA JAUHEPETISULATUKSELLA VALMISTETUN INCONEL 718 LÄMPÖKÄSITTELY

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TIIVISTELMÄ

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Laserpohjaisella jauhepetisulatuksella valmistetun Inconel 718 lämpökäsittely

Kandidaatintyö

2021

35 sivua, 14 kuvaa ja 10 taulukkoa

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Hakusanat: Lisäävä valmistus, Lämpökäsittely, IN718, Inconel 718 laserpohjainen jauhepetisulatus, 3D-tulostus.

Inconel 718:lla on erinomaiset mekaaniset ja kemialliset ominaisuudet. Korkean lujuuden ja lämmön- ja korroosionkeston vuoksi sitä käytetään paljon tekniikan sovelluksissa. Yksi Inconel 718:n käytön huonoista puolista on koneistuksen tarve, joka on hidasta ja työlästä. Tämän vuoksi lisäävä valmistus saavuttaa yhä enemmän huomiota vaihtoehtoisena tuotantotapana. Nopeasti vaihteleva lämpötila laserpohjaisen jauhepetisulatuksen aikana aiheuttaa IN718:sta tehtyihin kappaleisiin jäännösjännityksiä, huokoisuutta ja epähomogeenisen mikrorakenteen, jossa on haitallisia faaseja. Lämpökäsittelyllä voi vaikuttaa suuresti lisäävällä valmistuksella tuotetun Inconel 718:n ominaisuuksiin.

Tämän kandidaatintyön tarkoituksena oli selvittää eri lämpökäsittelymenetelmien vaikutusta laserpohjaisella jauhepetisulatuksella valmistetun Inconel 718:n mikrorakenteeseen ja mekaanisiin ominaisuuksiin. Työ suoritettiin kirjallisuuskatsauksena, jossa etsittiin ja koottiin uusinta tietoa aiheesta eri tietokannoista.

Kirjallisuuskatsauksen perusteella huomattiin, että lämpökäsittelyllä voidaan parantaa mekaanisia ominaisuuksia ja mikrorakenteen koostumusta. Lisäävällä valmistuksella tuotetulle Inconel 718:lle voidaan käyttää hyvin samanlaista lämpökäsittelyä, kuin perinteisillä menetelmillä tuotetulle Inconel 718:lle. Homogenisoinnilla ja liuotushehkutuksella yhdistettynä kaksinkertaiseen vanhennukseen päästään parhaisiin tuloksiin. Homogenisoinnin ja Liuotushehkutuksen lämpötilan täytyy olla korkeampi lisäävällä valmistuksella tuotetulla Inconel 718:lla, koska sen mikrorakenne eroaa perinteisesti valmistetusta Inconel 718:sta.

ABSTRACT

LUT University
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Heat treatment of Inconel 718 manufactured by laser powder bed fusion

Bachelor's thesis

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35 pages, 14 figures, and 10 tables

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Keywords: Additive manufacturing, heat treatment, IN718, Inconel 718 laser powder bed fusion, 3D-printing.

IN718 has excellent mechanical and chemical properties. Due to its high strength, and resistance to corrosion and heat, it is widely used in variety engineering applications. One of the drawbacks of using Inconel 718 (IN718) is machining which is difficult and slow to perform. For this reason, additive manufacturing (AM) gains more and more attention as an alternative production method for IN718. Rapid heating and cooling during the laser powder bed fusion process (L-PBF) causes residual stresses, porosity, and inhomogeneous microstructure containing detrimental phases. Heat treatment processes can have considerable effect on the mechanical properties of additively manufactured IN718.

The aim of this bachelor's thesis is to review the effect of different types of heat treatment methods on the microstructural and mechanical behavior of IN718 that is manufactured by L-PBF. To achieve this aim, a state-of-the-art literature review is performed.

It was found out that heat treatment can be used to improve the mechanical properties and the composition of microstructure. The heat treatment methods that are used for additively manufactured IN718 were similar to the ones used for conventionally produced IN718. Homogenization and solutioning combined with double aging provided the best result. The temperature used in homogenization and solutioning needed to be higher than the one used for conventionally produced IN718 due to the microstructural differences.

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Jesse Laakso
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LIST OF SYMBOLS AND ABBREVIATIONS

3D	Three-dimensional
AM	Additive manufacturing
CAD	Computer-aided Design
HT	Heat treatment
IN718	Inconel 718
L-PBF	Laser Powder Bed Fusion
STL	Standard Tessellation Language

1 INTRODUCTION

Laser powder bed fusion (L-PBF) is one of the additive manufacturing (AM) technologies. The basis of AM is adding the material to where it is needed, unlike conventional methods which rely on the removal of unwanted material. In L-PBF desired material is added layer by layer in powdered form and then melted by a heat source (Gibson et al. 2021, pp. 125-127). L-PBF has many advantages which are shown in figure 1. This makes it a tempting method to produce parts from high performance materials that are difficult to machine such as Inconel 718 (IN718) (Rahman et al. 1997, p. 199).

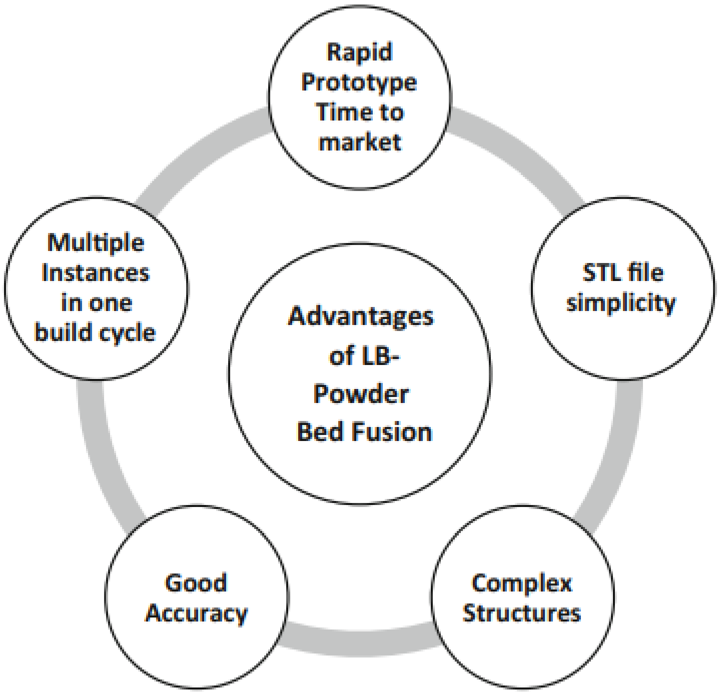

Figure 1. Advantages of L-PBF. (Milewski 2017, p. 139)

The industry however faces many challenges. As can be seen from figure 2. One issue is the ability to produce parts that have equal properties when compared to conventionally produced counterparts. Rapid heating and cooling during the AM process causes high surface roughness, anisotropic microstructure, porosity, and residual stresses. (Solberg 2020, p. 2326)

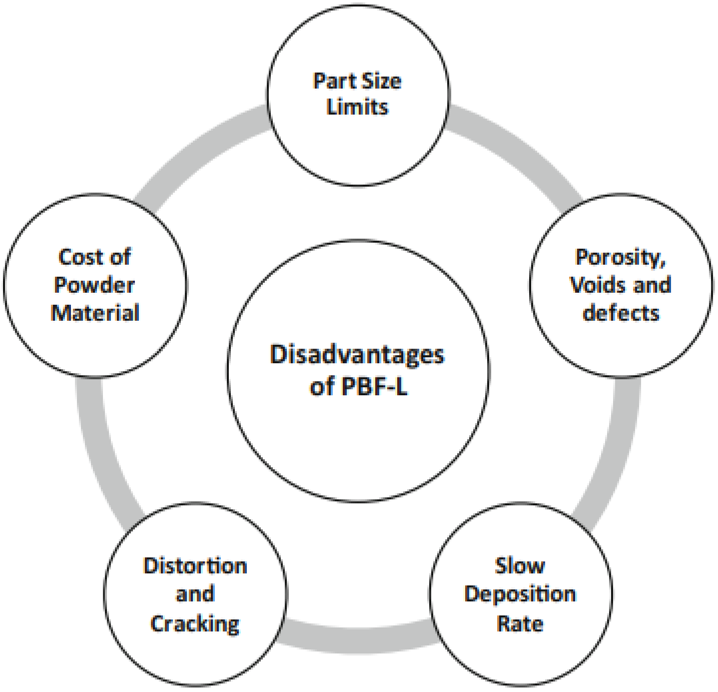

Figure 2. Potential disadvantages of L-PBF. (Milewski 2017, p. 144)

Heat treatment (HT) is used to address these issues. It is a process which aims to alter the mechanical and chemical properties of the material through cycles of heating and cooling which cause a change in microstructure (Banerjee 2017, p. 2). Since the structure of additively manufactured IN718 differs from conventionally produced one, parameters used in standard heat treatments need some adjustments (Fayed et al. 2020, p. 3). However, the topic is still new and rapidly evolving. As there is no comprehensive information available, this thesis aims to compile the available data from the latest research and to draw firm conclusions.

1.1 Aim of the thesis, research problem, and questions

The aim of this thesis is to find out what are the possible heat treatment methods for IN718 manufactured by L-PBF. Also, this thesis tries to find out how different parameters such as time, temperature, and cycles of heating and cooling affect the microstructural and mechanical properties of additively manufactured IN718.

The research questions of this thesis are then:

- Why L-PBF IN718 needs heat-treatment?
- How is the heat treatment of L-PBF IN718 performed?
- How do the heat treatment methods differ between additively and conventionally manufactured IN718?
- How do different parameters such as time, temperature, and cycles of heating and cooling affect the mechanical properties?
- How does heat treatment affect the microstructural features of L-PBF IN718?

1.2 Research methods

This thesis was carried out as a literature review by searching for latest information from different sources such as Google Scholar, LUT Finna and Scopus. Research was limited to IN718 manufactured by L-PBF. Properties of IN718 will be shortly introduced alongside theory of L-PBF in the chapters 2 and 3, as the focus of the thesis is on the heat treatment. This research provides novelty value as there are not any releases which combine the information about the heat treatment methods of IN718.

2 INCONEL 718

Inconel 718 is one of the nickel-chromium based superalloys. It has excellent mechanical and chemical properties in a wide range of temperature. Due to its high tensile, fatigue, creep and rupture strength and high resistance to oxidation and corrosion it is widely used in different industries. Aero engines, combustion chambers, cladding material and sheet metal parts for aircrafts are one of the many uses of IN718. (Milewski 2017, p. 70; Special Metals 2007, p.1)

Its mechanical and chemical properties however cause difficulties during machining. According to Gupta et al. (2017, p. 238) “Inconel 718 is one of the most difficult-to-machine materials.” Microstructure of IN718 contains hard carbides such as titanium carbide and niobium carbide. It work hardens rapidly and tends to weld with tool material. This results short tool lives. Machining requires high cutting forces which cause metallurgical damage to the workpiece while also increasing the work hardening, surface tearing and distortion. These properties in turn cause increased costs in machining and lower production efficiency. (Gupta et al. 2017, p. 238; Rahman et al. 1997, p. 199)
The chemical composition of IN718 makes it an age hardenable alloy. This process will be explained in the chapter 4. As can be seen from table 1, main elements in IN718 are nickel, chromium, and iron. Aluminum, titanium, and niobium is utilized during the aging process to achieve the full strength of the alloy. (Special Metals 2007, p. 2)

Table 1. Limiting chemical composition of IN718 (mod Special metals 2007, p. 1)

Composition	Weight (%)
Nickel (plus Cobalt)	50.00-55.00
Chromium	17.00-21.00
Iron	Balance
Niobium (plus Tantalum)	4.75-5.50
Molybdenum	2.80–3.30
Titanium	0.65–1.15
Aluminium	0.20–0.80
Cobalt	1.00 max
Carbon	0.08 max
Manganese	0.35 max
Silicon	0.035 max
Phosphorus	0.015 max
Sulphur	0.015 max
Boron	0.006 max
Copper	0.30 max

3 LASER POWDER BED FUSION

L-PBF is one of the AM technologies in which an object is built by alternating between spreading and melting powdered material (Gibson et al. 2021, p. 125-127)

First step in L-PBF process is modelling the desired part by using a CAD-software. As long as the output is three dimensional (3D) solid, or surface representation any CAD-software can be used. Using a reverse engineering is also a possibility. Information about the part is then converted into readable format for the AM-machine. Standard tessellation language (STL) file format has become the standard for AM industry. Preprocessing software is then used to slice the STL model into layers for the manufacturing operation. Final adjustments for size, location and building orientation can be done at this point. (Gibson et al. 2021, p. 3-5; Gu 2015, p. 4-6) The working principle of AM-machine is shown in figure 4.

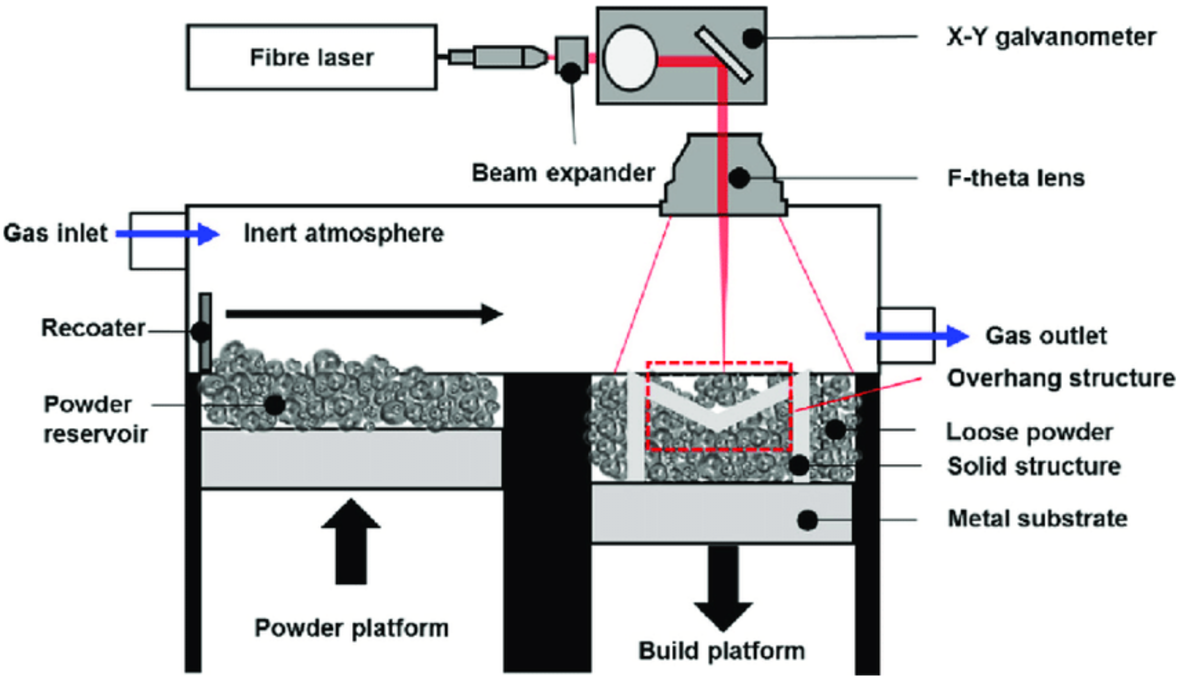

Figure 4. Schematic of typical L-PBF machine (Atwood et al. 2018, p. 648)

Thin layer of powder is spread from the powder reservoir to the build platform by the recoater or counter rotating roller. This layer is corresponding to the one slice in STL model. Laser is then used to melt the powder in a desired area. Build platform is lowered and powder platform rises so new layer can be spread. These steps are repeated until the part is complete (Kumar 2020, p. 41; Milewski 2017, p. 134).

Depending on the geometry of the part, a support structures may be required to prevent the part from falling over due to gravity as shown in figure 5. Supports also work as heat sinks and prevent the material from warping and distorting (Kumar 2020, p. 162; Milewski 2017, p. 135). Post processing such as cleaning, removal of support structures, cleaning, or heat treatment may be required before part can be used (Gibson 2021, p. 6).

Figure 5. Additively manufactured part with support structures pointed by red arrows (Materialise, 2021)

Any material that can be melted and resolidified can be used in L-PBF process. Most common ones are polymers, ceramics, metals, and their composites (Gibson et al. 2021, p. 127-130). Powders that are optimized for AM can be expensive. Unused powder can be utilized in the forming of next part but the possible changes in physical and chemical properties caused by the building process must be considered (Gibson et al. 2021, p. 398; Milewski 2017, p. 230). Possibility to reuse powder makes L-PBF a cost and material efficient method.

AM processes such as L-PBF make it possible to produce parts with complex internal structures. Internal cooling channels of the part shown in figure 6 would have been extremely difficult if not even impossible to produce using conventional casting and machining methods.

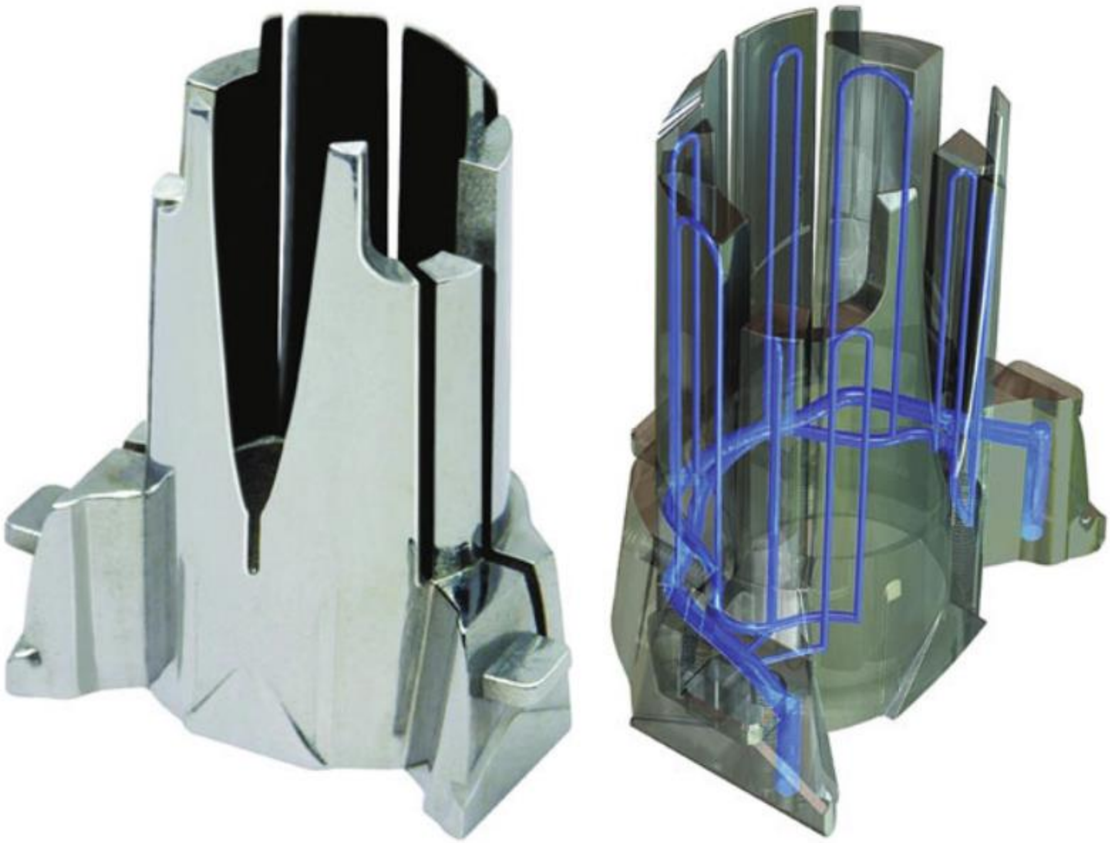

Figure 6. On the left post processed part fabricated by L-PBF (DMLS) and on the right model showing the internal conformal cooling channels. (Milewski 2017, p. 22)

4 HEAT TREATMENT

4.1 Basics of heat treatment

The defining factors in the mechanical and chemical properties of metals and alloys are their microstructure, atomic structure, and crystal structure. Magnitude of these properties are dependent on the shape, size, and distribution of different microconstituents. These can be altered by utilizing process called heat treatment. In this process, material is first heated to a specific temperature where it is held for a desired time followed by a cooling process. All these steps are done while the material is in solid state. (Rajan et al. 2013, p. 1; Tanzi 2019, p. 50)

The defining factors in the mechanical and chemical properties of metals and alloys are their microstructure, atomic structure, and crystal structure. Magnitude of these properties are dependent on the shape, size, and distribution of different microconstituents. These can be altered by utilizing process called heat treatment.

There exists multitude of different heat-treatment processes, but they can be sorted into five basic operations: hardening, tempering, annealing, normalizing and case hardening. While the result between these processes varies greatly each of them has the same three steps: heating, soaking, and cooling. (Tanzi 2019, p. 50)

In the first step, material is heated at constant rate to spread heat evenly. Highly heat conductive materials can be heated much faster than ones with low conductivity. Also, the shape of the object needs to be considered to avoid cracking and warpage since the outer parts heat up faster than the center. After reaching the desired temperature, material is held for a specified time so the structural changes can take place. This is called Soaking. Cooling is then applied, and cycle can be performed again. (Banerjee 2017, p. 3)

4.2 Heat treatment of Inconel 718

IN718 is a multiphase superalloy which means that on a microstructural level it contains multiple phases with different chemical and structural compositions. Modifying this composition is the basis of heat treatment. (Fayed et al. 2020, p. 2)

The major phases in IN718 are (Belan 2016, p. 941; Fayed et al. 2020, p. 2):

- Gamma (*γ*) austenitic matrix rich in solid-solution elements such as cobalt, iron, chromium, molybdenum, and tungsten.
- Gamma prime (*γ´*) where nickel forms a phase with aluminium and titanium that is coherent with the austenitic gamma matrix. Other elements such as niobium, tantalum and chromium also enter *γ´*.
- Gamma double prime (*γ´´*) Nickel and niobium form Ni_3Nb which is coherent with gamma matrix.
- Carbides such as MC, $M_{23}C_6$, M_6C where carbon combines with reactive elements such as titanium, tantalum, hafnium, and niobium.
- Delta (*δ*) phase has the same chemical composition as *γ´´* (Ni_3Nb) but different structure.
- Laves-phase $(Ni, Fe, Cr)_2$ (Nb, Mo, Ti).

γ´ and *γ´´* are the primary strengthening phases for IN718. *γ´* is the high-temperature one while *γ´´* provides very high strength at low to intermediate temperatures, it's unstable at temperatures above about 649 °C. Carbides may provide limited strengthening directly through dispersion hardening or indirectly by stabilizing grain boundaries against excessive shear. The *δ* and Laves phases are known to have degrading effects on mechanical properties of IN718. However appropriate amount of δ-phase along the grain boundaries may prevent undesired grain growth and improve the strength of the grain boundaries. (Belan 2016, p. 941; Fayed et al. 2020, p. 2)

Heat treatment processes used for IN718 are annealing and precipitation aging. These processes are also often referred as solution heat treatment and aging. When solutioning is done at high enough temperature it is referred to as homogenization (Cambpell 2006, p. 243-248). In this thesis and according to the literature, homogenization refers to the temperatures above 1030 °C.
Homogenization and solutioning is performed to dissolve detrimental phases such as Laves phases and to obtain uniformly distributed microstructure while releasing age-hardening constituents into the matrix for the aging treatment. After that aging is performed to form *γ´* and *γ´´* strengthening phases. These treatments increases the mechanical properties. (Campbell 2006, p. 243-248; Tucho et al. 2017, p. 220)

In general, homogenization and solutioning is done at temperatures between 980 °C and 1200 °C. For aging, the first step uses temperatures between 704 and 899 °C, and the second step temperatures between 593 and 704 °C. (Fayed et al. 2021, p. 2)

There are two industrial standard heat treatments for IN718. AMS 5383 for cast IN718 and AMS 5662 for wrought IN718 As shown in table 2 (AMS 5383, 2012; AMS 5662, 2009). IN718 manufactured by L-PBF differs considerably in microstructure, precipitates, and texture when compared to conventionally produced cast and wrought versions. Therefore, some adjustments need to be made to the standard heat treatments. (Fayed et al. 2020, p. 3)

Table 2. AMS standard heat treatments for cast and wrought IN718. (AMS 5383, 2012; AMS 5662, 2009)

	Designation	Homogenization	Solutioning	Aging
AMS 5383 (cast)	HSA	1080 °C/1,5 h/AC	980 °C/1 h/AC	720 °C/8 h/FC at 55 °C/h to 620 °C/8 h/AC
AMS 5662 (wrought)	SA	-	980 °C/1 h/AC	720 °C/8 h/FC at 55 °C/h to 620 °C/8 h/AC
AC = Air cooling, FC = Furnace cooling				

5 EFFECT OF HEAT TREATMENT

In this chapter the available literature about the effect of heat treatment on the mechanical and microstructural properties of IN718 is introduced. From each article, the applied heat treatment methods and most important findings are presented. Conclusions will then be made in chapter 6.

This first study which was performed by Fayed et al. (2021) investigated the effect of homogenization and solutioning treatment times on the elevated-temperature mechanical properties of L-PBF IN718. The heat treatment process of this study is shown in table 3. Mechanical properties and microstructure were investigated at 650 °C after combined heat treatment of homogenization, solution, and double aging. Specimens were labelled as HSA with letters referring to the heat treatment method used. Homogenization (H), solutioning (S), and aging (A) with number depicting the holding time used in homogenization treatment.

Table 3. Heat treatments performed on L-PBF IN718. (mod. Fayed et al. 2021, p. 4)

Specimen	Homogenization	Solutioning	Aging
As-printed	-	-	-
HSA1	1080 °C for 1 h/AC	980 °C for 15 min/AC	720 °C/8 h/FC at 55 °C/h to 620 °C + 620 °C/8 h/AC
HSA2	1080 °C for 1 h/AC	980 °C for 1 h/AC	
HSA3	1080 °C for 4 h/AC	980 °C for 37.5 min/AC	
HSA4	1080 °C for 7 h/AC	980 °C for 15 min/AC	
HSA5	1080 °C for 7 h/AC	980 °C for 1 h/AC	
AC = Air cooling, FC = Furnace cooling			

As can be seen in the table 4, the as-printed IN718 has the highest ductility and lowest strength. Reason to this is the absence of γ' and γ'' phases. All heat treatments increased the tensile and yield strength while ductility was lowered. When compared to the AMS standards 5662 and 5383 (cast and wrought IN718) strength of heat-treated AM-IN718 is higher and ductility is almost the same. (Fayed et al. 2021, p. 7) The effect of heat treatments on different properties are illustrated in figures 7 and 8.

Best results considering the strength properties were achieved with HSA1 and HSA2 after 1 h of homogenization. Increasing this time didn’t show any favorable changes in mechanical properties as both strength and ductility were lower when compared to HSA1 and HSA2. This was attributed to the increase of coarse MC carbide particles. (Fayed et al. 2021, p. 16)

Increasing the time when solutioning at 980 °C didn’t have significant effect on the grain structure. However, longer hold times resulted in increased precipitation of δ-phase. It was concluded that elevated-temperature mechanical properties were largely tied to the presence of *δ*-phase at grain boundaries. Amount of δ-phases was higher in HSA2 than HSA1 which resulted in higher strength. Same effect was observed in HSA4 and HSA5 (Fayed et al. 2021, p. 15-16)

Homogenization time of 1 h at 1080 °C resulted in mixture of columnar and equiaxed grains. Increasing this time to 4 h caused formation of more equiaxed recrystallized grains, and at 7 h coarse equiaxed grains form. Longer soaking time also increased the dissolving of Laves phase, as in HSA3 sample this phase was almost completely dissolved while in HSA1 and HSA2 amount was higher. Precipitation of coarse MC carbides was also increased with longer soaking times. It was concluded that hold time must be balanced with microstructure and mechanical properties in mind. (Fayed et al. 2021, p. 13-15)

Table 4. Mechanical properties of L-PBF, cast and wrought IN718 at 650 °C *after heat treatments. (mod. Fayed et al. 2021, p. 7)*

Mechanical properties (at 650 °C)			
Specimen	TS (MPa)	YS (MPa)	El (%)
As-printed	820 ± 2	617 ± 3	36 ± 3
HSA1	1038 ± 14	976 ± 9	11.2 ± 2
HSA2	1074 ± 28	1001 ± 12	11.8 ± 0.3
HSA3	1017 ± 11	927 ± 2	8.2 ± 0.2
HSA4	986 ± 14	901 ± 7	7.4 ± 0.9
HSA5	994 ± 11	909 ± 8	7.3 ± 1
AMS 5662	1000	862	12
AMS 5383	576	517	13.7
LPBF AMS 5662	1025	915	5.5
LPBF AMS 5383	1020	950	3.5
TS = Tensile strength, YS = Yield strength, El = Elongation			

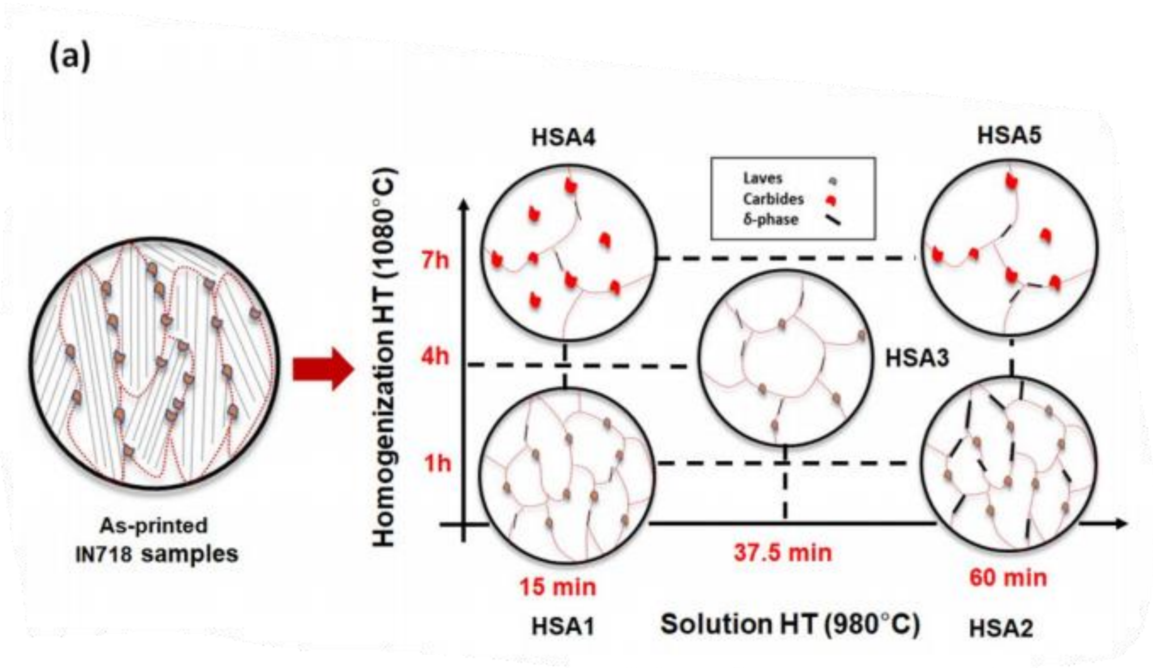

Figure 7. Illustration about the effect of heat treatment on L-PBF IN718. (Fayed et al. 2021, p. 15)

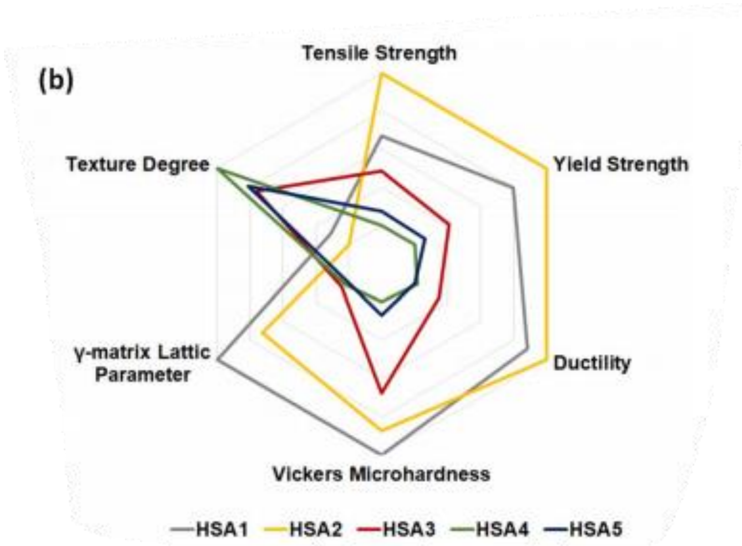

Figure 8. Radar chart summarizing the effect of heat treatment on L-PBF IN718. (Fayed et al. 2021, p. 15)

A study conducted by Zhao et al. (2020) investigated the effect of three-stage heat treatment on the microstructure and mechanical properties of AM-IN718. Table 5 shows the heat treatments done in the study. Specimens were labelled with letters depicting the heat treatments used. Homogenization (H), solutioning (S), and aging (A).

Table 5. Heat treatments used in the article. (mod. Zhao et al. 2020, p. 3358)

<table>
<tr><th>Specimen</th><th>Homogenization</th><th>Solutioning</th><th>Aging</th></tr>
<tr><td>As-printed</td><td>-</td><td>-</td><td>-</td></tr>
<tr><td>A</td><td>-</td><td>-</td><td rowspan="3">720 °C, 8 h/FC at 55 °C/h to 620 °C, 8 h/AC</td></tr>
<tr><td>SA</td><td>-</td><td rowspan="2">980 °C, 1 h/WC</td></tr>
<tr><td>HSA</td><td>1080 °C, 1.5 h/ WC</td></tr>
<tr><td colspan="4">WC = Water cooling, FC = Furnace cooling, AC = Air cooling</td></tr>
</table>

As can be seen from figures 9 and 10, heat treatments increased the hardness and strength, while the ductility was lowered. Hardness and strength changes were similar between A, SA, and HSA samples. Lower hardness of as-printed IN718 is caused by the rapid cooling rate during the L-PBF process as the time is insufficient for the strengthening phases to form. (Zhao et al. 2020, p. 3361)

Direct aged specimen had much lower ductility when compared to SA and HSA samples. Direct aging resulted in part of the strengthening phase precipitating between dendritic arms which lowered the ductility. Matrix in HSA and SA samples had long equiaxed grains and evenly distributed strengthening phases which resulted in higher ductility when compared to A Sample. HSA and SA samples meet the requirements for room temperature applications. (Zhao et al. 2020, pp. 3361-3366)

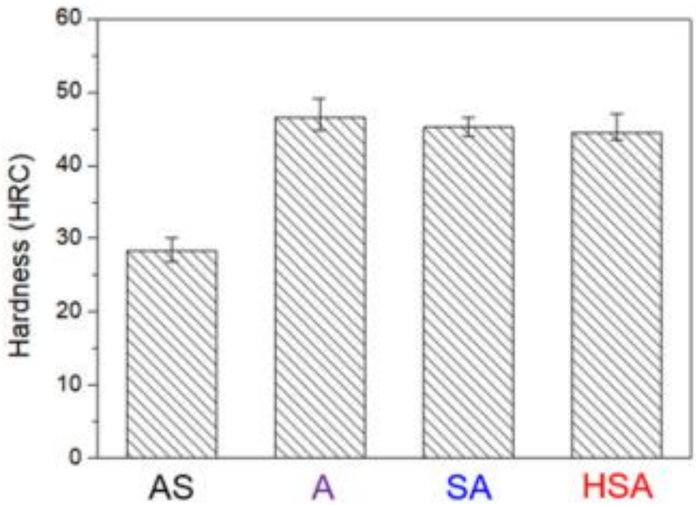

Figure 9. Hardness properties of specimens. (Zhao et al. 2020, p. 3361)

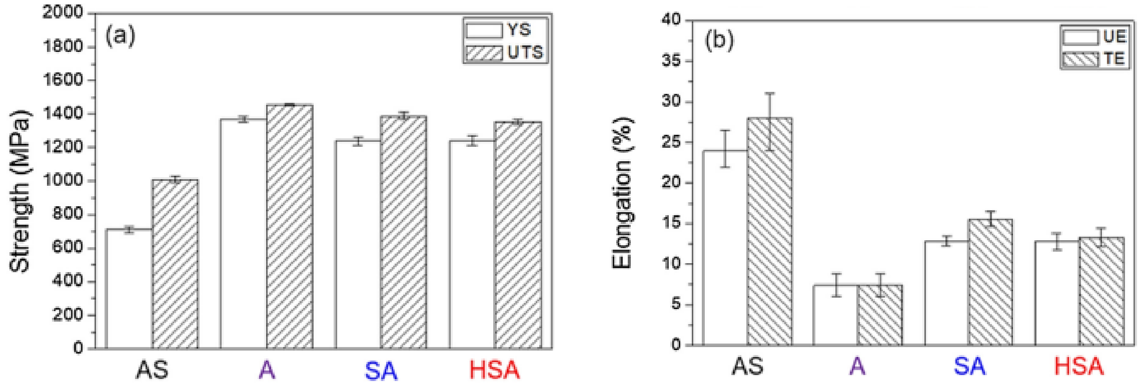

Figure 10. Strength and elongation properties of specimens in room temperature. YS: yield strength; UTS: ultimate tensile strength; UE: uniform elongation; TE: total elongation. (Zhao et al. 2020, p. 3361)

Effect of heat treatment on fatigue properties of L-PBF IN718 was investigated by Wan et al. (2018). Specimens were subjected to different combinations of heat treatments whereas time and temperature was kept constant. Heat treatment methods used are shown in table 6. Specimens were labelled with letters depicting the heat treatments used. Homogenization (H), solutioning (S), and aging (A).

All heat treatments increased the strength and fatigue properties of AM IN718. This was attributed to the dissolving of Laves phases and precipitation of strengthening phases γ' and γ''. In SA-specimen large number of Laves phases had dissolved back into the matrix with precipitation of fine acicular and granular δ phases in the interdendritic region. In HA-specimen Laves phase had completely dissolved back into the matrix. (Wan et al. 2018, p. 3-6)

As can be seen in figure 12, HA treated specimens had about 10 % increase in fatigue strength when compared to SA and HSA treated parts. It was hypothesised that width of δ-phases may be the cause of this, as phases with smaller width may have higher void-induced damage tolerance. Width of δ-phases were 30 nm and 70 nm in HA and HSA specimens, respectively. It was concluded that tailoring a proper heat treatment route can produce better results in strength properties when compared to conventionally produced IN718 parts. Controlling the size of δ-phase may provide a way to enhance the fatigue properties of AM-IN718 in the future. (Wan et al. 2018, p. 4-6)

Table 6. Heat treatments performed on L-PBF IN718. (mod. Wan et al. 2018, p. 2)

<table>
<tr><th>Specimen</th><th>Homogenization</th><th>Solutioning</th><th>Aging</th></tr>
<tr><td>As-built</td><td>-</td><td>-</td><td>-</td></tr>
<tr><td>SA</td><td>-</td><td>980 °C/1 h/AC</td><td rowspan="3">760 °C/10 h
FC at 55 °C/h to
650 °C/8 h / AC</td></tr>
<tr><td>HA</td><td rowspan="2">1065 °C/1 h/AC</td><td>-</td></tr>
<tr><td>HSA</td><td>980 °C/1 h/AC</td></tr>
</table>

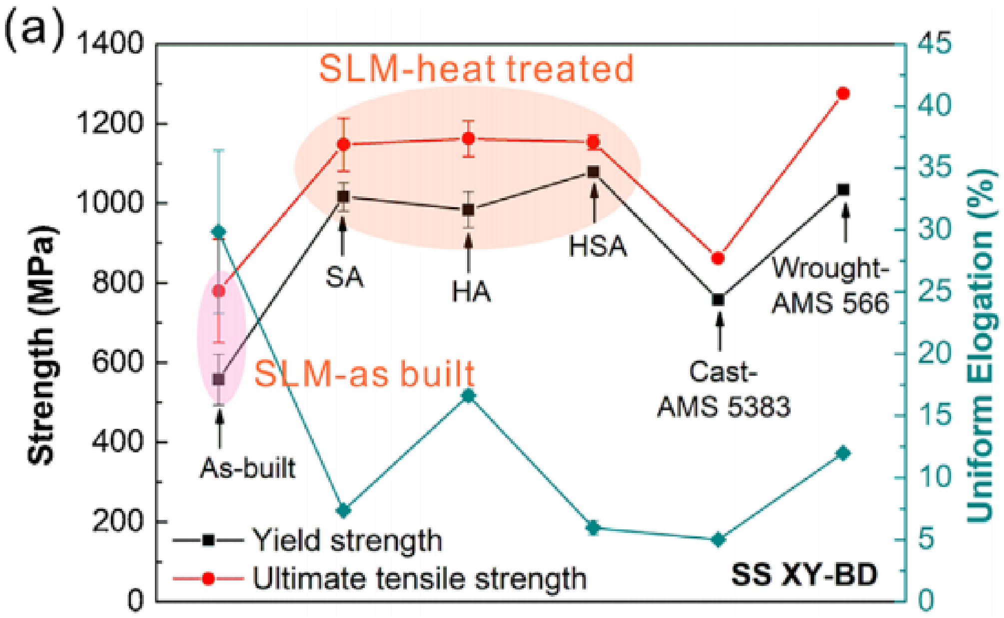

Figure 11. Strength and elongation properties of heat-treated AM, cast and wrought IN718 (Wan et al. 2018, p. 4)

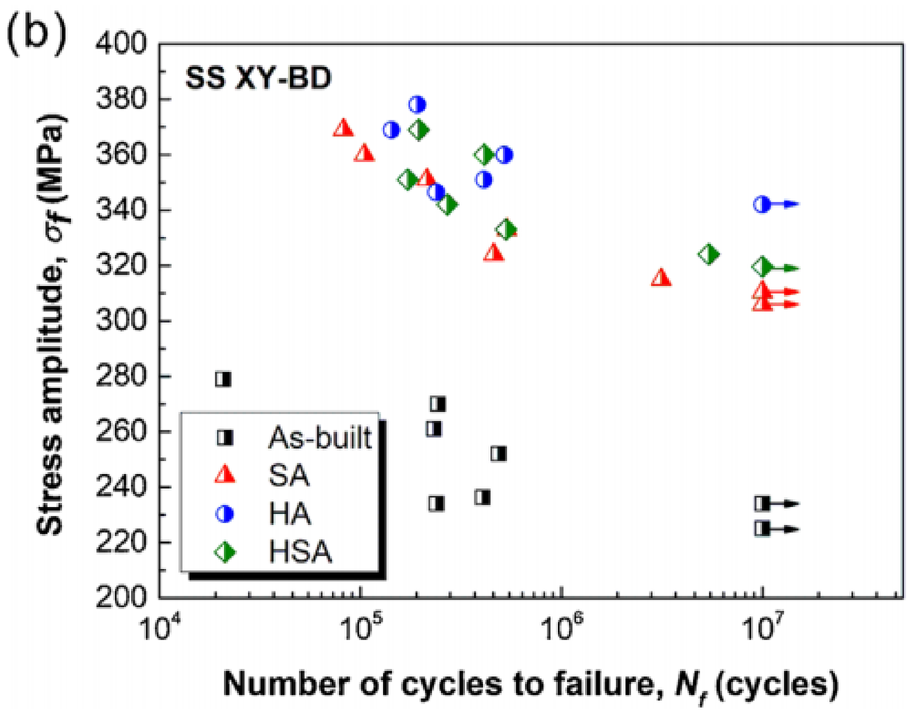

Figure 12. Fatigue performance of heat-treated AM IN718 specimens (Wan et al. 2018, p. 4)

Tucho & Hansen (2021) investigated the effects of solutioning hold time on microstructure, annealing twins, and hardness. ST-Specimens were solutioned using different hold times and then water quenched. STA-samples went through the same treatment but were also double aged. Heat treatment methods used are shown in table 7.

As can be seen in table 8. Hardness of ST-treated samples was lowered, whereas hardness of STA-samples increased by 36-49 % when compared to as-printed. Therefore, hardness is largely tied to the presence of γ' and γ'' phases formed during aging. Direct aged sample is as hard as STA-samples, but its microstructure is similar to the as-printed. Aging temperature is not high enough for microstructural changes to take place. Direct aging is not thus suitable as presence of δ and Laves phases is usually detrimental to other properties of material. On the microstructural level, significant changes were obtained after holding times of 3 h and longer. Complete annihilation of subgrains was observed after 9 h. Elimination of subgrain boundaries was preceded by the dissolution of Laves phase and annihilation of dislocations. (Tucho & Hansen 2021, p. 17-18)

Table 7. Heat treatments performed on AM IN718. (mod. Tucho & Hansen, 2021, p. 4)

Specimen	Solutioning			Aging
	T (°C)	Hold time (h)	Cooling	
ST1	1100	1	WC	None
ST3		3		
ST6		6		
ST9		9		
ST16		16		
ST24		24		
STA1		1		760 °C/10 h + 650 °C/10 h/AC
STA3		3		
STA6		6		
STA9		9		
STA16		16		
STA24		24		
DA		Direct aged		

Table 8. Hardness properties after heat treatment (mod. Tucho & Hansen, 2021, p. 16)

Scheme	Hardness [HV]			
	AP	ST	STA	%ΔHV
AP	308 ± 5	-	-	-
DA	-	-	446 ± 4	45
1 h	-	249 ± 4	456 ± 4	49
3 h	-	200 ± 3	444 ± 5	45
6 h	-	195 ± 3	439 ± 3	43
9 h	-	191 ± 3	442 ± 6	44
16 h	-	192 ± 3	439 ± 3	43
24 h	-	192 ± 5	420 ± 5	36
Note: %ΔHV is the percentage increment in hardness after aging compared to the hardness of the as-printed.				

Kuo et al. (2018) studied the effect of different post-processes such as heat treatment and hot isostatic pressing on the microstructure and creep properties. Heat treatments are shown in table 9. Samples were solutioned and then double aged.

Rapid heating and cooling during the manufacturing process induces thermal variations which cause high-density dislocations. The dendrite structure and interdendritic regions of as-built specimen were decorated with a continuous network of Laves phases and Carbides. Heat treatment at 980 °C was not suitable as Laves phases transform into δ phases which pinning effect result in similar grain morphology and grain size as in as-built specimen. Increasing temperature to 1120 °C and above dissolves Laves and δ phase back into the matrix but caused inhomogeneous grain growth and carbides to become coarse. Figure 13 depicts the changes in microstructure. (Kuo et al. 2018, p. 4-6)

Table 9. Heat treatments performed on the specimens. (mod. Kuo et al. 2018, p. 3)

Specimen	Solutioning	Aging
STA-980	980 °C/1 h/AC	720 °C/8 h/FC to 620 °C + 620 °C/10 h/AC
STA-1045	1045 °C/1 h/AC	
STA-1065	1065 °C/1 h/AC	
STA-1120	1120 °C/1 h/AC	
STA-1180 (1 h)	1180 °C/1 h/AC	
STA-1180 (4 h)	1180 °C/4 h/FC	
AC = Air cooling, FC = Furnace cooling		

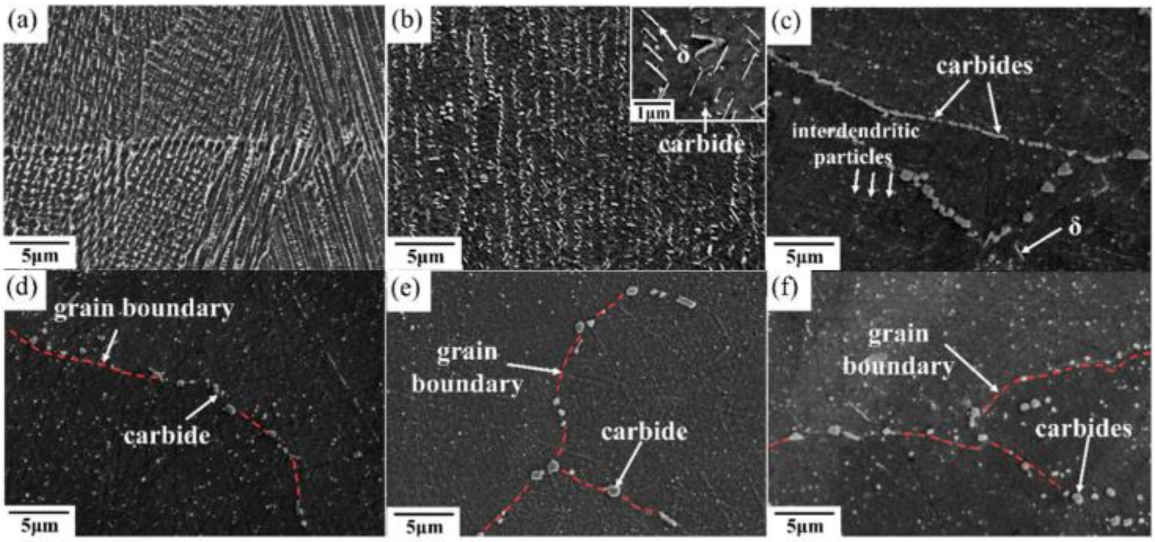

Figure 13. Scanning electron microscope images of samples with solutioning temperature from lowest (a) to highest (f). (Kuo et al. 2018, p. 6)

Ramakrishna et al. (2021) studied the effect of solutioning temperature on the microstructural evolution during double aging. Figure 14 shows an illustration about the changes in microstructure. Heat treatments used are shown in table 10. SL refers to solutioning and SA to solutioning combined with aging. HO is homogenization and HA homogenization combined with aging.

Main points of the study can be summarized as follows (Ramakrishna et al. 2021, p. 8-11):

- During manufacturing process thin discontinuous Nb layer forms along the interdendritic region of solidified IN718. During solutioning at 980 °C and aging this layer causes formation of coarse δ and γ'' phases, respectively.
- Formation of γ'' precipitates during double aging is affected by the solution treatment temperature. Solutioning at 1080 °C resulted in the formation of most homogenous γ'' precipitates.
- Response to heat treatment in terms of γ'' precipitation is similar between AM-IN718 and wrought IN718 when solutioning temperature is 1080 °C.
- As-built microstructure remains intact up to an hour during solutioning at 1030 °C. After solutioning for an hour at 1080 °C formation of new near equiaxed grains was observed.

- Microstructural analysis of 1030SL confirmed the dissolving of Laves phase although solvus temperature is higher. Amount of niobium present in the structure is known to determine the solvus temperature. Thus, the niobium concentration may be enough to lower the critical point so complete dissolution may take place at 1030 °C.
- Laves phase disappeared when solution treated at 980 °C. Though solvus temperature for such phase is higher. It was concluded that at temperature of 980 °C this increases the local niobium concentration which in turn increases the precipitation of δ phase. This was theorized as a PBF-AM specific effect and in need of further study. Finding was confirmed with transmission electron microscopy and scanning electron microscopy.

Table 10. Heat treatments performed on AM and wrought IN718. (mod. Ramakrishna et al. 2021, p. 3)

Specimen	Solutionization/ Homogenization	Double aging
As-built		
Direct aged		720 °C/8 h + 620 °C/8 h +WQ
980SL	980 °C/1 h + WQ	
980SA		720 °C/8 h + 620 °C/8 h +WQ
1030SL	1030 °C/1 h + WQ	
1030SA		720 °C/8 h + 620 °C/8 h +WQ
1080HO	1080 °C/1 h + WQ	
1080HA		720 °C/8 h + 620 °C/8 h +WQ
W-980SA	980 °C/1 h + WQ	720 °C/8 h + 620 °C/8 h +WQ
W-1030SA	1030 °C/1 h + WQ	
W-1080HA	1080 °C/1 h + WQ	
SL = Solutionization, HO = Homogenization, HA = Homogenization + aging, WQ = Water quench		

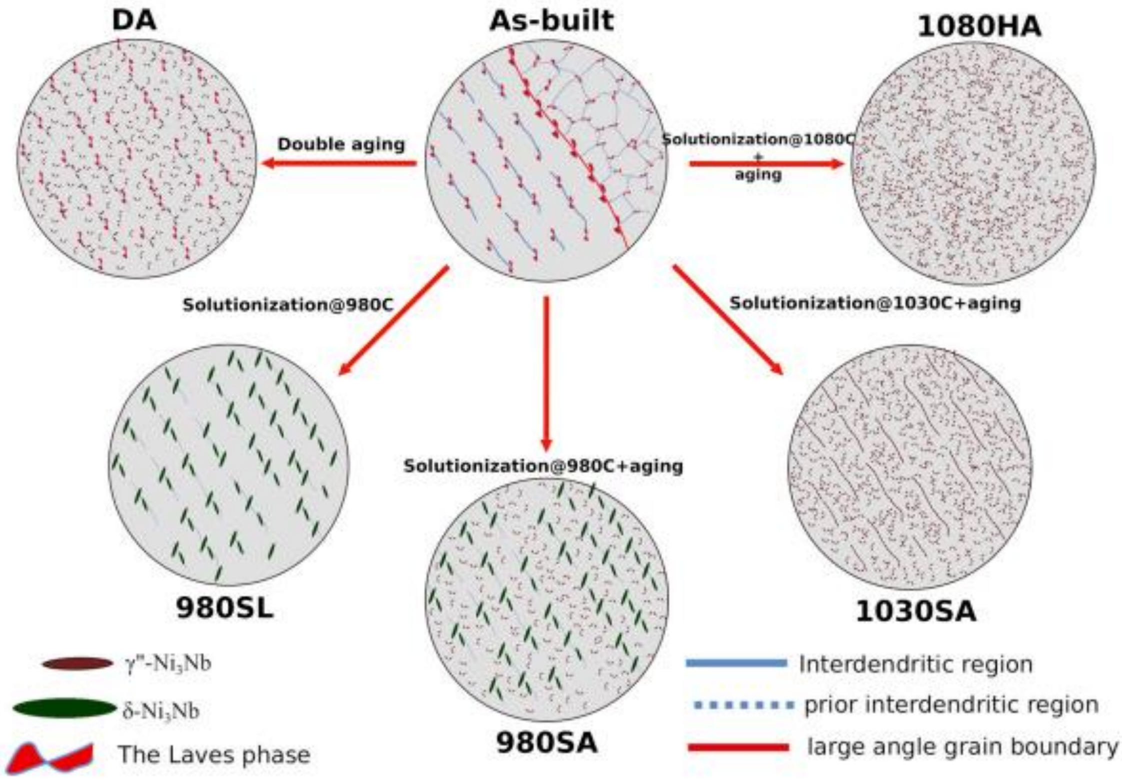

Figure 14. Illustration about the effect of different heat treatments on the phases present in additively manufactured IN718. (Ramakrishna et al. 2021, p. 11)

6 DISCUSSION AND CONCLUSIONS

The aim of this thesis was to review the current information available on the effect of heat treatment on the mechanical and microstructural properties of L-PBF manufactured IN718. In this chapter conclusions are made based on the reviewed literature.

Manufacturing parts made of IN718 using L-PBF suffer from defects such as inhomogeneous microstructure, residual stresses and phases such δ and Laves due to rapid heating and cooling present during the process. Therefore as-printed IN718 cannot be used straight after building process. Post-processing such as heat treatment is then required to improve the microstructure, consequently the mechanical properties. Heat treatment process begins with homogenization or solutioning. These processes are used to dissolve δ and Laves phases while transforming the microstructure into more homogeneous one. This step also releases the aging elements such as niobium, titanium, and aluminium into the matrix. Double aging is then performed to form the strengthening phases of γ' and γ''.

In the studied literature, the effects of different heat treatments and their combinations on AM-IN718 were studied. Homogenization and solutioning temperatures ranged from 980 °C to 1180 °C, with soaking times of 1 to 24 h. For double aging processes, temperature during the first aging was 720–760 °C, and 620–650 °C for the second, with soaking times of 8 – 10 h. For cooling, both water quenching and furnace cooling have been used. Effect of various cooling methods were not found in the literature.

The magnitude of the changes observed in microstructure increased with the temperature and soaking times used during homogenization and solutioning. Short soaking time resulted in only partially reformed microstructure. At the temperature of 980 °C longer soaking time resulted in increased precipitation of δ phase due to increased niobium concentrations. When temperature was 1080–1100 °C, the longer soaking time resulted in increased equiaxed recrystallized grains with precipitation of coarse carbides. Dissolving of Laves and δ preceded to the formation of recrystallized microstructure. At the temperature of 1120 °C and above, coarse carbides formed, and grain growth became activated.

Conflicting results can be observed in the studies about the effect of homogenization and solutioning on L-PBF IN718. Ramakrishna et al. (2021, p. 4) reported complete dissolving of Laves phases in all three temperatures: 980 °C, 1030 °C, and 1080 °C after soaking for 1 h. Though solvus temperature for such phase is above 1030 °C. In a study by Tucho & Hansen (2021, p. 5), Laves phases were largely dissolved after solutioning at 1100 °C for 1 h. Effect of Laves phases dissolving in a such low temperature was concluded to be a L-PBF IN718 specific effect and in need of further study.

All three heat treatments: homogenization, solutioning and aging increased the tensile, yield, and fatigue strength, while ductility was lowered. The hardness was lowered during homogenization and solutioning, while increased during aging. As can be seen from table 4, when proper heat treatment route was used, better mechanical properties were achieved in L-PBF IN718, even compared to cast and wrought IN718.

Heat treatment of L-PBF IN718 parts focused on obtaining the optimal combination between microstructural and mechanical properties based on the intended application. Too much concentration on the improvement of one property may negatively impact on the others. Homogenization at 1080 °C – 1100 °C for 2 – 3 h combined with solutioning at 980 °C for 1 h, and then applying same double aging treatment as is often used for cast and wrought IN718 seemed to result in the best balance between microstructural features and mechanical properties.

7 FURTHER STUDIES

The focus of studied literature in this thesis was on the temperatures and times used in homogenization and solutioning treatments. Studies about the effect of aging temperature and its time on the additively manufactured IN718 was not found. Although this could be attributed to the fact that the main issue of the heat treatment seems to be the preparation of microstructure for aging, yet further studies are needed to fill the knowledge gap.

Differences between the microstructural features and mechanical properties were observed when samples were heat treated, even when the same parameters were adopted. The reason for this could be attributed to the lack of unified parameters during the L-PBF process. Hence, further experiments on the L-PBF parts of IN718 are needed.

In the studied literature in this thesis, the effect of cooling method was not studied, and no articles were found on such topic for L-PBF IN718. Further studies are then needed to fill the knowledge gap.

LIST OF REFERENCES

AMS 5383. 2012. Aerospace Material Specification. Aerospace SAE International: Warrendale, PA, USA.

AMS 5662. 2009. Aerospace Material Specification. Aerospace SAE International: Warrendale, PA, USA.

Atwood, A., Bodey, A., Lee, P., Leung, C., Jones, J., Marussi, T., Towrie, M., Val Garcia, J. & Withers, P. 2018. Laser-matter interactions in additive manufacturing of stainless steel SS316L and 13-93 bioactive glass revealed by in situ X-ray imaging. In: Additive Manufacturing, Volume 24 p. 648.

Banerjee, M. 2017. Materials Science and Materials Engineering, Comprehensive Materials Finishing. Volume 2 p. 1-49.

Belan, J. 2016. GCP and TCP phases presented in nickel-base superalloys. In: Materials Today: proceedings. Volume 3. Pp. 936-941.

Campbell, F. C. 2006. Manufacturing technology for aerospace structural materials. Great Britain: Elsevier.

Fayed, E. M., Shahriari, D., Saadati, M., Brailovski, V., Jahazi, M., Medraj, M. 2021. Effect of homogenization and solution treatments time on the elevated-temperature mechanical behavior of IN718 fabricated by laser powder bed fusion. In: Scientific reports. Volume 11.

Fayed, E. M., Shahriari, D., Saadati, M., Brailovski, V., Jahazi, M., Medraj, M. 2020. Influence of Homogenization and Solution Treatments Time on the Microstructure and Hardness of IN718 Fabricated by Laser Powder Bed Fusion Process. In: Materials. Volume 13. p. 1-23.

Gibson, I., Rosen, D., Stucker, B. & Khorasani, M. 2021. Additive Manufacturing Technologies. Third edition. Switzerland: Springer.

Gu, D. 2015. Laser additive manufacturing of high-performance materials. Berlin: Springer.

Gupta, K. M., Sood, P. K., Singh, G., Sharma, V. S., 2017. 'Experimental Investigation and Optimization on MQL-Assisted Turning of Inconel-718 Super Alloy', in Gupta, K. (ed.) Advanced Manufacturing Technologies, Materials Forming, Machining and Tribology. Switzerland: Springer. p. 238-247

Kumar, S. 2020. Additive manufacturing processes. Switzerland: Springer.

Kuo, Y., Nagahari, T., Kakekhi, K. 2018. The effect of post-processes on the microstructure and creep properties of Alloy 718 built up by selective laser melting. In: Materials. Volume 9. Pp. 1-13

Materialise. 2021. Materialise Magics Support Generation Module for Metal 3D Printing (SG+) [Materialise webpage]. [Referred 17.5.2021] Available: https://www.materialise.com/en/software/magics/metal-support-generation-module

Milewski, J. 2017. Additive Manufacturing of Metals. Santa Fe: Springer. 258

Rahman, M., Seah, W. K. H., Teo, T. T., 1997. The Machinability of IN718. In: Journal of Materials Processing Technology. Volume 63. Pp. 199-204

Rajan, T. V., Sharma, C. P., Sharma, A. 2013. Heat Treatment, Principles and Techniques. 2nd ed. New Delhi: PHI Learning Private Limited.

Ramakrishna, M., Koppoju, S., Telasang, G., Korla, R., Padmanabham, G. 2021. Effect of solutionizing temperature on the microstructural evolution during double aging of powder bed fusion-additive manufactured IN718 alloy. In: Materials Characterization. Volume 172.

Solberg, K., Wan, D., Berto, F. 2020. Fatigue assessment of as-built and heat-treated IN718 specimens produced by additive manufacturing including notch effects. In: Fatigue & Fracture of Engineering Materials & Structures. Volume 43. Pp. 2326 – 2336

Special Metals. 2017. Inconel alloy 718 [web document]. Available: http://www.specialmetals.com/assets/smc/documents/inconel_alloy_718.pdf

Tanzi, M. C., Farè, S., Candiani, G. 2019. Foundations of Biomaterial Engineering. London: Elsevier Science.

Tucho, W. M., Cuvillier, P., Sjolyst-Kverneland, A., Hansen, V. 2017. Microstructure and hardness studies of IN718 manufactured by selective laser melting before and after solution heat treatment. In: Materials Science & Engineering A. Volume 689. Pp. 220-232.

Tucho, W. M., Hansen, W., 2021. Studies of Post-Fabrication Heat Treatment of L-PBF-IN718: Effects of Hold Time on Microstructure, Annealing Twins, and Hardness. In: Metals. Volume 11.

Wan, H., Zhou, Z., Li, C., Chen, G., Zhang, G. 2018. Enhancing Fatigue Strength of Selective Laser Melting-Fabricated IN718 by Tailoring Heat Treatment Route. In: Advanced Engineering Materials. Volume 20.

Zhao, J., Hung, F., Lui, T. 2020. Microstructure and tensile fracture behavior of three-stage heat treated IN718 alloy produced via laser powder bed fusion process. In: Journal of Materials Research and Technology. Volume 9. Pp. 3357-3367