LITERATURE REVIEW – ALLOY NI-718 ELABORATED BY POWDER BED FUSION TECHNOLOGIES: HEAT TREATMENTS

ANDDURO – WORKPACKAGE 2

Abstract

Powder Bed Fusion technologies induce specific microstructures presenting differences to those obtained through conventional elaboration processes. The cooling rates and the thermal cycles experienced by the produced materials generate microstructural and metallurgical features that are specific to each technology. This literature review aims at providing an overview on the different heat treatments commonly applied to alloy Ni-718 elaborated by these additive manufacturing processes, including considerations on their effects on microstructure and metallurgy and their impacts on mechanical properties.

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List of Acronyms

A	Ageing
CCT	Continuous Cooling Transformation Diagram
CHT	Continuous Heating Transformation Diagram
DA	Direct Ageing
EBM	Electron Beam Melting
EBSD	Electron Back Scattered Diffraction
FEG	Field Emission Gun
HA	Homogenized & Aged
HAADF	High Angle Annular Dark Field
HIP	Hot Isostatic Pressing
HIPA	Hot Isostatic Pressing & Ageing
HSA	Homogenized + Solution treated & Aged
LBM	Laser Beam Melting
PBF	Powder Bed Fusion
RT	Room Temperature
SA	Solution & Aged
SE	Secondary Electron
SEM	Scanning Electron Microscopy
STEM	Scanning Transmission Electron Microscopy
TTT	Time-Temperature-Transformation Diagram

Revision Table

Issue	Date	modified §	Evolution summary	Modified by
0	12/03/2019		Creation	All

1 Introduction

The application of heat treatments to alloy Ni718 have different purposes, that mainly lies in achieving optimized microstructure and metallurgical state adapted to confer to the alloy specific levels of final properties. The applied heat treatment sequences are dependent on the initial metallurgical condition, linked with elaboration and forming processes, and on the targeted mechanical properties and resistances regarding the type of service loading conditions.

Considering conventionally processed alloy Ni718, a great number of studies have been carried out on processing and heat treatments optimizations through the decades of development in the different fields of application of this alloys. Hence, this domain of activities is fruitful in terms of literature survey and available data to provide fundamental elements of reflexion to further optimize post fabrication heat treatments sequences on powder bed fusion manufactured (PBF-ed) alloy Ni718 parts.

This document is divided into three parts. The first one aims at providing synthetic information on the purpose, parameters, and effects of heat treatments currently applied on conventionally processed alloy Ni718. The second part summarizes the collected data on heat treatments applied to PBF-ed alloy Ni718, and largely inspired from those applied to conventionally processed alloy Ni718, with a distinction made on considered PBF technologies, Laser Beam Melting (LBM) or Electron Beam Melting (EBM). A final discussion based on the collected information on conventionally processed and PBF-ed alloy 718, whether abundant or scarce, will question the relevancy of applying “classical” heat treatments on radically different microstructures of PBF-ed alloy Ni718 parts, leading to consider the possibility further possible material optimizations.

2 Classical heat treatments for conventionally processed alloy Ni718

The long period of development and optimizations of elaboration conditions as well as of heat treatments on conventionally processed alloy Ni718 allowed great improvements of its microstructure and metallurgy providing it suitable resistances to critical structure and motor applications. As a function of elaboration processes, several unwanted detrimental inherited phases precipitate in the elaborated semi-products of alloy Ni718 that need to be removed before forming processes to ensure good and controlled formability of the alloy, as well final product properties conforming to the considered application’s specifications. Number of studies dealing with phases’ precipitation and kinetics have been carried out on alloy 718, which led to draw Time-Temperature-Transformation diagrams, highlighting the effect of global alloy’s composition on the position of precipitation noses, as shown in Figure 1. The specificity of alloy 718 lies in the great variety of phases likely to precipitate when the alloy is exposed to sufficiently high temperature. The temperature ranges of existence of the different phases are widespread and superimposed. This aspect is illustrated through Figure 2. The effective ranges of temperatures are actually dependent on the precise composition of the alloy, but the superimposition domains illustrate the extent to which heat treatments have to be precisely managed to trigger specific phases solutionizing or precipitation. In this goal, several heat treatments are commonly applied to control and optimize the alloy microstructure and metallurgy and are described thereafter.

2.1 Elaboration processes

Before addressing the purposes of each heat treatments currently performed on conventionally processed alloy 718, it seems essential to mention that the parameters of these heat treatments are correlated with the metallurgical states inherited from elaboration processes of the alloy. The elaboration is generally performed through two-steps melting (sometimes three, and even four for very specific applications) to provide ingots with suitable homogeneity and metallurgical state, ie. free from unwanted precipitated phases as well as sufficiently low levels of chemical segregations.

On the basis of these elaboration processes leading to very well controlled microstructures and segregations phenomena, hot and cold work steps are performed with subsequent precise heat treatments sequences. All these operations allow to produce high quality final products conforming to specific standards.

Reaching finely controlled microstructures and metallurgy on alloy 718 products required the development of a precise knowledge on the alloy's response to thermal exposure, which involved determining specific phases' existence domains. As an illustration, a classical Time-Temperature-Transformation (TTT) diagram for alloy 718 is shown in Figure 1, highlighting the effect of chemistry variations of precipitation noses' shift.

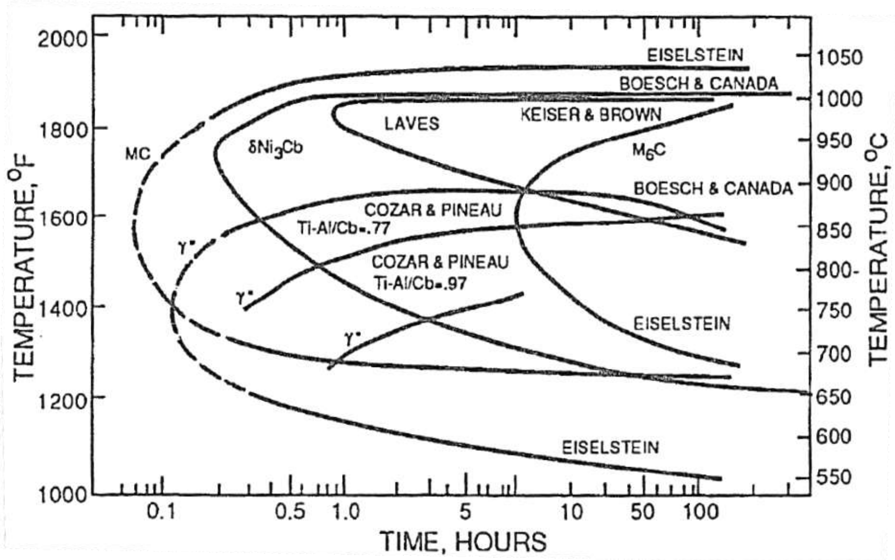


Figure 1- Time-Temperature-Transformation common diagram for alloy Ni718 with different compositions, based on data from different sources and provided by reference [1]

Hence, depending on the composition and the relative contents of the different alloying elements, the domains of existence of the different phases of interest in alloy 718 are likely to vary in a non-negligible manner. In addition to that, other parameters may influence the data collection dedicated to draw such a graphical representation, like local chemical variations, observation and analysis instruments, detection capability, identification of small size early precipitating phases. It is then worth mentioning that, through large surveys of literature in this field of research, the domains of existence of the different phases' are rather extended and the values of solvus temperatures remain quite scattered. The adapted schematic provided in Figure 2 shows the extent to which the domains of temperature existences of these different phases have been considered to range.

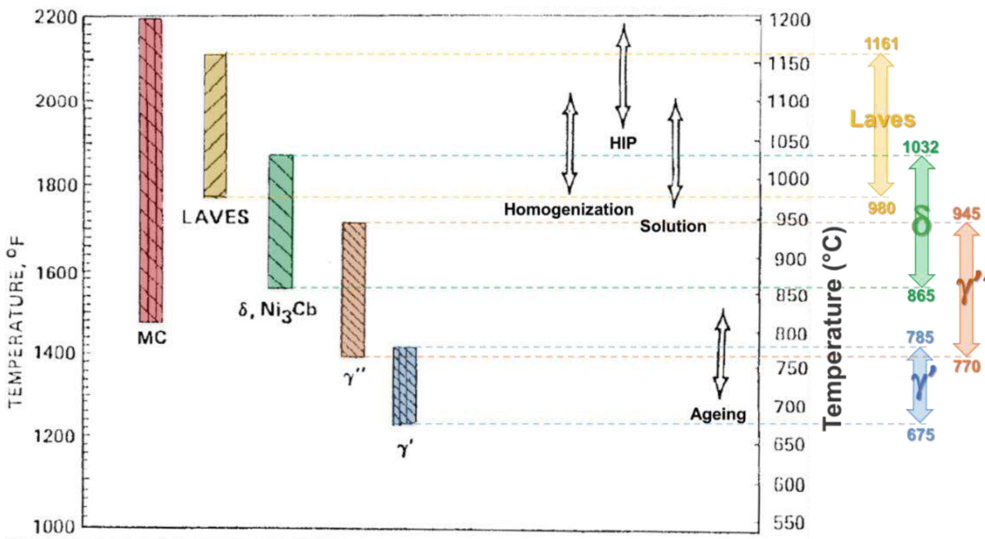


Figure 2- Representation of typical temperature domains of existence of the main phases existing in alloy Ni718, temperature values based on reference [2]

2.2 *Effects of the different heat treatments*

2.2.1 Stress relief

Stress relieving heat treatments are performed to remove or lower internal stresses in metallic materials. They are intended at the following specific operation with different purposes:

- Ensuring dimensional stability / preventing distortion during further machining steps,
- Increasing the capability of the alloy to undergo stress levels during subsequent forming operations likely to induce cracking,
- Avoiding cracking phenomena during subsequent heat treating steps.

The stress relief heat treatment has to be carried out at sufficiently high temperature to promote internal stresses reduction within an acceptable duration at the industrial scales of times, but should not be performed too high to prevent from excessive grain growth.

Concerning alloy 718, stress-relieving heat treatment is barely applied, and rather replaced by homogenization or solution heat treatment.

2.2.2 Homogenization (H)

Depending on the degree of chemical segregation phenomena induced by elaboration processes and on the characteristic dimensions of the elaborated ingot, a high temperature homogenization treatment is applied in order to solution Laves phases and to promote diffusion of Nb in dendritic areas without applying deformation.

As the dendritic structure exhibits important gradients of chemical composition, with "composite material" with different melting temperatures resulting from the presence of γ + Laves phase eutectics, the homogenization heat treatment is carefully performed by tempering with successive hold times at increasing temperatures, ensuring progressive reduction of segregation without inducing local liquation of the interdendritic areas. These successive tempering are generally performed at slow rates and can represent until 70 to 80 hours of heat treatment to ensure acceptable reduction of segregation and chemical heterogeneities levels reduction.

As Laves phase of different composition exhibits different solvus temperatures, heating to heat treating temperature is generally performed at slow rate.

2.2.3 Solution treatment (ST)

The solution treatment aims at solutionizing strengthening phases of alloy Ni718, namely γ'' and γ'. Two types of ST are commonly applied on alloy Ni718, depending on the required properties related to targeted applications.

(a) Standard Solution Treatment 1

The standard heat treatment for aerospace applications is ST1. It consists in maintaining the alloy during 1 to 2 hours in the range 927 to 1010°C. As this heat treatment is carried out under the δ solvus, this treatment promotes the precipitation of δ phase in the alloy that can develop either within the grains and/or at grain boundaries, acicular or globular. After this heat treatment, the alloy can be applied either fast cooling to "freeze" the microstructure, hot working steps and/or direct ageing. In all case, the presence of δ phase throughout the microstructure of the alloy constitutes a strong obstacle to interfaces motion, impeding grain boundaries sliding during high temperature forming and service exposure. The temperature at which the heat treatment is carried out in the considered range of temperature is very influent on the δ phase precipitation kinetics. Hence, the resulting δ precipitation state can be strongly affected by the solution treatment precise temperature and duration. This can be understood in the light of the T.T.T. diagram provided in Figure 1, taking into account the δ phase precipitation nose location regarding temperature in relation with the corresponding exposure duration triggering precipitation start. Further elements of information on the position variability of precipitation noses can be found out in the deliverable dealing with long term ageing of project alloys, referenced LIV-M031-L29-377.

The cooling rate from solution treatment temperature will also impact the nature and morphology of the phases likely to be observed. As an illustration, Figure 3 provides an established Continuous Cooling Transformation (CCT) diagram of alloy Ni718 after a 15 minutes exposure to 990°C. The β phase mentioned is another (older) denomination of δ phase. It is clear from this schematic that δ phase is very likely to precipitate during cooling, excepted for very high cooling rates. Of course, the effective CCT diagram will be affected, in shape and location, by the alloy chemical composition and by the time spent at prior heat treatment temperature.

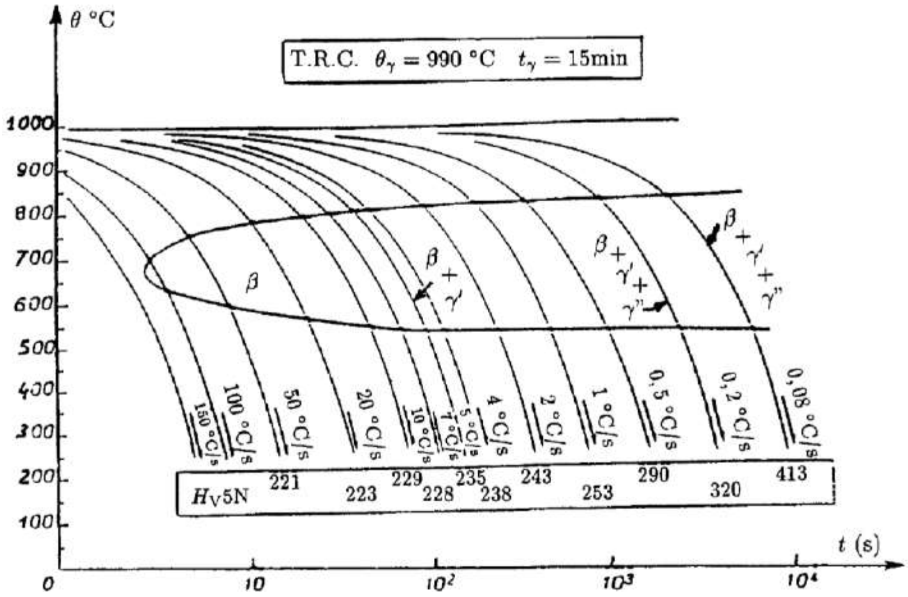


Figure 3- Continuous Cooling Transformation diagram for an initial maintain at 990°C, from reference [11]

(b) Standard Solution Treatment 2

For some applications, alloy Ni718 can be submitted to another solution heat treatment, performed above the δ solvus temperature, generally between 1038 and 1093°C during 1 to 2 hours, depending on the size of the treated part as well as on the initial δ phase dimension. In this high temperature range, the alloy reaches its "nil ductility" domain and is then not applied any hot working. The purpose here is to solutionize most of the precipitated phases in the alloy, including δ phase. In this case, the dissolution of all δ phase of the alloy induces progressive grain growth due to the disappearance of this pinning element impeding grain boundaries motion. Hence, the exposure to such high temperatures requires managing carefully the treatment duration to prevent excessive grain growth. This heat treatment is generally followed by rapid cooling to "freeze" the solutionized microstructure. This heat treatment is known to be suitable for tensile-limited applications, and provides higher impact strength and low-temperature notch tensile strength, as well as higher resistance for service conditions involving stress corrosion cracking or oxidation assisted intergranular cracking. It is commonly applied to cast alloy Ni718, but also to thin strips alloy 718 for nuclear applications for which δ phase constitutes an unwanted phase.

2.2.4 Ageing treatment (A)

The ageing heat treatment is the final element of the heat treating sequence which purpose is to ensure precipitation and growth of strengthening precipitates, namely γ'' and γ'. The ageing heat treatment parameters are optimized to follow a thermal path in the TTT diagram of alloy Ni718 likely to minimize the eventual δ phase precipitation and ensuring a maximum ageing state of the alloy. This state is commonly referred to "peak ageing". Further elements of information on the subject can be found out in deliverable dealing with long term ageing of the project alloys, referenced as LIV-M031-L29-377.

Two main ageing heat treatments have been developed and used on conventionally processed alloy Ni718, which can be described as follows:

1- Ageing heat treatment described through (AMS 5383, 5590)
   - 718°C +/- 8°C – 8 hours - 0 + 30 minutes;
   - Cool down to 621°C +/- 8°C at a cooling rate of 55°C +/- 8°C per hour
   - Holding at 621°C +/- 8°C for 8 hours - 0 + 30 minutes.
   - Final cooling at rate equivalent to air cool.

This treatment is known as the "conventional aeronautical ageing treatment". It provides the best compromise of properties and resistances regarding different types of service solicitations and degradation modes.

Several modifications in the description of this heat treatment appear in the different AMS considered:

- The description of the thermal cycles after first tempering step is sometimes replaced by a furnace cooling at any rate provided that the time at the second tempering step is adjusted to give a total precipitation heat treatment time of not less than 18 hours. (AMS 5596 – 5662)

2- Ageing heat treatment described through (AMS 5597, 5663, 5664)
   - 760°C +/- 8°C – 10 hours +/- 0.5 hour
   - Cool down to 650°C +/- 8°C at a cooling rate of 56°C +/- 8°C per hour
   - Holding at 649°C +/- 8°C until a total precipitation duration of 20 hours.

This ageing treatment held at higher temperature provides a higher thermal stability at the expense of tensile strength. Due to the presence of δ phase resulting from first tempering temperature, the resulting microstructure generally proves to be more sensitive to stress corrosion cracking and oxidation assisted intergranular cracking. In practice, this second ageing heat treatment is no longer used industrially.

In the framework of ANDDURO project, SAFRAN proposed an alternative ageing heat treatment which is likely to provide interesting levels of mechanical properties with a short heat treatment duration. This ageing heat treatment can be described as follow:

- 760°C – 5 hours +/- 0.5 hour
- Cool down to 650°C +/- 8°C at a cooling rate of 56°C +/- 8°C per hour
- Holding at 649°C +/- 8°C – 2 hours.

Further investigations and characterizations on the metallurgical state and mechanical properties provided by this specific ageing route are to be carried out during the project's activities.

Figure 4 illustrates the three aforementioned global heat treatments cycles .

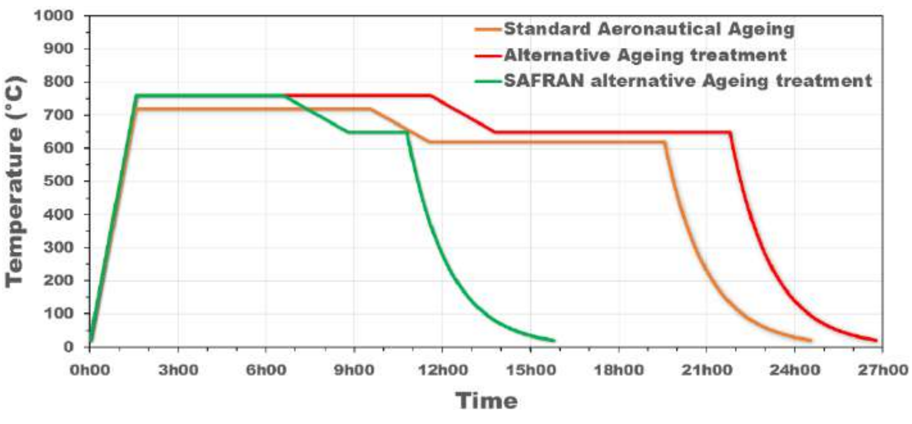


Figure 4- Comparative illustration of most commonly used ageing heat treatments sequences: (orange) standard aeronautical ageing treatment (AMS 5383, 5590, 5596, 5562, 5662) and (red) alternative ageing heat treatment (AMS 5597, 5564, 5663).

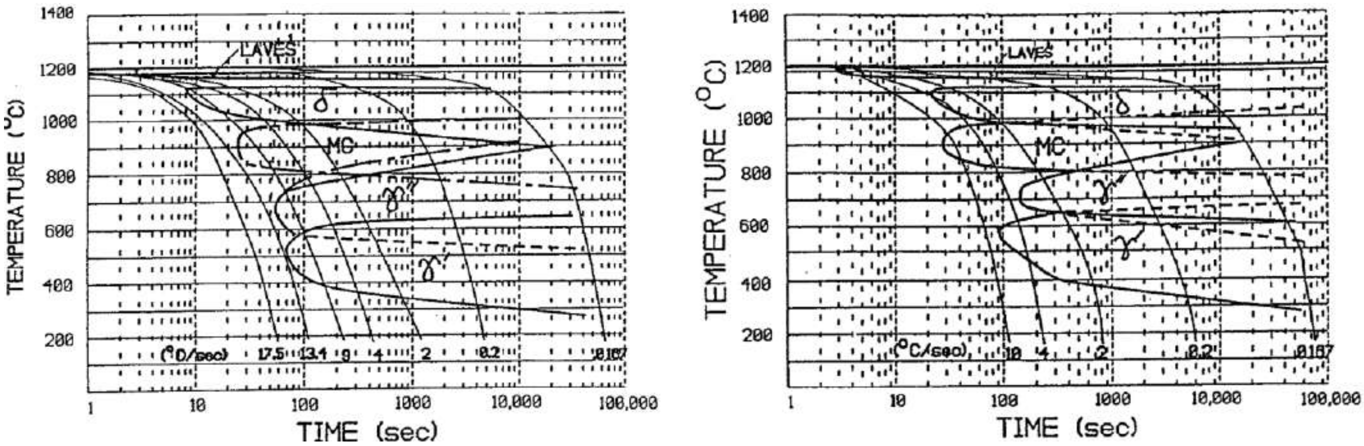
2.2.5 Hot Isostatic Pressing (HIP)

Hot Isostatic Pressing is mainly applied on cast alloy Ni718 or alloy Ni718 parts produced by powder metallurgy. It combines high temperature treatment (1100 to 1200°C) during few hours with important applied pressure (> 1000 bars). Whereas the main purpose of HIP lies in closing defects inherited from alloys processing steps, it can also offer the benefit of solving Laves phases and homogenizing the chemical gradients of the alloy, hence providing an interesting substitute to initial homogenization heat treatment. Considering its effect on defects, it is worth mentioning that HIP does not hold the same interest as a function of the processing atmosphere of the alloy: the presence of occluded gas defects type in the material make them unlikely to be enclosed by the application of pressure.

Regarding its impact on microstructure, the exposure to such a high temperature tends to solutionize all phases likely to pine grain boundaries and, thus, can induce a fast and important grain growth and has then to be managed carefully regarding thermal exposure duration.

Beyond these aspects, it is worth mentioning that, as shown in Figure 5 and Figure 6, precipitation after exposure to such a high temperature is likely to occur during cooling. These Continuous Cooling Transformation (CCT) diagrams are extracted from a publication, referenced as [12], dealing with the effects of homogenization treatments duration and precipitation during subsequent cooling on VIM + VAR material exhibiting important chemical segregations. The precipitation kinetics and magnitude are impacted by the tempering duration, correlated to the heterogeneities levels reduction, and the cooling rate from treatment temperature. Hence, being able to provide to the treated specimen cooling rates that are fast enough to manage and / or prevent unwanted precipitation likely to occur can be of important interest in the purpose of conferring to the alloy optimized properties.

This type of CCT diagram allows drawing another type of CCT representation showing when precipitation is likely to occur for different temperatures exposure, as shown on Figure 7 through the CCT like diagram representing in the temperature versus cooling rate plane the precipitation line of forming phases.



(a) (b)

Figure 5- CCT diagram of a highly segregated VIM + VAR alloy Ni718, homogenized at 1180°C during (a) 24 hours and (b) 72 hours before cooling, from reference [12]

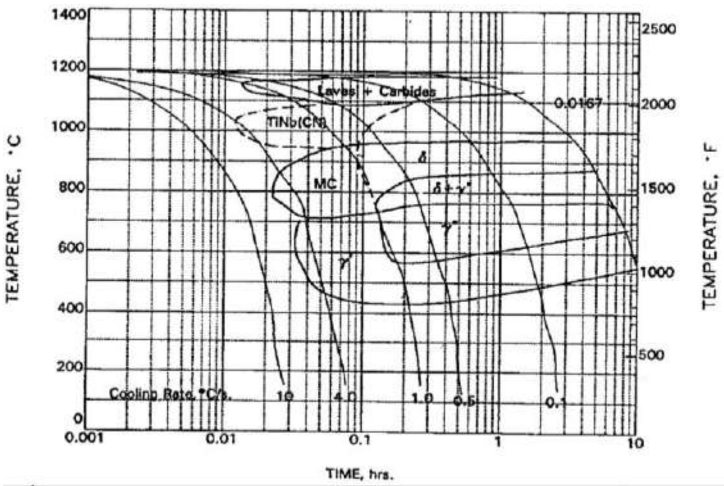


Figure 6- CCT diagram of alloy highly segregated VIM + VAR alloy Ni718, homogenized at 1180°C during 90 hours before cooling, from reference [12]

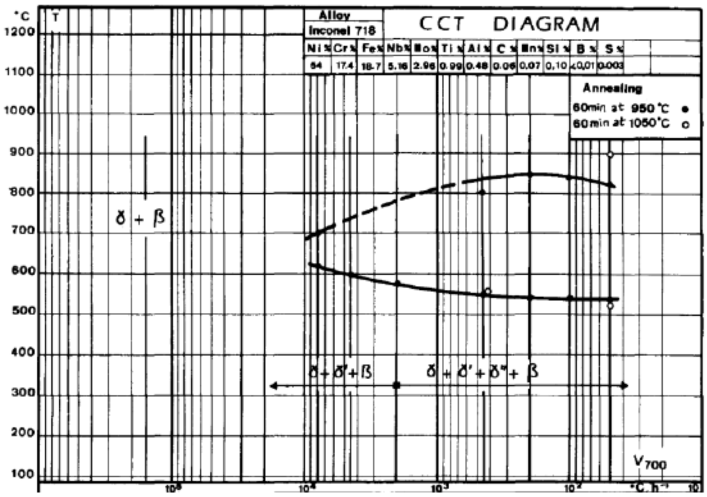


Figure 7- CCT representation of phases likely to appear in alloy Ni718, extracted from [13]

2.3 Targeted mechanical properties in tension

The standard minimum tensile properties Ni718 through the application of different aforementioned heat treatments sequences are reported in Table 1 of page 13 for room temperature testing and in Table 2 for high temperature (649°C) testing. These minimum specifications on tensile properties are provided on the basis of corresponding AMS norms, related either to cast or to wrought alloy Ni718 and depending on applied heat treatment sequence.

2.4 Discussion on conventionally processed alloy Ni718 heat treatments

The aforementioned heat treatments have been developed and optimized for alloy Ni718 produced by conventional processes. This involves specific elaboration steps including several remelting operations dedicated to lower chemical segregations, solutionize unwanted phases and generate microstructures of suitable workability for subsequent mechanical working routes to reach the suitable final shape, microstructure, metallurgical states. Hence, these heat treatment sequences are adapted to the specific features associated with conventional processing routes, in terms of characteristic microstructures, prior segregation phenomena intensities and precipitated phases dimensions. Furthermore, the applied heat treatments sequences are adapted to provide to the alloy levels of properties and mechanical resistances to specific types of solicitations suitable for the targeted applications of conventionally processed alloy parts.

Table 1- Minimum tensile properties of Ni718 with composition norm UNS N07718 at room temperature, related to different norms

Field of application	Homogenization	Solution Treatment	Ageing treatment	Testing temperature (°C)	YS (MPa)	UTS (MPa)	El. 4D (%)	RA (%)	Norm
Castings	1093°C - 1 to 2h / AC	954 to 982°C ≥ 1h - AC	718°C/8h + 621°C/8h	RT	758	862	3	10	AMS 5383™
Wrought	-	941 to 1010°C - AC	718°C/8h + 621°C/8h	RT	1034	1241	6	8	AMS 5662™
Wrought	-	941 to 1010°C - AC	718 to 760°C/8h + 621°C/8h	RT	1000	1241	10	12	AMS 5663™
Wrought	-	1052 to 1080°C - AC	718°C/8h + 621°C/8h	RT	1034	1241	10	12	AMS 5664™
Wrought	-	941 to 996°C - AC	-	RT	552	965	30	10	AMS 5596™
Wrought	-	941 to 996°C - AC	718°C/8h + 621°C/8h	RT	1034	1241	12	-	AMS 5596™
Wrought	-	1052 to 1080°C - AC	-	RT	517	965	30	-	AMS 5597™
Wrought	-	1052 to 1080°C - AC	760°C/10h + 621°C/5h	RT	1034	1241	15	-	AMS 5597™
Wrought	-	927 to 996°C / AC	-	RT	1069	1276	20	-	AMS 5950™
Wrought	-	927 to 996°C / AC	718°C/8h + 621°C/8h	RT	1020	1241	12	-	AMS 5950™

Table 2- Minimum tensile properties of Ni718 with composition norm UNS N07718 at high temperature (649°C), related to different norms

Field of application	Homogenization	Solution Treatment	Ageing treatment	Testing temperature (°C)	YS (MPa)	UTS (MPa)	El. 4D (%)	RA (%)	Norm
Wrought	-	941 to 1010°C - AC	718°C/8h + 621°C/8h	649°C	841	965	6	8	AMS 5662™
Wrought	-	941 to 1010°C - AC	718 to 760°C/8h + 621°C/8h	649°C	841	965	6	8	AMS 5663™
Wrought	-	941 to 996°C - AC	718°C/8h + 621°C/8h	649°C	793	965	5	-	AMS 5596™
Wrought	-	927 to 996°C / AC	718°C/8h + 621°C/8h	649°C	793	965	5	-	AMS 5950™

3 Powder bed additive layer manufaturing

Alloy Ni718 fabricated by powder bed additive layer manufacturing is known to exhibit specific microstructures and metallurgical states that are very different from what can be obtained through conventional processing, wrought or cast alloy Ni718. The powder used for powder bed technologies is obtained by atomization of ingots melted through conventional vacuum melting processes, VIM or VAR. The process of additive manufacturing induces a melting step of the as-deposited powder, and subsequent remelting due to layer-by-layer fabrication. During these operations, the alloy undergoes fast cooling rates and several reheating, which constitutes a very specific and complex thermal history. The characteristic features of the initial state will be largely dependent on the processing parameters, due to differences in undergone thermal cycles, building strategy and atmosphere. The fabrication process inherited phases are not managed as precisely as in the case of conventional elaboration processes, and the post fabrication treatments aiming at reductions of heterogeneities do not include working operations, and are exclusively based on the application of subsequent heat treatments.

4 Laser Beam Melting (LBM)

ALM parts in alloy Ni718 elaborated by LBM technology can exhibit relatively high mechanical properties in their as-built state, due to internal stresses resulting from fast cooling rates and precipitated phases inherited from thermal cycles during fabrication. The as-built microstructure of alloy Ni718 produced by LBM is dependent on several factors, which are mainly correlated with considered LBM device, ie. corresponding powder specification, building parameters and building strategy.

The application of stress relief heat treatment on as-built parts prior to their removal from their fabrication plates can be required for specific “thin” geometries to prevent deformation due to internal stresses, but is generally not mandatory. Then, a post fabrication thermal treatments sequence is applied to fulfil targeted mechanical performances through the achievement of specific microstructure ensuring suitable resistance to service loading conditions.

4.1 As-built LBM alloy Ni718 microstructure and metallurgical condition

LBM melting conditions and strategy induce quite specific as built alloy Ni718 microstructures that are inherited from high cooling rates and repeated re-heating of the deposited layers, generated complex fabrication thermal cycling histories. The general appearance of as-built material is dependent on fabricated parts size and orientation regarding building direction. Furthermore, the as-built strong texture effects have a strong incidence on resulting mechanical properties, inducing important anisotropy.

Further information on as-built alloy 718 produced by LBM technology can be found out in the deliverable dealing with microstructure and mechanical properties of project alloys, referenced as LIV-M031-L01-409.

Considering microstructure and metallurgy, the main specificities of LBM alloy 718 when comparing to conventional processes, casting and forging, lie in the very fine microstructure characteristic dimensions exhibited by the as-built material, inherited from the process specific thermal parameters. As to provide quantified figure, it can be mentioned that primary dendrites arm spacing are reduced by a typical factor of about 100 with respect to those observed in casting process. This has a direct impact on the characteristic sizes of precipitated phases (carbides, Laves), the magnitude of segregation between dendrites and interdendritic spaces, as well as the required duration, correlated to diffusion distances, to reach homogeneity when applying dedicated heat treatments.

4.2 Effects of conventional heat treatments on as-built LBM alloy Ni718 microstructure

4.2.1 Stress Relief (SR)

As the purpose of stress relief lies in preventing deformation of produced parts under the effect of internal stresses inherited from fast cooling after building process, it should be applied prior to removal from substrate of produced parts with fine geometries likely to undergo cracking during cutting operation.

As mentioned earlier in the document when dealing with conventional alloy 718, stress-relieving heat treatment is rather replaced by homogenization or solution heat treatment.

4.2.2 Solution treatment (ST)

The solution treatment aims at solutionizing several phases likely to precipitate during the alloy building process. Depending on the targeted properties, related to the considered applications of the produced part, this solution heat treatment can be carried out either above or under the δ solvus temperature. It can be considered that it will solutionize only strengthening precipitates, namely γ' and γ'', or solutionize both strengthening precipitates and δ phase as well. The survey of literature data on LMB alloy Ni-718 shows no consensus when considering ST temperature, and the explored domains of heat treatments are either above δ solvus ([14] [15]) or below δ solvus ([16] [17] [18] [19] [20] [21] [22] [23]).

(a) Solutionizing under the δ solvus temperature

Solutionizing the alloy under the δ solvus, generally at a temperature ranging from 920°C to 1020°C lead to the emergence of grains structure separated by grain boundaries. These grains are free from strengthening precipitates, γ' and γ'', but they are unable to reach an equilibrium shape due to the presence the aforementioned Nb-rich phases.

Metallographic observations at different scales show the traces of prior melt pools boundaries and evidence the former as-built dendritic structure. These observations highlight the partial effect of the heat treatment regarding the recrystallization of the alloy.

- Low temperature heat treatments (920°C < T < 1020°C)
    - Reduces internal stresses
    - Lowers texture intensity
    - Softens chemical heterogeneities
    - Solutionizes strengthening phases (γ', γ'')
    - Promotes δ phase precipitation
    - Promotes interfaces mobility and formation of grains structure

(b) Solutionizing above the δ solvus

Solutionizing the alloy above the δ solvus, generally at a temperature ranging from > 1020°C to 1100°C lead to the emergence of grains structure separated by grain boundaries. These grains are free from strengthening precipitates, γ' and γ'', and δ phase is no longer present to pine grain boundaries. Nevertheless, carbides and Laves phase are still sufficient to impede grain boundaries motion, preventing grains from important growing. Hence, the final microstructure resulting from such heat treatment is composed of elongated grains oriented in the same direction as the Nb-rich phases' initial alignment.

- High temperature heat treatment (1020°C < T < 1100°C)
    - Lowers internal stresses
    - Reduces texture intensity
    - Softens chemical heterogeneities
    - Solutionizes many phases (γ', γ'', δ)
    - Promotes interfaces mobility and formation of grains structure

4.2.3 Hot Isostatic Pressing (HIP)

Hot Isostatic Pressing is commonly applied in the purpose of closing the fabrication processes' inherent defects. These defects are of different types: pores of occluded gas, lacks of fusion, microcracks resulting from internal stresses. HIP is generally carried out in the temperature range 1100°C to 1200°C, under an applied pressure of about 1000 bars and for duration ranging from 2 to few hours.

The efficiency of HIP treatment is not the same regarding its ability to close all three types of defects. To precise, the greatest number of defects is composed of spherical porosities containing gas (argon from the fabrication environment). Their spherical morphology and internal gas pressure make them all the more difficult to be closed. An important aspect regarding HIP treatment is that it takes place at sufficiently high temperature to generate grain boundaries movement, involving a strong tendency to grain growth. This is well-known when dealing with conventionally processed alloy Ni718. Hence, on the basis of considerations dealing with the produced part targeted application and criticity, the type of loading conditions and the durability requirements, the choice of applying a HIP treatment results from a balance between benefits of defects closure and drawbacks associated with the important average grain size growth.

4.2.4 Ageing treatment (A)

The ageing treatment is classically applied to induce nucleation and growth of strengthening phases, γ'' and γ', which confer to the alloy its high levels of mechanical properties. This treatment does not impact the global microstructure, but leads to intergranular precipitation of a fine distribution of precipitates.

4.2.5 Typical heat treatments and effects on microstructures

As for conventionally processed alloy Ni718, several heat treatments might be applied sequentially to as-built LBM alloy Ni718 in order to fulfil desired microstructure, optimized to reach targeted mechanical properties suitable to service application's loading conditions. As a function of applied heat treatments sequence, the resulting microstructure is quite different:

- **As-built,** Figure 8 (α, β): Dendritic microstructure growing through successive melt pools. Presence of various precipitated phases: carbides, Laves phase, and some δ phase.
- **DA,** Figure 8 (a, b): Dendritic microstructure still present and prior melt pools still observable. Presence of as-built precipitated phases, and fine precipitation of strengthening precipitates, γ' and γ''.
- **SA,** Figure 8 (c, d): Dendritic microstructure less observable and unitary grains tend to develop. Grain boundaries' decoration by Laves phase and δ phase is more pronounced and takes the form of nearly continuous small platelets, which limit the ability of grains to reach their equiaxial equilibrium shape. Intragranular needle-shaped δ phase precipitates throughout the grains, and fine precipitation of strengthening precipitates, γ' and γ''. Strengthening precipitates free zones are surrounding δ phase due to niobium depletion in its vicinity.
- **HA,** Figure 8 (e, f): Dendritic microstructure no longer observable and unitary grains developed and grew. Grain boundaries are decorated by fine and nearly continuous mixture of Laves phase and some δ phase. The recrystallization process is more advanced, but grains remain elongated, oriented following precipitates distribution inherited from as-built structure. Intragranular fine and homogeneous precipitation of strengthening precipitates, γ' and γ'' occurred.
- **HSA,** Figure 8 (g, h): Dendritic microstructure no longer observable and unitary grains developed and grew. Grain boundaries are decorated by an abundant mixture of needle-shaped and small platelets δ phase. The recrystallization process is well advanced, but grains remain elongated oriented precipitates inherited from as-built structure. Some intragranular needle-shape delta phase is present, together with fine precipitation of strengthening precipitates, γ' and γ''.
- **HIP,** Figure 9 (c): Dendritic structure has totally disappeared, and unitary grains largely grew when compared to Figure 9 (a) and (b). The applied temperature of this treatment is high enough for grain boundaries to overcome pinning effect of remaining precipitated phases, enabling grains to grow and reach

a nearly equiaxial equilibrium shape. This heat treating temperature allows material recovery of a more equiaxial grains structure leading to more homogenous properties and reduction of anisotropy. The heterogeneous precipitation of additional carbides have been reported in reference [24] , preferentially located underneath the pressure exposed faces.

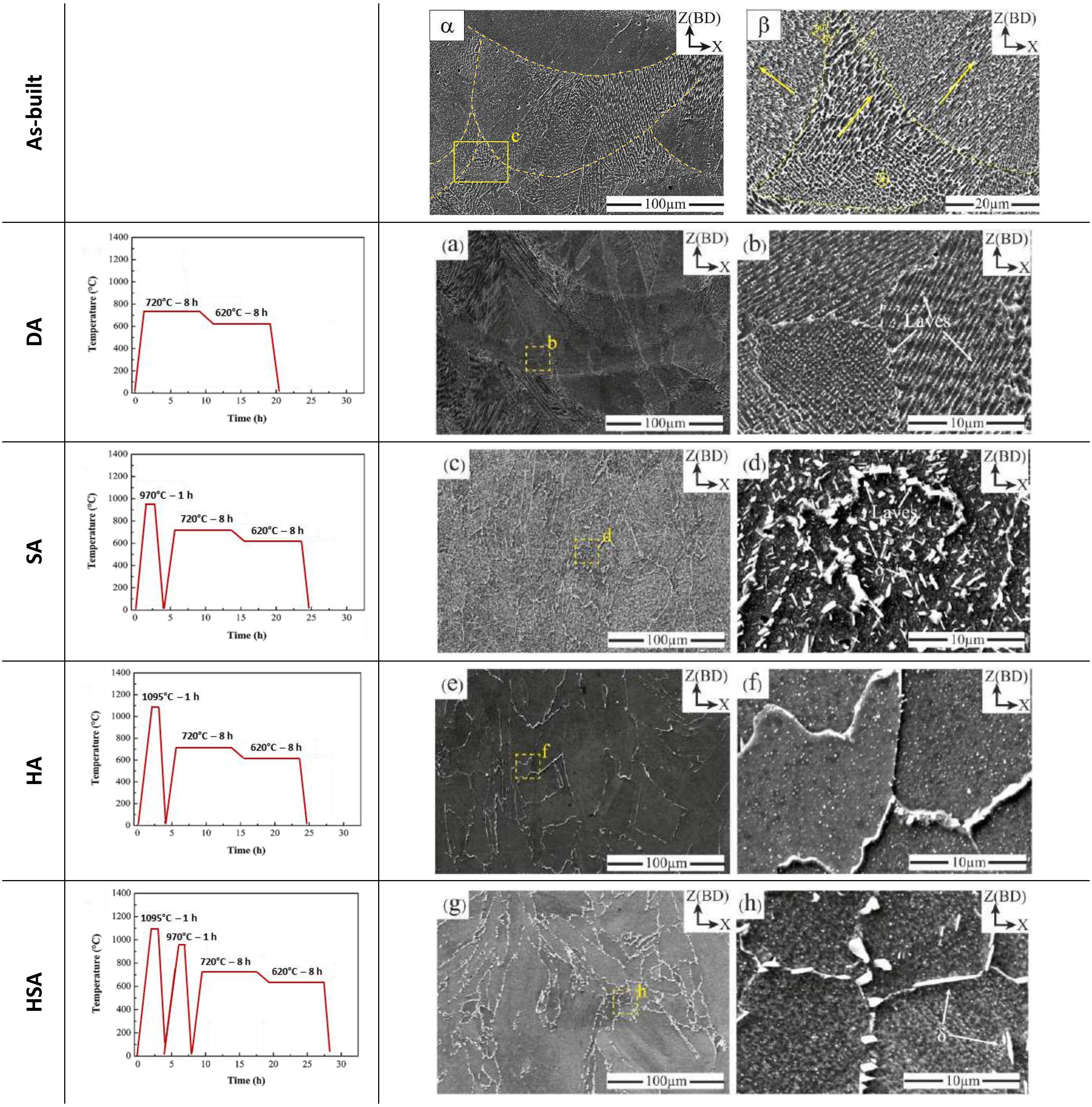


Figure 8- Microstructure of etched vertically built SLM IN718 samples in the heat-treated conditions: (α) As-built, (a) Direct Aged, (c) Solution treated below δ solvus and Aged, (e) Homogenized and Aged, (g) Homogenized, Solution treated below δ solvus and Aged, and (β) (b), (d), (f), (h) are the magnified regions indicated in (α), (a), (c), (e), (g) respectively, extracted from reference [20]

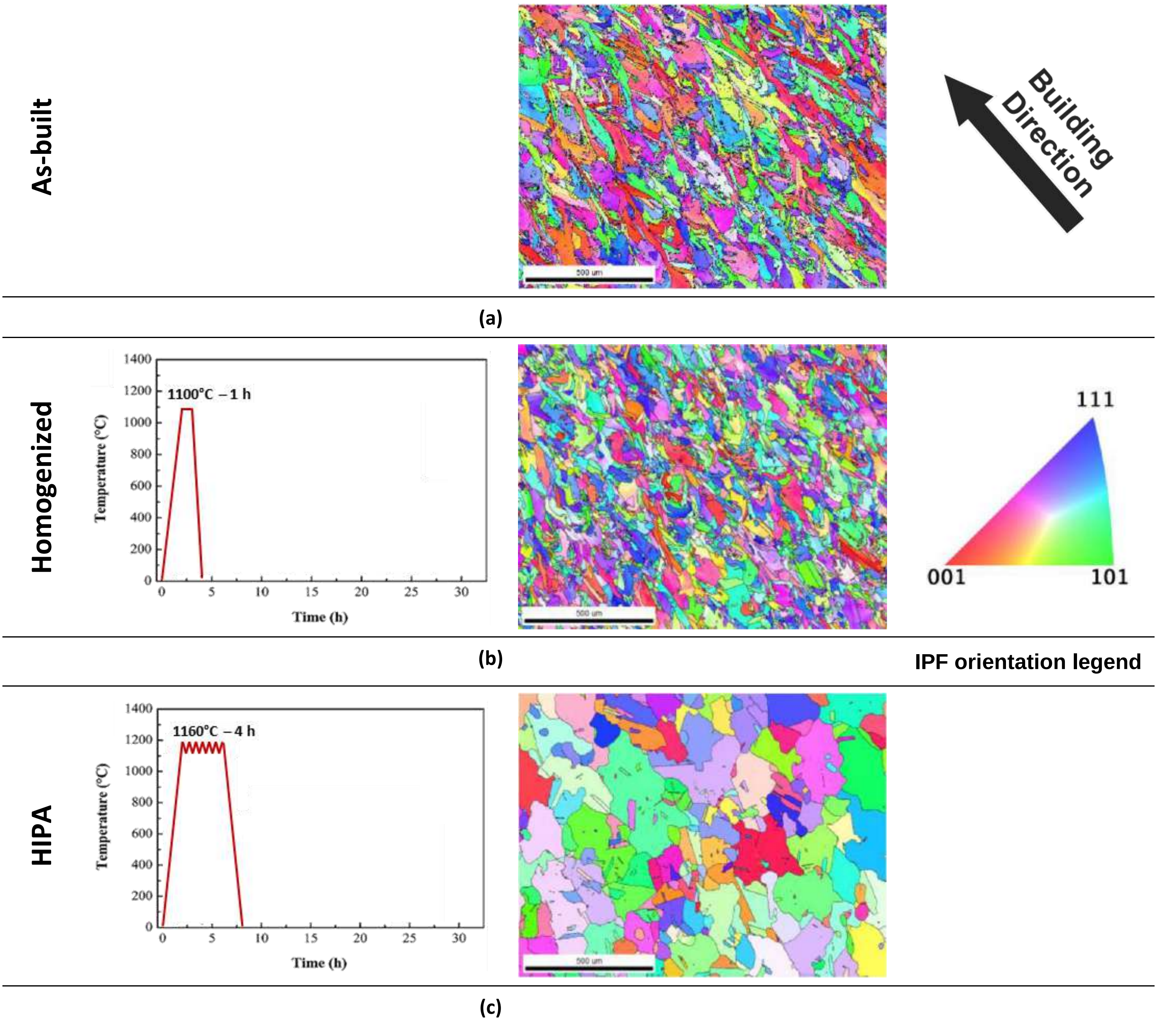


Figure 9- EBSD Inverse pole figure of LBM alloy Ni718 in different microstructural and metallurgical conditions (a) as-built, (b) Homogenized (1100°C/1h), and (c) HIP treated (1160°C/4h/1kbar), from reference [24].

4.3 *Effect of heat treatments sequences on tensile properties*

The available literature review on alloy Ni718 produced by LBM shows a great variety of performed post-fabrication heat treatments. The explored heat treatments sequences are different, and the temperature is different for single step of the sequence. Considering that the initial state of the alloys produced in the available studies is different, due to variability of processing parameters, building strategy and, eventually, basis powder used, the precise effect of each heat treatment step in the applied sequences is not immediate to infer from the collected data. Nevertheless, an attempt is made in this paragraph to highlight the main inferable trends. Table 3 is a synthetic table showing several available publications dealing with LBM-ed alloy Ni718, and detailing information on LBM device, applied heat treatment sequences with parameters, and obtained mechanical properties with respect to sampling directions.

Table 3- List of room temperature tensile properties of LBM alloy Ni718 as a function of LBM device, applied post heat treatment and sampling direction, as found out from a literature survey, ref. [14] [17] [15] [16] [19] [20] [21] [22] [25] [26].

	LBM Device	Known parameters	Stress Relieving / Q	Hot Isostatic Pressing / SC	Homo / Q	Solution Treatment / Q	Age step 1	Age step 2	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation to failure (%)
Schneider 2018 [14]	Concept Laser M2		-	-	-	-	-	-	698	995	33.2
			-	-	-	-	720°C - 8h	-	1204	1392	17.3
			-	-	-	-	720°C - 8h	620°C - 10h	1269	1740	15.4
			-	-	-	1010°C - 1 h	720°C - 8h	620°C - 10h	1238	1379	19.5
			1066°C - 1h30	-	-	-	-	-	860	1171	34.3
			1066°C - 1h30	-	-	-	720°C - 8h	-	1124	1331	21.3
			1066°C - 1h30	-	-	-	720°C - 8h	620°C - 10h	1201	1387	20.8
			1066°C - 1h30	-	-	1010°C - 1 h	720°C - 8h	620°C - 10h	1203	1390	22.0
			1066°C - 1h30	1163°C - 3h	-	954°C - 1 h	720°C - 8h	620°C - 10h	1087	1385	23.4
			1066°C - 1h30	1163°C - 3h	1163°C - 1h	954°C - 1 h	720°C - 8h	620°C - 10h	1111	1396	23.6
Zhang 2015 [12]	EOSINT M280		-	-	-	-	-	-	849	1126	22.8
			-	-	-	980°C - 1h	720°C - 8h	620°C - 8h	1084	1371	10.1
			-	-	1080°C - 1h30	980°C - 1h	720°C - 8h	620°C - 8h	1046	1371	12.3
Ströβner 2015 [15]	/								H // V	H // V	H // V
			-	-	-	-	-	-	816 // 737	1085 // 1010	19.1 // 20.6
			~ 980°C	-	-	980°C - 1h	760°C - 10h	650°C - 8h	1227 // 1136	1447 // 1357	10.1 // 13.6
			~ 980°C	-	1065°C - 1h	980°C - 1h	760°C - 10h	650°C - 8h	1222 // 1186	1417 // 1387	15.9 // 17.4
Raghavan 2016 [11]	EOS M400		-	-	-	-	-	-	612	962	28,8
			-	-	-	1040°C - 2h	720°C - 8h	620°C - 8h	1213	1404	18,0
			-	-	-	1040°C - 2h	700°C - 28h	-	1210	1401	18,8
			-	-	-	1100°C - 2h	720°C - 8h	620°C - 8h	1148	1305	17,8
			-	-	-	1200°C - 2h	720°C - 8h	620°C - 8h	822	1121	21,6
Scott-Em. 2015 [26]	EOSINT M280								H // V	H // V	H // V
			1065C° - 1h30	1177°C - 103MPa	-	968°C - 1h30	718°C - 8h	612°C - 8h	1034 // 1068	1309 // 1344	27 // 27
Trosch 2016 [22]	/								H // 45° // V	H // 45° // V	H // 45° // V
									1188 // 1194 //1186	1439 // 1449 // 1406	-
Aydinoz 2016 [13]	SLM 280^{HL}		-	-	-	-	-	-	580	845	-
						1000°C - 1h - AC			535	870	-
			-	-	-	1000°C - 1h - AC	720°C - 8h - FC	621°C - 8h - AC	1240	1400	-
				1150°C - 4h - FC					430	875	-
				1150°C - 4h - FC			720°C - 8h - FC	621°C - 8h - AC	1100	1315	-
Chlebus 2015 [13]	SLM Realizer II 250								H // 45° // V // 45°x45°	H // 45° // V // 45°x45°	H // 45° // V // 45°x45°
			-	-	-	-	-	-	643 // 590 // 572 // 723	991 // 954 // 904 // 1117	13 // 20 // 19 // 16
			-	-	-	1100°C - 1h - WQ	720°C - 8h - FC	620°C - 10h - AC	1159 // 1152 // 1074 // 1241	1377 // 1371 // 1320 // 1457	8 // 15 // 19 // 14
Deng 2018 [17]	EOS M290								H // V	H // V	H // V
			-	-	-	-	-	-	790 // 620	1060 // 1000	32 // 36.5
			-	-	-	-	720°C - 8h - FC	620°C - 8h - AC	1380 // 1198	1520 // 1415	15.5 // 15.5
			-	-	-	980°C - 1h - WQ	720°C - 8h - FC	620°C - 8h - AC	1220 // 1180	1500 // 1400	19 // 23.5
			-	-	1080°C - 1h - WQ	-	720°C - 8h - FC	620°C - 8h - AC	1250 // 1190	1450 // 1390	19.8 // 25.2
			-	-	1080°C - 1h - WQ	980°C - 1h - WQ	720°C - 8h - FC	620°C - 8h - AC	1250 // 1200	1450 // 1390	20 // 24

	LBM Device	Known parameters	Stress Relieving / Q	Hot Isostatic Pressing / SC	Homo / Q	Solution Treatment / Q	Age step 1	Age step 2	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation to failure (%)
Brenne 2016 [18]	SLM 280^{HL}		-	-	-	-	-	-	449	784	29.1
			-	-	-	550°C - 1h	-	-	498	858	25.0
			-	-	-	700°C - 1h	-	-	741	1055	19.5
			-	-	-	1000°C - 1h	-	-	380	719	46.3
			-	-	-	-	720°C - 8h - AC	621°C - 8h - AC	993	1290	15.7
			-	-	-	550°C - 1h	720°C - 8h - AC	621°C - 8h - AC	944	1209	8.7
			-	-	-	700°C - 1h	720°C - 8h - AC	621°C - 8h - AC	964	1284	18.0
			-	-	-	1000°C - 1h	720°C - 8h - AC	621°C - 8h - AC	1105	1287	23.2
									H // V	H // V	H // V
Seifi 2018 [19]	SLM 280		-	-	-	-	-	-	762 // 608	1058 // 933	26.5 // 33
				1120°C - - Fast C		980°C - 1h - AC	720°C - 8h - FC	621°C - 8h - AC	1037 // 1053	1271 // 1271	24 // 20
Popovitch 2017 [20]	SLM 280^{HL}	250 W	-	-	-	-	-	-	668	752	22,0
			-	-	-	850°C - 2h - AC	-	-	875	1153	17,0
			-	1180°C - 3h- 150 MPa - FC	-	-	-	-	645	1025	38,0
			-	1180°C - 3h- 150 MPa - FC	1065°C - 1h - AC	-	760°C - 10h - FC	650°C - 8h - AC	1145	1376	19,0
		950 W	-	-	-	-	-	-	531	866	21,0
			-	-	-	850°C - 2h - AC	-	-	668	884	7
			-	1180°C - 3h- 150 MPa - FC	-	-	-	-	481	788	34
			-	1180°C - 3h- 150 MPa - FC	1065°C - 1h - AC	-	760°C - 10h - FC	650°C - 8h - AC	1065	1272	15

On the basis of the data collected throughout this bibliographical review, the tensile properties obtained on as-built alloy Ni718 or after the application of different heat treatments sequences have been extracted and synthetized by the graphical representations provided in Figure 10. On these graphics, several AMS norms properties specifications have been superimposed to simplify the reading. From these graphical representations, several aspects worth noting:

- As-built LBM-ed alloy Ni-718 properties, Figure 10 (a), exhibit an important variability, particularly in terms of yield strength and ductility, their comparison with the AMS specifications for solutionized alloy Ni718 is intended to highlight the fact that are far from being in a solid solution state.
- The solution treated and aged LBM-ed alloy Ni718 properties, Figure 10 (b), shows that most of the represented treated alloys are complying with targeted properties of AMS specifications for STA conventionally processed alloy Ni718. This remains true for solution treatments carried out either below or above the δ solvus temperature. The treated alloy that does not comply with AMS norm has been solutionized either at very high temperature (ie. 1200°C [14]), or at very low temperatures (ie 550°C and 700°C [21]).
- Hot Isostatic pressed and aged alloy Ni718 properties, Figure 10 (c), comply with AMS specifications whatever the HIP temperature ranging from 1080°C to 1180° and the duration ranging from 1 to 4 hours, and independently of applied subsequent treatments (homogenization and/or solution treatment) provided that a final ageing treatment is applied.
- Direct aged alloy Ni718 seems to provide interesting tensile properties as shown on Figure 10 (d). Nevertheless, it is noteworthy that the available data on this metallurgical state of the alloy are scarce, and further investigations might be of interest to ensure its viability regarding, for example, different types of loading conditions or its response to thermal ageing.

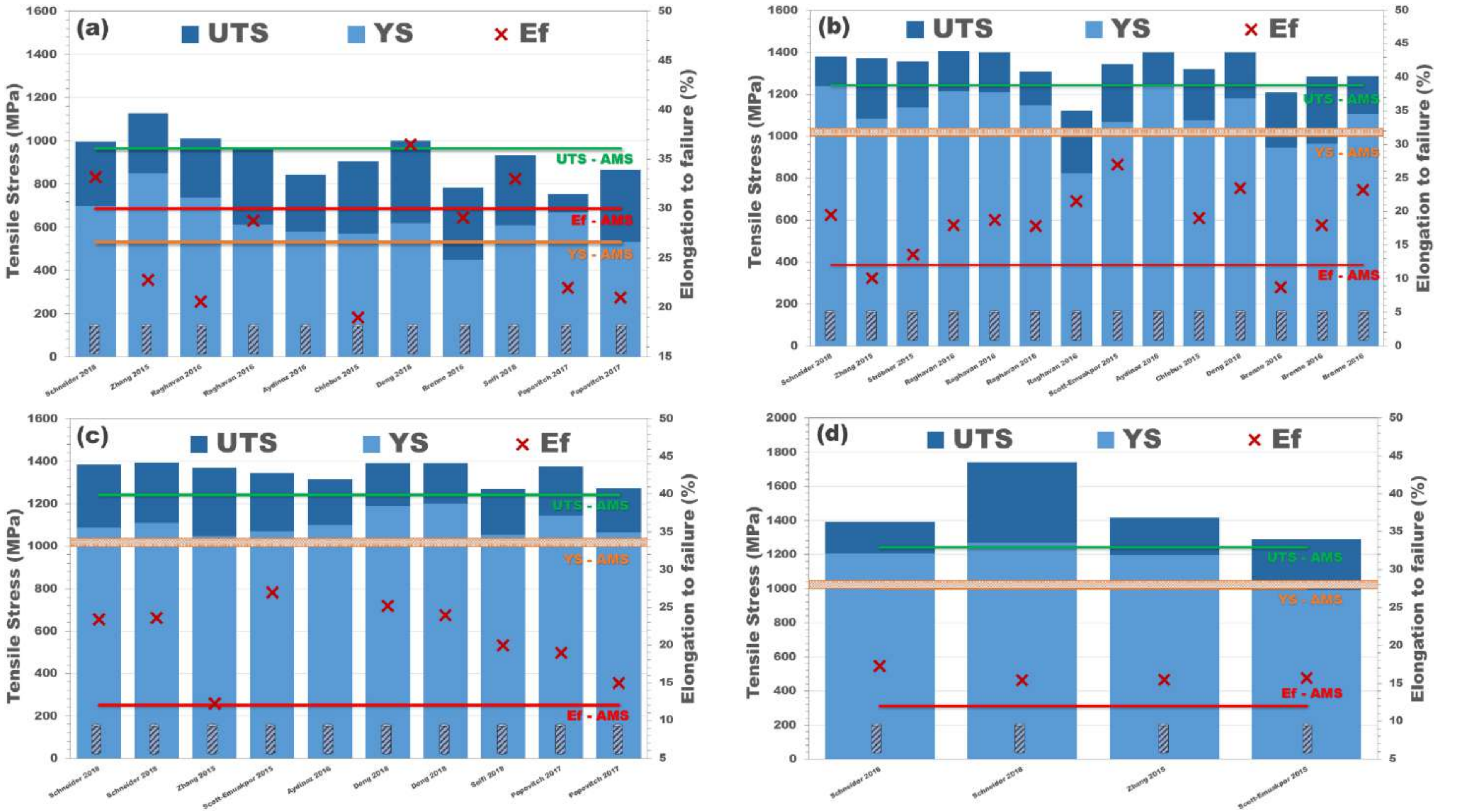


Figure 10- Graphical synthesis of RT tensile properties for different states of LBM-ed alloy 718 presented in Table 3, and superimposed specifications of AMS norms. (a) As-built alloy Ni718, (b) Solution Treated and Aged alloy Ni718, (c) Hot Isostatic Pressed and aged alloy Ni718, and (d) Direct Aged alloy Ni718.

5 Electron Beam Melting (EBM)

After fabricated EBM parts are removed from their support material, post-thermal treatment are applied to fulfil targeted mechanical performances through the achievement of specific microstructure ensuring suitable resistance to service loading conditions.

One of the specificity of EBM lies in the heating of the substrate during building process. This pre-heating induces much lower cooling rates than in the case of LBM, and the global fabrication resulting thermal cycle is quite different.

5.1 As built EBM microstructures

From the literature survey on the characteristics of alloy 718 produced by EBM technology, it appears that the accessible melting conditions and strategy offer wider ranges to manage the alloy's as built microstructures. Another specific aspect of EBM fabrication lies in the variability of microstructure and metallurgical state throughout the height of the produced parts resulting from different local thermal histories.

Hence, applying an optimized post fabrication heat treatments sequence on EBM alloy 718 requires several elements of information on the effective as-built microstructure as well as the extent of local microstructure and metallurgical state variability.

5.2 Effects of conventional heat treatments on as-built EBM alloy Ni718 microstructure

The available information throughout a large literature survey shows the scarcity and the variability of bibliographic data on EBM alloy 718 post fabrication heat treatments.

5.2.1 Stress Relief (SR)

As a result preheating of the material during fabrication, the cooling rates experienced by EBM-ed alloy Ni718 are much slower than those encountered in LBM. These cooling rates are slow enough to induce low internal stresses, and as final cooling to room temperature takes place under vacuum atmosphere, the eventually present internal stresses are sufficiently lowered so that alloy Ni718 specimens produced by EBM do not require SR treatment.

5.2.2 Solution treatment (ST)

Due to prolonged fabrication process thermal cycling and prolonged exposure to high temperatures, several unwanted precipitated phases are likely to be present in the as-built material. As mentioned earlier in this document, the application of a solution treatment has two main interests : solutionizing phases inherited from fabrication to allow interfaces mobility necessary to promote partial recrystallization and reduction of properties' anisotropy, and/or ensuring more homogeneous distribution of strengthening precipitates forming during subsequent ageing heat treatment. The applied solution heat treatment parameters, temperature and duration, are dependent on the initial metallurgical state, which are, in the case of EBM, largely correlated with pre-heating temperature, and is also function of the targeted properties and resistances to specific loading conditions. Hence, the solution treatment temperature can be adapted to dissolve selected undesired phases before applying the ageing heat treatment and its duration should be correlated with the precipitated phases' characteristic dimensions. The effects of different solution heat treatments temperature will considered in the paragraph 5.2.2 of page 24, dealing with general global heat treatment sequences, on the basis of available literature on the subject.

5.2.3 Hot Isostatic Pressing (HIP)

The application of HIP treatment is often considered as mandatory when considering parts dedicated to high loading structure application. Its essential purpose is to close bulk porosity, fusion defects and internal cracks inherent to additive manufacturing processed materials. It is worth mentioning that, the fact that EBM fusion is performed under dynamic helium primary vacuum environment, induces porosities that are inherently different from those resulting from LBM processing condition. Hence, their low internal gas pressure makes them all the more likely to be closed through the application of HIP treatment.

The main drawback of the HIP treatment lies in the significant coarsening of the initial grains structure [27] [31], reducing the benefit of fine structures potentially accessible through additive manufacturing technologies, as shown in Figure 11.

Due to the specificity of EBM for which building can be done at different pre-heating temperature, as-built microstructures can exhibit different characteristics as a function of this parameter. HIP will produce different effects regarding as-built microstructure evolution depending on the initial microstructure. This can be illustrated by the EBSD maps provided in Figure 11 and showing the effect of 1200°C – 2 hours HIP treatment application on different microstructures inherited from several EBM processing parameters builds. These EBSD characterisations highlight the great variability of microstructures response to such a high temperature exposure, resulting either in an important grain coarsening and reduction in their inherited texture, or in nearly no observable effect on as-built microstructure or texture.

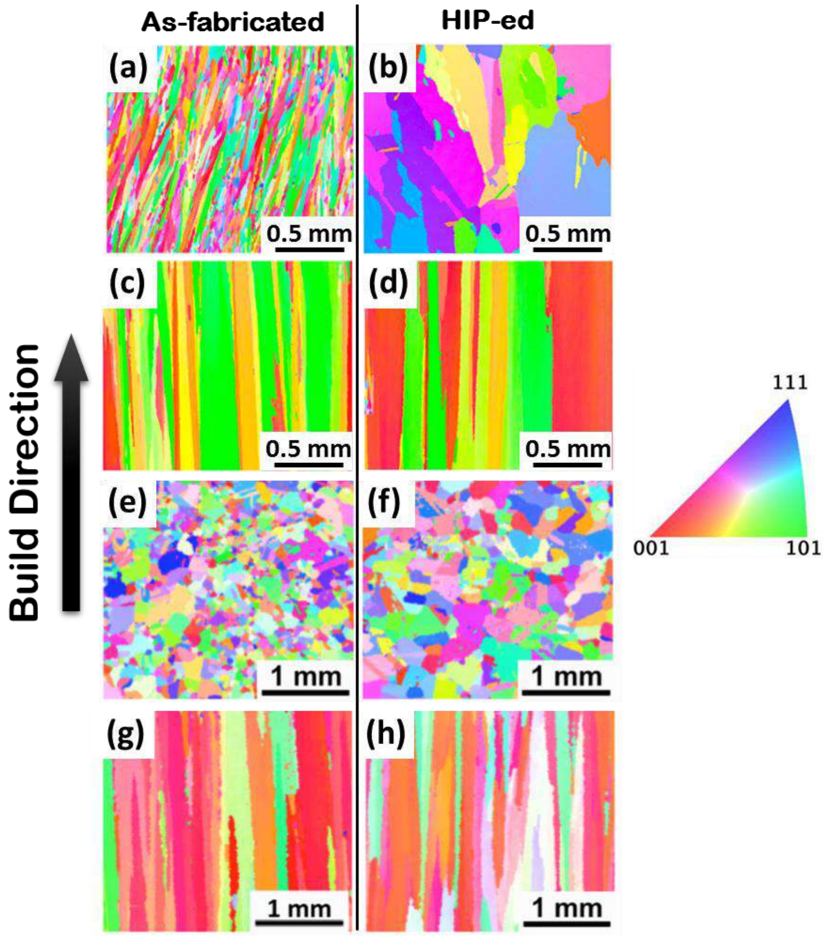


Figure 11- IPFz EBSD scanning maps of the EBM builds exhibiting [left column] different initial microstructures and [right column] their evolution through the application of HIP treatment:

- **(a) and (c) are respectively processed at low temperature (915°C) and high temperature (990°C). The application of [HIP treatment at 1200°C/100 MPa/2 h] on (a) and (c) generates respectively (b) an important grains coarsening and (d) no observable evolution of as-built microstructure, taken from [31]**
- **(e) and (g) are respectively equiaxial material fabricated through the point heat source fill and columnar material fabricated through the standard raster scan strategy. The application of [HIP treatment at 1200°C/120 MPa/4 h + Homogenization 1066°C/1h + Ageing] on (e) and (g) generates respectively (f) an important grains coarsening and (h) no observable modification of as-built microstructure, taken from reference [27].**

5.2.1 Ageing treatment (A)

The ageing treatment is the final part of any alloy Ni718 heat treating sequence as it induces nucleation and growth of strengthening phases, γ'' and γ', which confer to the alloy its high levels of mechanical properties. This treatment does not impact the global structure of the material, ie. average grain size, but lead to intragranular precipitation of a fine distribution of precipitates in the austenitic γ Ni matrix. The available literature related to performed ageing heat treatments on EBM-ed alloy Ni718 deals exclusively with the standard aeronautical heat treatment [32] [28] [33].

5.2.2 Heat treatments sequences

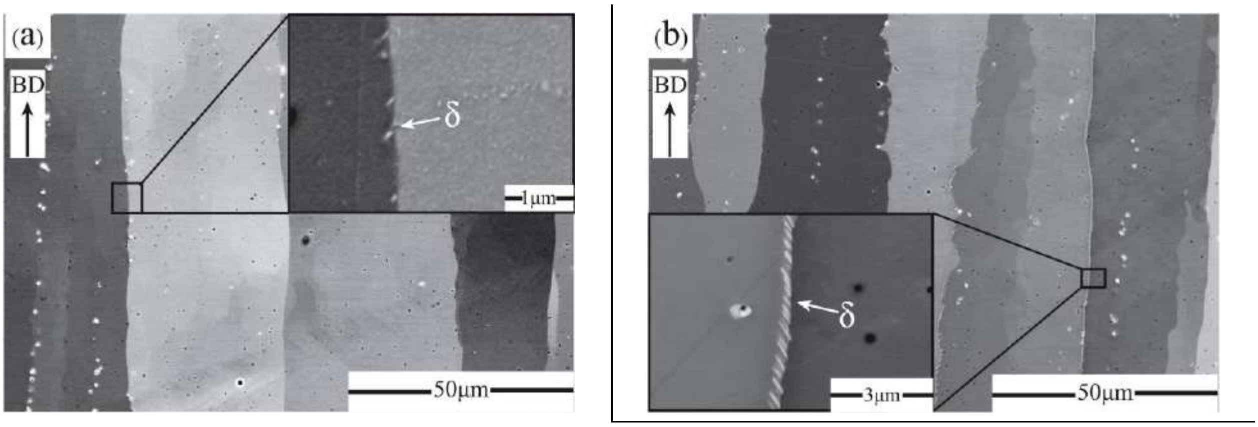
As mentioned earlier, specific heat treatments sequences can be performed so as to fulfil the desired microstructure regarding the produced part service application. These sequences are dependent on the initial microstructure and on the metallurgical state of the alloy, but also on the targeted mechanical properties and resistances to specific types of loading conditions. Hence, as highlighted in this part dedicated to the variety of accessible as-built EBM alloy Ni718 microstructure, processing parameters are likely to generate radically different microstructures, which require adapted post fabrication heat treatments sequences to get the best out of the alloy's potentialities.

Considering the limited available bibliography dealing with EBM alloy Ni718 and post fabrication heat treatments, no "classical" heat treatment sequence can be highlighted as commonly performed. Nevertheless, in the light of collected information on LBM post fabrication heat treatments sequences and on the basis of the microstructure and metallurgical states of EBM as-built material, the general orientations of adapted heat treatments can be foreseen.

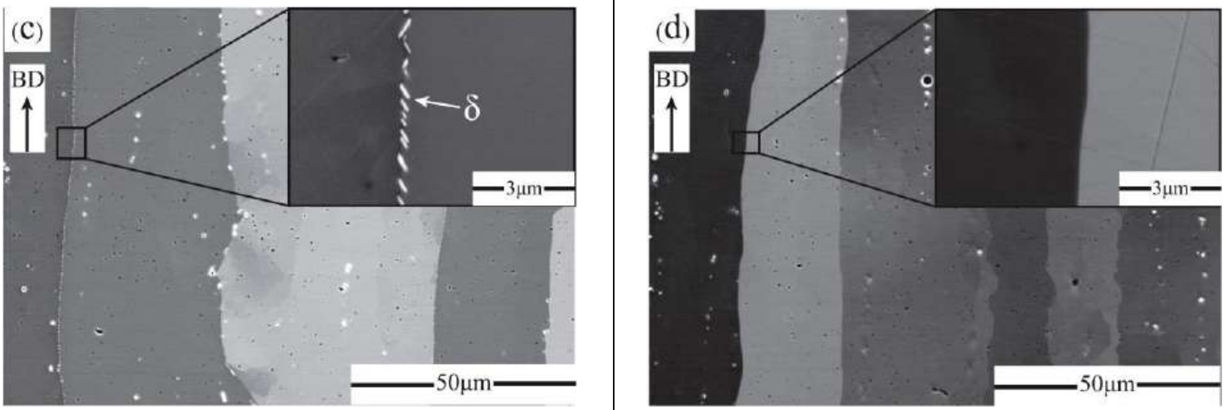
The effect on different solution heat treating temperatures before standard heat treatment are shown through the different pictures of Figure 12. In the considered study, reference [33], the as-built directly aged structure exhibits scarce δ phase precipitation at the grain boundaries of the fabrication inherited columnar structure. The effect of solutionizing at different temperatures can be evidenced by the provided other SEM observations:

- "930°C ST & ageing" induces the fine precipitation of numerous needle-shaped δ precipitates regularly dispersed along the grain boundaries as shown on Figure 12(b),
- "980°C ST & ageing" provides a less homogenous distribution of δ precipitates at grain boundaries and the precipitates are slightly larger as shown on Figure 12 (c),
- "1080°C ST & ageing" solutionizes grain boundaries δ phase Figure 12 (d).

Nevertheless, even if general trends of post fabrication heat treatments can be inferred, their precise effects on mechanical properties and resistances through their microstructural and metallurgical induced modifications is far from being fully understood. A comprehensive study on the effects of specific heat treatment sequences and the way properties are impacted appears to be of relevant interest.



(a)	(b)



(c)	(d)

Figure 12- Precipitates observed at grain boundaries on EBM alloy Ni718 in different metallurgical conditions: (a) As-built and aged, (b) 930°C solution treated and aged, (c) 980°C solution treated and aged, (d) 1080°C solution treated and aged, taken from reference [33]

5.3 Effect of heat treatments sequences on tensile properties

The available literature on alloy Ni718 produced by EBM is very scarce and the resulting information on the effects of post-fabrication heat treatments is rather limited. What should be highlighted is that the preheating temperature has an important impact on the resulting as-built properties, but the provided information in the bibliographic references exhibits serious lack in quantified elements. The following Table 4 gathers the collected data on obtained tensile properties of alloy Ni718 produced by EBM, in the as-built state and after different heat treatment sequences.

An easier to read view of these data is provided in Figure 13 which shows graphical representations of tensile properties alloy Ni718 produced by EBM in the as-built state, Figure 13 (a), and after different temperatures of solution heat treatments, either below or above the δ phase solvus temperature, and ageing, Figure 13 (b). AMS specifications regarding tensile properties for conventionally processed alloy Ni718, are superimposed to these graphical representations. Sampling direction of tested specimen are represented by the presence of hatched dark colour cylinder on each histogram.

Table 4- Short list of room temperature tensile properties of EBM alloy Ni718, applied post heat treatments and sampling direction, as found out from the scarce available literature references [32], [28], [33].

	EBM Device	Known parameters	Stress Relief / Q	Hot Isostatic Pressing / SC	Homo / Q	Solution Treatment / Q	Age step 1	Age step 2	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation to failure (%)
Strondl 2011 [34]	EBM'	Arcam AB - optimized for process stability							H // V	H // V	H // V
			-	-	-	-	-	-	822 // 744	1060 // 929	22 // 5.5
			-	-	-	1000°C/1h/WQ	718°C - 8h	621°C - 8h	1154 // 1187	1238 // 1232	7 // 1.1
Unocic 2014 [29]	EBM'		-	-	-	-	-	-	669	1207	21
			-	1200°C/2h/FC	-	-	-	-	476	765	54,1
			-	1200°C/2h/FC	-	-	720°C - 8h	620°C - 8h	1034	1151	12,5
Deng 2017 [33]	Arcam A2X								H // V	H // V	H // V
						-	-	-	765 // 920	1000 // 1110	40 // 31
						-	720°C - 8h	620°C - 8h	1080 // 1120	1200 // 1265	32 // 22
						930°C/1h/WQ	720°C - 8h	620°C - 8h	940 // 1130	1125 // 1220	14 // 25
						980°C/1h/WQ	720°C - 8h	620°C - 8h	965 // 1100	1090 // 1180	38 // 22
						1080°C/1h/WQ	720°C - 8h	620°C - 8h	930 // 1125	1070 // 1200	35 // 28

The tensile properties of as-built EBM alloy Ni718 specimen provided in Figure 13 (a) clearly shows that whatever the manufacturing parameters, the material is far from being a solid solution due to the high levels of yield strength. It has to be mentioned that the only material exhibiting YS below the AMS specification for solutionized alloy Ni718 has not been tested as-built, but has been submitted to a 1200°C post fabrication heat treatment for 2 hours with relatively slow cooling rate, which probably explains its very low level of tensile properties.

Figure 13 (b) synthetized the tensile properties for alloy Ni718 fabricated by EBM and submitted to different solution heat treatments, ranging from 930°C to 1080°C during 1 hour with final water quenching and then aged following standard aeronautical ageing treatment. From this representation, several elements can be highlighted:

- The vertical specimen systematically exhibit reduced ductility when compared to similar production batch horizontal specimen.
- An important variability in ductility values is shown by scattering of red crosses related to elongation to failure of the produced alloys. And some materials exhibit very low ductility, even below the minimum AMS values.
- The different tested specimens hardly reach the AMS specifications for conventionally processed alloy Ni718 related to UTS, and several have lower yield strength than the minima AMS requirements.

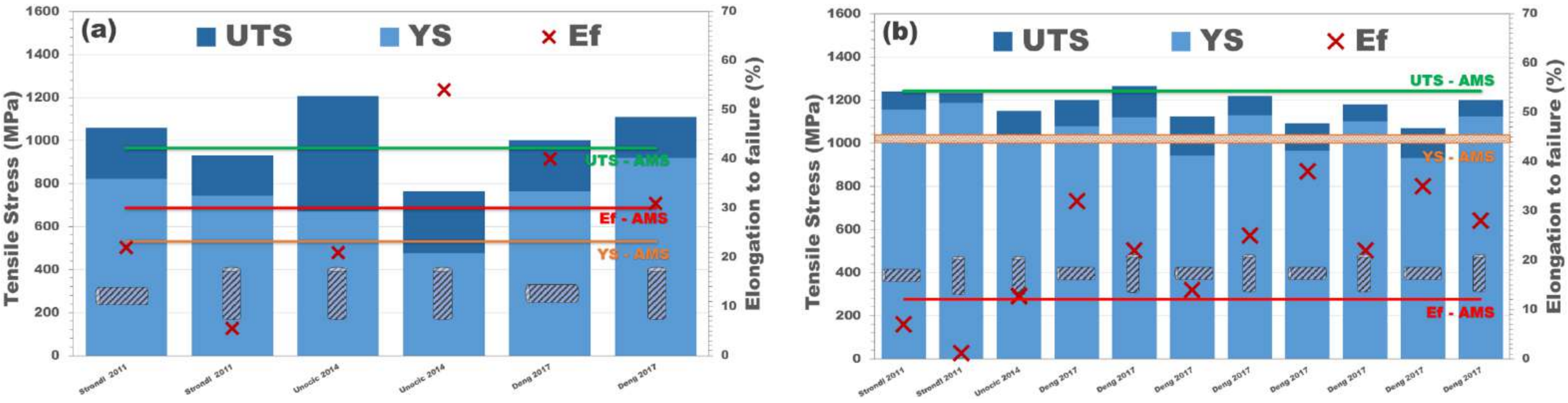


Figure 13- Graphical synthesis of RT tensile properties for different states of EBM-ed alloy 718 presented Table 4, and superimposed specifications of ASTM norms. (a) As-built alloy Ni718 and (b) Solution Treated and Aged alloy Ni718

6 Conclusion

As-built microstructures of powder bed additively manufactured alloy 718, whether it is produced by LBM or EBM, are very different from those generated by conventional processes. The main reason being related to the specific thermal histories the material is subjected to during fabrication processes, including several high cooling rates from melted phase and re-heating following complex cycles.

When considering LBM alloy Ni718, post fabrication heat treatments have extensively been studied and important amount of bibliographic references dealing with their effects on microstructures and resulting mechanical properties are available. Hence, even if initial microstructures can prove to be slightly different as a function of fabrication parameters sets or building strategy, the present document gathers data providing important information on the impacts of different post fabrication heat treatments sequences on room temperature tensile properties.

Nevertheless, several other fields of information are clearly missing regarding the effects of applied post treatments on the material high temperature static properties, its room and high temperatures dynamic properties, as well as its response to long term thermal ageing.

On the contrary, the available information concerning EBM alloy Ni718 is very scarce, and very few are the research teams working on it. This means that the gathered information on EBM alloy Ni718 does not allow to provide a wide scope of microstructures and properties likely to be accessed by this technology, and, hence, does not provide a synthetic overview on the associated optimization latitude. It mainly provides a state of the art approach, highlighting the lack of information and emphasizing the relevancy of developing activities on this insufficiently mature field of research.

On the basis of collected elements from the literature, several main directions for possible PBAM alloy Ni-718 durability optimizations can already be considered of relevant interest to explore. It is obvious that the final heat treatments generating the most suitable properties for a given application will result from a compromise between different guidelines and targets.

Depending on the considered technology and build parameters, a range of available as-built states can be observed – which are always characterized by very fine dendritic structures or fine crystallized structure, with grains either elongated or equiaxed. This induces naturally chemical heterogeneities and segregation magnitudes that are not of the same order than those observed in conventionally processed alloy 718. In all case, the characteristic dimensions of the as-built microstructures are much smaller than those observed in forged or cast alloy 718. Consequently, depending the fabrication parameters, the phases likely to precipitate in the interdendritic areas, like carbides or Laves phase, are also extremely fine if present in the produced material.

These specific characteristics of PBAM-ed alloy 718 lead to emphasize the importance of evaluating the effects of post fabrication heat treatments. Indeed, it has to be mentioned that heat treatments duration showing effectiveness in reducing chemical segregations and/or ensuring specific phases' solutioning at a given temperature are correlated to the characteristic diffusion distances of alloying elements through classical diffusion laws. As mentioned earlier in this document, several characteristic microstructural features of as-built PBAM-ed alloy 718 can be so far as two orders of magnitude smaller than what is observed in conventionally processed alloy 718. This observation lead naturally to consider that the required post fabrication heat treatments durations to homogenize the alloy, to solution or induce precipitation of specific phase are very likely to be different from conventionally processed alloy 718.

When dealing with conventionally processed alloy 718, elaboration steps, thermo-mechanical processes and heat treatments sequences applied to the products, have been subject to decades of improvements and optimizations. This was done through fine analysis of the response of the alloy to applied heat treatments parameters - like heat treatments temperatures, durations, heating and cooling rates - and led to build specific schematic representations highlighting the response of the material to static thermal exposure, Time-

Temperature-Transformation diagram, or to dynamic temperature evolution, Continuous Heating Transformation (CHT) or Continuous Cooling Transformation (CCT) diagrams.

When considering additive manufacturing of alloy 718, it is noteworthy to mention that no transformation diagram, TTT, CHT or CCT, is currently available. This lead PBAM alloy 718 users to rely on old thermal treatments and thermal treatment sequences that did demonstrate effectiveness on products that are totally different from what is presently heat treated.

7 Discussion

The purpose of post-fabrication heat treatments lies in achieving the desired microstructure and metallurgical state to reach the targeted mechanical properties. The specific heat treatments sequences performed on alloy Ni-718 elaborated by Powder Bed Fusion technologies have different main purposes, which are dependent on the considered technology, the as-built material state and on the targeted service properties. The choice of heat treatments sequences likely to optimize the alloy's properties is then of first order interest.

The present approach basically consists in the application of heat treatments sequences directly inspired from what has been developed and optimized on conventionally processed alloy Ni718. Nevertheless, PBF processed alloy 718 proves to be radically different in terms of microstructures and metallurgical sates when comparing to conventionally processed alloy 718. Hence, the relevancy of transposing classical heat treatments on a material which is radically different, and for which elaboration issues and targeted service applications are not the same can legitimately be questioned.

With the aim of proposing relevant orientations towards post-treatments optimizations, improving knowledge on the alloy's initial state and on its response to thermal exposure is mandatory. Furthermore, the physical metallurgy of the alloy and the correlations between microstructure and mechanical properties have to be better understood.

These elements of information are necessary to achieve the purpose of providing adapted and optimized post fabrication heat treatments sequences taking into account the initial state and variability of the material, the targeted mechanical properties and resistance to service loading condition.

The collected bibliographical data and highlighted specific features of PBF processed alloy Ni718 in the framework of ANDDURO project tend to bring out the extent of missing knowledge on number of essential aspects which can be summarized as follows:

- Variability of microstructural and metallurgical throughout a produced part due to local thickness gradient,
- Chemical heterogeneities and their evolution through heat treatments application,
- Effective detrimental effects of undesired phases on the alloy's durability,
- Phases transformations and dissolution kinetics as a function of thermal exposure to build phases transformations diagrams,

Considering these aforementioned fundamental aspects, the development of activities dedicated to produce data and collect information on the response of alloy 718 produced by powder bed additive manufacturing technologies appear is essential. In this view, the activities planned in AnDDurO Work Package 2 aim at providing a further insight in the accessible optimization possibilities regarding post-fabrication heat treatments, either by improving the final material properties through optimized microstructures, or by reducing the required duration of post treatments sequences.

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