Effect of heat treatment on the ductility of Inconel 718 processed by laser powder bed fusion

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Abstract. Inconel 718 is a precipitation-hardening alloy, with a typical heat treatment consisting of solution annealing before a two-stage ageing process. Depending on the heat treatment procedure, strength or ductility can be enhanced, typically at the cost of the other. This study aims to determine a heat treatment procedure suitable for applications that require high ductility. Tensile tests of Inconel 718 processed by laser powder bed fusion additive manufacturing has been carried out on specimens subjected to different heat treatment procedures. The results show that solution annealing above 1010°C followed by a two-stage ageing at 720°C/8 h with furnace cooling to 620°C/2 h and final holding at 620°C/10 h, produce elongation at break of above 30% tested at room temperature and above 24% tested at 650°C.

Keywords: Additive Manufacturing, Powder Bed Fusion, Selective Laser Melting, Inconel 718, Ductility, Elongation at break

1 Introduction

Inconel 718 is a high temperature, high strength, corrosion-resistant nickel base superalloy that has been widely used in aerospace and energy industries [1]. The microstructure consist of a γ matrix, and is preferentially strengthened by precipitation of γ''-Ni3Nb phase [2]. Other phases, such as brittle γ', δ, Laves, and σ phases, will form if the material is exposed to high temperatures for extended periods of time [2]. The formation of these phases is what effectively limits the service temperature of the alloy.

Inconel 718 has proven to be a suitable material for laser powder bed fusion (LPBF) additive manufacturing, with excellent material properties and high relative density [1]. Heat treatment for cast and wrought Inconel 718 has been thoroughly researched and standardized, but the recommended heat treatment for LPBF Inconel 718 in e.g. ASTM F3301-18 refers to SAE AMS2774E, which is developed for conventionally manufactured Inconel 718, and does not take into account the unique properties of LPBF materials. The microstructure of LPBF Inconel 718 is typically fine grained compared to its

cast and wrought counterparts [3], with traces of microsegregation, MC carbides and Laves-phase as a result of the LPBF process [4-8].

Several studies have been conducted to investigate the influence of different heat treatment procedures on the microstructure and mechanical properties of Inconel 718 processed by LPBF. Aydinöz et al. [4] investigated the effect of, amongst other, solution annealing (1000°C/1 h, air cooling) and ageing (720°C/8 h, furnace cool to 621°C/2 h, 621°C/8 h), and reported elongation at break of approximately 10% at room temperature. Chlebus et al. [5] conducted experiments with an identical ageing scheme, but with different annealing temperatures prior to ageing. The reported elongation at break for specimens built parallel to the build direction and solution annealed at 1100°C is 19±2% at room temperature. Hovig et al. [2] reports elongation at break of 5±2% at room temperature for specimens manufactured parallel to the build direction, solution annealed at 980°C/1 h, and aged following the same scheme as Aydinöz et al. and Chlebus et al. Hot isostatic pressing (HIP) at 1160°C prior to ageing increased the elongation at break up to 9±4%. Schneider et al. [9] compared the mechanical properties of 10 different heat treatment variations, with reported elongation at break ranging from 15.44±2.00% to 34.34±1.52% depending on the heat treatment condition. Amongst the aged conditions, stress relief at 1066°C/1.5 h, solution treatment at 954°C/1 h, followed by ageing at 720°C/8 h and 620°C/10 h, produced the highest elongation at break with a reported value of 21.96±0.37%. Common for all the mentioned studies are yield strengths upwards of 1000MPa, and in some cases exceeding 1300MPa.

When conducting tensile tests at elevated temperatures the yield strength, UTS, and elongation at break is reduced when comparted to testing at room temperature. Trosch et al. [3] compared the tensile properties of cast, forged and additively manufactured Inconel 718 tested at room temperature, 450° and 650°C. The material was heat treated with solution treatment at 980°/1h followed by ageing at 720°C/8h and 620°C/8h. The elongation at break dropped from 20.4% at room temperature to 14.2% at 650°C for the LPBF specimens built in parallel to the build direction. The elongation at break of LPBF specimens were reported to drop further than that of forged and casted samples at 650°C compared to room temperature, which is attributed to the presence of δ-phase within the grains.

Zhang et al. [10] conducted a study where three different levels of δ-phase was present in the material. The material was tested at 950°C and the material condition without δ-phase display the highest elongation at break. As the δ-phase content is increased the elongation at break is significantly reduced. By increasing the level of δ-phase from 0% to 3.79%, the elongation at break is reduced by 20%. If the level of δ-phase is further increased from 3.79% to 8.21%, the elongation at break is reduced by an additional 20%. The yield strength and UTS was not as greatly affected by the alteration of δ-phase content.

This study focuses on understanding the mechanisms that influence the elongation at break, in order to increase the ductility while maintaining acceptable strength.

2 Experimental method

Twenty tensile specimens were manufactured out of Inconel 718 powder using an EOS GmbH EOSINT M280. The processing parameters are denoted as In718 Performance 2.1 by the vendor, and argon shielding gas was used with a Grid Nozzle type 2200 5501. The specimen geometry is shown in **Fig. 1**, with the specimen orientation with respect to the build direction indicated.

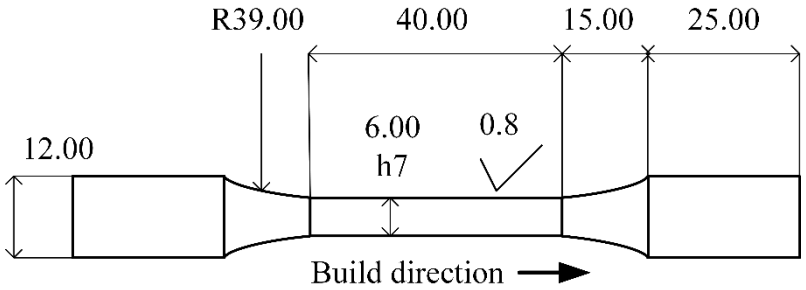

Fig. 1. Specimen geometry. All dimensions in mm, except for roughness (μm).

The chemical composition of the powder feedstock as given by the material vendor is shown in **Table 1**.

Table 1. Chemical composition of the Inconel 718 powder feedstock as supplied by the material vendor.

	Ni	**Cr**	**Nb**	**Mo**	**Ti**	**Al**	**Co**	**Cu**	**C**	**Fe**
wt-%	50-55	17-21	4.75-5.5	2.8-3.3	0.65-1.15	0.2-0.8	<1.0	<0.3	<0.08	Bal.

Prior to machining, eight specimens were stress relieved at 980°C followed by furnace cooling to room temperature. After machining, the specimens were heat treated according to the recommendation by the material supplier (AMS 5664). The recommended heat treatment consists of a solution annealing at 1065°C/1 h followed by air cooling. After solution annealing, precipitation ageing was carried out at 760°C/10 h, followed by furnace cooling to 650°/2 h, where it was held at 650°C/8 h before air cooling to room temperature. This is later referred to as heat treatment condition 1 (HT1).

Twelve specimens were solution annealed and then aged according to AMS 5663 at 720°C/8 h, followed by furnace cooling to 620°C/2 h, and held at 620°C/10 h. Prior to ageing, six specimens were solution annealed at 1010°C/1 h, while the last six were solution annealed at 1065°C/1 h. These are denoted as HT2.a, and HT2.b, respectively.

The tensile specimens were tested in an MTS3 tensile machine with a 100kN load cell at a displacement rate of 0.6 mm/min, at either room temperature, 550°C, 600°C, or 650°C (induction heating).

3 Results and discussion

All the Inconel 718 samples display a microstructure typical for LPBF. The microstructure is cellular, with fine grains in a narrow area close to the laser scan trajectory, and equiaxed grains growing between the laser paths. This complies well with the findings of e.g. Rodgers et al. [11].

In the stress relieved condition, the microstructure is decorated with needle shaped δ-phase in the interdendritic region, with additional δ-phase on the grain boundaries, and Al-oxides distributed across the microstructure, as shown in **Fig. 2 (a)**.

3.1 Heat treatment condition 1

The tensile properties for tensile testing at 550°C and 600°C are tabulated in **Table 2**.

Table 2. Mechnical properties of Inconel 718 tested at 550°C and 600°C, heat treated according to HT1. The values in parenthesis are the reported expected values by the material supplier tested at 650°C [12].

	$R_{p0.2}$ (MPa)	UTS (MPa)	Elongation at break (%)
		550°C	
Mean	1101 (1010 ± 50)	1267 (1210 ± 50)	11.49 (20 ± 3)
Std. Dev.	4.61	9.01	0.96
		600°C	
Mean	1076 (1010 ± 50)	1220 (1210 ± 50)	11.53 (20 ± 3)
Std. Dev.	2.59	9.55	0.33

The tensile strength properties in HT1 are about 30-40 MPa lower when tested at 600°C compared to 550°C. The elongation at break in HT1 did not seem to be affected by the different testing temperatures. The elongation at break compares to reported values in the literature [4], with elongation at break of 11.5%, but the elongation is not satisfactory when compared to the material data sheet provided by the material supplier, which suggests an elongation at break of 20 ± 3% [12]. The material in HT1 was subjected to a stress relief prior to the recommended heat treatment, however. The influence of the stress relief on the microstructure is discussed later in this section.

Fig. 2 (b) shows the microstructure of Inconel 718 after HT1. δ-phase was observed on the grain boundary, an Al-rich spherical phase was observed within the grains, and evidence of Laves-phase was observed on the grain boundaries. Some δ-phase was observed within the grains as well.

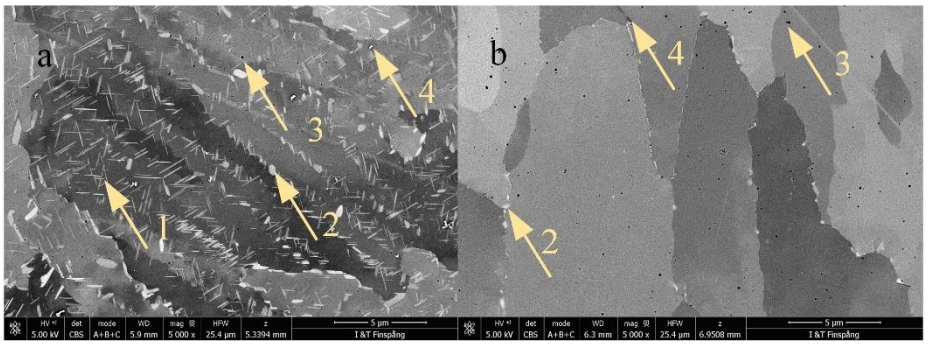

Fig. 2. SEM micrograph of stress relieved (a) and HT1 (b) Inconel 718. The arrows indicate 1) interdendritic δ-phase, 2) grain boundary δ-phase, 3) Al-oxides, and 4) Irregular particles, believed to be either Laves, carbides, or inclusions because of powder impurity or introduced during the sample preparation.

3.2 Heat treatment condition 2

The tensile properties for tensile testing at room temperature and 650°C under argon atmosphere for heat treatment condition HT2.a and HT2.b are tabulated in **Table 3** and **Table 4** respectively.

Table 3. Mechanical properties of Inconel 718 heat treated according to HT2.a tested at room temperature and 650°C.

	$R_{p0.2}$ (MPa)	UTS (MPa)	Elongation at break (%)
		Room temperature	
Mean	1160	1402	32.2
Std. Dev.	10.10	10.10	0.50
		650°C	
Mean	978	1098	25.6
Std. Dev.	0.9	3.0	2.8

Table 4. Mechanical properties of Inconel 718 heat treated according to HT2.b tested at room temperature and 650°C.

	$R_{p0.2}$ (MPa)	UTS (MPa)	Elongation at break (%)
		Room temperature	
Mean	1188	1425	33.0
Std. Dev.	4.5	4.46	0.3
		650°C	
Mean	1047	1164	24.1
Std. Dev.	16.8	7.93	0.5

The elongation at break in HT2.a and HT2.b far exceeds the reported values in the literature [2, 4, 5, 9], while at the same time keeping a satisfactory yield strength. The

heat treatment procedure was based on findings by Schneider et al. [9] in order to maximize the elongation at break. In HT2.a and HT2.b the material was not subjected to a stress relief prior to solution annealing, and the ageing temperature was lower compared to HT1. HT2.b was solution annealed at a slightly higher temperature (1065°C) compared to HT2.a (1010°C). The higher temperature seems to increase both strength and ductility. **Fig. 3** shows the microstructure after HT2.a and HT2.b heat treatments respectively. δ-phase was observed both on the grain boundaries and within the grains. The Al-rich phase is also observed, but no Laves-phase was detected. Solution treatment at a higher temperature reduces the amount of δ-phase observed.

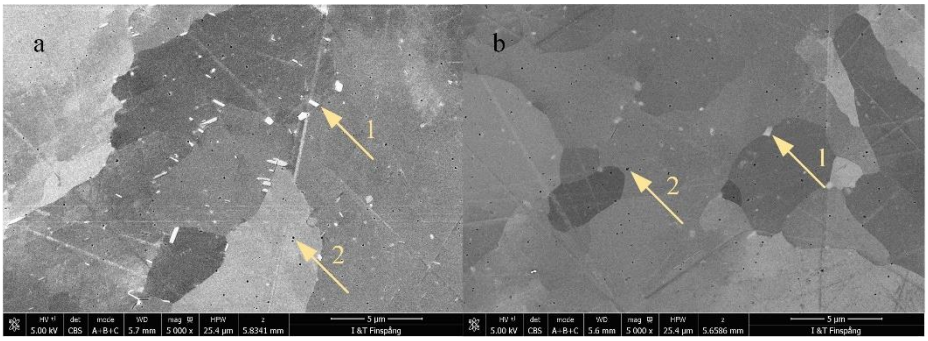

Fig. 3. Microstructure of Inconel 718 with heat treatment HT2.a (a) and HT2.b (b). The arrows indicate 1) δ-phase (white) and 2) Al-oxide with Ni3Nb phase (black dots).

4 Discussion

Based purely on the tensile results in this study, it is apparent that HT2 is superior to HT1 with respect to elongation at break. There is one main difference between the two heat treatment procedures; HT1 was stress relieved prior to solution annealing and ageing.

The elongation at break after HT1 of 11.5% at 550°C and 600°C is comparable to the findings of Aydinöz et al. [4], where the material was solution annealed at 1000°C prior to ageing. Similar results are reported by Hovig et al. [2], where the material was solution annealed at 980°C prior to ageing. In the reported cases from the literature with solution annealing temperatures above 1000°C the elongation at break for tensile specimens built parallel to the build direction in LPBF is in the region of 20% or higher [5, 9]. The elongation at break after HT2.a and HT2.b is 25.6% and 24.1% at 650°C, which compares favourably to previous studies with similar solution annealing temperatures [5, 9].

In an effort to explain why a change in solution annealing temperature can more than double the elongation at break, an isothermal transformation diagram (TTT diagram) is consulted. The TTT diagram for Inconel 718 published in Hovig et al. [2] shows that unwanted phases such as δ and σ, form at temperatures between 650°C and 1000°C. δ-phase can form if the material is held at temperatures between 900°C and 1000°C in excess of one hour. Grain-boundary δ can form after just minutes if held at the same

temperature. In order to form σ-phase the material must be held at temperatures in excess of 660°C for extended periods of time (>1000 hours). The high amount of δ-phase in the stress relieved condition (**Fig. 2 (a)**) is likely a result of the slow cooling rate. Solution treatment at sufficiently high temperature reduces the content of δ-phase [13], which can be seen in the reduction of δ in HT2.b compared to HT2.a (**Fig. 3**).

In other words, when solution annealing (or stress relief) is performed at temperatures below 1000°C unwanted phases such as δ are allowed to form. The same would be the case if the cooling rate from temperatures above 1000°C is not sufficiently high. This is supported by the microscope image in **Fig. 2 (a)**, where a high density of both interdendritic and grain-boundary δ is evident. Most of the interdendritic δ-phase dissolve in the solution annealing that follows the stress relief in HT1, but the microscope analysis still shows traces of δ-phase within the grains (**Fig. 2 (b)**). There is still evidence of δ-phase, especially on the grain boundaries, in HT2 (**Fig. 3**) as well, but to a far lesser extent. Zhang et al. [10] demonstrated how an increased δ-phase content reduces the elongation at break, which supports the findings in this study. In order to increase the elongation at break, the amount of δ-phase, especially within the grains, should be reduced through careful selection of heat treatment parameters.

5 Summary and conclusions

In an effort to increase the ductility of Inconel 718 processed by LPBF the effects of different heat treatment procedures have been investigated. The following conclusions can be drawn from this work:

LPBF Inconel 718 subjected to stress relief or solution annealing at temperatures below 1000°C, or with insufficient cooling rates from higher temperatures, exhibit an unsatisfactory elongation at break. This is attributed to an excessive formation of δ-phase.

The heat treatment denoted HT2.a and HT2.b resulted in elongation at break of 32.2% and 33.0% at room temperature and 26.6% and 24.1% at 650°C respectively. This heat treatment procedure more than doubled the elongation at break at high temperature compared to HT1, while maintaining satisfactory strength.

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