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RESEARCH PAPER

Unveiling the Effect of Annealing Temperature on the Phase Transformations of Inconel 718 Super alloy Manufactured by Additive Manufacturing

Sajad Ghaemifar, Hamed Mirzadeh*

* *hmirzadeh@ut.ac.ir*

School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran

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Abstract: *Phase transformations and the evolution of hardness during elevated-temperature annealing of Inconel 718 superalloy manufactured by the laser powder bed fusion (L-PBF) were investigated. The microstructural evolution, elemental analysis, phase formation, and hardening were characterized by scanning electron microscopy, energy-dispersive spectroscopy, X-ray diffraction, and Vickers indentation test, respectively. It was observed that the effect of annealing treatments is directly governed by the annealing parameters (i.e. time and temperature), for which the hardness measurement as a fruitful and convenient tool can reveal this effect. The increase of the hardness, which was obtained by the annealing (aging) treatments at the temperature range of 800-900°C, indicated precipitation of the Ni_3Nb γ″ strengthening phase; while owing to the coarsening of precipitates as a result of overaging at this temperature range, the hardness decreased. For instance, the length and aspect ratio of precipitates in the aged sample at 800°C for 1 h are 67.14 nm and 0.32, respectively; while these values in the aged sample at 800°C for 8 h are 78.34 nm and 0.44, respectively. On the other hand, the decrease of the hardness at temperatures of 950 and 1000°C was attributed to the decrease of dislocation density in conjunction with the Ni_2Nb Laves phase dissolution. Hence, it is crucial to determine the annealing parameters according to the required microstructure and properties.*

Keywords: *Nickel-based superalloys, Additive manufacturing, Aging heat treatment, Homogenization treatment, Precipitation strengthening.*

1. INTRODUCTION

Ni-based superalloys are well-known engineering materials, which are widely used in various industries such as petroleum, energy generation, and aerospace [1, 2]. The Inconel 718 (IN718) superalloy is one of the most utilized alloys in this category, which is known as an age-hardening superalloy with favorable weldability [3-5].

Owing to the good weldability of IN718, the additive manufacturing (AM) process is a convenient method for the fabrication of IN718 parts with complex geometry [6, 7]. Laser powder bed fusion (L-PBF) is one of the most important AM methods for manufacturing metallic parts [8-13]. Recently, the fabrication of IN718 parts using the L-PBF method has been reported in many studies [14, 15]. Furthermore, performing the post-heat treatment is crucial to obtain favorable properties in the AM parts [16, 17].

Various annealing treatments as the post-processing of the IN718 manufactured by the L-PBF method have been reported in recent studies, including homogenization [18, 19] and aging treatment [20, 21]. It has been reported that these heat treatments might result in the potential phase transformation such as Laves phase dissolution as well as the precipitation of γ′, γ″, and δ phases, which also depends on the temperature and time of the heat treatment process. The main strengthening phases are γ′and γ″ phases; while the growth of the δ phase occurs at the expense of γ" phase, leading to the deterioration of the precipitation hardening effect, and generally impairing the creep resistance [22, 23]. Moreover, the importance of the time and temperature and their effect on the kinetics of the phase transformation has been studied in recent studies on the IN718 fabricated by cast [24], wrought [25], and L-PBF [18] processes. For instance, the activation energy for the dissolution of Laves phase in a cast sample was determined by the JMAK model as ~274.5 kJ/mol [24]; while this activation energy in an L-PBF sample was determined as ~160 kJ/mole [18], which shows the effect of the manufacturing process. Furthermore, it has been reported that utilizing hardness measurements is a useful tool to trace the evolution of phase transformations [26, 27].

Although many investigations have been performed on annealing at a specific temperature range, the effect of annealing at a wide

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temperature range to reveal its effect on the phase transformation and hardness values are not scrutinized yet and needs systematic investigation. Hence, the current study is dedicated to achieving these aims by determining the annealing parameters according to desirable microstructure and properties.

2. EXPERIMENTAL PROCEDURES

The L-PBF process was performed by using IN718 powder (EOS NickelAlloy IN718, EOS Company, Germany) with particle diameters in the range of 15-50 µm (Fig. 1a) and with adopting a commercial LPBF machine (SLM machine, ST). The process was performed by the above machine, equipped with a 100 W Nd-YAG laser, with a laser spot diameter of 50 µm. The protective atmosphere was argon, and the oxygen content inside the chamber was kept below 0.2%. Moreover, the process parameters are as follows: laser power of 95 W, scanning speed of 800 mm/s, hatching distance of 0.05 mm, layer thickness of 0.025 mm, and scanning strategy of stripe/67°. The annealing treatment was performed at the temperature range of 800-1000°C for a duration of 0.5 to 8 h. The samples were etched with the waterless Kaling's 2 reagents. Afterwards, a field-emission scanning electron microscope (MIRA3 TESCAN FESEM) was utilized for microstructural analysis. Image analysis was performed by the ImageJ software version 1.54 on the FESEM images. The Vickers indentation test (Wilson Tukon 1202 hardness tester) was utilized for the hardness measurements with a load of 5 kg, for which at least five indentations were averaged. It is noteworthy to state that for each heat treatment condition, 3 samples were prepared and the hardness testing was performed on them to check the reproducibility of the results and avoid any potential bias. X-ray diffraction (using a PHILIPS diffractometer with Cu-ka radiation) was performed for phase identification using the scan rate of 2°/min.

3. RESULTS AND DISCUSSION

3.1. As-built Microstructure

The FESEM image of the as-built sample is shown in Fig. 1b. It can be seen that the microsegregation (occurring during solidification [28, 29]) resulted in the formation of Laves phase (bright regions labelled as S2), which is consistent with the previous studies [16-18]. In other words, the microstructure of the as-built sample consists of the austenite phase (dark regions labelled as S1) and the mentioned phase. Furthermore, adopting the EDS analyses, Table 1 shows that the leaves phase is in the form of Ni_2Nb due to the atomic ratio of Ni/Nb ~1.99. The aforesaid form of the Laves phase has been reported in previous studies [30, 31].

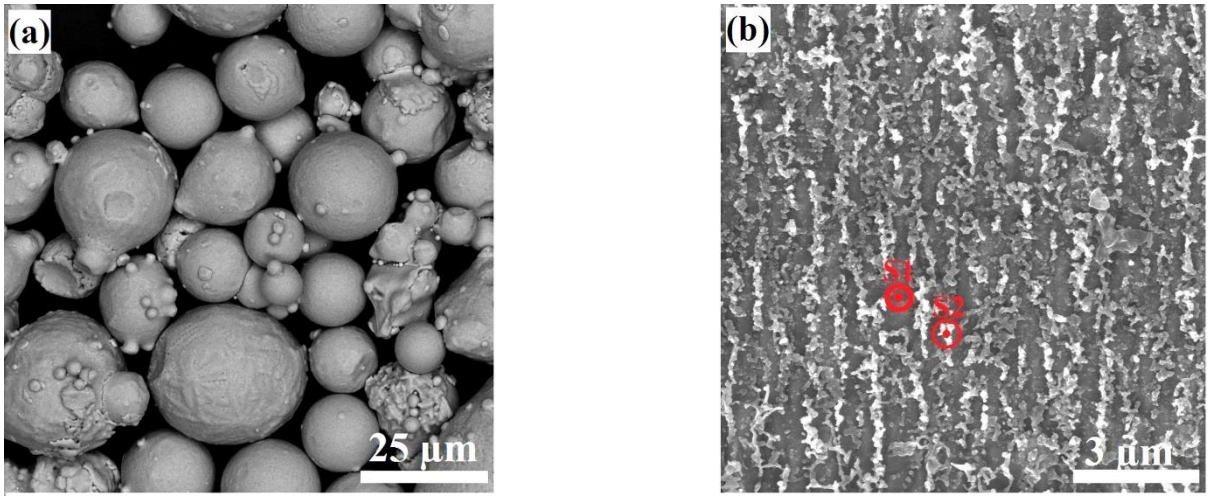

Fig. 1. (a) Atomized powder particles and (b) microstructure of the as-built sample.

Table 1. EDS results representing the elemental analysis (at %) of spots shown in Fig. 1

Analyzed Spot	Ni	Cr	Nb	Mo	Ti	Al	Fe
Spot 1 (Austenite matrix)	54.02	20.95	2.98	2.37	1.03	0.63	18.02
Spot 2 (Laves phase)	32.94	23.45	16.54	1.04	1.26	8.98	15.79

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3.2. Evolutions of the Hardness and Microstructure during Annealing

Fig. 2 shows the evolution of the hardness during annealing treatment at 800-1000°C. The results are also summarized in Table 2, for which the assigned name to the sample follows the pattern of Ax-y, where x and y represent the annealing temperature (°C) and time (h), respectively. Vividly, the variation of hardness values during annealing is directly related to the annealing temperature: At the temperature range of 800-900°C, the hardness increased during annealing; while at the temperatures of 950 and 1000°C, the reverse behavior can be seen. Therefore, two approximate temperature ranges of T< 950°C and T ≥950°C can be considered, as will be discussed in the following sections.

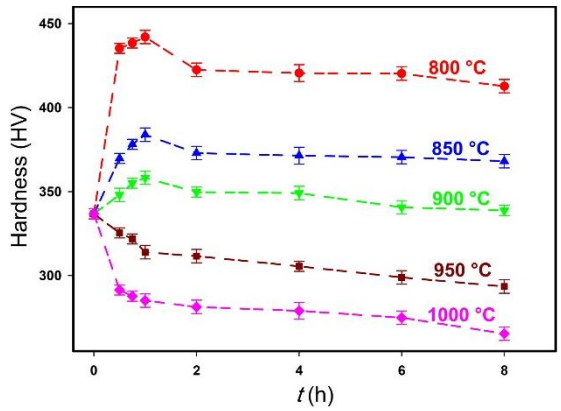

Fig. 2. Evolution of hardness during annealing at different temperatures.

3.3. Microstructural Evolution during Annealing at T< 950°C

As mentioned above, annealing at this temperature range leads to a significant increment of hardness, especially at the lower temperature of 800°C. Moreover, after reaching the peak hardness value, it decreases at long holding times. To find the reason behind these observations, a microstructural analysis should be performed. The microstructure of the A800-1 and A800-8 samples are shown in Fig. 3. It can be seen that along with the Laves phase with decreasing amount, new disk-shaped precipitates can be seen. Conducting EDS analysis on the observed disk-shaped precipitates shows that the atomic ratio of Ni/Nb in this phase is ~3.16 (Table 3), which is comparable to the ratio of 3 based on the stoichiometry of the γ″ phase (Ni_3Nb). It should be noted that the mentioned phase is reported in previous studies as the main precipitating phase during the aging of the IN718 superalloy processed by conventional methods [25, 32]. As shown in Fig. 4, the XRD pattern of the A800-1 sample confirms the precipitation of γ″ phase. Since the aging process is directly performed on the as-built sample, the Laves phase remained in the microstructure as confirmed by EDS analysis (Table 3). It has been stated that conducting aging treatment in the range of 650-900°C results in the precipitation of strengthening phases which leads to an increase in hardness [26, 33, 34]. Hence, the formation of these precipitates led to higher hardness values (for the annealing in the temperature range of 800-900°C) in comparison with the as-built sample. It can also be seen that the incremental behavior of the hardness values is sharply obtained at the first hour of aging treatment by reaching the peak hardness; while a longer aging time resulted in a decrease in hardness due to the occurrence of overaging, which is the consequence of the precipitate coarsening.

Table 2. Hardness values and the assigned names of different samples.

Sample Name	Hardness (HV)	Sample Name	Hardness (HV)	Sample Name	Hardness (HV)	Sample Name	Hardness (HV)	Sample Name	Hardness (HV)
A800-0.5	435.2 ± 2	A850-0.5	369.7 ± 2	A900-0.5	348 ± 3	A950-0.5	325.3 ± 2	A1000-0.5	291.3 ± 2
A800-0.75	438.5 ± 2	A850-0.75	378.1 ± 2	A900-0.75	355 ± 2	A950-0.75	321.7 ± 2	A1000-0.75	287.6 ± 2
A800-1	442.1 ± 3	A850-1	383.8 ± 2	A900-1	358.2 ± 3	A950-1	313.8 ± 2	A1000-1	285.1 ± 3
A800-2	422.4 ± 3	A850-2	372.9 ± 3	A900-2	349.6 ± 2	A950-2	311.5 ± 3	A1000-2	281.2 ± 3
A800-4	420.5 ± 4	A850-4	371.4 ± 4	A900-4	349.1 ± 3	A950-4	305.4 ± 3	A1000-4	278.9 ± 4
A800-6	420.3 ± 3	A850-6	370.5 ± 3	A900-6	340.5 ± 3	A950-6	298.8 ± 4	A1000-6	274.8 ± 3
A800-8	412.7 ± 3	A850-8	368.1 ± 3	A900-8	338.7 ± 2	A950-8	293.4 ± 3	A1000-8	265.3 ± 3

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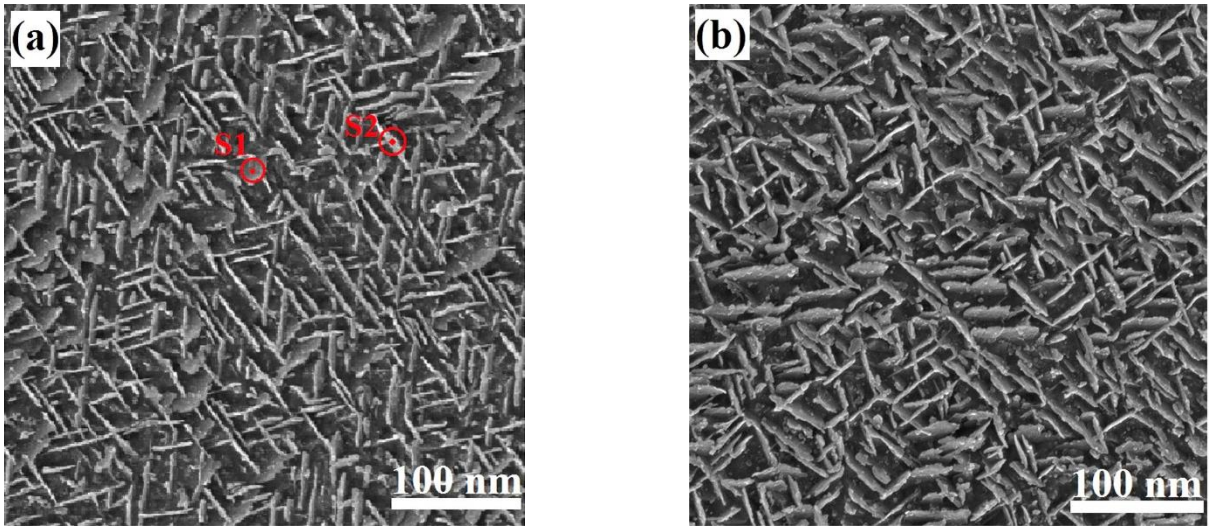

Fig. 3. Microstructure of the a) A800-1 and b) A800-8 samples.

Table 3. EDS results representing the elemental analysis (at %) of spots shown in Fig. 3

Analyzed Spot	Ni	Cr	Nb	Mo	Ti	Al	Fe
Spot 1 (Laves phase)	32.12	23.56	16.33	1.13	1.32	9.11	16.43
Spot 2 (gamma double prime)	36.08	22.86	11.42	1.64	1.41	10.06	16.53

In other words, the length and aspect ratio of precipitates in the A800-1 sample (peak aged sample, Fig. 3a) is 67.14 nm and 0.32, respectively; while these values in the A800-8 sample (overaged sample, Fig. 3b) is 78.34 nm and 0.44, respectively.

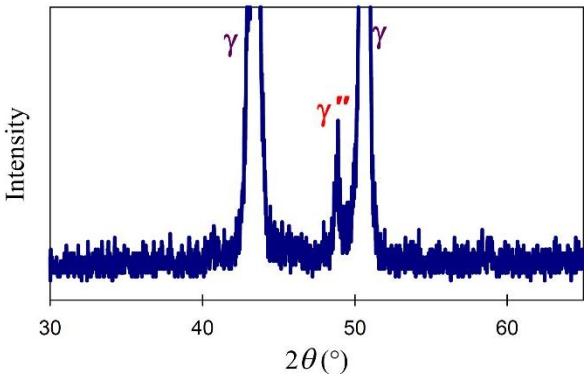

Fig. 4. XRD patterns of the A800-1 sample.

3.4. Microstructural Evolution during Annealing at T ≥950°C

As shown in Fig. 2, annealing at the temperatures of 950 and 1000°C led to a decrease in the hardness values, which implies that precipitation of the strengthening phases has not occurred. Moreover, it has been reported that performing annealing at the temperature range of 950-1250°C leads to the dissolution of the Laves phase [35, 36].

Fig. 5 shows the microstructure of the A1000-1 sample. It can be seen that the Laves phase is completely dissolved and a new phase has been precipitated.

The EDS analysis of this precipitate is shown in Table 4, which reveals the presence of a high amount of C and Nb atoms in this phase. Therefore, it can be inferred that the precipitated phase is the niobium carbide (NbC).

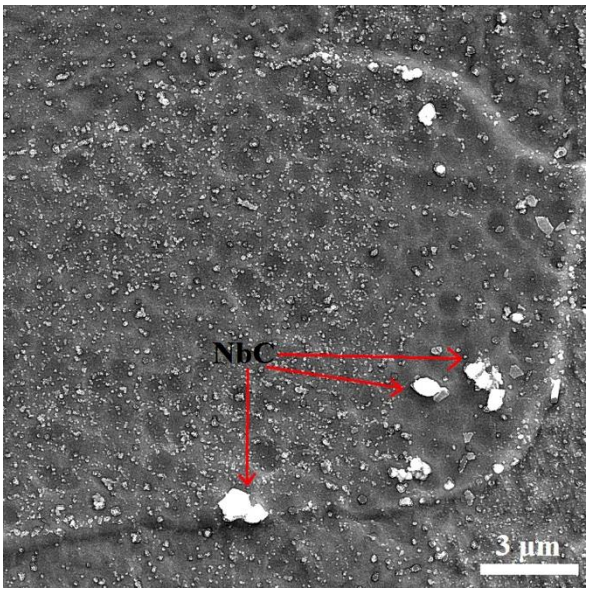

Fig. 5. Microstructure of the A1000-1 sample.

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Table 4. EDS results representing the elemental analysis (at %) of niobium carbide (NbC)

Analyzed Spot	Ni	Cr	Nb	Mo	Ti	Al	Fe	C
Niobium carbide (NBC)	17.91	1.89	22.13	0.61	5.03	1.28	1.28	49.87

The precipitation of the NbC during homogenization heat treatments in additively manufactured IN718 superalloy has been reported previously, while it is noteworthy to state that the volume fraction of the precipitated NbC (~0.4 %) is much less than values that can affect the hardness values [15, 37]. Moreover, it has been reported that the homogenization heat treatment, as a post-processing treatment, leads to a decrease in the initial dislocation density, which exists in the as-built sample owing to the fast solidification during the AM process [38-40]. Hence, it can be concluded that the decrease in the dislocation density along with the dissolution of the Laves phase resulted in the decrement in hardness values during annealing at temperatures of 950 and 1000°C.

In conclusion, it can be stated that the effect of annealing treatments is directly governed by the annealing parameters (i.e. time and temperature), for which the hardness measurement as a fruitful and convenient tool can reveal this effect. In other words, the increase of the hardness, which was obtained by the annealing (aging) treatments at the temperature range of 800-900°C, indicates precipitation of strengthening phases; while the decrease of the hardness is attributed to the decrease of dislocation density in conjunction with the Laves phase dissolution. Hence, it is crucial to determine the annealing parameters according to the required microstructure and properties.

4. CONCLUSIONS

The effects of annealing temperature on the phase transformation of Inconel 718 superalloy manufactured by additive manufacturing were investigated. The following conclusions can be drawn:

1) The microstructure of the as-built sample was composed of Laves phase in the austenite matrix. The stoichiometry of the Laves phase was in the form of Ni_2Nb due to the atomic ratio of Ni/Nb ~1.99.
2) The effect of annealing treatments is directly governed by the annealing temperature and time, for which the hardness measurement as a fruitful and convenient tool can reveal this effect. Two approximate temperature ranges of T< 950°C (associated with the increment of hardness) and T ≥950°C (associated with the decrement of hardness) were considered.
3) The increase of the hardness, which was obtained by the annealing (aging) treatments at the temperature range of 800-900°C, indicated precipitation of the Ni_3Nb γ'' strengthening phase. Moreover, after reaching the peak aged condition, overaging led to a decrement in hardness. In other words, as the annealing time increased, the size of the strengthening precipitates increased.
4) The decrease of the hardness at temperatures of 950 and 1000°C was attributed to the decrease of dislocation density in conjunction with the Laves phase dissolution.

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