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ARTICLE

Effects of Heat Treatment on Microstructure and Mechanical Properties of Selective Laser Melting IN718

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Abstract: Selective laser melting (SLM) is a laser cladding/deposition based technology, which can fabricate and repair near-net-shape high-performance components directly from metal powders. Nickel-based superalloys such as IN718 are the most commonly used metal materials in aircraft engine high-performance components. The pre-alloyed and rapidly solidified IN718 powder was used as a precursor for the production of additive manufactured components using SLM. The laser deposition process was optimized through a set of designed experiments to reduce the porosity. The microstructure and mechanical properties of laser-deposited IN718 were studied and compared under the heat treatment conditions of as-deposition, direct aging (DA), solution treatment and aging (STA), and full homogenization followed by STA. Tensile test results show that the direct aging treatment produces the highest tensile strength for the wrought material, while the homogenization followed by STA treatment exhibits good ductility. Failure modes of the tensile samples were analyzed by fractography. Considering the room temperature and high temperature tensile test results of the three heat treatments, the homogenized STA samples not only have higher strength than the AMS wrought specifications, but also have good plasticity. Therefore, homogenized STA is a suitable heat treatment method for LSM IN718 alloy. Then, stress rupture properties at 650 °C/700 MPa and 650 °C/725 MPa, and low cycle fatigue properties at 455 °C of the homogenized STA samples were investigated, and the fracture mode was analyzed and compared to that of the wrought IN718 alloy.

Key words: IN718; heat treatment; SLM; microstructure; mechanical properties.

Selective laser melting (SLM), also referred as laser engineered net shaping (LENS), direct light fabrication (DLF), etc., is a promising technology based on a new additive manufacturing principle, which combines laser cladding with rapid prototyping into a solid freeform fabrication process for manufacturing fully dense metallic parts with high performance. During SLM, by moving the laser beam and CNC working table to generate certain trajectories, the fine metal powders are laser deposited onto the substrate to fabricate three-dimensional components layer by layer in near-net shape without die, thus saving delivery time and manufacturing cost [1-5]. One of the important applications of the SLM technology is to provide an economic and flexible method for fabricating and repairing high-performance components in aircraft engines, namely blisk blades, compressor blades, and turbine components [6-9].

IN718 has been the most widely used nickel-based superalloy in the aircraft engine industry over the past 40 years. IN718 was designed to retain high strength, high creep resistance, and good fatigue life at high temperature up to 650 °C [10-12]. It can be strengthened by precipitating γ'' (Ni_3Nb) and γ' ($Ni_3(Al,Ti)$) phases in the γ matrix at normal volume fractions of approximately 16vol% and 4vol%, respectively, after a full heat treatment[4,5]. IN718 has good weld ability due to its relatively slow precipitation strengthening kinetics; however, the solidification process of cast or welded IN718 is often associated with the segregation of high concentration refractory elements, such as Nb and Mo [6]. As a result, a Nb-rich brittle intermetallic compound called Laves phase, represented as (Ni, Cr,

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Fe)$_2$(Nb, Mo, Ti), often forms in the interdendritic regions. Laves phase is detrimental to the material tensile ductility, fatigue, and creep rupture properties, as it depletes the principle elements needed for precipitation strengthening and aids in easy crack initiation and propagation[13,14].

Previous literatures have reported the tensile properties of laser-deposited IN718 (typically after solution and precipitation heat treatment) and claimed that its properties are equivalent to or superior to the wrought properties[6,15-18]. This is due to the fine grain size and small dendrite arm spacing as the results of the rapid solidification rate associated with laser deposition. However, research direction of these literatures is restricted to the tensile properties, and does not address the important rupture stress and fatigue properties for long-term service in high temperature environments.

In the present study, the pre-alloyed and rapidly solidified IN718 powder serves as a precursor for the production of additive manufactured components using selective laser melting (SLM). Consequently, different process parameters produce cooling variations, which can affect the microstructure and mechanical properties. This study represents the comprehensive microstructure and mechanical behavior characterization using optical metallography (OM), scanning electron microscopy (SEM), electron probe microanalysis (EPMA) and electron backscattered diffraction (EBSD). The mechanical properties (room-temperature and high-temperature tensile, stress rupture, low cycle fatigue) of as-fabricated and heat-treated components were also investigated.

1 Experiment

The precursor and pre-alloyed atomized IN718 powder has the composition of 53.5Ni-19Cr-5Nb-3Mo-1Ti-0.43Al. The average powder particle size is 50~80 μm. LSM IN718 samples were represented in the EOS M270 SLM system which used a 200 W Yb:YAG fiber laser. Cylindrical components with a size of 1.3 cm (diameter)×6.7 cm (length) were fabricated in both the vertical and horizontal build orientations, and the cylinder axis is perpendicular to the beam direction. The 100 μm diameter laser beam was used to scan at 800 mm/s in either argon or nitrogen gas environments surrounding the building components. The build platform was preheated to 80 °C and maintained at this temperature. The formed Re-coater and melted layers were alternately scanned along the *x*- or *y*-axis. The as-fabricated cylindrical specimens were performed in argon for 4 h.

Post weld heat treatment is necessary to relieve the residual stresses and enables the precipitation of strengthening phases. Microstructures from a laser deposition process are usually different from those from the conventional cast and wrought processes due to the inherent rapid solidification rate associated with laser deposition. It is necessary to investigate how the industrial standard heat treatment methods (usually used for cast and wrought IN718) affect the laser-deposited microstructures and the eventual mechanical properties of the material. In this study, three heat treatment methods conforming to Aerospace Material Specifications (AMS) were used to evaluate the laser-deposited IN718 samples, namely direct aging, solution treatment and aging (STA), and homogenization followed by STA. Table 1 presents the detailed conditions for each heat treatment method used in this study.

SLM IN718 samples for microstructure observation were machined by wire electrolytic-discharge machine. The microstructure of the material was revealed using an etchant of a mixture of H_2O, HCl and H_2O_2 with the volume ration of 4:2:1. The microstructure was examined by optical microscope. The fracture surface was characterized by scanning electron microstructure (SEM) to correlate the fracture characteristics with microstructure and properties. Electron probe microanalysis (EPMA) was also utilized to characterize the chemical composition of the SLM IN718.

2 Results and Discussion

2.1 Microstructure

IN718 alloy is a Ni-Fe-based alloy that is extensively applied in structural applications up to approximately 650 °C. A special characteristic of the alloy is that, in its wrought form, it normally exhibits three intermetallic precipitation phases, namely, γ' having a composition of $Ni_3(Al,Ti)$ and cubic ($L1_2$) crystal structure, γ'' having a composition of Ni_3Nb and bct ($L0_{22}$) crystal structure, and δ having a composition of Ni_3Nb and orthorhombic ($D0_a$) crystal structure. The strengthening phase in IN718 is γ''-Ni_3Nb appearing as disc-shape. A small amount of γ' phase $Ni_3(Al,Ti)$ with fine spherical particles dispersed in IN718[10-12]. Because of the morphology and size of δ phase, it does not significantly improve the hardening of the alloy. The presence of the δ

Table 1 Three heat treatment methods for laser-deposited IN718

Step	Process
1 homogenization	Heating to 1080 °C, holding for 1 h, air cool
2 solution	Heating to 960 °C, holding for 1 h, air cool
3 aging	Heating to 720 °C, holding for 8 h, furnace cooling to 620 °C, holding for 8 h, air cool

* According to AMS-5383D for cast IN718 and AMS-5662M for forged IN718; homogenized STA (steps 1+2+3); STA (steps 2+3); direct aging (step 3)

phase implies the loss of hardenability due to the depletion of γ'' phase. However, its presence has certain beneficial effects. For example, δ phase with appropriate volume fractions can inhibit grain growth during solution treatment, and δ phase with an appropriate morphology at grain boundary has been shown to provide resistance to grain boundary creep fracture [19].

Fig.1 demonstrates the microstructures of the transverse section of the as-deposited IN718 sample. Staggered individual deposit layers can be observed from the low-magnification microstructure. The deposit layers are delineated by bands of thin remelted nucleation zones formed at the layer interface. Extensively dendritic grains that grow across a number of deposit layers can also be seen occasionally. A high-magnification SEM micrograph in Fig.1b shows the detailed microstructure of the as-deposited material, where fine secondary dendrites are formed within a grain with an average dendrite arm spacing around 1 μm. In contrast to the dark austenite matrix, small white particles in globular and irregular shapes precipitated along the interdendritic boundaries. They are identified as the Laves phase and some minor MC and TiN phases that are segregated during the rapid solidification process. Fig.2 shows the EPMA analysis of the major phase appearing in the as-deposited microstructure. It can be observed that the matrix of the dendrite's core area is rich in Fe, Cr and Ni, while the white segregation particles are rich in Nb, Mo and Ti, which are the major composition elements of the Laves phase.

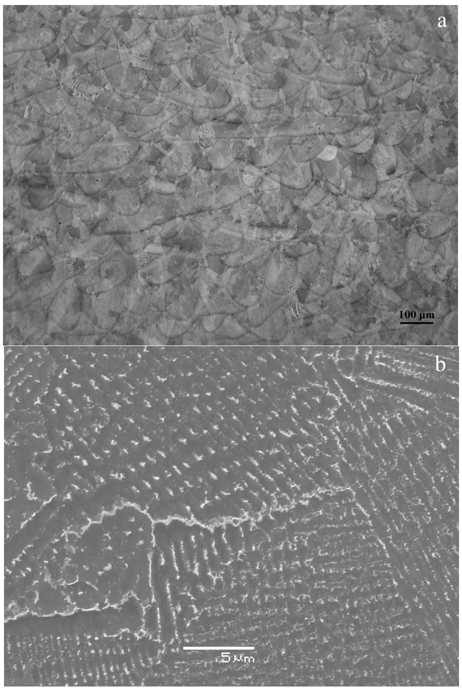

Fig.1 Microstructures of transverse cross section of the as-deposited IN718 sample: (a) OM and (b) SEM

Fig.3 indicates the microstructure of the transverse section of the as-solutioned IN718 sample. As can be seen, most Laves phase are dissolved into austenite matrix though 1080 °C high-temperature solution. Small white particles in globular and irregular shapes precipitate along the interdendritic boundaries. They are identified as some minor MC and TiN phases. Compared to the Φ508 mm industrial cast ingot of IN718 alloy, solution temperature of Laves phases is decreased to 80 °C, because the Nb, Mo, Ti, Cr etc elements are easily diffused in the fine dendrites. Fig.4 shows the EPMA analysis of the major phase appearing in the as-solutioned microstructure. It can be observed that the grain area rich in Fe, Cr and Ni is austenite matrix, while the small white particles rich in Nb and C at grain boundary are MC carbide. Compared to the Φ508 mm industrial cast ingot of IN718 alloy, the size of MC carbide is very small, less than 1 μm, because of the SLM rapid solidification process.

Fig.5 indicates the microstructure after homogenization at 1080 °C and solid solution at 960 °C. The high temperature of homogenization results in considerable grain coarsening. The average grain size is up to 10~20 μm. Most Laves phases dissolve into the matrix. The primary MC phase, which appears in the white small particles, is a relatively stable phase and can be found in the homogenized STA samples. Additionally, most lamellar δ phases precipitate along grain boundaries. Although the δ phase is not a strengthening phase, it can inhibit grain growth and avoid notch sensitivity.

2.2 Tensile properties

The average ultimate strength, yield strength, and plastic elongation of the room temperature tensile tests are shown in Table 2. These results under different heat treatment conditions were compared to the minimum properties from the AMS for cast and wrought IN718. The as-deposited material produces low yield strength (641 MPa) and ultimate strength (962 MPa) but relatively high plastic elongation (35%). After direct-aging heat treatment, the ultimate strength increased to 1431 MPa, while the yield strength reached 1283 MPa. However, the plastic elongation of the direct-aged material compared with the as-deposited material dropped significantly from 35% to 12.5%. The direct-aged elongation value is still above the wrought AMS property. The STA treated material has a slightly higher tensile strength (ultimate strength 1374 MPa) than the wrought material, but it produced a much better plastic elongation of 17% compared to the direct-aged material. The material that underwent homogenization treatment produced a slightly lower tensile strength (ultimate strength 1348 MPa) but had an even better elongation (17.5%) than the STA-treated material. In all cases, the heat-treated materials exhibited better tensile properties than the AMS casting properties, but they

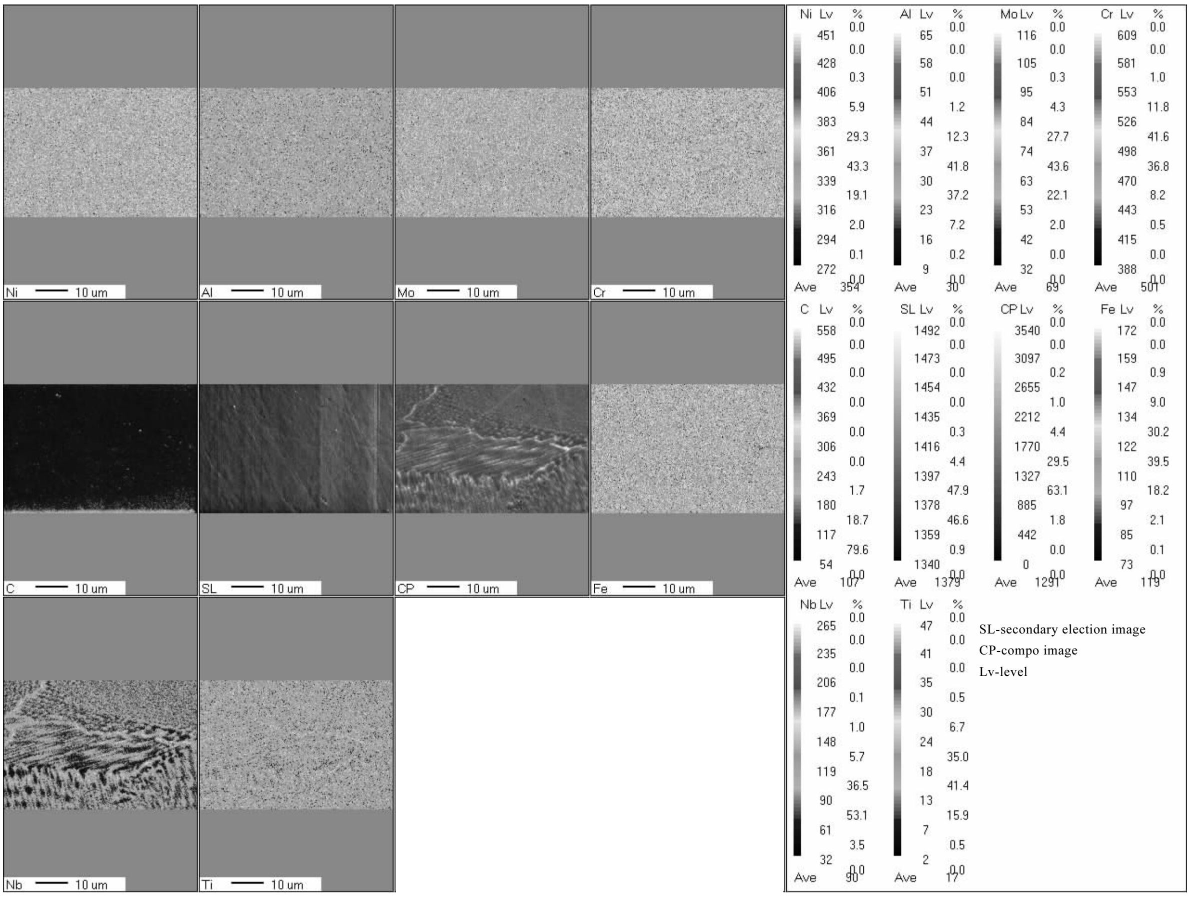

Fig.2 EPMA analysis of the transverse cross section of the as-deposited IN718 sample

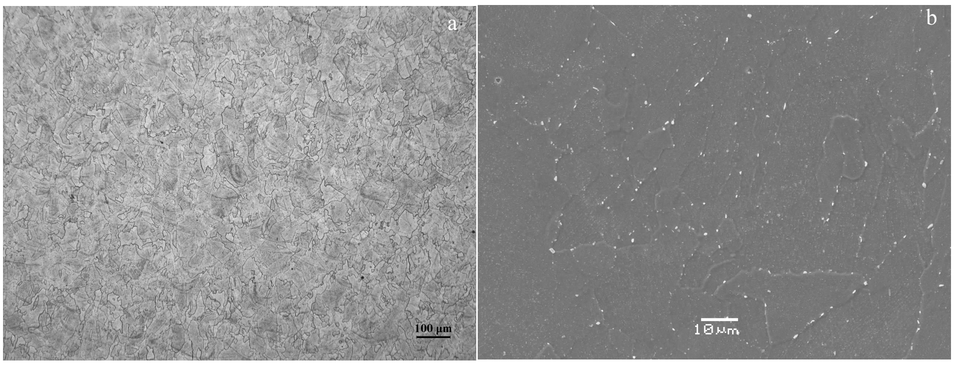

Fig.3 Microstructures of the transverse cross section of the as-homogenized IN718 sample: (a) OM and (b) SEM

only approached the wrought AMS properties. Direct-aged material's tensile stress results were comparable to that of the wrought alloy, but they showed an inferior ductility. The STA and homogenized STA treatments generated good ductility, which exceeded the minimum wrought elongation, but their tensile strength values were slightly higher than the AMS of wrought properties.

The average ultimate strength, yield strength, and plastic elongation of tensile test at a high temperature of 650 °C are shown in Table 3. These results under different heat treatment conditions were compared with the minimum properties from the AMS for wrought IN718. After direct-aging heat treatment, the ultimate strength increased to 1190 MPa, while the yield strength almost reached 1050 MPa. However, the plastic elongation of the direct-aged material is 21%. The STA treatment condition caused a higher tensile strength (ultimate strength 1150 MPa) than the wrought material, while it produced a much better plastic elongation of 22% compared to the direct-aging condition. The material that underwent homogenization

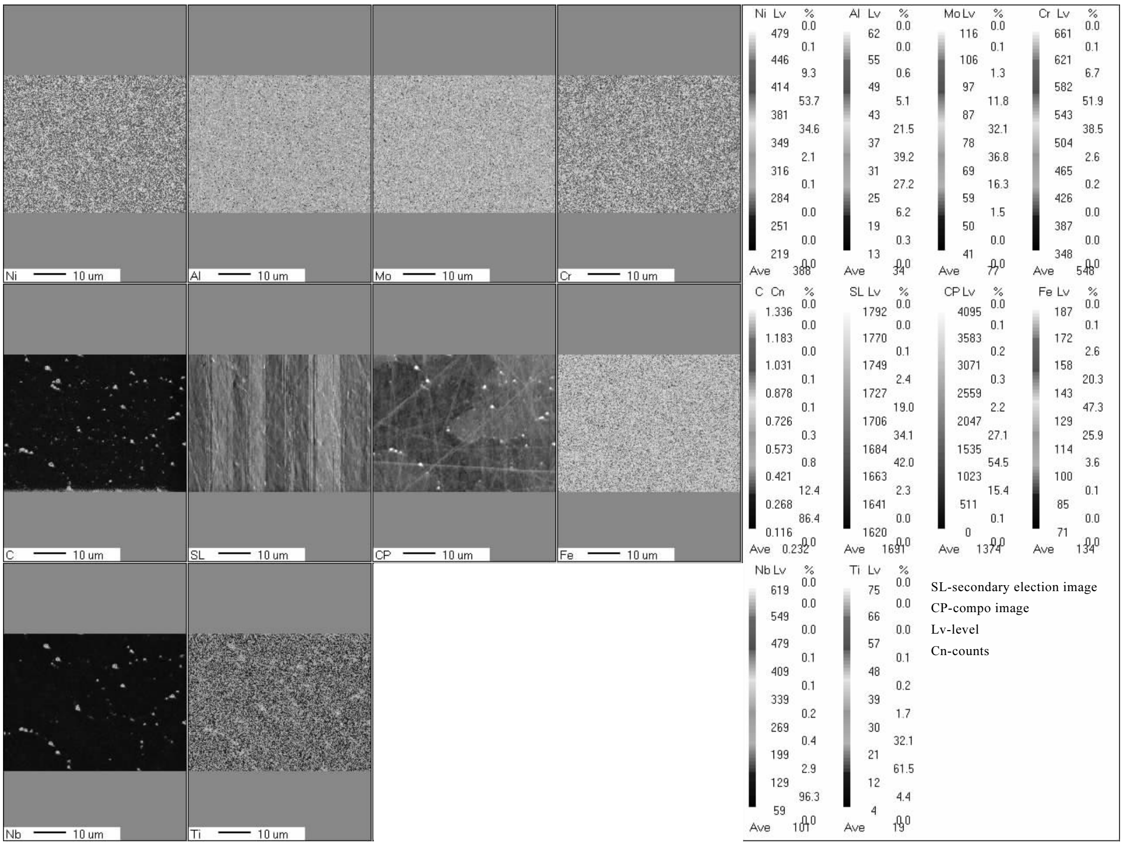

Fig.4 EPMA analysis of the transverse cross section of the as-homogenized IN718 sample

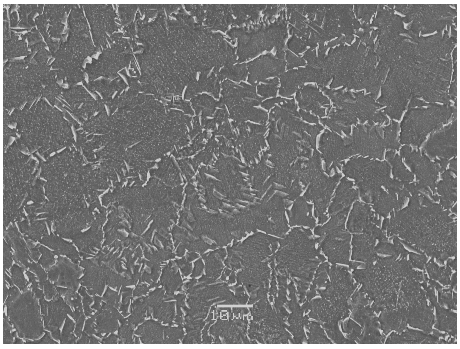

Fig.5 SEM micrograph of homogenized STA sample

treatment showed a slightly lower tensile strength (ultimate strength 1120 MPa). Whether it is direct-aging heat treatment, or the STA and homogenized STA treatments, high temperature tensile properties at 650 °C are higher than the wrought AMS.

The microstructure of laser-deposited IN718 was obviously caused by the dynamic heat transfer of the moving heat source and the formation of layered material. During laser deposition, the material is built up layer by layer. When a new layer is deposited, a thin top portion of the previous layer is remelted. These interfacial layer regions are usually associated with sharp changes in grain size and degree of microsegregations, and therefore, they may be the weak sites in tensile test [6,13].

The fracture surfaces of the room temperature tensile tested specimens were investigated by SEM. The as-deposited fracture surface exhibited a fine dimpled surface, as shown in Fig.6a, indicating a transgranular ductile mode of failure associated with good elongation. Fig.6b shows the obviously visible dendritic shape in the fracture of the direct aged samples. This suggests that the failure took place at the interdendritic regions or grain boundaries and was relatively brittle. The STA fracture surface, as shown in Fig.6c, exhibited some shallow dimples with protuberances composed of white particles, which are believed to be the Laves phase and MC carbides. The homogenized STA fracture surface, as shown in Fig.6d, exhibited coarse dimples with voids and broken carbides. It can also be observed that some white particles, mostly δ phase and carbide particles, are present inside the dimples on the fracture surface, which suggests that these particles are the microvoid initiation site associated with parti-

Table 2 Room temperature tensile properties for laser-deposited IN718

State	Yield strength/MPa	Ultimate strength/MPa	Elongation/%
As-deposited	641	962	35
Homogenization+STA	1198	1348	17.5
STA	1165	1374	17
Direct aging	1283	1431	12.5
Cast AMS	758	862	5
Wrought AMS	1034	1276	12

Table 3 Tensile properties for laser-deposited IN718 at 650 °C

State	Yield strength/MPa	Ultimate strength/MPa	Elongation/%
Homogenization+STA	1010	1120	21
STA	980	1150	22
Direct aging	1050	1190	21
Wrought AMS	862	1000	12

cle-matrix decohesion or particle fracture, leading to the final transgranular fracture caused by void coalescence

According to the tensile test results at room temperature and high temperature, the mechanical properties of all three heat treatments, including direct aging, homogenized STA and STA, are higher than the AMS for wrought IN718. Strength of direct aged samples is the highest and plastic elongation of homogenized STA sample is the best. However, due to the lack of solution process, no δ phases pre--cipitate in the direct aged samples, leading to the notch sensitivity. High temperature homogenization at 1080 °C can promote the solution of brittle Laves phase and increase plasticity. Compared to both homogenized STA and STA treatment, although homogenization treatment accelerates grain growth, the effect on the strength is smaller. Therefore, homogenized STA is a suitable heat treatment method for LSM IN718 alloy.

2.3 Stress rupture and low cycle fatigue properties

As shown in Fig.7, stress rupture life at 650 °C/700 MPa of the homogenized STA samples is 40.7 and 32.7 h, which is higher than AMS specifications for wrought IN718 (25 h). Stress rupture life of the homogenized STA samples at 650 °C/725 MPa is 174 and 69.8 h, respectively, which has a larger fluctuation. The stress rupture fracture at 650 °C/700 MPa, as shown in Fig.8, indicates an intergranular fracture mode with a preferred orientation. Under high temperature

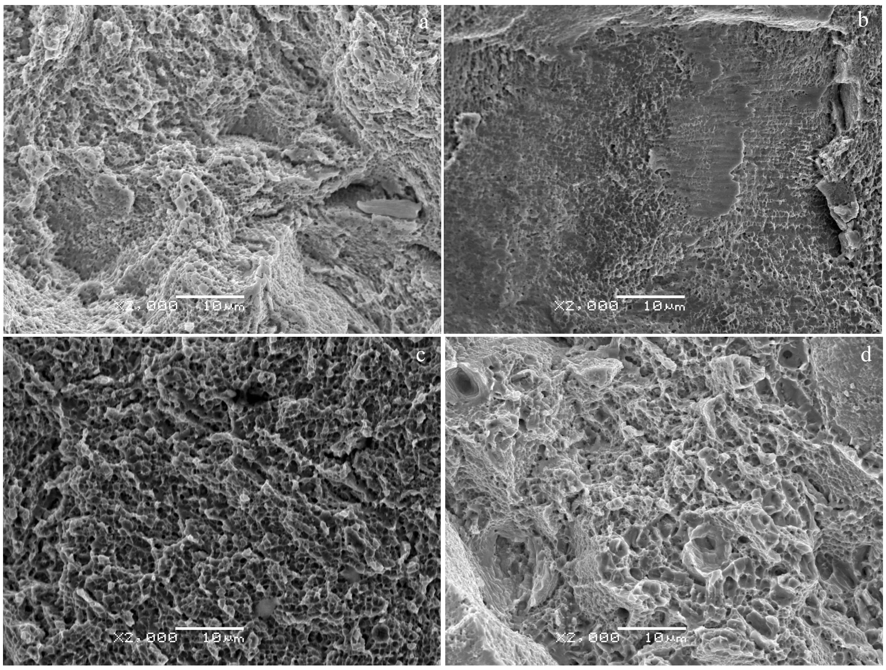

Fig.6 SEM micrographs of the room temperature tensile fracture surface of as-deposited (a), direct aged (b), STA (c), and homogenized STA (d) samples

deformation conditions, grain boundary is the weak site. Dislocation slip and grain boundary slide are the main methods of plastic deformation. As the degree of deformation increases, the stress concentration at the grain boundaries continuously increases. When the stress exceeds the bond strength of the grain boundaries, the cracks initiate at the grain boundaries and extend along the grain boundaries until the final break [19]. As shown in Fig.7, stress rupture life at 650 °C/725 MPa has significant dispersion, which is probably due to the preferred orientation characteristics of the LSM IN718 alloy [17]. It can be confirmed from the EBSD pattern of the homogenized STA sample, as shown in Fig.9. In addition, due to the fine microstructure accompanied to the rapid solidification process, the size of the δ phase particles is also very small during the solution treatment, and the material has a notch sensitive possibility. Through appropriately decreasing the solution treatment temperature or prolonging the solution treatment time, this tendency can be reduced.

As shown in Fig.10, low cycle fatigue life under 455 °C, σ_{max}=1050 MPa, R=0.1, f=10 Hz test conditions is 59 641, 61 771 and 62 998. Compared to specification of wrought IN718, LCF life of homogenized STA samples is slightly lower than AMS specification for wrought IN718 (7000).

Fig.11 shows the LCF fractography under 455 °C, σ_{max}=1050 MPa, R=0.1, f=10 Hz test conditions, indicating the fatigue source site and fatigue crack propagation site. In the fatigue source site, there are cleave facet features, and no pores and carbides are found. Moreover, fatigue cracks do not start at the edge of the specimen, which is quite different from the wrought IN718 specimens [20]. As shown in Fig.12, in the LCF fractography of wrought IN718, carbides with a size of 20 μm are visible, and fatigue cracks start at the edge of the specimen. The alternating effect of cyclic loading promotes the initiation of fatigue cracks on the carbides near the edge of the specimen, which leads to the final fracture. Therefore, microstructure with fine grains and small carbides of SLM specimen has better resistance to the big stress low cycle fatigue for wrought IN718 under 455 °C.

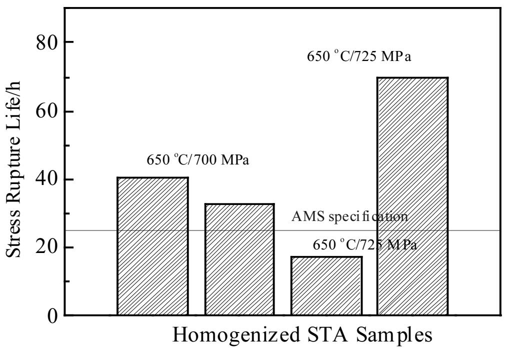

Fig.7 Stress rupture life of the homogenized STA samples of LSM IN718 alloy

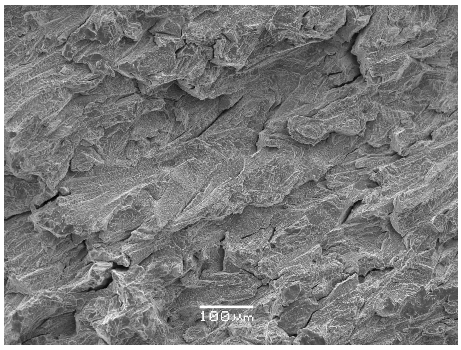

Fig.8 SEM micrographs of the stress rupture fracture at 650 °C/700 MPa

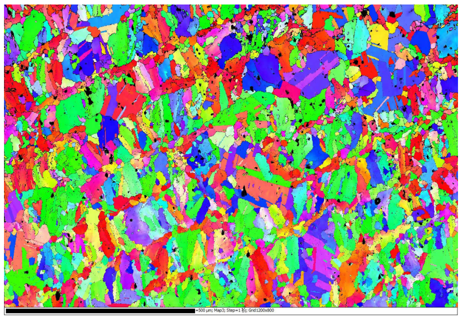

Fig.9 EBSD pattern of the preferred orientation of homogenized STA IN718 alloy

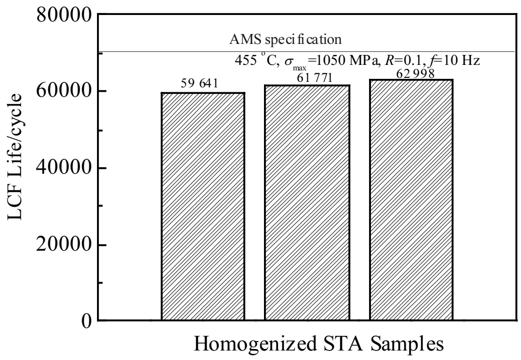

Fig.10 Low cycle fatigue life of the homogenized STA samples of LSM IN718 alloy

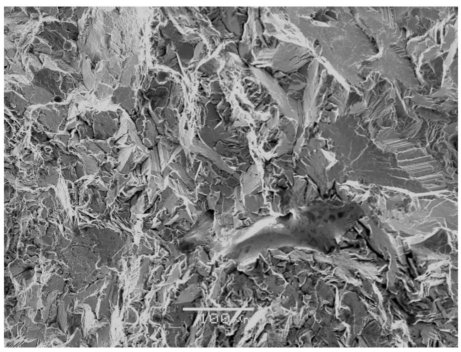

Fig.11 SEM micrograph of the LCF fracture of homogenized STA IN718 at 455 °C

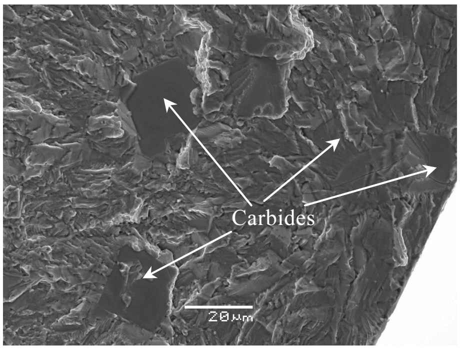

Fig.12 SEM micrograph of the LCF fracture of wrought IN718 at 455 °C

3 Conclusions

1) Room temperature and high temperature tensile properties of SLM IN718 alloy with all three heat treatments, including direct aging, homogenized STA and STA, are higher than AMS for wrought IN718. Strength of direct aged samples is the highest and plastic elongation of homogenized STA sample is the best. Homogenized STA is a suitable heat treatment method for LSM IN718 alloy.

2) Stress rupture life of homogenized STA samples is higher than AMS for wrought IN718 at 650 °C, while it presents a notch sensitivity phenomenon. Low cycle fatigue life of the homogenized STA samples is lower than AMS.

References

1 Schwendner K I, Banerjee R, Collins P C *et al*. *Scripta Mater*[J], 2001, 45: 1123
2 Zhong M, Yang L, Liu W. *Proc SPIE*[C]. Washington: SPIE, 2002, 5629: 59
3 Lewis G K, Schlienger E. *Mater Design*[J], 2000, 21: 417
4 Liu W, DuPont J N. *Acta Mater*[J], 2004, 52: 4833
5 Kobryn P A, Moore E H, Semiatin S L. *Scripta Mater*[J], 2000, 43: 299
6 Qi H, Azer M, Ritter A. *Metall Mater Trans A*[J], 2009, 40: 2410
7 Marchese G, Lorusso M, Calignano F *et al*. *Superalloy* 2016[M]. Pennsylvania: TMS, 2016: 1013
8 Gong J D, Hallee Z, Deutchman A P *et al*. *Superalloy 2016*[M]. Pennsylvania: TMS, 2016: 1031
9 Zhang B C, Lee X H, Bai J M *et al*. *Mater Design*[J], 2017, 116: 531
10 Coste S, Andrieu E, Huez J. *Mater Sci Eng A*[J], 2005, 396: 92
11 Prasad K, Sarkar R, Ghosal P *et al*. *Mater Design*[J], 2010, 31: 4502
12 Kuo C M, Yang Y T, Bor H Y *et al*. *Mater Sci Eng A*[J], 2009, 510-511: 289
13 Feng K Y, Liu P, Li H X *et al*. *Vacuum*[J], 2017, 145: 112
14 Panwisawas C, Sovani Y, Anderson M J *et al*. *Superalloy 2016*[M]. Pennsylvania: TMS, 2016: 1021
15 Zhao X M, Chen J, Lin X *et al*. *Mater Sci Eng A*[J], 2008, 478: 119
16 Amato K N, Gaytan S M, Murr L E *et al*. *Acta Mater*[J], 2012, 60: 2229
17 Blackwell P L. *J Mater Proc Tech*[J], 2005, 170: 240
18 Benn R C, Salva R P. *7th Inter Symp Super 718 Deriv*[M]. Pennsylvania: TMS, 2011
19 Du J H, Lu X D, Deng Q *et al*. *Rare Metal Mater Eng*[J], 2017, 46: 2359
20 Lu X D, Du J H, Deng Q. *Mater Sci Eng A*[J], 2013, 588: 4

热处理对选择性激光熔炼 IN718 显微组织和力学性能的影响

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摘 要：选择性激光熔炼（SLM）建立在激光熔覆/沉积基础上，能够由粉末直接制备或修复近成形高性能部件。通过优化激光沉积过程试验参数，以最大限度地降低气孔率。对沉积态、直接时效态、固溶时效态、均匀化后固溶时效态 4 种状态激光沉积 IN718 合金的显微组织和力学性能进行了对比分析。拉伸试验结果显示，直接时效态合金强度最高，均匀化后固溶时效态合金塑性最好。综合考虑 3 种热处理状态的室温和高温拉伸试验结果，均匀化后固溶时效态试样不仅具有高于锻态 AMS 标准的强度，而且有很好的塑性。因此，选择均匀化后固溶时效处理作为选择性激光熔炼 IN718 合金的热处理方式。考察了该种热处理状态合金的 650 ℃/700 MPa 和 650 ℃/725 MPa 的持久性能和 455 ℃的低周疲劳性能，并与锻态 IN718 进行了对比。

关键词：IN718；热处理；SLM；显微组织；力学性能

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