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Effects of Heat Treatment on Microstructures and Tensile Properties of IN718/TiC Nanocomposite Fabricated by Selective Laser Melting

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Selective laser melting (SLM) is a powder-bed fusion additive manufacturing process that fabricates metallic parts in a layer-by-layer manner. IN718 is a nickel-based austenitic (γ) superalloy with broad applications in aerospace, chemical, and nuclear industries. In this study, fully-dense IN718-matrix composite reinforced by TiC nanoparticles were fabricated in-situ by SLM. As-built and heat-treated IN718/TiC nanocomposite was compared against pure IN718 in terms of microstructures and tensile properties. Columnar and cellular microstructure morphologies in as-built pure IN718 and IN718/TiC nanocomposite were demolished by heat treatment. Microstructure refining and grain boundary dislocation pinning effect of TiC nanoparticles improved the tensile strength of as-built IN718/TiC nanocomposite. In both as-built IN718/TiC and pure IN718, γ and Laves phase are the major constituents while the other precipitates were suppressed by rapid solidification in SLM. Heat treatment dissolved Laves precipitates and grew γ'/γ'' , δ , and $MC/M_{23}C_6$ carbides that strengthened both pure IN718 and IN718/TiC nanocomposite. In heat-treated IN718/TiC, the TiC nanoparticles and $M_{23}C_6$ carbides degenerated from TiC counteracted the deficiency of δ precipitates. Therefore, heat-treated IN718/TiC nanocomposite had similar tensile strength but lower ductility than heat-treated pure IN718.

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1. Introduction

Additive manufacturing (AM) is new manufacturing technology based on the principle of layer-by-layer material fusion¹. Compared with traditional manufacturing processes, AM provides higher design freedom, better cost-effectiveness in complex part fabrication², and new possibilities in new material developments^{3,4}. Among different AM techniques, selective laser melting (SLM) is a powder-bed fusion process that manufactures parts by melting metallic powders using a laser beam⁵. Compared with cast and wrought metals, different material microstructures and properties can be obtained by SLM due to the rapid cooling and highly directional solidification in the process⁶. Besides metals, ceramic particle reinforced metal matrix composites (MMC) have also been manufactured *in-situ* by SLM. Fe, Al, and Ti-based matrix alloys in their powder forms were blended with fine ceramic particles; and the metal-ceramic mixtures were fused into bulk-form composites after being processed by SLM⁷⁻⁹.

IN718 is a Ni-Cr-Fe superalloy that finds many applications in aerospace, chemical, and nuclear industries¹⁰. Due to its good weldability, IN718 is suitable for AM fabrication¹¹. Studies of

additive manufactured IN718 have been reported in literatures¹²⁻¹⁴. Improvements in material hardness, wear resistance, and oxidation resistance were achieved by tuning AM process parameters for IN718 fabrication¹⁵. *In-situ* additive manufacturing of ceramic-reinforced Ni-based MMC was attempted in previous research. SiC were blended with IN625 respectively and processed by additive layer manufacturing (ALM), which resulted in higher hardness but a large content of porosity and cracking.¹⁶ The mixture of TiC and IN625 was processed in laser aided additive manufacturing (LAAM), and the fabricated composite had enhanced hardness and tensile strength due to grain refinement¹⁷. IN718-based MMC reinforced by TiC nanoparticles was fabricated by SLM, and the effects of laser energy density on hardness and wear rate were investigated¹⁸. However, in order understand the combined effect of heat treatment and ceramic particle reinforcements in additive manufactured IN718-based MMC, further research is needed.

In this research, both pure IN718 and IN718/TiC nanocomposite are fabricated by SLM, followed by heat treatment on the as-built materials. As-built/heat-treated pure IN718 and IN718/TiC nanocomposite are characterized by microscopy investigations, X-ray

diffraction analysis, and tensile testing. The effects of heat treatment on microstructural morphologies, phase formations, and tensile properties are analyzed.

2. Experimental Setup

2.1 Powder Preparation

Gas atomized spherical IN718 powders (21 – 55 μ m) were used as the matrix material. The chemical compositions of IN718 powders are listed in Table 1. 2.0wt% of spherical TiC nanoparticles (80 – 150nm) were blended with IN718 powders in an Inversina Tumbler Mixer (Bioengineering, Inc.). SEM micrographs of pure IN718 powders and the IN718/TiC powder mixture are shown in Fig. 1. It can be seen in the magnified view (i.e. the lower half image) of Fig. 1 (b) that the bright TiC nanoparticles are homogeneously spread over the surface of the IN718 powder.

Table 1 Chemical compositions (wt%) of IN718 powders

Ni	Cr	Nb	Mo	Co	Ti	Mn	Si	Al	Cu	Fe
50-55	17-21	4.75-5.5	2.8-3.3	1.0	0.65-1.15	0.35	0.35	0.2-0.8	0.3	Bal

Fig. 1 Pure IN718 powders (a) and the IN718/TiC powder mixture showing bright TiC nanoparticles (b)

2.2 Selective Laser Melting Process

The SLM250HL machine (SLM Solutions GmbH) was used to fabricate both pure IN718 and IN718/TiC nanocomposite. The machine setup is illustrated in Fig. 2. A ytterbium fiber laser with the wavelength of 1070nm was used as the energy source. The focal diameter of the laser beam is 0.08mm. The laser absorptivity is 0.45 for IN718 powders¹⁹ 0.82 for TiC particles²⁰. The carbon steel substrate was preheated to 100°C before the process starts. As shown in Fig. 3, during the SLM process, the scanning laser selectively melted the powders into liquid, which re-solidified into bulk metal. The cooling rate in SLM is in the order of 10⁶ K/s^{21,22}. After the laser scanning on each layer finished, the build platform was lowered by the layer thickness (0.05mm). Powders were dispensed from the

hopper and spread over the previous layer, and then the next iteration of laser scanning started. The laser scanning–powder recoating process repeated until the entire part had been built. The “strip” scanning strategy was applied in the SLM process, as illustrated in Fig. 4. Each strip has a width of 4mm and contains parallel hatch lines representing the laser path. The hatch lines are represented by arrows in Fig. 4. The strips rotate 79 degrees between neighboring layers.

Fig. 2 The schematic of the SLM machine setup

Effects of SLM process parameters on microstructures and properties of IN718 have been discussed in several literatures^{15,23}. However, in this research, the material composition and heat treatment were the only control variables, while the SLM process parameters were set as constants in the fabrication of both pure IN718 and IN718/TiC nanocomposite. A series of preliminary experiments were conducted to find the suitable parameters that are listed in Table 2. According to Jia and Gu¹², the porosity was significantly reduced and the surface smoothness was improved as the linear energy density (defined as the ratio of laser power to scan speed) reached the maximum value of 330J/m. The 98.4% density was observed in the aforementioned study. In this research, the energy density was set at 375 J/m, and a near full density (> 99.9%) was achieved in both pure IN718 and IN718/TiC nanocomposite, which showed a better result than the previous research.

Fig. 3 Laser scanning in SLM

Fig. 4 The “strip” scanning strategy in SLM

Table 2 SLM process parameters

Laser power	Scan speed	Layer thickness	Hatch space
300 W	800 mm/s	0.05 mm	0.13 mm

2.3 Heat Treatment

Heat treatment based on the AMS6554 standard was carried out on as-built pure IN718 and IN718/TiC nanocomposite. The as-built materials went through solution heat treatment at 1070°C (1 hour, water quenching) followed by a double-aging at 720°C (8 hours, furnace cooling) and 620°C (18 hours, air cooling).

2.4 Material Characterization

Four types of materials, i.e. as-built pure IN718, as-built IN718/TiC, heat-treated pure IN718, and heat-treated IN718/TiC, were analyzed and compared in terms of microstructures and tensile properties. X-ray diffraction (XRD, Bruker D8 Discover) with Cu K α radiation (40 kV, 40 mA) was used to identify crystal phases. The specimens were polished and then electro-etched in 70% phosphoric acid. Optical microscopy (OM, OLYMPUS MX51) and scanning electron microscopy (SEM, ZEISS EVO 50) were used to investigate the microstructures. Element concentrations were analyzed by energy dispersive X-ray spectroscopy (EDS, Oxford Instrument X-Max). Tensile coupons were machined from SLM fabricated blocks according to the ASTM E8 standard. An Instron Universal Tester was used to perform the tensile tests, from which yield strength (YS), ultimate tensile strength (UTS), and elongation were obtained.

3. Results and Discussion

3.1 Densification

Near fully-dense (> 99.9% of theoretical densities) pure IN718 and IN718/TiC nanocomposite were fabricated. As shown in Fig. 5, the as-built samples have smooth surfaces with clear features (i.e. the “A1” and “D4” characters on the top faces). No defects (such as balling and cracks) are present on the surfaces.

Fig. 5 Samples of pure IN718 (a) and IN718/TiC nanocomposite (b) fabricated by SLM

Optical microscopy (OM) images of SLM fabricated pure IN718 and IN718/TiC nanocomposite are shown in Fig. 6. Micro-sized pores can be found, due to entrapped gas bubbles and the liquid metal’s viscosity in the melt-pools during rapid solidification²³. Although pores cannot be completely avoided in SLM, the porosity level and pore sizes observed in this experiment are smaller than the results in the literatures²³. This is believed to be achieved by applying a high laser energy density (375J/m) that reduces the dynamic viscosity of molten IN718¹².

Fig. 6 OM images of pure IN718 (a) and IN718/TiC nanocomposite (b) fabricated by SLM

Fig. 7 Microstructural morphologies of: as-built pure IN718 in the melt-pool (a), as-built pure IN718 in the vicinity of the melt-pool boundary (b), heat-treated pure IN718 (c), as-built IN718/TiC in the melt-pool (d), as-built IN718/TiC in the vicinity of the melt-pool boundary (e), and heat-treated IN718/TiC (f)

3.2 Microstructures

Fig. 7 shows the microstructural morphologies of pure IN718 and IN718/TiC nanocomposite before and after heat treatment. As-built pure IN718 is characterized by prominent columnar grains in parallel with the build direction (+Z), which is caused by the highly directional laser induced thermal gradient in SLM. In as-built IN718/TiC nanocomposite, although columnar traces are still present, more refined cellular microstructures are formed, which is believed to be caused by unmolten TiC nanoparticles (melting

temperature: 3050-3230°C) dispersed in the melt-pool that act as nucleation sites during re-solidification. Due to the inhomogeneity of SLM fabricated materials' microstructures, the grain refinement behavior in as-built IN718/TiC nanocomposite is illustrated by comparing IN718/TiC with pure IN718 in different regions. Fig. 7 (a) and (d) show the solidified melt-pool regions, whereas Fig. 7 (b) and (e) show the regions in the vicinity of the melt-pool boundary. In both regions, large elongated grains are dominating in pure IN718, although more complicated morphologies are formed near the melt-

pool boundary²². In contrast, finer grains with less continuous boundaries are found in both regions of IN718/TiC nanocomposite. For both pure IN718 and IN718/TiC nanocomposite, columnar and cellular morphologies formed during the SLM process are demolished after heat treatment, as shown in Fig. 7 (c) and (f), due to homogenization and recrystallization during heat treatment.

As shown in the high magnification SEM micrographs (Fig. 8), both as-built pure IN718 and IN718/TiC nanocomposite contain significant amounts of Laves precipitates along the grain boundaries of the γ matrix. The EDS result shows a high Nb concentration (10-20 wt%) in the Laves phases. The SEM and EDS observations agree with the previous literatures on laser processed IN718²³⁻²⁵. As-built pure IN718 has thick and continuous Laves precipitates, while as-built IN718/TiC nanocomposite has thinner Laves with decreased continuity due to faster solidification and weaker Nb segregation²⁶. During the heat treatment, the Laves precipitates are dissolved in both pure IN718 and IN718/TiC nanocomposite. Meanwhile, the dissolution of the Nb-rich Laves phase has released Nb atoms that promote the formation of needle-like δ (orthorhombic Ni_3Nb) precipitates along grain boundaries. The δ phase precipitation occurs during the aging process between 700°C and 1000°C for less than 100 hours. The uniformly distributed δ precipitates start nucleating at grain boundaries and then grow into elongated thin platelets²⁷. Smaller Laves precipitates (and hence

less available Nb atoms upon Laves dissolution) in as-built IN718/TiC result in fewer grain boundary δ precipitates in heat-treated IN718/TiC nanocomposite than in heat-treated pure IN718. In comparison, δ phase is not observed in as-built pure IN718 and IN718/TiC nanocomposite. Due to the high temperature gradient and cooling rate in the SLM process, Nb segregation at grain boundaries and interdendritic regions rarely occurs, and hence the δ phase precipitation is inhibited²⁸. During the heat treatment, MC/ M_{23}C_6 (M=Nb, Mo, Ti) carbides are formed in both pure IN718 and IN718/TiC nanocomposite; while the formation of MC/ M_{23}C_6 carbides is suppressed during SLM.

As shown in Fig. 9 (a), in as-built IN718/TiC nanocomposites, unmolten TiC nanoparticles (80-100nm) are dispersed in the γ matrix. At the solution heat treatment of 1000-1070°C, a few TiC nanoparticles and MC carbides degenerate into the more stable form of M_{23}C_6 , while Ti atoms in TiC nanoparticles may be substituted by the other M (Nb, Mo) atoms²⁹. No M_6C carbide is formed due to low Mo content (1.97at%) in IN718. As shown in Fig. 9 (b) for heat-treated IN718/TiC nanocomposite, spheroidal γ' precipitates (5-10nm) are found around the degenerated M_{23}C_6 carbides, which is the result of the carbide reaction³⁰ expressed as:

Fig. 8 SEM micrographs showing Laves, δ , and carbides in as-built pure IN718 (a), heat-treated pure IN718 (b), as-built IN718/TiC (c), and heat-treated IN718/TiC (d)

In as-built pure IN718 and IN718/TiC nanocomposites, the precipitation of γ' and γ'' phases cannot be observed. The rapid solidification rate in SLM inhibits the segregation of alloying atoms (Al, Ti, Nb) for γ' and γ'' precipitation²³. The γ' and γ'' are the strengthening phases in Ni-based superalloys, hence they are desirable in the microstructure through heat treatments¹⁰. As shown in Fig. 9 (b) for heat-treated IN718/TiC nanocomposite, the spheroidal γ' and columnar γ'' precipitates can be observed. During the solution heat treatment, γ' and γ'' phases are dissolved in the austenite γ matrix. And then in the following double-aging treatment, γ' and γ'' phases precipitate from the matrix²⁸.

Fig. 9. SEM micrographs showing TiC nanoparticles, $M_{23}C_6$ carbides, γ' / γ'' precipitates in as-built (a) and heat-treated (b) IN718/TiC nanocomposite

Crystal phases of as-built/heat-treated pure IN718 and IN718/TiC nanocomposite are confirmed in the XRD spectra (Fig. 10). γ , γ' , and γ'' peaks are identified. However, since the γ' / γ'' precipitates are coherent with the γ matrix, γ and γ' / γ'' peaks overlap in the XRD spectra and can hardly be distinguished. TiC peaks are not detected in IN718/TiC nanocomposite, due to the small size and quantity of TiC nanoparticles compared to the γ matrix. Fig. 11 shows a magnified view of the diffraction peaks in the (111) crystal plane. For both pure IN718 and IN718/TiC nanocomposite, the positions of the (111) peaks shift to the right after heat treatment. According to Bragg's Law as shown in Eq. (2), as the scattering

angle (θ) increases, the interplanar lattice spacing (d) decreases, indicating the lattice distortion in the heat-treated materials.

$$2d \sin \theta = n\lambda \quad (n = 1, 2, 3, \dots) \quad (2)$$

The observed lattice spacing decrease is caused by the precipitation of γ' / γ'' , δ and $MC/MC_{23}C_6$ carbides from the γ matrix during the heat treatment. In both as-built and heat-treated conditions, IN718/TiC nanocomposite have larger interplanar lattice spacing than pure IN718, indicated by the smaller 2θ values at the left-shifted (111) peaks. The increase in lattice spacing is caused by the unmolten TiC nanoparticles embedded in the γ matrix.

Fig. 10 XRD spectra of as-built/heat-treated pure IN718 and IN718/TiC nanocomposite

Fig. 11 XRD spectra at the (111) peak

3.3 Tensile Properties

Tensile testing results are shown in Fig. 12. Compared with as-built pure IN718, as-built IN718/TiC has higher YS and UTS but lower elongation. Microstructure refinement and the dislocation pinning effect of TiC nanoparticles are the two major strengthening mechanisms in as-built IN718/TiC nanocomposite. The TiC nanoparticles in the IN718/TiC nanocomposite impede the movement of dislocations in the IN718 matrix under tensile loadings. According to George et al.³¹, the local stress fields surrounding the TiC nanoparticles due to lattice distortions further enhance the tensile strength of IN718/TiC nanocomposite compared with pure IN718. Furthermore, during the melting-solidification process of SLM, the mismatch in the thermal expansion coefficient of IN718 and TiC results in elevated stress at the interface of these two constituents. The thermally-induced internal stress also contributes to the strengthening of IN718/TiC nanocomposite³². However, TiC nanoparticles increase the number of failure initiation sites, which causes the ductility decrease in as-built IN718/TiC compared with pure IN718.

Fig. 12 Tensile properties

Heat treatment has significantly increased the YS and UTS of both pure IN718 and IN718/TiC nanocomposite. γ'/γ'' precipitation is the major strengthening mechanism in the heat-treated materials^{10, 14}. Meanwhile, dissolution of the brittle Laves phase and precipitation of δ particles along grain boundaries are also contributing to the strength improvement from heat treatment. However, on the other hand, precipitation hardening of γ'/γ'' phases and the crack initiating effect of δ and MC/M₂₃C₆ particles cause the ductility decrease compared with the as-built materials. Although heat-treated IN718/TiC nanocomposite has fewer grain boundary δ particles than heat-treated pure IN718, the extra strengthening γ' precipitated from TiC-to-M₂₃C₆ transformation in IN718/TiC nanocomposite counteracts with the deficiency of grain boundary δ .

Therefore there is only a minor strength difference between heat-treated IN718/TiC and pure IN718, indicated by 2.0% decrease in YS and 5.6% decrease in UTS. Additional TiC nanoparticles and M₂₃C₆ carbides also reduced the ductility (38.8% decrease in elongation) of heat-treated IN718/TiC compared to pure IN718, due to failure initiation at carbide/matrix interfaces.

Fracture surfaces from tensile testing are shown in Fig. 13. All four types of materials are characterized by dimpled fracture surfaces, indicating ductile failures. Heat-treated pure IN718 and IN718/TiC nanocomposite have larger and slightly shallower dimples than their as-built counterparts, indicating the lower ductility of heat-treated materials^{3, 33}.

4. Conclusions

In this research, IN718/TiC nanocomposite was fabricated by selective laser melting process. As-built and heat-treated IN718/TiC specimens were characterized and compared with pure IN718 in terms of microstructures and tensile properties. The following conclusions can be drawn.

- Columnar and cellular microstructure morphologies in as-built pure IN718 and IN718/TiC nanocomposite disappeared after heat treatment due to homogenization and recrystallization.
- As-built IN718/TiC nanocomposite had higher tensile strength but lower elongation than as-built pure IN718, due to grain refinement and dislocation pinning of unmolten TiC nanoparticles.
- In both pure IN718 and IN718/TiC nanocomposite, heat treatment dissolved the brittle Laves phase and promoted the precipitation of strengthening γ'/γ'' , δ , and MC/M₂₃C₆ carbides from the γ matrix. Therefore, heat treatment improved the tensile strength but decreased the ductility of both pure IN718 and IN718/TiC nanocomposite.
- IN718/TiC nanocomposite had larger crystal lattice space than pure IN718, since TiC nanoparticles were embedded in the γ matrix. Crystal lattice space decreased after heat treatment due to the precipitation of strengthening phases.
- TiC nanoparticles and extra M₂₃C₆ carbides in heat-treated IN718/TiC nanocomposite counteracted its deficiency of grain boundary δ precipitates. Therefore, heat-treated IN718/TiC nanocomposite had similar YS and UTS but significantly lower elongation than heat-treated pure IN718.

In future research, other reinforcement nanoparticles, such as oxides, borides, and carbon nanotubes (CNT), can be applied in SLM-fabricated IN718-based metal matrix composites. The effects of reinforcement content and heat treatment on thermal and chemical properties can also be investigated in the future.

Fig. 13 Fracture surfaces of as-built pure IN718 (a), heat-treated pure IN718 (b), as-built IN718/TiC (c), and heat-treated IN718/TiC (d) nanocomposite

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