Investigations of γ′, γ″ and δ precipitates in heat-treated Inconel 718 alloy fabricated by selective laser melting

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Abstract

Inconel 718 alloy samples were fabricated by selective laser melting (SLM). Microstructure and precipitation in solution-heat-treated- and double-aging-SLM-made Inconel 718 were studied by scanning and transmission electron microscopy. Electron microscope observations showed that disc-shaped and cuboidal γ″, and circular γ′ precipitates with an average size of 10 - 30 nm developed within cellular γ austenite matrix. The simulated, experimentally observed electron diffraction patterns, and dark-field imaging further revealed that the precipitation of three variants of γ″ in the γ matrix occurred. The coarser needle-like γ″, and globular as well as plate-like δ phases precipitated at grain boundaries and also within the

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interior of austenite matrix. The morphology, distribution and crystallography of these precipitates and their formation mechanisms were analyzed and discussed.

Keywords: Nickel alloys; Selective laser melting; Inconel 718; Microstructure; Transmission electron microscopy (TEM)

1. Introduction

Most heat-resistant nickel-based superalloys are hardened by precipitation of γ' phase, an intermetallic phase based on $Ni_3(Al,Ti)$ with an ordered face-centered cubic (fcc) $L1_2$ structure. The γ' phase is coherent with the γ-Ni solid-solution matrix. The principal strengthening mechanism is the antiphase boundary hardening arising from the coherent and ordered γ' phase [1,2]. A few nickel-based alloys such as Inconel 718 and 625 are hardened by precipitation of coherent, ordered metastable γ'' phase, which has a body-centered tetragonal (bct) DO_{22} structure and is based on the composition Ni_3Nb [1-4]. The metastable γ''-Ni_3Nb phase is susceptible to transformation during processing or service to the equilibrium δ-Ni_3Nb (orthorhombic DO_a structure) [5].

Inconel 718 alloy has found many industrial applications because of its excellent corrosion, thermal and strength properties under extreme thermal and mechanical conditions [6,7]. However, Inconel 718 is difficult to machine and weld due to excessive tool wear and low material removal rate, and the segregation of alloying elements in the heat-affected zone around welds degrades strength. Moreover, many Inconel 718 components have complex shapes with mazy inner channels or overhangs making them difficult to manufacture by conventional techniques [7,8]. Selective laser melting (SLM) is a rapid prototype, 3D printing or additive manufacturing (AM) technique used to directly produce metallic parts from powder [9,10]. SLM fabrication enables fabrication of components with complex geometries and overcomes geometric restrictions that exist for conventional manufacturing processes such as casting and forging [11].

During SLM fabrication each layer undergoes rapid solidification when the metal powders are melted by the laser, and the successive layers experience a variety of heating and cooling cycles. The microstructural features after AM processing are different from those of forging and casting [12,13]. Prior studies have shown that a laminar material structure and columnar microstructure are observed in as-fabricated SLM Inconel 718 samples [6-8,11,14]. The predominant texture component is a relatively strong <100> fibre in the build direction [6,7]. For the heat-treated specimen, the plate-like δ-Ni_3Nb precipitates appear at grain boundaries and inside the grains [6,8,11,14], and the γ' and γ'' phases are too small to be clearly resolved even by high-resolution scanning electron microscopy (SEM) [8,15]. The TEM work by Amato et al. [6] has shown γ'' precipitates sized 25 - 50 nm in SLM-processed Inconel 718 specimens. However, rather limited information has been published on the precipitate morphologies, their crystallographic structures and the orientation relationships of the γ', γ'' and δ phases with the γ matrix in Inconel 718 alloy prepared by SLM.

The work reported in this paper is to investigate the microstructure of heat-treated Inconel 718 processed by SLM via X-ray diffraction (XRD), SEM and

TEM. Emphasis is placed on studying the precipitation of intermetallic γ′, γ″ and δ phases in Inconel 718. Such a study is expected to be helpful in understanding the microstructural development and mechanical behavior of Inconel 718 fabricated by SLM.

2. Experimental procedure

Commercial Inconel 718 powders with a particle size of ~ 50 μm were used as the starting materials in SLM experiments. The manufacturing of the samples was performed in an EOSINT M280 machine operated at a power of ~ 285 W with a scanning speed of 0.96m/s, laser spot size of 45 μm, and laser thickness of 40 μm [16]. The as-built cylindrical samples were 10 mm in diameter and 100 mm long in total, which were solution heat-treated at 1065˚C for 1 h, followed by air cooling. Then, the double aging was carried out at 760˚C for 10 h followed by furnace cooling for 2 h to 650˚C and holding at 650˚C for 8 h, and finally air cooling to room temperature. The chemical compositions (at.%) of the heat-treated Inconel 718 alloy analyzed by inductively coupled plasma optical emission spectrometry is 54.22Ni-20.21Cr-3.33Nb-2.33Mo-1.09Ti-1.72Al-Fe (balance)

Fig. 1 is a sketch of a cylindrical sample. To investigate the microstructure, a cubic specimen (Fig. 1) with a side length of ~8 mm was prepared by electrical-discharge machining (EDM) and mechanically polished via a standard metallographic procedure using 1 μm diamond powder in the final stage of polishing. After final electropolishing the specimen was etched in a solution composed of 70 vol.% H_3PO_4 and 30 vol.% H_2O at 5 V and room temperature. The microstructure of the etched specimen was examined by a JSM-7001 SEM. The phase constitutions of the alloy were determined by a D/MAX-3C X-ray diffractometer with Cu K_α radiation in the range 2θ = 20 - 100° with a step size of 0.02° and a counting time of 1 s per step. A small pin (Fig. 1) with a diameter of 3 mm was prepared by EDM with the pin's cylindrical axis parallel to the longitudinal building direction. Discs 3 mm in diameter (*xy* plane) and ~ 0.2 mm in thickness (*z* axis) were cut from the pin by EDM. TEM specimens were prepared by grinding discs to a thickness of ~ 80 μm and electropolishing in a twin-jet apparatus using an etchant consisting of 10 vol.% perchloric acid and methanol solution at 25 V and a temperature of −25˚C. TEM bright-field (BF) imaging and selected area electron diffraction (SAED) were performed in Philips CM 200 ST and JEOL 2010F electron microscopes equipped with field emission guns and operated at an accelerating voltage of 200 kV.

3. Results

The XRD diffractograms of the horizontal (*xy*) and vertical (*yz* and *xz*) planes from heat-treated specimen are shown in Fig. 2. The profiles have similar XRD patterns with strong <200> textures. Since there is a nearly exact γ(111)/γ′(111) peak match as well as matches for γ(200)/ γ′(200)/γ″(200), γ(220)/ γ′(220)/γ″(220), and γ(311)/γ′(311)/ γ″(033), it is not possible to determine the γ′ and γ″ precipitates from XRD data. Peaks at 2θ = 47.5° could be indexed as the (211) peak of the δ phase. Fig. 3 shows a three-dimensional (3D) SEM image composite corresponding to a section of Fig. 1 illustrating three different types of precipitates. The thickest precipitates with the morphology of short platelets were identified as δ-phase according to the literature

[5,15]. The γ″ phase precipitates in two directions with acicular morphology. Cuboidal and circular precipitates could be γ″ and γ′phases [2,4]. Therefore, unambiguous identification of the γ′, γ″ and δ precipitates must rely on TEM observations, especially the SAED patterns.

Fig. 4a is a TEM BF image showing fine cellular substructures in heat-treated SLM Inconel 718 specimen. Fig. 4b-d are the SAED patterns of the region marked as A along the [011], [$10\bar{3}$], and [001] zone axes, respectively. Indexing indicated that the austenite (γ) matrix has a fcc (A1) crystal structure with lattice parameter *a* = 0.360 nm. The SAED patterns have been indexed with respect to the fcc (Al) austenite such that the fractional index can be used to identify the γ″ (DO_{22}) variants [2-5]. For the superlattice reflections (100), (010), and (110) in Fig. 4d, the first two types are thought to arise from γ′ precipitates, and the third type could arise both from γ′ and γ″ phases. The fractional indexes, (1 1/2 0) and (1/2 1 0) reflections in Fig. 4d, belong only to γ″ precipitates. The [001] SAED patterns are similar to those of other TEM studies on Inconel 718 alloy in the literature [2,4,17]. The observation of the forbidden ($01\bar{1}$) reflection in Fig. 4b is due to the effect of dynamic double diffraction. Electron diffraction reveals that the cellular structure marked as B in light color is still the γ matrix in another crystallographic orientation. From Fig. 4a, it was observed that coarser plate and needle-like precipitates characterized in the next sections were distributed within the austenite and at the grain boundaries.

Since three variants of the γ″, and γ′ phase could form in the γ matrix during heat-treatment, the nano-scale precipitation of those structures results in the overlapped SAED patterns at some specific orientations. So, the calculated electron diffraction patterns could help to identify the individual structure. Based on the DO_{22} structure, we considered that DO_{22} Ni_3Nb has two Nb atoms and six Ni atoms in the unit cell. The Nb atoms occupy the positions of (0, 0, 0) and (1/2, 1/2, 1/2), and those of Ni (1/2, 1/2, 0), (1/2, 0, 1/4), (0, 1/2, 1/4), (1/2, 0, 3/4), (0, 1/2, 3/4), and (0, 0, 1/2) [4,17]. The calculated electron diffraction patterns of the γ, γ′, and γ″ phases are shown in Fig. 5. The calculated diffraction patterns of the [001] γ (Fig. 5a), [001] γ′ (Fig. 5b), [001] γ″ (Fig. 5c), [100] γ″ (Fig. 5d), [010] γ″ (Fig. 5e), and composite <001> γ″ ([001]+[100]+[010], Fig. 5f) are displayed. The (100), (010), and (001) spots for γ″ structure in Figs. 5c-e are systematic extinctions. The (110) spots of γ′ (Fig. 5b) and γ″ (Fig. 5c) phases, and the (100) or (010) spot of γ′ (Fig. 5b) and (002) spot of γ″ (Figs. 5d and e) have remarkable coincidences. Figs. 5d-f indicate that the (011) and (101) spots of γ″ could also be fitted to the fractional indices of the (1/2 1 0) and (1 1/2 0) spots indexed with respect to the fcc matrix. Therefore, the nano-precipitation of three variants of γ″ and γ′ in the γ matrix can produce the electron diffraction patterns shown in Fig. 4d.

Fig. 6a is a high-magnification BF TEM image showing precipitates within γ. The SAED patterns shown in the inset of Fig. 6a is the same as that of Fig. 4d. Figs. 6b-f are dark-field (DF) TEM images by using the appropriate superlattice spots labelled（1）–（5）in the inset of Fig. 6a, respectively. Comparing the DF TEM images shown in Fig. 6b (taken from spot 1, possibly (010) γ′ or (002) γ″) with Fig. 6e (taken from spot 4, (011) γ″ or indexed as (1 1/2 0)), the precipitates shown both in

Fig. 6b and e are the [100] variant of γ″. A similar case exists for Fig. 6c (taken from spot 2, (101) γ″ or indexed as (1/2 1 0)) and Fig. 6f (taken from spot 5, possibly (100) γ′ or (002) γ″), revealing that the precipitates shown in both Fig. 6c and f are the [010] variant of γ″. The cuboidal particles shown only in Fig. 6d (taken from spot 3) correspond to the [001] variant of γ″. The circular particles shown in Figs. 6b, d and f are γ′ phase. The spots 1 and 5 could be composed of a point which is a {100}γ′ reflection and a streak which is a (002)γ″ reflection [3]. The [100] and [010] γ″ precipitates lying end-on with an average size of 10 - 30 nm form as thin discs accounting for the visible streaks of the superlattice reflections shown in Fig. 4d and the inset of Fig. 6a.

Fig. 7a shows the morphologies of the precipitates in other area of the TEM specimen. The γ matrix still displays cellular substructure. It is clear from the SAED pattern in the inset of Fig. 7a that the γ (indicated by white γ font) also has [001] orientation. Figs. 7b-d are TEM BF magnified images of γ in two-beam conditions of (200), (020), and (220) when the electron beam is close to γ [001] zone axis, respectively. Variants of the γ″ lying end-on along the [100] (Fig. 7b) and [010] (Fig. 7c) directions were also clearly present. The strain field contrasts at γ/γ″ interfaces were observed due to the retained coherency. The variant of the [001] γ″ could not be resolved by the two-beam condition at g = (220) in Fig. 7d.

Micrograph (Fig. 8a) from the heat-treated SLM Inconel 718 alloy displays coarser acicular precipitates. The precipitates nucleated at grain boundaries and within the interior of the γ phase. Figs. 8b and c are the SAED patterns of the precipitates marked as C and D. Indexing indicated they are bct (DO_{22}) γ″-Ni_3Nb with lattice parameters *a* = 0.363 nm, and *c* = 0.741 nm. The needle-shaped precipitates correspond to two γ″ variants of [100] (C, Fig. 8b) and [010] (D, Fig. 8c) for which the tetragonal axes were normal to the electron beam direction. The experimentally observed patterns of γ″ phase along [100] and [010] zone axes are in good agreement with the simulated electron patterns shown in Fig. 5d and e. When compared with the SAED pattern of the austenite (marked as E) along the [001] zone axis (Fig. 8d), the orientation relationship of γ″-Ni_3Nb with the γ matrix was determined to be $(001)[100]_{\gamma''}$ // $(100)[001]_{\gamma}$. The weaker reflections in Fig. 8d (same as Fig. 4d and the inset of Fig. 6a and 7a) are from the nano-scaled γ″ variants and γ′ in γ matrix, which are the same as reflections of precipitates C ([100] γ″ variant) and D ([010] γ″ variant) shown in Fig. 8b and c. The strain field contrast around a γ″ precipitate (C) at the inset is shown in Fig. 8a at two-beam conditions of (200) when the electron beam is close to the γ [001] zone axis.

A TEM BF image, Fig. 9a, shows that globular and plate-like precipitates are found to form within the interior of the austenite matrix and at grain boundaries of the heat-treated SLM Inconel 718. The globular precipitate F is analyzed in more detail by SAED. Figs. 9b-d show SAED patterns along the [100], [110], and [302] zone axes, respectively. From these patterns, it is deduced that this precipitate has an orthorhombic (DO_a) crystal structure with lattice parameters *a* = 0.514 nm, *b* = 0.423, and *c* = 0.453 nm. Thus, we conclude that the precipitate F is δ-Ni_3Nb. Fig. 9e is the

SAED pattern of the γ marked as G along the [011] zone axis. The superposition of the SAED patterns (Fig. 9f) of the δ-Ni_3Nb (F) and γ (G) reveal the crystallographic orientation relationship: $(010)[100]_{\delta} // (11\bar{1})[011]_{\gamma}$. Examples for plate-like precipitates are observed in the region marked by δ in Fig. 9a.

4. Discussion

As shown in Fig. 4a and 7a, a micron-scale, cell-shaped structure was observed in heat-treated SLM Inconel 718. The fine structures may result from the rapid solidification of the melted materials during SLM fabrication and subsequent heat-treatment by air cooling [9]. The SLM microstructure was reported to be a factor of 10 finer than the forged and cast microstructure (factor 100) [11], which could increase the strength by impeding dislocation glide. The cell-like substructures were observed in Inconel 939 processed by SLM [13]. Similarly, Amato et al. [6] observed such substructure by TEM images in SLM-processed Inconel 718. The compacted M2 tool steel (Fe-6W-5Mo-3.8Cr-2.1V-lC, wt.%), APK-1 Ni-based alloy (Ni-14.9Cr-3.5Ti-4AI-16.9Co-5.lMo, wt.%), and Al-6wt.%Cu alloy synthesized by using the dynamic powder compacting process all show cellular structures. A cellular structure would change to equiaxed microcrystalline structure for cooling rates in excess of a certain range [18]. Further work is needed to illustrate the formation mechanism of these cellular substructures. In addition, the resistance of γ to coarsening may be improved by the precipitation of nano-sized γ′ phase and γ″ variants, and thus the cellular substructure could be retained even after heat-treatment.

Non-equilibrium defects such as grain boundaries, dislocations, stacking faults, and inclusions are suitable heterogeneous nucleation sites, which reduce the activation energy barrier [19]. TEM observations indicate that γ′, γ″ and δ phases precipitate at both grain boundary and intragranular matrix. SAED patterns revealed that the orientation relationships of γ″ with γ is $(001)[100]_{\gamma''}$ // $(100)[001]_{\gamma}$, and orientation relationship of δ with γ is $(010)[100]_{\delta}$ // $(11\bar{1})[011]_{\gamma}$. These orientation relationships mean that the lattices of the γ″ and δ phases in the [100] directions are superimposed on the lattices of the γ phase in [001] and [011] directions. The lattice mismatches are $\delta_{\gamma''\text{-}\gamma} = (d(100)_{\gamma''} - d(001)_{\gamma})/\ d(001)_{\gamma} \approx 0.83\%$, and $\delta_{\delta\text{-}\gamma} = (d(100)_{\delta} - 2d(011)_{\gamma})/\ 2d(011)_{\gamma} \approx 0.78\%$, indicating lower internal stresses in the precipitation of γ″ and δ phases from γ during aging. Since the γ″ precipitate forms coherently, its tetragonal distortion (*c/a*) results in considerable coherency strains. The strain field contrasts, as indicated in Fig. 7 and by arrows in Fig. 8a, were observed. In the present research, the compact morphology, in which the cuboidal γ′ particles are coated on all six faces with a shell of γ″ precipitates [3], or the composite γ′-γ″ morphology (coffee-bean-shaped co-precipitates, γ″ was in contact with γ′) [20-22] has not been observed. The ratio of the combined atomic contents of Al and Ti to the atomic content of Nb, (Al+Ti)/Nb, is a measure of the compact morphology proposed by Cozar and Pineau [3]. If (Al+Ti)/Nb is between 0.9 and 1.0, the compact morphology is expected to form in Inconel 718-type alloys. The calculated ratio of (Al+Ti)/Nb is 0.84. Cozar and Pineau’s ratio for the heat-treated SLM Inconel 718 is in good agreement with the observed precipitate morphology since the occurrence of the compact γ′-γ″ morphology was not observed.

5. Conclusions

The microstructure and precipitation of the heat-treated Inconel 718 allloy samples manufactured by selective laser melting have been studied by XRD, SEM, and TEM. It is observed that the γ austenite matrix shows a fine cellular substructure. The simulated electron diffraction patterns and dark-field image analysis reveal that γ′ phase and three variants of γ″ phase around 10 - 30 nm precipitate within cellular γ matrix, no composite or compact γ′-γ″ precipitate morphologies were observed. At grain boundaries and intragranular matrix, coarser needle-like γ″, and globular as well as plate-like δ precipitates occurred. The orientation relationships of γ″ with γ, and of δ with γ are $(001)[100]_{\gamma''}$ // $(100)[001]_{\gamma}$ and $(010)[100]_{\delta}$ // $(11\bar{1})[011]_{\gamma}$, yielding small lattice mismatches. The present research provides a direct examination of the precipitation of γ′, γ″ and δ phases in heat-treated Inconel 718 alloy fabricated by selective laser melting.

Acknowledgments

This work was financially supported by Siemens Ltd., China, and the Shanghai Committee of Science and Technology, China under Grant No. 16520721700.

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Figure captions

Fig. 1. Sketch showing cylindrical sample and the positions of the cube and pin taken for microstructural analysis.

Fig. 2. XRD diffractogams from heat-treated Inconel 718 alloy at horizontal (*xy*) and vertical (*yz* and *xz*) planes.

Fig. 3. 3D SEM image composite view for a heat-treated specimen at the horizontal (*xy*) and vertical (*yz* and *xz*) planes.

Fig. 4. (a) TEM BF image showing the cellular substructure of the heat-treated SLM Inconel 718 specimen, SAED patterns of γ austenite (region marked A) along the (b) [011], (c) $[10\bar{3}]$, and (d) [001] zone axes.

Fig. 5. Simulated electron diffraction patterns of (a) γ along the [001], (b) γ' along the [001], (c) γ'' along the [001], (d) γ'' along the [100], (e) γ'' along the [010], and (f) composite along <001> γ'' ([001]+[100]+[010]) zone axes.

Fig. 6. TEM BF image displaying the precipitates, the inset shows a SAED pattern of γ along the [001] zone axis. TEM DF images taken from the labeled spots (b) (1), (c) (2), (d) (3), (e) 4, and (f) 5.

Fig. 7. (a) TEM BF image displaying the morphologies of the precipitates and cellular substructure of γ matrix, the inset shows a SAED pattern of γ (indicated by white γ font) along the [001] zone axis. TEM BF magnified images showing precipitates inside γ at two-beam conditions of (b) g = (200), (c) g = (020), and (d) g = (220) when beam is close to γ [001] zone axis.

Fig. 8. TEM BF image showing the precipitation of the needle-like γ″-Ni_3Nb. The inset shows the strain-field contrast around a γ″ precipitate (C) at two-beam conditions of g = (200) when beam is close to γ [001] zone axis. SAED patterns of γ″ precipitates marked as C and D along the (b) [100], (c) [010] zone axes, and (d) γ matrix (region marked E) along the [001] zone axis.

Fig. 9. (a) TEM BF image showing the precipitation of globular and plate-like δ-Ni_3Nb. SAED patterns of δ precipitate (region marked F) along the (b) [100], (c) [110], and (d) [302] zone axes, (e) γ matrix (region marked G) along the [011] zone axis, and (f) δ and γ show the orientation relationship.