Materials Science & Engineering A 689 (2017) 220–232

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Microstructure and hardness studies of Inconel 718 manufactured by selective laser melting before and after solution heat treatment

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ARTICLE INFO

Keywords:
Microstructure
Hardness
Microsegrgation
Precipitation
Dislocation

ABSTRACT

The microstructure of Additive Manufactured (AM) Inconel 718 in general and Selective Laser Melting (SLM), in particular is different from the material produced by conventional methods due to the rapid solidification process associated with the former. As a result, the widely adapted standard solution heat treatment temperature (< 1100 °C) for conventional material is found to be not high enough for materials fabricated with SLM method in order to dissolve Laves and other microsegregated phases for releasing the ageing constituents (Nb, Ti, Al) sufficiently into the alloy matrix. In this study, sample of Inconel 718 fabricated with SLM method were solution heat-treated to 1100 °C or 1250 °C at different hold times to investigate the dissolution of macro- and micro-segregated precipitates. Investigations of microstructure and segregation in as-printed and solution heat-treated states have been studied using Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM) and Optical Microscopy (OM). Measurement of material hardness was performed with Vickers hardness tests. The microstructure of the as-printed parts exhibit non-columnar grains, but contain well-shaped columnar/cellular sub-grains. The intergranular boundaries are decorated with high density of dislocations and segregated particles. Tremendous stress relief and grain coarsening were observed with solution heat treatment. In particular, at 1250 °C annealing, the sub-grains, including precipitates and dislocation networks along the sub-grain boundaries, were entirely dissolved. However, the 1100/1250 °C solution heat treatment scheme could not dissolve microsegregated precipitates and carbides completely. Details of the analysis on microstructure, dissolution of precipitates and hardness are presented.

1. Introduction

Inconel 718 is a Ni-based superalloy known for its ability of retaining mechanical stability at elevated temperature up to 650 °C, excellent resistant to oxidation and good fatigue life [1,2]. Hence, it is widely used in aeronautics and in energy industries for various applications. In oil industries, for instance, Inconel 718 is used for downhole tools such as subsurface safety valves, packers, and flow control devices and for wellhead components and valve bodies [3], where combination of high stability in mechanical properties and good corrosion resistivity at high temperature conditions are the requirements. The microstructure of Inconel 718 constitutes γ solid supersaturated solution matrix rich in Ni, Cr and Fe with precipitates of coherent phases of γ″ (Ni_3Nb) and γ′ Ni_3 (Al,Ti). The primary strengthening mechanism of the superalloy is by heat treatment that involves (i) solid solution annealing for 1 h at a temperature between 980 - 1200 °C, (which is below the fusion temperature ≈1300 °C), to dissolve age hardening constituents into the matrix and (ii) ageing treatment at relatively lower temperature (between 650 and 900 °C) to form both γ′ and γ″ precipitates [4–6]. The precipitation of the phases are generally obtained after performing two successive steps of heat treatment aging, at 750 °C and 650 °C. The coherent γ′ and γ″ phases form a morphology that realizes the microstructure stability of the alloy if proper reaction takes place during precipitation. Nb is one of the age hardening constituents of Inconel 718 which is highly susceptible to segregation and tend to form some undesirable phases, including NbC, δ-Ni_3Nb and the Laves phases, known for degrading tensile ductility, fatigue and creep rapture properties [3,7]. Laves is a brittle intermetallic compound represented by (Ni, Cr, Fe)(Nb, Mo, Ti) and the content of Nb, Mo and Ti are higher in Laves than in the γ matrix [2,8]. If not properly solution annealed, the Laves phase and the carbides are obviously harmful to the mechanical properties.

Inconel 718 is well known for its good weldability due to its slow precipitation strengthening kinetics [2] and thus creates a minimal cracking possibility, which is a conducive property that enable the production of the material with Selective Laser Melting (SLM) techni-

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http://dx.doi.org/10.1016/j.msea.2017.02.062
Received 17 December 2016; Received in revised form 14 February 2017; Accepted 17 February 2017
Available online 20 February 2017
0921-5093/

que. SLM is one of the Additive Manufacturing (AM) methods, utilizing a 3D layer-by-layer fabrication to produce a solid, simple or geometrically complex objects from a series of layers where each layer of the initial material (in the form of a powder) is melted and added (or "printed") on to the top of the previous one [9]. This technique has a number of advantages over the conventional so-called "subtractive" manufacturing techniques. SLM allows, among others, production of very precise parts, recycling of existing parts and saving a high quantity of initial material. In SLM method, the strong temperature gradient induced by the high cooling rates during the fabrication process mainly characterizes microstructure and physical properties of the alloy. To achieve the desired mechanical and microstructure properties, post-fabrication heat treatment are commonly required for relieving residual stresses and for facilitating the precipitation of strengthening phases as mentioned above. The rapid solidification process at the standard solid solution temperature normally prevents macrosegregation of the likes of Nb completely, but not microsegrgation that can be associated with the formation of dendritic/cellular growth of columnar grains [10]. For optimal precipitation hardening, an effective solid solution thermal treatment is an important step. This may involve tuning of the thermal treatment temperature and hold time for homogenizing the microsegregated particles, including Laves so that abundant amounts of Nb, Ti and Al could be released in the matrix for the precipitation hardening.

Previous reports showed that solution heat treatment under 1100 °C is not sufficient to dissolve segregated particles/phases completely to enhance precipitation of the γ′ and γ″ phases for AM Inconel 718. Zhang et al. [11], for instant studied quantitative analysis of Laves morphology of Laser-Cladded-fabricated sample and obtained about 37% of undissolved Laves phase after standard solution heat treatment (980 °C) and ageing as compared to the concentration of the Laves in as-deposited sample. Similarly, the study by Qi et al. [2] on Laser Net Shape fabricated sample indicates that, solution heat treatment at 980 °C did not dissolve completely the Laves phase. After heat treatment at 1093 °C for homogenization. The same group however found dissolution of most of the Laves phase into the matrix accompanied with recrystallization that led to grain growth with bands of annealing twins. Amato et al. [12] have also reported a considerable volume (> 50%) of recrystallization after annealing SLM fabricated specimen at 1160 °C for 4 h. It is however seldom raised in the literature the effects of solution heat treatment at higher temperature than the standard temperature on the resulting microstructure and hardness in relation to macro- as well as micro-segregation.

In this work, microstructure and hardness of Inconel 718 fabricated by SLM method have been studied prior to and after solution heat treatment at 1100 °C or 1250 °C for different hold times. The aim is generally to analyze the effect of high temperature (1100 °C or 1250 °C) solution heat treatment on the segregated particles and the possible consequences on the microstructure and thus on the mechanical strength of SLM fabricated Inconel 718. The microstructure and composition analysis were studied with Optical Microscope (OM), Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) techniques. Hardness measurement was done with Vickers Hardness tests.

2. Experimental methods

Inconel 718 parts were fabricated from pre-alloyed powder served as precursor using an SLM metal printing machine. The chemical composition of the initial powder was adopted from the standard specification for Additive Manufacturing of Ni alloy (UNS N07718) designated by the ASTM F3055 – 14a [13]. The compositional range in wt% is [13]: Ni(50–55), Fe(11–22.4), Cr(17–21) Nb(4.8–5.5), Mo(2.8–3.3), Ti(0.7–1.2), Al(0.2–0.8), Co(1.0), Si(0.4), Mn(0.4), Cu(0.3). The particle size distribution of the powder is between 10 to 45 µm. A laser beam of 175 W power at a scanning speed of 619 mm/s was used for melting the powder. The printing was done in argon atmosphere to minimize oxidation of the molten pool and to minimize the risk of introducing contaminants. In order to minimize the stresses developed during printing, the building platform was preheated to 200 °C. The layers of the slices processed were about 0.03 mm thick. Inconel 718 cylindrical parts were fabricated in two different directions of printing, i.e., horizontal and vertical. In the horizontal printing (h-print), the printing axis was set perpendicular to the longest axis of the part while in vertical printing (v-print) the longest axis of the part was parallel to the printing axis.

The as-printed samples, h-print and v-print parts have been studied and compared with each other and with that of the solution heat-treated samples. In order to analyze the differences in microstructure and physical properties between the starting and finishing layers of the fabricated parts, two samples were prepared from the v-print parts - one sample from the top region, that corresponds to the layers deposited last and the other sample was sliced from the bottom region which relates to the layers deposited at the beginning of the printing. The results may demonstrate differences in microstructure and physical properties between the starting and finishing layers of the manufactured parts.

In order to explore the effects of elevated temperatures, four h-print samples were solution heat-treated and studied. Two of the samples were heated to 1100 °C – one for 1 h (1100 °C/1 h) and the other for 7 h (1100 °C/7 h). Similarly, the other two samples were heated at 1250 °C, for different durations, one for 1 h (1250 °C/1 h) and the other for 7 h (1250 °C/7 h). The thermal heat treatment was performed in Nabertherm furnace equipped with a K type thermocouple for monitoring the temperature. The samples were water quenched immediately after heat treatment. Descriptions of the samples and heat treatment scheme are given in Table 1.

Hardness tests were performed using a Vickers Struers DuraScan testing machine with 5 kg and 10 kg loads. The samples for this measurement were sliced perpendicular to the longest axis of the parts and polished according to the requirements for Vickers hardness testing. Two samples were prepared from the v-print parts – one from the bottom region and the other from the top region. A minimum of ten tests performed across the surface of each sample at approximately even intervals. The microstructure of the samples was studied with optical microscope and scanning electron microscope, Gemini SUPRA 35VP equipped with EDAX Energy Dispersive X-ray Spectroscopy (EDS) detector. JEOL-2100 (LaB_6 filament) TEM, operating at 200 kV, equipped with EDAX and annular bright and annular dark-field detectors. For TEM analysis, thin foils were prepared, first by thinning down mechanically to a thickness of about 50 - 100 micrometer, and then punched 3 mm disks from the foils. These disks were then electro polished using a dual jet polishing system, Struers TENUPOL-3 operated at −30 °C and 13 V in an electrolyte solution of 80% methanol and 20% perchloric acid. Gatan-made Precision Ion Polishing System (PIPS) that operate at 5 keV with gun angle of +/−8° was also used for preparation of TEM samples.

Table 1
Description of the samples studied and average values of Vickers hardness of the as-printed and solution heat-treated Inconel 718.

Printing orientation/ region	Solution heat-treatment T (°C)/ duration	HV loads (kg)	Vickers hardness (HV)
h-print	As-printed	10	304 ± 9
h-print	1100 °C/1 h	10	258 ± 8
h-print	1100 °C/7 h	5	217 ± 4
h-print	1250 °C/1 h	5	210 ± 7
h-print	1250 °C/7 h	5	207 ± 6
v-print, top	As-printed	10	288 ± 7
v-print, bottom	As-printed	10	301 ± 6