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Metastable carbides and their impact on recrystallisation in IN738LC processed by selective laser melting

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ABSTRACT

Selective laser melting of nickel based superalloys opens up new possibilities for gas turbine engine manufacturers; namely, efficient production of low component volumes, decreased component cost through reduced post-machining procedures and access to new component geometries that cannot be fabricated by conventional processing methods. However, processing high performance nickel-based superalloys components via additive manufacturing, without the occurrence of defects, is challenging and requires a better understanding of the resulting microstructure, especially as different compositions can lead to significant changes in the microstructure obtained. In addition, carefully selected post-processing heat-treatments are required to alter the microstructure and attain the required mechanical properties. In this study, the microstructure of the nickel base superalloy IN738LC has been characterised in the as-deposited and stress relieved heat-treated states, as well as following high temperature heat treatments. The data acquired highlights the influence of the stress relief step on the recrystallisation temperature and its relation with the distribution of carbide particles present in the alloy's microstructure.

1. Introduction

The additive manufacturing (AM) of nickel base superalloys is of great interest to the aerospace industry due to its ability to efficiently fabricate components in low volumes, minimise subtractive processing steps and access complex geometries. Among the wide family of additive manufacturing techniques available, selective laser melting (SLM) has become a preferred technique for the AM of nickel-based superalloys [1–3]. This process works by local melting and solidification of a powder bed in a layer wise manner [4,5]. Compared to other additive techniques, it is particularly suitable for the production of very fine features, such as air cooling channels [6]. This potentially allows the creation of advanced cooling configurations that would permit gas turbine engines to operate at the higher service temperatures required for greater thermodynamic efficiency.

Over the last 20 years, research has been carried out to develop the technology and optimise the AM building parameters for a range of commercial alloys, mainly through changes in laser power, velocity and scan parameters [7,1,2,8,3]. However, the critical deficiencies of AM products are the high levels of residual stresses and high defect densities generated as a result of the extreme temperature gradients associated with this process. In addition, the layer-wise deposition of material leads to the development of high crystallographic anisotropy, which complicates component design and may adversely affect the properties in some directions. Understanding the occurrence of deposition-related defects and how they may be modified or controlled through powder deposition processing is therefore necessary if components fabricated by such methods are to ultimately find use in critical applications in an engine.

As these issues cannot be entirely mitigated through process parameter optimisation, it is generally accepted that post deposition heat treatments are required to tailor the microstructure and achieve the required mechanical properties. It is therefore unsurprising that significant efforts have been directed towards understanding the effects of post-processing treatments [9–14], as well as compositional modifications that make alloys more amenable to processing by additive manufacturing [15]. More recently, demonstration of *in situ* heat-treatments during deposition has been presented by Sames et al. [16]. However, the refinement of the precipitates within the microstructure as a result of the *in situ* heat-treatment was not sufficient to counterbalance the large grain size and presence of cracks which lead to poor macro-mechanical properties. To facilitate these efforts further, studies are

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needed to gain an improved understanding of the origins of the microstructural changes that take place during the overall processing.

Among the nickel-based superalloys, IN738LC offers excellent high temperature strength and hot corrosion resistance [17]. During conventional processing, the mechanical properties of this alloy are controlled through the application of heat-treatments that optimise the γ′ morphology and particle size distribution [18]. However, this alloy contains comparatively high concentrations of aluminium and titanium (> 3 wt% each), which render it difficult to weld and prone to form cracks [19,20]. Whilst cracking is also observed during the direct energy deposition (DED) of IN738LC, it has been reported that a lower laser power can reduce the concentration of defects (interface cracking and grain boundary liquation) [9]. In addition, the use of lower laser powers has led to the identification of parameters that enable the production of defect-free components [9]. In other cases where SLM is used for another crack prone alloy (i.e. CM247LC), the microstructure of this alloy has been reported to be composed of non-uniform coarse columnar grains with very fine directionally solidified dendrites of a supersaturated γ′ phase growing parallel to the build direction [11]. Similarly to the above paper, cracks were also identified in the microstructure and that high laser power was found to be particularly detrimental for crack occurrence. Additional heat-treatments were subsequently applied to precipitate the γ′ phase, which also resulted in removal of the dendritic structure, but the retention of the elongated grain shape.

The high crystallographic anisotropy produced by SLM strongly affects the alloy's properties. Kunze et al. [14] investigated the mechanical response of laser deposited IN738LC after heat-treatment at 1180 °C, just above the γ′ solvus. The resultant microstructures showed no evidence of recrystallisation and retained the original cube texture present in the as-deposited specimens. Consequently, recovery was identified as being the dominant process in the temperature range investigated. The retention of a strong cube texture was also observed to result in a lower yield strength parallel to the build direction than perpendicular to it. In comparison, as-cast specimens exhibited yield strengths intermediate between these two values. Complementary heat-treatment studies on the same alloy and process by Geiger et al. [21] have since shown that recrystallisation occurs at higher temperature, around 1250 °C. This significantly reduced the crystallographic anisotropy originally present in the laser deposited state.

Extending these studies, work has also been undertaken to ascertain the relation between the remnant crystallographic anisotropy and the mechanical properties of SLM heat-treated IN738, and how it is influenced by the scan strategy [14]. Such studies have also shown that scan strategies similar to that used in the present study lead to minimal texture after recrystallisation of other superalloys, e.g. [13].

Selective laser melting has also been shown to generate high residual stresses [22,23], which may influence the integrity of the component and lead to cracking during post-processing [11]. To reduce or eliminate the occurrence of such cracking, additional heat-treatments have been considered that may serve to reduce the residual stress level before subsequent processing is carried out. However, little is currently known about the effect of these heat-treatments on the microstructure and properties. The present study seeks to address this by studying the effect of a stress relieving treatment on the microstructure of SLM IN738LC and its propensity to recrystallise following subsequent high temperature heat treatment. The relation between the extent of recrystallisation and carbides present throughout the microstructure has also been assessed.

2. Experimental techniques and procedure

The nickel base superalloy IN738LC was gas atomised to form a pre-alloyed powder. The nominal composition and powder size distribution are provided in Table 1. Selective laser melting was carried out using a 200 W IPG Yb:YAG fibre laser. Henceforth, the build-up direction will be identified as the *Z*-direction and the laser scan transverse directions contained in the *X*–*Y* plane. The entire process was carried out under an argon atmosphere and the base-plate was a DIN 1.1730 tool steel maintained at 80 °C. Rectangular cuboids of 8 mm × 5 mm × 12 mm were sectioned from the deposited material by electro discharge machining (EDM). More details about the deposition strategy can be found in the work of Divya et al. [24].

Before performing the post-deposition heat-treatments, the as-SLM specimens were cut into smaller, rectangular cuboids measuring 8 mm × 5 mm × 6 mm. Two sets of heat-treatments were carried out on the as-SLM IN738LC. The first set consisted of a standard heat-treatment at high temperature around the γ′ solvus temperature, from 1120 °C to 1230 °C for 2 h. This was followed by an aging heat-treatment of 24 h at 840 °C. Samples in this condition have been referred to as SLM-*HT*, where *HT* is the temperature of the initial heat-treatment performed around the γ′ solvus temperature. The second set, named SLM-SR-*HT*, was identical to the first one, but an initial stress relieving (SR) step of 24 h at 840 °C was included between the deposition and the aforementioned heat-treatments. Every sample was encapsulated in argon-backfilled quartz ampoules to minimise oxidation during the heat treatments and, for each step, the specimens were air-cooled inside their ampoules. To minimise inconsistencies between the thermal cycles experienced by the samples, the heat-treatments were performed in the same furnace and its temperature was controlled with a type-K thermocouple. Hence, the temperatures quoted are accurate to ± 4 °C, corresponding to the variation existing within the furnace. One sample was subjected to heat-treatment at 1220 °C for 4 h following the SLM processing to assess the effect of longer duration heat-treatment on the recrystallisation behaviour.

Conventionally cast (CC) IN738LC, supplied by Access e.V, was acquired in order to permit direct comparison between the SLM and CC microstructures. This material was subjected to a standard heat-treatment of 1120 °C for 2 h, followed by 845 °C for 16 h; both steps were followed by air-cooling [25,26].

2.1. Differential scanning calorimetry

Differential scanning calorimetry (DSC) was carried out to determine the phase transformation temperatures of IN738LC in the different microstructural states using a Netzsch 404 calorimeter. Samples for the thermal analysis consisted of circular discs, ~5 mm in diameter and ~1 mm thick. The measurements were carried out in an argon atmosphere, with a flow rate of 50 mL min^{-1}. The heat flux was recorded as the samples were heated at 10 °C min^{-1} from room temperature to 1400 °C, before cooling back to room temperature at the same rate. To differentiate between effects associated with the thermo-mechanical history to those related to alloy composition, the specimen was tested a second time with the same parameters. For each result, the data acquired was corrected against data acquired from an empty crucible. The phase transformation temperatures were determined during the heating cycle following the methodology defined in [27].

2.2. X-ray diffraction

X-ray diffraction was performed to investigate the phases present in the alloy during the various heat-treatments using a Bruker D8 X-ray diffractometer equipped with a Cu-K_α source operated at 40 kV and 40 mA. A nickel filter, 0.0012 mm thick, was used to suppress the K_β peaks. The specimens were spun at 30 RPM during data acquisition in order to improve counting statistics. The scans were performed across the same angular range (2θ) from 20° to 80° with a step size of 0.05° and a dwell time of 5 s. Pawley refinement of the data acquired was performed to evaluate the lattice parameters of the phases present. This method was rendered necessary due to the presence of texture in the specimens.