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Microstructure and yield strength of SLM-fabricated CM247LC Ni-Superalloy

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ABSTRACT

Specimens of Selectively Laser Melted (SLM) CM247LC powder have been characterised using analytical scanning and transmission electron microscopy. In specimens from the bulk, it has been found that longitudinal sections consist mainly of columnar γ grains, containing virtually identically oriented cells, approximately 700 nm in width and length up to hundreds of microns. These cells are separated from adjacent cells and from adjacent grains by γ′/γ eutectic, by high densities of Hf/Ti/Ta/W-rich precipitates and high densities of dislocations. The eutectic γ′ is up to about 50 nm in diameter but up to 10 nm within the cells. The microstructure in the top layer is similar to that taken from the bulk, but single-track samples are heterogeneous along the track length. The microstructures are interpreted in terms of the precipitation sequence, the volume fraction of eutectic and partitioning of the solute elements during solidification and the influence of subsequent laser-tracks. The cooling rate during solidification, calculated from the observed cell diameters is about 10^6 K/s, but the value obtained from the size of γ′ within the bulk is about 10^4 K/s. It is suggested that the discrepancy is due to the limited accuracy of this approach. Tensile tests on as-fabricated samples show that the yield strength is comparable with that of cast samples after standard heat treatments. The high strength of the as-fabricated samples is interpreted in terms the high densities of precipitates and dislocations in cell boundaries, the fine cell structure and the γ′ within cells.

1. Introduction

The significant advantages, in producing near-net shape components with short lead time and low wastage of materials, have driven research into the application of Selective Laser Melting (SLM) in many areas such as aero-engine components and medical implants [1,2]. A considerable amount of work has been published on the response of a range of alloys to SLM [3–5] and a great deal of work has been carried out aimed at optimising the process conditions to minimise the density of pores and cracks formed during SLM. Studies have also shown that both cracks and pores can be further minimised by post-process Hot Isostatic Pressing (HIPping) [6–8].

In Ni-base superalloys, the precipitation of large amounts of γ′ provides precipitation hardening and excellent high-temperature mechanical properties [9–11]. Radis et al. [9] reported that the higher cooling rate during continuous cooling of UDIMET 720 Li from above the solution temperature, gives finer γ′ precipitates, which are more spherical and are of uniform size. In a similar study carried out on the CM247DS alloy [12], it was found that water-quenched (~10^3 K/s) samples show a much higher density, finer size (~40 nm), more spherical and a more uniform distribution of γ′ precipitates than those in samples cooled at 75 K/s. Kusabiraki et al. [13] reported that the effective cooling rate to avoid the precipitation of γ′ in Nimonic 105 needs to be higher than 10^4 K/s.

In the case of high γ′-fraction Ni-base superalloys processed by SLM (e.g. CM247LC), where it is accepted that the cooling rates are high, the occurrence of cracking is a dominant feature due to the high residual stresses that result from rapid cooling and the susceptibility of these alloys to various cracking mechanisms [14–18]. In contrast to comprehensive studies on SLM process-optimisation to reduce the cracks and pores observed in the builds, only a limited amount of work has been published on the development of the microstructure in laser-processed materials. The work that has been published has focused on optical microscopy and scanning electron microscopy (SEM), although very recently, Divya et al. [4] used transmission electron microscopy

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(TEM) to investigate the microstructure of CM247LC formed during SLM processing, but their interpretation of the observed microstructures differs from that in the present paper. There is also a lack of information concerning the cooling rates and data on the size and distribution of γ′ precipitates in SLM-processed CM247LC, although several studies suggest cooling rates $>10^5$ K/s [19,20]. Since precipitates, in the as-fabricated condition, can range in size down to a few nm [21], characterisation of the structures requires TEM. Earlier studies have related the primary Dendrite Arm Spacing (DAS) λ_1 (equivalent to cell spacing) to the cooling rate ($\dot{T}$) experimentally and theoretically, deriving Eq. (1) [22]. Harrison et al. found $|\dot{T}|$ ~3×10^5 K/s with primary DAS of 0.9–1.2 μm during SLM of Hastelloy X [1].

$$\lambda 1 = 97(\pm 5)\dot{T}^{-0.36(\pm 0.01)} \quad (1)$$

The terminology, which will be used throughout this paper, will be explained, as was done in the recent paper [23] on AlSi10 Mg, because different authors have used “cells” to describe different microstructures in SLM-processed samples. The terminology here will follow that used to describe the microstructures developed during solidification of alloys at different cooling rates [24]. These cells are similar to dendrites with effectively no secondary arms (very short secondary arms may well be formed) that grow into the liquid, forming an array of parallel cells of the same orientation if they originate from the same nucleus. Grain boundaries are formed when groups of cells growing from different nuclei meet.

The aim of this investigation is to characterise the microstructure and tensile properties of SLM-processed CM247LC by carrying out a detailed assessment of the microstructure in SLM-processed samples. This work has allowed the cooling rates occurring during solidification to be estimated through measurement of the cell width (equivalent to primary dendrite arm spacing) and the cooling rate in the solid (below the γ′-solvus) by measurement of the γ′-size in Jominy end-quenched samples, in samples that were water quenched with a thermocouple insert and in as-fabricated samples. The yield strength of these as-fabricated samples have been measured and compared with cast and heat treated CM247LC [25].

2. Experimental

The CM247LC argon gas atomised powder (+15–53 μm), the composition (wt%) of which is listed in Table 1 was supplied by LPW Technology Ltd. (UK). Samples were fabricated using a ‘Concept Laser M2 Laser powder Bed’ machine [7], with a laser power of 150 W, a scan speed of 1500 mm/s, dimensionless scan spacing factor, (scan spacing (μm)/laser track width (μm)) of 0.3 and island scanning strategy with an island size of 5 mm. The track width is assumed to be 150 μm. These conditions selectively melt successive 20 μm thick layers of powder.

To assess the role of cooling rate on the size of γ′, Jominy end quenching tests [26,27] have been carried out on samples with dimensions according to [26]. The test bar was heated up to a solution treatment temperature of 1533 K and held for 2 h before quenching. The Jominy quenching apparatus directs a water column to quench the end of the test bar. The very end of the test bar (<500 μm distance from surface) was prepared for TEM observation. Water quenching of a 10 mm cubic sample with an embedded K-type thermocouple, with an acquisition rate of 100 Hz, was also performed to measure the actual cooling rate. Measurements, of the size of γ′, was used to assess the cooling rate below the γ′-solvus in SLM-processed samples.

Table 1
The chemical composition of supplied powder (wt%).

Cr	Co	Mo	W	Ta	Ti	Al	Hf	C	B	Zr	Ni
8.31	9.15	0.54	9.4	3.2	0.73	5.62	1.28	0.07	0.02	0.01	Bal.

Metallographic samples were prepared for both the longitudinal and transverse planes by sectioning both along the building direction (Z) and the laser-scanning plane. Samples, which were ground and polished to 0.05 μm oxide suspension finish, were observed using a Philips XL-30 SEM operated at 20 KV to obtain Electron Back Scatter Diffraction (EBSD) maps and a JEOL 7000 operated at 20 KV to acquire secondary and backscattered electron (BSE) images. 3 mm diameter discs from longitudinal and transverse sections for TEM were spark eroded and ground down to 150 μm which were twin-jet electro-polished at −20 °C with a solution of 10% perchloric acid and 90% methanol using a Struers Tenupol-5 system.

Since SLM is a layer-wise process, transverse samples were prepared both from the bulk of SLM-processed samples and from the uppermost surface layer in order to assess the influence of the repeated thermal cycles on the initial microstructure by comparing the as-solidified microstructure of the top layer with the remelted/reheated microstructure taken from the bulk samples. Since the microstructure of the top layer sample may result from remelting of tracks by adjacent laser tracks during processing, the microstructure of a single track sample has been characterised and compared with that in the top layer and bulk. A rectangular sample of CM247LC (20 × 20 × 5 mm) was built as a substrate, and single track (10 mm in length) was scanned across the top of substrate, which had been recoated with a layer of powder prior to the single laser track. The single-track specimen (with the substrate) was sectioned perpendicular to the laser scanning direction followed by mounting, grinding and polishing. The cross section of single track is around 150 μm thick and FIB (Focused ion beam) thinning was used to extract a TEM sample (14 × 14 μm) from the single-track sample.

A 200 kV JEOL TEM interfaced to an Energy Dispersive X-ray Spectroscopy (EDX) facility has been used for assessment of the orientation relationships between cells and for assessment of the detailed microstructures. Diffraction patterns were taken either using an aperture and a defocused electron beam to define the area selected (selected area diffraction S.A.D.) when high angular resolution patterns were required, or using a small focused probe when the area had to be accurately defined (micro-diffraction patterns), to determine the orientations of adjacent cells. Some EDX analyses were carried out in the JEOL 2100, using a focused probe on selected areas.

A TALOS Scanning Transmission Electron Microscopy (STEM) operating at 200 kV has also been used to carry out microstructural assessment and to obtain X-ray maps to assess the compositions of the precipitates and to measure the extent of segregation to cell and grain boundaries. To complement X-ray maps, the probe was focused on specific areas for longer counting times than are used when obtaining X-ray maps in order to obtain more accurate local data. Measurements of the size of γ′ precipitates in bulk, top layer and single track samples, used to estimate cooling rates below the γ′ solvus, were carried out using ‘feret’ in ImageJ software where all precipitates are considered to be spherical of diameter defined by the largest dimension. To avoid background noise, no attempt was made to measure precipitates smaller than 3 nm.

Tensile tests at room temperature were carried out by Westmoreland Mechanical Testing & Research, Ltd. on as-fabricated samples, according to ASTM E8-15a [28], to allow comparison with the strength of cast and heat-treated CM247LC. Three tensile specimens were tested in the Z-direction.