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Influence of heat treatments on microstructure evolution and mechanical properties of Inconel 625 processed by laser powder bed fusion

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ABSTRACT

This study investigated the mechanical behaviour and microstructure of as-built and heat-treated Inconel 625 (IN625) samples processed by laser powder bed fusion (LPBF). This process offers freedom in design to build complex IN625 components in order to overcome extensive machining. However, post heat treatments must be performed to obtain specific mechanical properties to match industrial requirements. For this purpose, different heat treatments were performed on IN625 samples, and through hardness measurements, three different heat treatments were selected, as optimised conditions. A direct ageing, a solutioning and a solutioning followed by ageing treatments were chosen to study the effects of these specific heat treatments on the microstructure and tensile properties, comparing them to those of as-built condition. The tensile properties of as-built and selected heat-treated IN625 samples showed superior values to minimum requirements for wrought IN625 alloys, whereas the investigation on the microstructures and fracture surfaces of as-built and heat-treated IN625 contributed to an understanding of the tensile properties evolution. The high tensile strength of as-built samples essentially derived from very fine dendritic structures mainly below 1 µm with high dislocation density and nanometric MC carbides. The high tensile properties of ageing treatments performed at 700 °C for 24 h, whether directly aged or post-solutioning, were found to be primarily dependent on γ" phases (10–30 nm) and $M_{23}C_6$ carbides formation. By contrast, the tensile properties of solution-treated IN625 samples at 1150 °C for 2 h showed higher ductility coupled to lower strength than other conditions, due to the grain growth.

1. Introduction

Inconel 625 (IN625) is a solid-solution strengthened nickel-based superalloy derived from refractory elements including niobium and molybdenum, in a nickel-chromium matrix. The high temperature strength, corrosion resistance as well as an excellent weldability offered by IN625 has led to its application in different areas such as aeronautics, aerospace, marine, nuclear, chemical and petrochemical industries [1–3]. However, machining IN625 components into elaborate shapes is remarkably difficult and expensive by subtractive manufacturing, due to the material is characterised by high hardness and high-temperature strength as well as low thermal diffusivity [4–6].

Nowadays, it is possible to overcome the obstacles and problems related to traditional subtractive manufacturing by means of additive manufacturing (AM) technologies [7]. AM in fact has attracted massive interest due to the possibility to use a layer by layer process to build near-net shape components with very complex geometries combined to a high efficiency of material usage [8]. One of the main AM technologies used to build nickel-based superalloy components is laser powder bed fusion (LPBF) process, also known as selective laser melting (SLM). In this process, consecutive layers of loose powders are melted by a focused laser beam in specific areas according to the computer-aided design (CAD) data [8–10]. In literature, several works are concentrated on understanding the phenomenology of the process as well as the influence of process parameters to generate dense components [10–14]. In particular, for IN625 alloy produced by LPBF, Sateesh et al. [15]

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studied the interaction of different parameters on dimensional accuracy, micro hardness and densification levels. Carter et al. [12] focused their attention on the energy density necessary to obtain IN625 components with a relative density close to 100%, whereas Wang et al. [16] fabricated IN625 samples with a relative density of 99.95%. A previous work presented by Marchese et al. [17] investigated the effect of a combination of various parameters on densification level and Brinell hardness, which led to a relative density as high as 99.96% and IN625 microstructures with very fine dendrite structures. Yadroitsev et al. [18] mainly evaluated the impact of different hatching distance values and scanning strategies, reaching a residual porosity lower than 1%. Moreover, they assessed that as-built IN625 samples had higher tensile strength and lower ductility compared to wrought IN625 alloy.

The high tensile properties of LPBF superalloys can be attributed to the extremely fast solidification rates (10^5–10^6 °C/s) involved in the LPBF, generating a fine dendritic architectures and the suppression of macro segregation or formation of large Laves phases or carbides that can be detrimental to the mechanical properties [19–21]. On the other hand, these microstructural features as well as the high thermal residual stresses induced by the process are not desired for high temperature applications [19,21,22]. It is therefore necessary to perform post-processing heat treatments [10,21–24]. In fact, heat treatment is a key factor to reduce residual stresses, to eliminate the segregation of elements, to promote grain growth and phase precipitation, thus obtaining specific microstructure and mechanical properties suitable for different conditions. For instance, Li et al. [22] studied the microstructure, texture, grain morphology and hardness of as-built and heat-treated IN625 for short heat treatments. Brown et al. [25] investigated the tensile properties of stress relieved IN625 specimens built by different LPBF machines, showing that the tensile properties can change based on the basis of the different building strategies of the used machines, because of different thermal flows. Witkin et al. [26] studied the tensile and fatigue properties of as-built and hot isostatically pressed (HIPed) IN625 LPBF specimens compared with standard wrought IN625. They showed that HIPed IN625 samples have lower strength and higher ductility than as-built ones and that their fatigue behaviour can be clearly improved by machining or shot peening of the surface. Kreitcberg et al. [27] studied the microstructure of the as-built, three heat-treated and HIPed IN625 samples, mainly showing the grain size and morphology evolution correlated to the tensile properties. In a successive work Kreitcberg et al. [28] chiefly focused on the microstructure of heat-treated LPBF IN625 samples and their impact on the elevated temperature tensile properties.

However, so far very little attention has been paid to the microstructural evolution of LPBF as-built IN625 parts under different heat treatments, investigating the microstructure development coupled with the influence on hardness and tensile properties. In fact, a similar investigation is crucial to better understanding whether the very particular microstructures of LPBF parts require different heat treatments with respect to the traditional IN625 alloys. Therefore, in the present study, the mechanical behaviour at room temperature of as-built and heat-treated IN625 samples were correlated to their microstructural features and tensile fracture surfaces. The tensile properties of these IN625 samples were compared to those of wrought IN625 alloys in order to show the effect of selected heat treatments on rapidly solidified IN625 derived from LPBF process.

2. Materials and methods

2.1. IN625 specimens built by LPBF

In this study, gas atomised IN625 powder by EOS GmbH (Germany) company was employed. The powder chemical composition declared by the supplier is reported in Table 1, and it is in accordance with UNS N06625.

The powder consisted of fairly spherical particles with some satellites and clusters as shown in Fig. 1a. The particle size distribution had a d_{10} of 16 µm and d_{90} of 48 µm and the powder presented a good flowability, as reported in a previous study [17]. Test samples were built using an EOSINT M270 Dual Mode machine, which is equipped with a 200 W Yb fiber laser with a spot diameter of 100 µm. The process was carried out in a protective argon atmosphere and the building platform was held at 80 °C. The optimised operative parameters were: laser power of 195 W, scan speed of 1200 mm/s, hatching distance of 0.09 mm and layer thickness of 0.02 mm. The EOS scanning strategy using stripes of 5 mm and an overlapping of 0.12 mm with the laser scanning direction rotated of 67° between subsequent layers was employed [17]. IN625 cubic ($15 \times 15 \times 15$ mm^3) and cylindrical (length of 110 mm and diameter of 15 mm) samples were fabricated. The applied parameters enabled the production of cubic and cylindrical specimens with a residual porosity less than 0.1%. The cubic samples were used to study the microstructure of as-built and heat-treated IN625 and also to determine Brinell hardness. The cylindrical samples, on the other hand, were machined to obtain tensile specimens, with a gauge length of 40 mm, a diameter of 8 mm a length of reduced section of 51 mm as well as radius of fillets of 8 mm, consistent with ASTM E8/E8M-09. It should be noticed that the cylindrical samples were built parallel to the building platform (xy plane), as illustrated in Fig. 1b.

2.2. Heat treatment conditions

The as-built IN625 samples were subjected to various heat treatments: direct ageing, solution treatments as well as solution followed by ageing treatments for different temperatures and times, as schematised in Fig. 2. The direct ageing and ageing treatments were carried out at 600, 700, 800 and 900 °C for different times up to 24 h, whereas the solutioning were carried out at 1000 °C and 1150 °C up to 2 h.

It should be noticed that all these heat treatments were performed using a muffle furnace with a ± 5 °C precision, equipped with a K-type thermocouple located close to the samples. Furthermore, all the heat-treated IN625 samples were water quenched (WQ) in order to avoid any microstructure evolution during cooling, as recommended in the literature for large sections [29].

2.3. Microstructure and mechanical characterisation

The as-built and three chosen heat-treated LPBF-IN625 samples were cut along the z plane (parallel to the building direction), subsequently ground and polished down to 1 µm using SiC abrasive papers and diamond suspensions, and then etched using Kalling's No.2 etchant (5 g $CuCl_2$ in 100 ml HCl and 100 ml CH_3CH_2OH) or mixed acids (15 ml HCl, 10 ml CH_3COOH and 10 ml HNO_3). Afterwards, the microstructures were investigated by means of optical microscope (OM - Leica DMI 5000 M), by Field Emission Scanning Electron Microscope (FESEM - Zeiss SupraTM40), and by Field Emission Transmission Electron Microscope (FETEM - JEOL JEM-2100F operating at 200 kV) equipped with Energy Dispersive X-ray Spectroscopy (EDS) detector. The fracture surface of tensile samples were observed by Scanning Electron Microscope (SEM - Leo 1450VP).

A Brinell hardness test was applied to provide a good understanding of the relative level of strength of the IN625 alloy underwent different heat treatments. The Brinell hardness tests were carried out on all the samples polished up to 4000 fine grit SiC paper along the z plane, by means of an EMCO TEST M4U test machine with a load of 62.5 kg_f for 15 s (HBW2.5/62.5), in accordance with the ASTM E10–14 standard. The hardness values were assessed using three samples, five indentations on each sample.

Tensile specimens were tested by a Zwick-Roell BT1 - FR100 testing machine at room temperature using a strain rate of 8.10^{-3} s^{-1} following the ASTM E8/E8M-09 standard. The tensile tests were performed at least on three specimens for each condition.

Engineering strain and stress obtained by tensile test at a fixed strain