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Effect of heat treatment and hot isostatic pressing on the microstructure and mechanical properties of Inconel 625 alloy processed by laser powder bed fusion

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ABSTRACT

The effect of different heat treatments and hot isostatic pressing on the microstructure and mechanical properties of laser powder bed fusion IN625 alloy was studied. The heat treatments were: stress relief annealing, recrystallization annealing and low-temperature solution treatment. The resulting microstructure and crystallographic textures were studied using optical and scanning electron microscopy. The mechanical properties of the as-built and post-treated IN625 alloy were obtained after tensile testing at room temperature and at 760 °C (1400 °F), and compared to those of an annealed wrought alloy of the same composition.

1. Introduction

Laser powder bed fusion (L-PBF) technology allows the production of parts on demand with complex geometry and is applicable to different classes of metallic materials including Ti, Ni, Co, Al and Fe-based alloys [1–5].

The L-PBF processing of metallic parts involves three main phases: (1) preparation phase, (2) L-PBF of metallic powders and (3) post-treatment. The first phase involves the orientation of a 3D CAD part model with respect to the building platform, data conversion into an STL format, and model slicing. The L-PBF of metallic powders (second phase) comprises layer-by-layer powder consolidation, a complex thermal processing involving localized melting, solidification and cooling of spread powder particles. This non-equilibrium laser processing triggered by a high localized heat input, and involving short interaction times, determines the material integrity and homogeneity, microstructure and mechanical properties, which are different from those resulting from conventional manufacturing processes [6–11]. Therefore, the final phase, post-treatment, serves to attenuate the negative consequences of the layer-by-layer selective laser powder consolidation, such as high level of internal stresses and anisotropy of mechanical properties, while preserving the capacity to produce components with complex geometry.

This post-treatment phase is especially important for high-performance components used in the aeronautics, nuclear, chemical and petrochemical industries. One of the most promising alloys used in these fields is Inconel 625 due to its high corrosion resistance and mechanical properties at elevated temperatures [12,13]. Since, according to [1], the mechanical properties of L-PBF IN625 components are highly anisotropic, significant effort must be made to optimize post-L-PBF operations with the objective of decreasing the level of anisotropy while preserving the high mechanical properties of this alloy under service conditions.

Four post-treatment schedules can be recommended for L-PBF IN625: 1) stress relief (SR) annealing, 2) recrystallization annealing (RA), 3) solution treatment (ST) and 4) hot isostatic pressing (HIP).

- Stress relief annealing is recommended for L-PBF parts to reduce residual stresses and therefore decrease distortions before cutting the parts from the building platform, but without significantly affecting the grain structure. For nickel alloys, stress relief temperatures range from 650 to 870 °C, but this operation runs the risk of experiencing carbide precipitation phenomena and other phase formation [14,15].
- Recrystallization annealing of L-PBF parts is intended to produce a recrystallized grain structure and reduce the anisotropy of mechanical properties. For nickel alloys, the recrystallization annealing requires temperatures ranging from 930 to 1040 °C [15–17].
- For nickel alloys, a solution treatment at 1040–1200 °C serves to dissolve carbides and δ-phase precipitates before age-hardening treatment [14,15].
- Hot isostatic pressing (1120–1240 °C, 100–165 MPa, 3–4 h) allows

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the reduction of the microporosity and a decrease in anisotropy of the alloy's mechanical properties [18,19].

The initial (as-built) microstructure, level and distribution of residual stresses in L-PBF-processed parts influence the precipitation kinetics and grain structure evolution triggered by post-L-PBF thermal treatments and HIP, and, therefore, impact the resulting mechanical properties. To date, the effects of thermal treatments and HIP on the anisotropy of the mechanical properties of L-PBF-processed IN625 alloy have undergone much less intense study than those of other nickel-based superalloys, such as IN718 [1]. Thus, the main purpose of the experiments described in this paper was to study the structural changes occurring during thermal and HIP post-L-PBF treatments of the IN625 alloy and assess their influence on the mechanical properties of this material. The experimental results should allow the optimization of the post-treatment schedule of L-PBF-processed IN625 alloy components, with the main objective being to improve the homogeneity of their microstructure and mechanical properties. Note that in this work, the exact heat and HIP-treatment conditions are omitted and the stress values measured are normalized to protect proprietary partner information.

2. Material and methods

An EOSINT M290 (EOS GmbH, Munich Germany) selective laser melting machine equipped with a Ytterbium 400 W fiber laser and an EOS IN625 Performance Setup were used to manufacture two types of specimens: 10×10×10 mm^3 cubic specimens for microstructure evaluation and 85×18×3 mm^3 rectangular prismatic coupons with three build orientations (ZX/vertical, XZ/horizontal and 45°/inclined), for mechanical testing (Fig. 1a).

Next, directly after L-PBF, some specimens were cut from the platform to evaluate the as-built microstructure and mechanical properties. The specimens remaining on the building platform were subjected to stress relief annealing (SR) [15,20]. Then, all the SR-annealed specimens were cut from the platform, and the rectangular prismatic coupons were machined to obtain dumbbell-shaped tensile testing specimens shown in Fig. 1b. Some specimens were then reserved for future study. The other specimens were subjected to three post-treatments: recrystallization annealing (RA), solution treatment (ST) [15,20], and HIP [18,19,21]. All the treatments were conducted under argon atmosphere. After post-treatment, RA and ST specimens were water-quenched, SR specimens were air-cooled and HIP specimens were furnace-cooled.

It was hypothesized that the RA and ST post-treatments would reduce the anisotropy of the as-built alloy's mechanical properties, but to a different extent, whereas HIP post-treatment would additionally strengthen the material by eliminating the L-PBF-generated microporosity [16,17]. All the concurrent thermomechanical treatment schedules are schematically presented in Fig. 2. For reference, annealed wrought IN625 alloy (ASTM B443) with a grain size of 20 µm provided by McMaster Corp. was also mechanically tested in the framework of this study.

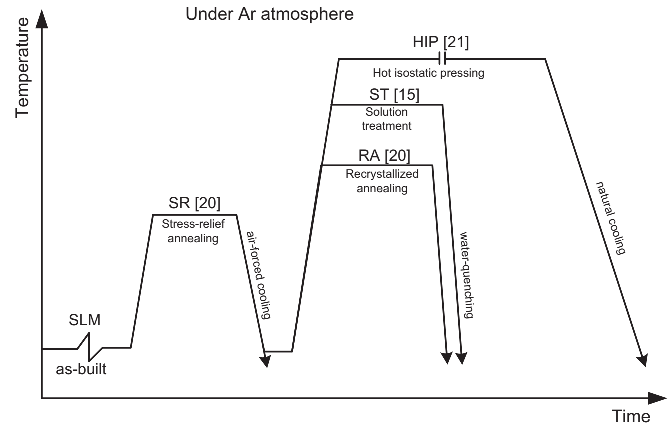

Fig. 2. Specimens designation and corresponding thermomechanical treatment (TMT) routes.

For microstructure analysis, an optical microscope (OM, Olympus 3D laser microscope) and scanning electron microscope (SEM, Hitachi SU8230) equipped with an electron backscatter diffraction (EBSD) unit were used. The horizontal (XY) and vertical reference (ZX) faces of the cubic specimens were polished manually (1 µm grit size), and then by using a vibrometer and colloidal silica (0.05 µm grit size). For OM, the specimens were electro-etched for 1–4 min under 2–5 V potential in a 70 mL HPO_3+30 mL water solution. Grain size was measured according to ASTM E112–13 (linear intercept method, 700–1000 grains for statistics), using OM images for the horizontal plane (XY). The texture evolution was characterized using EBSD, orientation contrast imaging and integrated ARGUS FES/BSE imaging. Specimens were tilted by 70° and scanned at 20 kV, with a 1–2 µm step. This analysis was performed using 1×1 mm^2 and 0.5×1 mm^2 mapping areas for horizontal and vertical planes, respectively. Inverse pole figures were calculated using a 1×1 mm^2 area to eliminate local heterogeneities of the specimens.

The dumbbell-shaped specimens (Fig. 1b) with a 2×4 mm^2 gauge section were tested in tension at room temperature (RT) and at 760 °C (1400 °F). The tensile tests were performed under a constant strain rate of 10^{-3} s^{-1} using an MTS 810 system. For RT testing, this system was coupled with an MTS 634.12 contact extensometer (initial gauge length of 20 mm). For high-temperature testing, the specimens were heated up to 760 °C with a 1 °C/s rate, using an infrared radiant heating chamber (Fig. 3a), and then maintained for 10 min at this temperature before testing. The thermal field uniformity was assessed using three K-type thermocouples distributed along the specimen gauge length and placed in contact with the specimen, the central thermocouple being used for temperature control (Fig. 3b). The high-temperature testing was performed under Ar atmosphere with a flow rate of 5–18 L/h.

Three specimens were tested for each build orientation. The relative orientation dependence of mechanical properties as a function of the

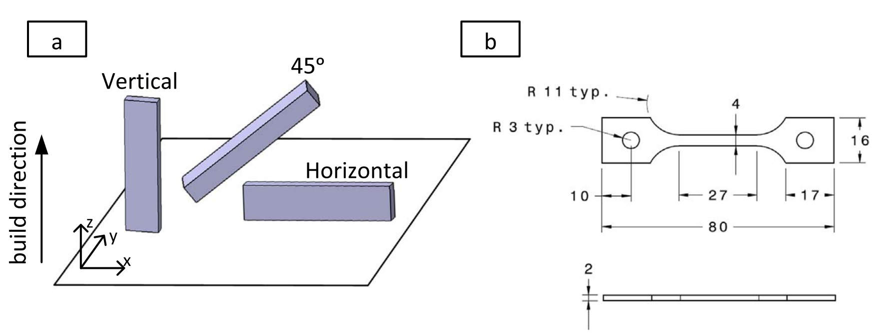

Fig. 1. (a) Specimen orientations during processing; (b) Tensile specimens (dimensions in mm).