On the hot isostatic pressing of Inconel 625 structures built using laser powder bed fusion at higher layer thickness

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Abstract
This paper reports the effect of hot isostatic pressing (HIP) on the porosity, microstructure and mechanical properties of laser powder bed fusion (LPBF) IN625 structures built at a higher layer thickness of 100 µm. It is observed that the process-induced pores/voids of volume fraction ($V_f$) 0.43% in as-built IN625 structures are reduced significantly to ~0.01% after HIP treatment. The microstructure is changed from fine columnar dendrites to coarse equiaxed dendrites. The microstructural analysis of as-built structures reveals the presence of cellular/dendritic growth along with elemental segregation of Nb, Si and C and precipitation of Nb-rich carbides, whereas coarse recrystallized microstructure along with elemental segregation of Si and precipitation of Nb, Mo and Cr rich carbides is observed in hot isostatic pressed (HIP) samples. HIP structures exhibit lower tensile strength, higher ductility and lower anisotropy as compared to LPBF built structures. There is a reduction in the Vickers micro-hardness of IN625 samples after HIP, and the values are observed to be similar to their conventional counterparts. Further, an increase in the energy storage capacity of the material is observed after HIP treatment through Automated Ball Indentation (ABI®) studies. The study paves a way to develop ~100% dense, defect-free and isotropic engineering components using LPBF.

Keywords Laser additive manufacturing · Laser powder bed fusion · Inconel 625 · Hot isostatic pressing (HIP) · Characterization

1 Introduction

Ni-based superalloys are the preferred choice of deployment in applications involving extreme duty conditions due to high mechanical strength, corrosion resistance, oxidation resistance and creep strength at elevated temperature (up to ~0.7 times of the melting point) [1]. Inconel 625 (IN625) is one of the Ni-based superalloys that finds wide applications in marine, automotive, aerospace and nuclear industries [2]. The alloy is a solid solution strengthened with the addition of Fe and Nb into the Ni matrix and contains Cr and Mo for improving the corrosion resistance and high-temperature strength, respectively [2, 3]. IN625 has excellent weldability with minimal cracking susceptibility and can be deployed for applications ranging from 0 to 1000 °C [2]. However, one of the issues associated with the use of IN625 for many engineering applications is its difficulty in machining due to work-hardening during metal-cutting operation. Machining of IN625 block needs machining parameters in narrow window and selection of appropriate tools for circumventing the effect of work hardening and chatter, for reduction of residual stress build-up during machining and for slowing down the wear of the tools and machines [2]. It necessitates adopting advanced manufacturing techniques, like additive manufacturing for building near-net-shaped engineering components and avoiding excessive machining of IN625. There are many additive manufacturing techniques...
including laser-directed energy deposition [4], laser powder bed fusion [5], electron beam-based additive manufacturing [6] and wire-arc-based additive manufacturing [7] that can be deployed for these applications.

Laser powder bed fusion (LPBF) is one of the most commonly used additive manufacturing processes in industries to build complex-shaped engineering components due to the shape design freedom offered by the technology [5]. It starts with a 3D model sliced into several layers (2D sections), and material addition is carried out in a layer-by-layer sequence by selective fusion of the pre-placed material using high power lasers [8]. LPBF provides the additional advantages of reduced tooling, geometric freedom and mass customization over conventional manufacturing processes (i.e. machining or forming) [9, 10]. However, LPBF suffers from inherent processing defects, such as internal cracking, porosity, high residual stress and high surface roughness, which can reduce the mechanical integrity of LPBF built parts [11]. These defects in LPBF can be resolved by adopting an appropriate manufacturing strategy including manoeuvring process parameters/conditions and deploying suitable post-processing techniques [2, 12]. As it is not viable to mitigate all the defects only by manoeuvring process parameters/conditions, various post-processing techniques are deployed [13].

HIP is one of the post-processing techniques that are often employed for laser additively manufactured components to eliminate defects (such as lack of fusion and cracks) and to reduce the gas generated porosity to a great extent [14]. In this technique, the LPBF built parts are subjected to an elevated temperature and high pressure simultaneously for a pre-defined time period. This facilitates the plastic deformation and the visco-plastic flow of the material resulting in the closure of the pores and the cracks present in the part [15, 16]. Argon (Ar) is generally used for pressurizing due to its inert nature. The pressure is applied from all directions equally making the process ‘isostatic’. The primary governing parameters of HIP are inert atmosphere, temperature, pressure and cycle time. The pores having trapped gases are not mostly closed completely by the HIP. This is because as the pores get smaller, the pressure exerted by the trapped gases on the inner walls of the pores builds up. The closure of pores stops when the net force due to the internal pressure from trapped gases equals the force due to the externally applied pressure. On the other hand, the defects without trapped gases, i.e. the voids or cracks formed during the LPBF, can be closed completely by HIP [17]. Depending on the temperature of the process, HIP can also affect the microstructure and dislocation density in the as-built part, which in turn contributes to the change in its mechanical properties. The list of potential components that can use LPBF followed by HIP are gas turbine blades, fuel rocket engines, etc. requiring specific properties enabling their operation in hostile conditions [2].

Many researchers are reporting the deployment of LPBF built IN625 parts with post-processing for various engineering applications. Li et al. found that no precipitation of carbides or other phases occurred in the as-built samples (layer thickness = 20 μm). The heat treatment (annealing performed at 700, 1000 and 1150 °C, respectively, for 1 h followed by still air-cooling) caused a reduction in the lattice constant due to the precipitation of a large number of NbC and MoC carbides. The MC carbides formed due to heat treatment resulted in the formation of a zig-zag grain boundary that was crucial for the alloy [18]. Marchese et al. investigated the effect of various heat treatments on the mechanical behaviour and microstructure of the IN625 samples built by LPBF (layer thickness = 50 μm) and compared the mechanical properties of the as-built samples and the heat-treated samples. It was observed that in the as-built samples, the presence of high dislocation density and the eutectic reactions resulted in the formation of very fine Nb-rich MC carbides (10–50 nm) in dendritic cores. Samples that are directly aged at 700 °C for 24 h did not show any significant changes in the microstructure. However, very fine (10–30 nm) discoidal γ′ phases and discontinuous and elongated Cr-rich M23C6 carbides were detected at grain boundaries. It was also observed that the strength and ductility of the material increased and decreased, respectively. Solution treatment at 1150 °C for 2 h resulted in grain coarsening due to recrystallization, recovery of dislocations and dissolution of dendrites yielding a decrease in strength and increase in ductility. Fine intergranular Nb, Ti-rich MC carbides were also detected. Heat treatment involving solutionizing (1500 °C, 2 h) followed by ageing (700 °C, 24 h) resulted in recrystallized microstructure along with γ′ and Cr-rich M23C6 carbides leading to increased strength and decreased ductility as compared to the solutionized samples [12]. Kretsch et al. studied the effects of stress relieving + HIP on the microstructure and mechanical properties of LPBF built IN625 (layer thickness = 50 μm). It was observed that HIP of LPBF built samples resulted in the dissolution of M23C6 carbides during stress relief treatment and formation of MC carbides along with equiaxed microstructure. It was observed that the anisotropy in the microstructure resulted in the lowest strength and highest elongation to failure in the vertically built specimens. However, the highest strength and lowest elongation to failure was observed for samples oriented along 45° to the build direction and along the laser scan direction, respectively. The room temperature strength (yield strength and ultimate tensile strength) and elongation to failure of as-built samples were found to be higher or equal to wrought-annealed (WA) samples. However, the elevated temperature strength (at 760 °C) of as-built samples was found to be comparable to that of WA samples, and elongation at failure was found to be lesser than that of WA samples [19]. Post-processing techniques, like HIP and ST, were used primarily to close defects (cracks and
pores) and dissolve undesirable phases by hindering diffusive transformation (layer thickness: 20–50 µm) [20, 21]. Applying HIP or ST could dissolve the δ phase and M₆C carbides, respectively. HIP + ST reduced the anisotropy in mechanical properties by reducing the difference in the mechanical properties between the vertical and horizontal samples for stress-relieved LPBF built IN625 samples and improved the ductility at room temperature. This was mainly due to the homogenization of microstructure during HIP. At elevated temperatures, the presence of carbides along grain boundaries caused embrittlement of the solution treated and HIP sample [19]. Hyer et al. carried out microstructural examinations on LPBF built IN625 thin and bulk samples before and after post-processing treatment (stress relieving + HIP + solution annealing). It was observed that Nb and Mo segregated along the intercellular boundaries along with the A₂B Laves phase and a small number of Al-rich oxide particles were uniformly distributed. Stress relief + HIP + solution annealing led to the formation of undesirable M₆C carbides. Repeating the heat treatment at a higher HIP temperature resulted in the dissolution of M₆C carbides and the formation of MC carbides [22]. Guo et al. built IN625 samples using LPBF (layer thickness = 40 µm) and subjected them to two different post-processing techniques. One set of the samples was subjected to only solution treatment (LPBF + ST). The second set of the samples was subjected to solution treatment followed by HIP (LPBF + ST + HIP). Tensile tests were conducted on these samples at both room temperature (RT) and elevated temperature (815 °C). Hardness tests were then conducted on these deformed samples. It was also observed that the hardness of the LPBF built samples decreased after HIP mainly due to the depletion of Nb and Mo from the matrix to the interdendritic regions [23]. Son et al. performed creep tests on LPBF built IN625 in stress relieved + HIP and as-built condition and detected γ” along with Al₂O₃ ceramic inclusions in the as-built sample which were assigned the reason for lesser ductility during creep tests as compared to HIP Wrought IN625 samples [24]. Poulin et al. investigated the effect of stress relieving + HIP on the room temperature fatigue crack propagation behaviour of LPBF built IN625 (layer thickness = 40 µm). It was observed that the crack propagation behaviour of the HIP specimens was nearly unaffected for both the build and the crack orientations [25].

In one of our recent endeavours, IN625 structures are successfully developed using LPBF at a higher layer thickness of 100 µm [5]. Though there are many published literature about the deployment of HIP on LPBF parts, these studies are pertaining to the lower layer thickness (layer thickness ≤ 50 µm). Our literature survey shows that the HIP of LPBF built IN625 samples built at higher layer thickness (> 50 µm) is not available in the open domain. HIP at higher layer thickness is relatively difficult due to a reduction in the ease of dislocation movement along the grain boundaries. This is mainly because of the reduction in the cooling rate during LPBF at higher layer thickness resulting in relatively coarser dendrites and hence fewer boundaries for the dislocations to travel along at high temperature and pressure during HIP. Therefore, it is important to investigate the effect of HIP on LPBF components built at higher layer thickness. Additionally, the literature reports HIP on LPBF built samples being preceded by other heat treatments such as stress relief or ST [26, 27]. This causes a change in the number of dislocations in the LPBF built part at the beginning of HIP and can affect its response to HIP. Further, limited comprehensive studies are available to the best of the author’s knowledge explaining the effect of the HIP on the mechanical properties of LPBF IN625 structures, viz. the tensile properties along and normal to the laser scanning direction in the planes normal to the build direction.

The objective of the present study is to investigate the effect of the HIP on the IN625 samples built using LPBF at a higher layer thickness (100 µm). Using a higher layer thickness (100 µm) in LPBF contributes towards improved build rate as compared to using lower layer thickness (≤ 50 µm). At the same time, using higher layer thickness will affect the melt-pool cooling rate, which in turn will affect the grain morphology and dislocation density. The grain morphology and the dislocation density will affect the dislocation movement during HIP. This study will contribute towards the understanding of the efficacy of HIP on IN625 samples built using LPBF at higher layer thickness (100 µm). This in turn will help in building a dense complex metallic component at a higher build rate. Thus, in this manuscript, a systematic investigation of the direct effect of HIP on the porosity, microstructure and mechanical properties of LPBF built IN625 structures at a higher layer thickness of 100 µm is presented. In addition, the effect of HIP and laser scan direction on the tensile properties are analysed and reported in this manuscript.

2 Materials and methods

2.1 Materials

IN625 metal powder (Make: Powder Alloy Corporation, USA) with particle size ranging 15–45 µm is used as the feed material for the LPBF process. The chemical composition of IN625 powder provided by the powder manufacturer is presented in Table 1. Sandblasted SS 304L substrate of 75-mm diameter and 10-mm thickness is used for processing.
Table 1 Chemical composition of IN625 powder used for LPBF

| Element | Composition (wt%) |
|---------|------------------|
| Cr      | 22.3             |
| Fe      | 4.8              |
| Mo      | 9.2              |
| Nb+Ta   | 4.08             |
| C       | 0.10             |
| Al      | 0.38             |
| Ti      | 0.38             |
| Ni      | Bal              |

2.2 LPBF and HIP of IN625

IN625 is deposited using an in-house developed open architecture LPBF system having a build volume of 250 mm×250 mm×250 mm. The photograph of the LPBF system is presented in Fig. 1a, and the front view schematic and the top view schematic of the LPBF system have been presented in Fig. 1b and c, respectively. The key components of the LPBF system are a 500-W fibre laser, galvano-scanner, build plate, powder-hopper and powder re-coater controlled by a control unit. The movement of the laser beam on the powder bed/build platform is achieved by the galvano-scanner. The diameter of the laser beam spot on the top surface of the powder bed is ~ 500 µm, which is larger than the beam diameter used in most of the commercial LPBF systems. The motivation for using a larger beam diameter for the present system was for achieving a higher build rate. IN625 blocks of size 25 mm×25 mm×15 mm are built at optimized process parameters of laser power 300 W, scan speed 0.05 m s⁻¹ and hatch spacing 0.250 mm with a higher layer thickness of 100 µm. More details on the process parameter optimization and selection are reported elsewhere [5]. The samples are built using a unidirectional scanning strategy. The unidirectional build pattern is used as it offers a uniform local temperature gradient near the beam-powder interaction zone over the entire laser scanning region. This results in uniform cooling and solidification conditions across the laser scanning region during the multi-track deposition.

Wire electrical discharge machining (WEDM) (Make: Concord, Model: DK-7732) is then used to extract the samples from the LPBF built structures. HIP (Make: ASACO, Model: V50) is then used to extract the samples. The HIP cycle was chosen as per ASTM F3301 standard. HIP cycle used in the present study is one of the standard recipes developed by the Indian Space Research Organisation, India, for aerospace-grade alloys. The temperature, pressure and dwell time were chosen, to suit the clubbing of other SS316L components in the HIP cycle. Argon gas of purity grade 1 (purity ≥ 99.99%) is used to apply pressure and prevent unwanted oxidation.

2.3 Material characterization and testing

LPBF built IN625 samples in as-built and HIP conditions are sectioned using WEDM. The sectioned samples are cold mounted and subjected to polishing using emery paper with grit sizes ranging from 200 to 1000. Subsequently, the mirror finish of the sample surfaces is obtained by cloth polishing with the help of a colloidal silica solution. An optical microscope (Make: Omnitech, Model: Metagraph) is used to obtain the images of the mirror-polished surfaces for porosity analysis. The porosity is analysed using the area fraction technique with the help of ImageJ software. After porosity analysis, the samples are electrolytically etched at 12 V for 15 s in an etching solution with 10 g of oxalic acid and 100 ml distilled water solution. The etched sample surfaces are studied using the optical microscope (Make: Omnitech, Model: Metagraph) and scanning electron microscope (SEM) (Make: Zeiss, Model: Supra55). Elemental mapping is carried out to understand the effect of the HIP on the precipitation and segregation of elements in LPBF built IN625 samples.

X-ray diffraction (XRD) of the samples is carried out using an X-ray diffractometer (Make: BRUKER, Model: D8 Advance) from 30 to 110° for identifying phases and estimating the size of the crystallites. Crystallites are sub-grains possessing lower misorientation angles and are equivalent to the microstructure domains that scatter X-rays coherently. Dwell time of 0.5 s and a step size of 0.02° are used for the present XRD studies. Cu Kα radiation (λ = 1.54 Å) is used at 40 mA and 40 kV in a continuous scanning mode. From the XRD curves, Williamson-Hall (W–H) plots for the samples are obtained. W–H plot from XRD is obtained by plotting β cos (θ) in Y-axis against 4 sin(θ) in X-axis and linearly interpolating them. The Y-axis intercept ‘c’ can be used to obtain the crystallite size ‘D’ using Eq. 1 [28].

\[
D = \frac{0.9\lambda}{c}
\]  

(1)

The slope of the W–H plot gives lattice strain ‘η’.

The crystallite size and lattice strain can be used for estimating the average dislocation density ‘ρ’ using Eq. 2.

\[
\rho = 2\sqrt{3}\frac{\eta}{D\ast b}
\]

(2)

where b represents the Burger’s vector, and for IN625, it is equal to 0.179 nm [29].

Figure 2 shows the schematic of LPBF building strategy indicating the front section and the side section with respect to the scan direction on the built sample. The build...
direction, laser scan direction, front section and side section are labelled in Fig. 2.

Micro-tensile test specimens are extracted from both as-built and HIP samples normal to the laser scan direction (Y) and along the laser scan direction (X). The dimensions of the micro-tensile test specimens are given in Fig. 3 [5]. A Vickers micro-hardness tester (Make: Omnitech, Model: MVH-S Auto) is used to measure the micro-hardness of both as-built and HIP samples. The load and dwell time of the micro-hardness tester is set at 0.98 N and 10 s, respectively.
Average micro-hardness is obtained from the indentations taken at six different positions on the polished surfaces of the samples. Automated Ball Indentation (ABI®) studies for the single cycle are carried out using an Automated Ball Indenter (Make: BISS, Model: AC-04–2370-05) machine to determine the indentation depth and estimate the material’s energy storage capability [4]. The maximum load and a loading rate of 80 N and 0.1 mm/min, respectively, are used for carrying out ABI® studies. Before performing the actual testing, a 5 N preload is used. The applied load is unloaded to 10 N after the dwell time.

3 Results and discussion

3.1 Porosity analysis

Figure 4a and b present the typical cross-section (front section (ZX)) images of the as-built samples showing irregular porosity and HIP samples, respectively. The measured average porosity in the as-built sample using area fraction technique is 0.43%, which primarily are irregularly shaped pores (marked 1 in Fig. 4a), and after HIP, the average porosity is found to be ~0.01% (Fig. 4b). The number of irregular pores is more in as-built samples as compared to the circular pores. The irregular shaped pores are primarily generated due to the incomplete coalescence of molten metal powder or due to the melt-pool turbulence (Marangoni effects, recoil pressure, etc.). A similar observation is reported by Qiu et al.
As the present work is focused on higher layer thickness, predominantly irregular porosity in the LPBF built samples can be attributed to two possible reasons. The first one can be attributed to the higher layer thickness increasing the probability of improper bonding of the molten material with the previously built layer resulting in pore/void formation. In addition, when the laser beam strikes the top surface of the powder, it gets melted instantly, and the powder particles beneath are melted due to the conduction of heat. The relatively lower packing density of metal powder (~ 80%) results in poor heat conduction. Poor heat conduction results in a highly turbulent melt-pool on the powder bed leading to the generation of voids or pores [31]. The accumulated heat during laser scanning enhances the recoil pressure and Marangoni effect and leads to the generation of voids/pores. Marangoni effect is the phenomenon, where the gradient in the surface tension causes transfer of fluid mass from low to high surface tension region. This change in surface tension can be due to gradient in temperature inside the melt-pool [31].

Figure 5 shows the postulated mechanism of void formation from recoil pressure effects during LPBF. As shown in Fig. 5, the generation of recoil pressure creates a localized depression over the melt-pool at the point of consideration. However, once the laser moves away from the point under consideration, the melt-pool temperature goes below the boiling point yielding no recoil pressure. Thereafter, the Marangoni convection causes the molten metal to flow and fill the depression created earlier due to the recoil pressure. If the cooling rate is very high, sufficient time will not be available for the complete filling of the depression, resulting in the formation of voids.

When the as-built samples are subjected to HIP, the high pressure at elevated temperature causes the visco-plastic flow of the metal in the samples. This flow of metal tries to close the pores/voids generated during LPBF. The pores that have Ar gas trapped inside them will not be closed completely (unless Ar is soluble in metal). HIP will only be able to reduce the pore size until the force exerted by trapped Ar gas as internal pressure equals the force by the externally applied pressure. As HIP is carried out at an elevated temperature, the LPBF built IN625 experiences a reduction in yield stress. Plastic deformation of IN625 begins when the yield stress at the elevated temperature decreases below the inert gas pressure during HIP. The plastic flow occurs on a microscopic level at very high rates following different creep deformation mechanisms such as Nabarro-Herring creep, Coble creep mechanisms that can be attributed to diffusion through grain interiors, around grain boundaries and dislocation creep [32]. In this way, HIP results in a significant reduction in void/pores in the as-built samples.

### 3.2 Microstructure examination

Figure 6a–c present the microstructures of the front section (YZ) of the as-built samples obtained using an optical microscope. In Fig. 6a, the etched surfaces of the as-built samples show visible melt-pool boundaries. These melt-pool boundaries in the as-built sample are mainly generated due to the re-melting and re-solidification of
the previously built layers during LPBF process. Also, the microstructure growth of the as-built samples is columnar with cellular/dendritic growth. The mixed cellular and dendritic growth are primarily due to the favouring thermal gradient at the solidification front during LPBF. Initially, the build plate/substrate is at room temperature, and it acts as a sink. During the first layer deposition, the thermal gradient is very high causing cellular growth at the bottom layers as shown in Fig. 6b. However, as the layers build up, dendritic growth with classical secondary arms is also seen due to the lower thermal gradient as shown in Fig. 6c. Generally, finer microstructures are observed in the LPBF built samples due to the high cooling rate during LPBF. The typical cooling rate in LPBF is around 10$^6$ °C/s [12]. It is also seen that the microstructural growth spans across multiple melt-pool boundaries at several locations as marked in Fig. 6a. This epitaxial growth can be attributed to the unidirectional build pattern causing similar local solidification conditions across the layers. This effect can also be attributed to the higher thermal energy retained in the melt-pool for a relatively long period because of the higher layer thickness that results in relatively lower effective thermal conductivity.

Figure 7 presents the microstructure of the front section (YZ) of the HIP samples. It can be seen that there are no melt-pool boundaries as they are annihilated during HIP treatment due to solutionizing effects. The solutionizing effect involves recrystallization and grain growth. It begins from the grain boundaries because these are high energy sites. The grains grow and dissolve the earlier formed microstructure. This is evident from the transformation of equiaxed growth to cellular/dendritic growth. The measured average grain size in the HIP sample is 31 µm. During HIP, the coalescence of dendrites leads to the formation of coarse equiaxed grains. It can also be seen that the second phase precipitates of variable sizes are observed along the grain boundaries.

Figure 8 illustrates the elemental mapping of the front section (YZ) of as-built and HIP samples. The segregation
of Nb, Si and C is observed in the as-built sample as presented in Fig. 8a. However, the combination of Ni-Nb is not observed in as-built samples due to the depletion of Ni at Nb-rich regions. Thus, the generation of the δ (Ni₃Nb) phase could not be confirmed in the as-built samples. Further, a combination of Nb-C is observed at some locations, which may be due to the formation of Nb-rich carbides during the LPBF of IN625. The formation of Nb-rich carbides can be because of thermal cycles during the successive layer by layer fabrication. This is in line with the work by Keller et al. in which thermodynamic calculations are carried out for determining the driving force for nucleation of secondary phases in the interdendritic zones [33]. Nb-rich carbides can nucleate first either from the γ-matrix that is supersaturated with Nb or precipitate directly from the liquid phase. Dupont et al. explained that Nb segregates strongly during solidification and its formation can be understood with the help of the following reaction [34]:

\[ \text{L} \rightarrow \gamma + \text{NbC} \]  

(3)

Figure 8b presents the elemental mapping of the front section (YZ) of the HIP samples. Elemental segregations of Mo, Si, Cr, Nb and C are observed. The tendency for higher segregation in the HIP samples is primarily due to a lower cooling rate during HIP. The formation of carbides rich with Nb, Mo and Cr is also detected in the HIP samples. This can be due to the lower cooling rate after HIP, which provides sufficient time for Nb, Mo and Cr to react with C to form carbides and precipitate.

Figure 9 represents the XRD plots obtained for as-built and HIP IN625 samples. It is observed that XRD of as-built samples and HIP samples shows peaks corresponding to the γ-Ni matrix. However, the presence of Nb-rich carbides detected in the as-built samples and Nb, Mo, Cr based carbides present in the HIP samples are not observed during XRD analysis. This can be due to the lower volume fraction of these phases. Further, Eq. 2 is used to quantify the effect of HIP on the dislocation density, which is a function of crystallite size and microstrain in the material. The crystallite size and the microstrain are estimated using the W–H. Figure 10a and b present the W–H plot obtained from the XRD of LPBF built samples in as-built and HIP conditions.

From the W–H plot, the crystallite sizes are calculated using Eq. 1 for as-built IN625 samples and HIP samples, and they are found as 40 nm and 207 nm, respectively. The
larger average crystallite size is observed for the HIP samples mainly due to the coalescence of fine crystallites into coarse crystallites during HIP. Further, the reduction in the number of low angle grain boundaries during the HIP process may also result in the formation of larger crystallites as the samples are kept at an elevated temperature for a sufficiently long period.

It is also observed that the dislocation density calculated using Eqs. 2 and 3 for as-built IN625 samples and HIP samples are $6.74 \times 10^{14}$ m$^{-2}$ and $2.06 \times 10^{14}$ m$^{-2}$, respectively. Thus, the dislocation density of the as-built samples is greater than three times that of the corresponding HIP samples. This is because the elevated temperature during HIP facilitates the easy movement of the dislocations. The dislocations with opposite burger vectors annihilate each other, and in turn, the number of dislocations reduces [35].

### 3.3 Effect of HIP on mechanical properties

Figure 11 presents the stress–strain curves obtained from the tensile tests for LPBF built IN625 samples in both as-built and HIP conditions for samples extracted along (X) and normal (Y) to the laser scan direction. Table 2 presents the yield stress (YS), ultimate tensile stress (UTS) and the percentage elongation/ductility (%El) obtained from tensile test results.

It is observed that the YS and the UTS of IN625 in the WA condition are lower than that of the as-built samples and higher than that of the HIP samples. This is because the HIP samples have coarser microstructure relative to the WA samples and the WA samples have coarser microstructure than the as-built samples. As mentioned earlier, the grain size of the HIP sample is 31 µm, while the grain size of the WA sample reported in the literature is 15 µm [36]. It is observed that the YS and the UTS of the HIP samples are lower than that of the corresponding as-built sample values. At the same time, an increase in ductility after HIP is also observed. This can be primarily because of the reduction in the dislocation density after HIP. The coarse grains in the
HIP sample as compared to the fine dendrites in the as-built sample also contribute to the decreased mechanical strength and increased ductility of the HIP samples. The decrease in the dislocation density after HIP yields less resistance to the external load resulting in lower strength and higher ductility after HIP. It is expected that the decrease in strength after HIP will be reflected in the hardness tests and the ABI® tests. It should be noted that although HIP decreases YS and UTS of the as-built sample, it leads to an increase in the toughness of the as-built samples in both the directions X and Y by 22.51% and 13.74%, respectively. Ductility of the as-built samples along the X and Y directions also increase by 46.26% and 57.28%, respectively, after HIP. Additionally, HIP can improve the fatigue life of the as-built samples. As shown earlier, the as-built samples can contain pores that can act as crack initiation zones during dynamic loading conditions. Under dynamic loading conditions, stress concentration around pores will result in crack initiation and propagation. This may decrease the endurance limit of an as-built part and lead to an early failure under dynamic loading conditions [37, 38]. The ductility (%El) of the HIP samples taken along the laser scan (X) (17.45%) and taken normal to the laser scan (Y) (12.70%) is more than that of the corresponding samples in as-built samples. This can be attributed to the reduction in porosity, presence of coarser grains and reduction in the dislocation density after HIP. The effect of coarser grains and reduced dislocation density on the mechanical properties of the HIP sample in comparison to the as-built sample is already discussed earlier. These cooling rates are high enough to result in finer grains as compared to the WA samples, but at the same time, the grain size is coarser than the as-built samples as LPBF processed samples experience relatively higher cooling rates.

It is observed that the elongation of the sample taken along the laser scan (X) (11.9%) is larger than that of the sample taken normal to the laser scan (Y) (8.11%) for the as-built samples. This can be attributed to the direction of the applied load concerning the boundaries formed from the overlapping tracks in an individual layer. For the samples taken normal to the laser scan direction (Y), when the tensile load is applied, the dislocations have to encounter the melt-pool boundaries formed from the overlapping zones of the adjacent tracks, which act as sinks for void coalescence. For the samples taken along the laser scan direction (X), the coolings do not encounter the melt-pool boundaries with overlapping tracks and can have relatively easy movement avoiding the sinks for void coalescence and hence give rise to the relatively higher ductility. In the case of the HIP samples, YS, UTS and elongation for the sample taken along the laser scan direction (X) and the sample taken normal to the laser scan direction (Y) show lesser variation within 10%. This can be due to the equiaxed nature of the grains formed after HIP, which results in isotropy in the sample.

Figure 12 presents the micro-hardness of the as-built and HIP samples taken on both front (YZ) and side (ZX) sections of the samples along with that of the WA samples obtained from the literature [36]. Micro-hardness of the samples subjected to HIP is found to be lower than that of the as-built samples but similar to the WA samples. The lower hardness of HIP samples can be attributed to the coarser grain size and lower dislocation density as compared to the as-built samples. The coarser grain size and the lower dislocation density provide lower resistance during plastic deformation, which leads to a reduction in micro-hardness after HIP. As mentioned earlier, this reduction in the hardness after HIP is also reflected in the tensile test studies. It is also observed that

![Stress–strain curves obtained for as-built and HIP IN625 samples](image)

### Table 2 Yield strength, ultimate tensile strength and ductility of as-built and HIP IN625 samples

| Sample                                      | YS (MPa) | UTS (MPa) | %El        |
|---------------------------------------------|----------|-----------|------------|
| WA [36]                                     | 482      | 955       | 41 (20 mm gauge length) |
| As-built extracted along the laser scan (X) (present work) | 671      | 969       | 11.9 (3 mm gauge length) |
| As-built extracted normal to the laser scan (Y) (present work) | 786      | 1053      | 8.11 (3 mm gauge length) |
| HIP and extracted along the laser scan (X) (present work) | 466      | 846       | 17.45 (3 mm gauge length) |
| HIP and extracted normal to the laser scan (Y) (present work) | 457      | 804       | 12.70 (3 mm gauge length) |
the deviation in the hardness values reduced after HIP treatment. This can be attributed to the isotropic nature of the HIP sample. Also, the micro-hardness of HIP samples is found to be close to the micro-hardness of conventionally built IN625 bar in the annealed condition. The micro-hardness values are similar on the front section (YZ) and side section (ZX) of the as-built samples as well as HIP samples. The similar values of micro-hardness between WA and HIP samples are primarily due to the presence of coarse equiaxed grains and reduced dislocation density in HIP samples.

Figure 13 presents the load vs. displacement curve obtained from single-cycle automated ball indentation for IN625 as-built and HIP samples on both front and side sections. The area under the curve is representative of the amount of energy that can be stored by a material, while the maximum displacement of the indenter at a particular load is a function of the hardness or the resistance to deformation. It is observed that the maximum indenter displacement is higher for HIP samples in comparison to that of as-built samples. The obtained maximum displacement values for the as-built front, as-built side, HIP front and HIP side are 0.0450 mm, 0.0476 mm, 0.0495 mm and 0.0491 mm, respectively. It is also observed that the area under the curve for the as-built samples is lower than the area under the curve for HIP samples. The measured area under the curve for the as-built front, as-built side, HIP front and HIP side are 0.286 N-mm, 0.341 N-mm, 0.408 N-mm and 0.391 N-mm, respectively. The reason for higher indenter displacement and the area under the curve for HIP samples can be attributed to its coarse grain structure and relatively reduced dislocation density. The presence of coarse grains reduces the number of grain boundaries encountered by the dislocations for HIP samples. Further, due to lower dislocation density, the hindrance for dislocation movement is also reduced in HIP samples. The relatively unhindered motion of the dislocations in the HIP sample results in higher indenter displacement and higher area under the curve. In contrast, the reason for such lower indenter displacement and the area under the curve for the as-built sample can be attributed to its fine grain structure and relatively higher dislocation density. Because of fine grains, the dislocations have more sites or grain boundaries for them to get annihilated during plastic deformation. The dislocation movement during plastic deformation is also hindered by the relatively larger dislocation density. Both these reasons lead to relatively lower resistance or lower indentation depth and lower area under the curve for the as-built sample. The maximum displacement values and the area under the curve obtained from single-cycle automated ball indentation on the front section and side section are closer for the HIP samples as compared to that for the as-built samples. This can be attributed to the formation of equiaxed grains in the HIP sample yielding improved isotropy as opposed to the columnar grain structure in the as-built sample.

4 Conclusions

In the present work, LPBF built IN625 samples are subjected to HIP treatment, and the effect of the HIP on the porosity, microstructure and mechanical properties is investigated using a systematic study, and the following is concluded:

i. The as-built samples revealed the presence of process-induced pores/voids, which reduced significantly in size and numbers after HIP treatment. The estimated porosity in as-built and HIP samples is 0.43% and 0.01%, respectively.
ii. The microstructure of as-built samples reveals melt-pool boundaries and a combination of cellular growth and dendritic growth, while the HIP sample reveals coarse equiaxed grains without melt-pool boundaries.

iii. Elemental mapping reveals that Si gets elementally segregated and the Nb-rich carbides get precipitated in the as-built samples, while elemental segregations of Si are observed along with precipitation of Nb-, Mo- and Cr-rich carbides in the samples subjected to HIP. XRD reveals the presence of γ phase in both cases with no peaks corresponding to precipitates due to their presence in low volume fractions. The estimated dislocation density from XRD analysis is $6.74 \times 10^6 \text{m}^{-2}$ and $2.06 \times 10^6 \text{m}^{-2}$ for the as-built and HIP samples, respectively.

iv. Tensile tests are performed on the samples extracted from the XY plane from both as-built and HIP samples. The samples are extracted along the laser scan direction (X) and normal to the laser scan direction (Y). Irrespective of being extracted either along X or Y directions, HIP samples show lower strength and higher ductility than the corresponding as-built samples. Additionally, the YS and UTS of the HIP samples are found to be slightly less than those of the WA samples. However, anisotropy is observed in the mechanical properties of as-built samples, while the HIP samples show isotropic behaviour.

v. Vickers micro-hardness and ABI® studies reveal that the HIP samples have lower resistance to plastic deformation than the as-built material due to grain coarsening and reduction in dislocation density. Also, the ability to store energy during deformation is observed to be higher for HIP samples in comparison to the as-built samples. The hardness values of the HIP samples are found to be close to the hardness of the WA samples reported in the literature.

Thus, HIP is a suitable method for building dense and isotropic IN625 near net-shaped components. This study will pave a way for the fabrication of defect-free, isotropic and dense functional Ni-based superalloy components using higher layer thickness LPBF followed by the HIP. Further studies on the fatigue behaviour of the IN625 samples built using LPBF in the as-built condition and after HIP will be performed as future scope of work to determine its capability under dynamic loading conditions.

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Code availability Not applicable.

Declarations

Ethical approval No ethical approval was required for this research.

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