Microstructural effects on properties of as-fabricated Inconel 625 with direct energy deposition process

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Abstract. Three-dimensional printing (3D), also known as metal additive manufacturing (MAM), fabricates parts or components from different feedstocks: wires, powders or sheets. This process differs from traditional manufacturing techniques such as casting, moulding, or subtracting existing materials. In the development and improvement or fabrication of new materials for higher strength and various applications, the type or character of a material is very important as this will ascertain the strength of the finished product. Direct energy technology can be used to fabricate and repair parts or components with the following two fabrication methods: laser wire-directed energy deposition (LW-DED) or laser powder-directed energy deposition (LP-DED). In this research, laser powder-directed energy deposition (LP-DED), a MAM process method, was employed to fabricate Inconel 625. The LP-DED process uses a laser as a heat source and rapidly melts metallic powders of different chemical compositions to fabricate complex structures, which is an innovative three-dimensional material processing technology. The as-fabricated (AF) sample specimens were investigated to determine the microstructural development, microhardness and sample defects. The microstructural features were analysed using two experimental surface microscopy methods: light optical microscopy (LOM) and scanning electron microscopy (SEM). The morphological grain structure within the samples was predominantly cellular, columnar and columnar-dendritic. Energy dispersive X-ray (EDX) and X-ray diffraction (XRD) analysis were performed to determine the chemical composition and crystallographic structures of virgin gas atomisation (GA) powder and as-fabricated sample. The XRD peaks in samples composed of face-centred-cubic (FCC) γ-nickel phase. The material microhardness was studied by performing Rockwell hardness test (HRB) with a fluctuated trend averaging 98.9 – 101.6 HRB. The relationship between processing, microstructure, grain structure and material hardness was systematically summarised and

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established. The study concluded with research suggestions on LP-DED of Inconel 625.

1 Introduction

Metal additive manufacturing (MAM) has revolutionised the metal manufacturing industry as it allows the fabrication of complex near net three-dimensional (3D) metal components or parts with both minimal retooling during the fabrication process and post-processing compared with traditional methods (cast and wrought) [1]. Understanding process development and the prediction of microstructure and mechanical properties of MAM materials is fundamental. Machine and computational learning techniques in MAM have proven to be exceptionally useful tools for accumulating insight into MAM processes and conditions [1]. However, the MAM process has challenges, including thermo energy and the molten process of metallic powders or wire during melting and resolidification. The thermal aspect during heat transfer subsequently affects the solid and solid-to-solid phase transformation and macroscale of any change in temperature [2, 3]. The information from microscale models will predict important characteristics of the material microstructure, including grain morphology, grain size, aspect ratio, precipitate volume fraction, and size and build-up of plastic strain [1-3]. This process must be developed to translate these microstructural characteristics into mechanical property predictions [1-2]. The focus has been on process parameters to understand MAM materials and not so much on the mechanical response of MAM.

In this study, MAM techniques for producing metal components from metal powders with a focus on microstructure science of metals and mechanical properties, heat transfer, solidification and post-processing metallurgy are compared and analysed. The metallurgical component manufactured using additive manufacturing is determined with a chemical reaction at a certain temperature experienced by the material. Heat transfer has different applications for powder bed fusion (PBF), direct energy deposition (DED), and additive manufacturing, and the metallurgical principle for both processes yields similar outcome [2].

Materials are subjected to some kind of loading, and their prediction to produce reliable designs must be correct and the properties with their numerous metallurgical variables have to be understood [2]. A combination of metals has been produced for commercial production using additive manufacturing, but recent efforts have made it possible for the continuous development of new materials suitable for MAM processes [2, 4]. This paper focuses mainly on as-fabricated (AF) laser powder-directed energy deposition (LP-DED) Inconel 625 samples and the relationship between processing, microstructure, grain structure, and material hardness [2].

1.1 Why Inconel 625?

Inconel is group of superalloys. Particularly, Inconel 625 is one of the most recognised nickel-based superalloys and is ideal for high-temperature applications. The key factor of nickel-based alloy strength can be explained by precipitation hardening, which is the most useful process at high temperatures [2]. Inconel 625 is a type of solid solution-strengthened nickel-based super alloy, with molybdenum and niobium in a nickel chromium matrix [2-5]. Inconel 625 is resistant to wear, corrosion and fatigue, and further displays good weldability. Table 1 lists the chemical element composition ranges of Inconel 625.
Table 1. Element composition with percentage ranges and limits of Inconel 625 (wt%)[2,7].

<table>
<thead>
<tr>
<th>Element</th>
<th>Percentage Range</th>
<th>Percentage Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel</td>
<td>58% min</td>
<td></td>
</tr>
<tr>
<td>Chromium</td>
<td>20–23%</td>
<td></td>
</tr>
<tr>
<td>Iron</td>
<td>5% max</td>
<td></td>
</tr>
<tr>
<td>Molybdenum</td>
<td>8–10%</td>
<td></td>
</tr>
<tr>
<td>Copper</td>
<td>0.5% max</td>
<td></td>
</tr>
<tr>
<td>Aluminium</td>
<td></td>
<td>0.4% max</td>
</tr>
<tr>
<td>Niobium</td>
<td>3.2–4.1%</td>
<td></td>
</tr>
<tr>
<td>Silicon</td>
<td>0.5% max</td>
<td></td>
</tr>
<tr>
<td>Carbon</td>
<td></td>
<td>0.10% max</td>
</tr>
<tr>
<td>Sulphur</td>
<td>0.015% max</td>
<td></td>
</tr>
<tr>
<td>Manganese</td>
<td>0.5% max</td>
<td></td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.02% max</td>
<td></td>
</tr>
<tr>
<td>Titanium</td>
<td>0.4% max</td>
<td></td>
</tr>
</tbody>
</table>

This superalloy is currently used in aviation gas turbines, the chemical and medical industries, petrochemical equipment, rocket engines, nuclear reactors, submarines and other applications that operate at high temperatures with chemical resistance [2]. MAM of Inconel 625 is appropriate for developing custom or complex parts for the aerospace, marine and possibly biomedical fields [2]. The shape of most Inconel 625 components is complex and expensive to manufacture using traditional methods due to extensive machining. The microstructure of MAM nickel-based superalloys Inconel 625 is dependent on specific processes with different applications, such as process parameters and deposited geometry [2, 8]. When compared with traditional and wrought fabrication methods, solidification in the MAM process occurs more rapidly as the finer grain size is modified to suit a particular grain structure [2]. LP-DED uses a laser as feedstock to heat and melt the metallic powder, which solidifies rapidly. It is a rapid fabrication method for fabricating high-quality metallic 3D parts or components. This fabrication is possible without the aid of any tools that require retooling during fabrication processing. MAM is a remarkably innovative process that opens up opportunities for companies to improve their manufacturing efficiency. The technology allows design freedom with minimum constraints compared with traditional manufacturing methods.

1.2 Materials and processing

The Inconel 625 metallic powder was supplied by Carpenter Additive, a business unit of Carpenter Technology Corporation. The gas-atomisation (GA) powder particles have a particle size distribution of between 20 μm and 50 μm with almost spherical shapes as observed with scanning electron microscopy (SEM). Figure 1a illustrates the use of Inconel 625 as a feedstock material. The Inconel 625 sample specimen was fabricated using an Optomec application workstation, an LP-DED system with motion numerical control with a five-axis operation option, a coaxial metallic powder feeder, and an automatic feeding device as illustrated in Figure 1b. Figure 1c illustrates the fabricated sample specimen with dimensions 40 mm × 20 mm × 10 mm. Table 2 lists the fabrication process parameters.
Fig. 1.a) GA metallic Inconel 625 powder particles; b) LP-DED process (courtesy [10]); and c) AF Inconel 625 on substrate.

Table 2: Fabrication process parameters.

<table>
<thead>
<tr>
<th>Laser power</th>
<th>Laser type</th>
<th>Laser scanning speed</th>
<th>Powder feed rate</th>
<th>Layer thickness</th>
<th>Operation atmosphere argon approx.</th>
<th>Scanning pattern</th>
</tr>
</thead>
<tbody>
<tr>
<td>400 W</td>
<td>IPG fibre laser</td>
<td>10.58 mm/s</td>
<td>5.23 g/min</td>
<td>20 μm</td>
<td>50–100 ppm</td>
<td>Bidirectional</td>
</tr>
</tbody>
</table>

1.3 Sample preparation

The AF Inconel 625 sample specimens were wire-cut from the mild steel substrate and embedded in Bakelite hot mounting powder resin using a thermal-compression mounting machine [2]. A standard thermal moulding process was applied as illustrated in Figure 2. Thereafter, the specimens were ground and polished in line with standard procedure. The samples were immersed in freshly mixed acid – 15 ml hydrochloric acid (HCl), 10 ml acetic acid (CH₃COOH), and 10 ml nitric acid (HNO₃) – for ±35 seconds [2, 5]. Ethanol was used to clean the surface, whereafter the samples were dried using hot air ready for light optical microscopy (LOM) and SEM microstructure analysis.
1.4 Microstructural characterisation

In this study, LOM and SEM were used for the microstructural characterisation of LP-DED AF Inconel 625 samples. LOM analysis was carried out on a Moticam 1080 BMH equipped with Motic software plus 30 ML for image analysis [2]. This machine has a spatial resolution of 2.8 µm and a focusable lens of 12 mm with a 1 920 × 1 080 high-resolution monitor [2]. LOM was used to characterise the grain structure and the porosity of the Inconel 625 samples. It is the most common and basic instrument for analysing surface microstructures in metallic sample specimens. To quantify the porosity of the Inconel 625 samples, four images were taken at 5× magnification mid-thickness of each sample.

SEM analysis was performed with a Tescan Mira3 machine equipped with a secondary electron detector and a backscattered electron detector for further microstructural analysis [2]. The application of quantitative metallography to SEM mainly focuses on backscattered electrons, electron backscatter diffraction, chemical analysis energy dispersive X-ray (EDX), and volume fraction analysis [6]. The X-ray technique analyses the crystal structure, which identifies the crystalline phases in a particular place of the sample and determines the chemical composition information [7]. An EDX spectrometer was used to determine the chemical composition of the Inconel 625 samples in terms of percentage by analysing the characteristics of the X-rays.

1.5 X-ray diffraction

X-ray diffraction (XRD) analysis is a non-destructive experimental technique for analysing the molecular structures of organic and inorganic compounds [11]. XRD analysis is a rapid technique used for identifying the phases of a crystalline material and determining the crystal orientation, chemical composition and physical properties thereof. Therefore, XRD was used to identify and characterise the phases in the virgin powder and the AF Inconel 625 sample. The microstructural phase was performed by a multipurpose X-ray diffractometer D8 ADVANCE coupled with Cu-Kα radiation at 40 kV and 40 mA operating with a continuous scan mode. The measurements ran within a range of 2θ with a typical step size of 0.034° in
2θ. The scanning was performed over the range and was recorded at a speed of 0.5 sec/step of the $2\theta = 20^\circ–100^\circ$ range to determine the diffraction peaks.

### 1.6 Mechanical properties characterisation

MAM materials are still not completely understood and MAM literature mostly focuses on tensile behaviour and microhardness. These mechanical properties are the most common properties used for comparison with traditionally manufactured materials (wrought and cast). Both the orientation and location of sample specimens are important during testing and should thus always be mentioned and reported when evaluating experimental and MAM mechanical results. Additional factors to consider include porosity, residual stress, thermal history, and microstructural characteristics. Porosity – both spheres and irregular shapes – in MAM components or parts reduces the cross-sectional area of the materials, affecting the mechanical properties, which is notable in the reduction of elongation [4-6]. Residual stress can also lead to early failure in MAM parts if a post-non-stress-relieving process is not applied; furthermore, part of the thermal history is important for precipitate-hardened alloys. The columnar grains in most MAM alloys indicate that grain orientation and mechanical properties have a relationship and mechanical anisotropy, which must be considered [14].

MAM nickel-based alloys are complex alloys due to the substantial number of chemical elements, which have different secondary phases that affect the mechanical properties of the product. Thus, the AF microstructure of nickel-based alloys is practically dependent on their chemical composition and thermal history thereof [15]. Hardness is classified as a mechanical property that withstands a material’s plastic deformation. A material’s hardness measurement can be classified using three methods, namely indentation hardness, rebound hardness, and scratch hardness [16]. Indentation hardness was a concern for the AF Inconel 625 specimens – both parallel and perpendicular to the building direction.

Indentation hardness measurements were conducted to determine the strength of these sample specimens. Hardness was measured using the Rockwell hardness scale. For each sample, both parallel and perpendicular, a load of 10 kg and a dwell time of $\pm 10$ seconds were applied, and at least six indentations were performed to obtain reliable results.

### 2 Results and discussion

The polished samples were made conductive using a coating in order to enable or improve the imaging of the samples. Applying the correct amount of coating reduces thermal damage, which improves the secondary electrons during analysis. Figure 3 and Figure 4 illustrate the LOM and SEM micrographs of sample specimens of Inconel 625, respectively, both parallel and perpendicular to the building direction.

#### 2.1 Metallographic studies

##### 2.1.1 Light optical microscope

Micrographs of Inconel AF sample specimens are helpful to gain an insightful understanding of the MAM process for layer-by-layer orientation. Figure 3a and Figure 3b respectively illustrate the Inconel 625 parallel and perpendicular to the building direction with laser movement direction, porosities (spherical and irregular shapes) and layer-by-layer building orientation. Micropores with small spherical and larger irregular shapes were observed in
samples – both parallel and perpendicular to the build direction as illustrated in Figure 3a and Figure 3b, respectively [2].

![Fig. 3. LOM micrographs of Inconel 625: a) Parallel with arc shaped beads and porosities (irregularly shaped); and b) Perpendicular with melted track layers patterns and porosities (spherical and irregular shapes) denoted with arrows.](image)

Figure 3a illustrates the bead morphology parallel to the building direction. Bead morphology is common in multi-pass welds in laser powder bed fusion (L-PBF). The bead size with non-uniform multi-pass welds is slightly larger close to the substrate with a smaller bead size at the top layers. The difference in the slightly larger bead size may be attributed to the rapid cooling rates in the first two layers, which are closer to the substrate.

The laser-melted track layers are visible in the scanning pattern in Figure 3b perpendicular to the building direction [2]. The individual melt pool boundaries are visible where the two tracks meet. The overlaps between the tracks were caused by the laser movement forming each layer during the layer-by-layer building process [2]. The overlaps in each layer may be considered high-stress regions because the overlaps face rapid reheating and solidification [2].

The lack of fusion porosity transpired when the molten pool that formed during fabrication could not melt fully in the neighbouring tracks, which resulted in flat cracks perpendicular to the building direction [17]. The small spherical porosity was caused by insufficient fusion and is related to the use of inert gas during the melting pool stage and gas trapped inside the powder, which cannot escape from the melting pool [2], [12-15]. The larger irregular shaped porosity occurred due to an insufficient energy intake to melt the powder for LP-DED, selective laser melting and wire for laser wire-directed energy deposition (LW-DED) during the layer-by-layer fabrication process and may lead to unmelted regions [2], [14-15]. This happened due to the reduction of heat from the feedstock energy and scanning rate, mass flow rate accumulation, or a consolidation of these variables [19]. The tips of irregularly shaped pores may introduce local stress during loading, which could result in early fracture or failure of parts, especially for the side perpendicular to the build direction [2, 20]. Both types of porosity can be reduced by selecting the process parameters carefully. The proper scan speed, scan pattern and laser power parameters must be chosen to have a melt pool that melts at least two to three layers of the substrate to avoid the keyhole pores effect [2], [13-14].
2.1.2 Scanning electron microscopy

The distinction in grain size and direction of the sample specimens shown by SEM analysis in Figure 4a and 4b, respectively, indicate that the grains have a preferred growth direction [2]. The average grain size parallel to the building direction is smaller than the average grain size perpendicular to the building direction illustrated in Figure 4a and Figure 4b, respectively [2]. This indicates that the preferred grain growth direction of these MAM samples is normal to the building direction, which means that the grains are smaller in the direction of the laser motion [2]. This suggests that there may be a connection between the crystallographic orientation and mechanical properties [2, 24]. The grain orientation of Inconel 625 is parallel and perpendicular, as illustrated in Figure 4a and Figure 4b. The grain has texture, meaning it has a preferred direction [2].

Figure 4a parallel to the building direction illustrates two distinct regions, and both cellular and columnar grain structures can be observed. The melting pool boundaries indicate that these two regions have different grain structures due to their different histories and thermal cooling rates during solidification [25]. Figure 4b perpendicular to the building direction illustrates a columnar grain structure with elongated cells. This grain morphology causes a heat-affected zone (HAZ) or laser overlapping zone with a relatively lower cooling rate. Materials with smaller grain sizes increase tensile strength and may increase ductility, whereas materials with larger grain sizes improve high-temperature creep properties [2, 12].

Figure 4c illustrates the HAZ in Inconel 625 perpendicular to the build direction. The HAZ near or in the substrate was neglected because the focus was on the HAZ between the overlapping layers. The dotted lines (region) in Figure 4c indicate the melting pool contours that affect the grain structure and orientation during the next layer process indicated in melted pool 1 and melted pool 2, respectively. HAZ and liquation cracking transpire when the solidified material alongside the fusion zone is exposed to different temperatures, which results in partial melting of the material microstructure or partially melted zone [26].

Two mechanisms cause HAZ liquation cracking, namely melt penetration and solute segregation [27]. Furthermore, the grain structure in Figure 4c illustrates melted pool 1 with columnar grains and melted pool 2 with columnar-dendritic grains. The solidification structure in MAM with metals can be cellular, columnar-dendritic, equiaxed-dendritic, or planar. This is determined from the temperature gradient ($G$) and grain solidification rate ($R$) during fabrication processes with respect to the relationships derived from Equations 1, 2 and 3, respectively [19].

The relationships for the cooling rate, thermal gradient and solidification front velocity are as follows:

\[
\text{Cooling rate:} \quad \frac{\partial T}{\partial \tau} \quad (1)
\]
\[
\text{Thermal gradient:} \quad G = |\nabla T| \quad (2)
\]
\[
\text{Solidification front velocity:} \quad R = \frac{1}{G} \frac{\partial T}{\partial \tau} \quad (3)
\]

where $T$ is temperature and $\tau$ is time.
planar. Structure in MAM with metals can be cellular, columnar grain structure in solidification results in partial melting of the material microstructure or partially melted zone. Solidified material alongside the fusion zone is exposed to different temperatures that overlapping layers. HAZ near or in the substrate was neglected whereas rate. Materials with smaller grain sizes increase tensile strength and may increase ductility, causes direction illustrate thermal cooling rates during solidification. Scanning electron microscopy suggests that there may be a connection between crystallographic orientation and mechanical properties. Furthermore, the grain structure in Figure 2.1.2 has texture, meaning it has a preferred direction. Inconel 625 is parallel and perpendicular, as illustrated in the direction of the laser motion. The average grain size perpendicular to the building direction illustrated in Figure 2.1.2 indicates that the grains have a preferred growth in the direction of the laser motion. The relationships for Scanning Electron Microscopy (1) with Equation \( G \) in solidification (2), with Equation \( R \) in solidification (3) and Equation \( T \) in solidification (4) is temperature and solidification front velocity. Thermal Cooling rate, thermal gradient and solidification front velocity affect the grain structure and orientation during the next layer process indicated in melted pool. The melting pool boundaries indicate that the grains have a preferred growth direction. The grain orientation of Inconel 625 in Figure 5 illustrate spectra 1–5, respectively, in different areas of the AF samples. EDX analysis was used to determine the combined chemical element averages listed in Table 3. The chemical elements prominent in Inconel 625 were 55.76% nickel, 20.72% chromium, and 9.65% molybdenum. Nickel-molybdenum-chromium alloys in this category are known for their high strength despite their high temperatures and corrosive environments [2].

**2.2 Energy dispersive X-Ray**

The results of Inconel 625 in Figure 5 illustrate spectra 1–5, respectively, in different areas of the AF samples.
Fig. 5. EDX of Inconel 625: a)–b) parallel, and c)–d) perpendicular to spectra 1–5 in different areas of each sample to the building direction, respectively, all with the same magnification.

Table 3. Chemical results for Inconel 625 virgin powder, 400 W with 10.58 mm/s AF sample compared with nominal composition (wt%) [20, 21].

<table>
<thead>
<tr>
<th>Element wt%</th>
<th>Al</th>
<th>C</th>
<th>Co</th>
<th>Cr</th>
<th>Cu</th>
<th>Fe</th>
<th>Mn</th>
<th>Mo</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Virgin powder</td>
<td>&lt;0.01</td>
<td>0.01</td>
<td>&lt;0.1</td>
<td>21.4</td>
<td>0.03</td>
<td>3.4</td>
<td>0.25</td>
<td>8.7</td>
<td>0.09</td>
</tr>
<tr>
<td>Nominal</td>
<td>0.4 max</td>
<td>0.1 max</td>
<td>–</td>
<td>20–23</td>
<td>0.5 max</td>
<td>5 max</td>
<td>0.5 max</td>
<td>8–10</td>
<td>–</td>
</tr>
<tr>
<td>Element wt%</td>
<td>Nb</td>
<td>Ni</td>
<td>O</td>
<td>P</td>
<td>S</td>
<td>Si</td>
<td>Ta</td>
<td>Ti</td>
<td></td>
</tr>
<tr>
<td>Virgin powder</td>
<td>3.23</td>
<td>62.34</td>
<td>0.0185</td>
<td>0.001</td>
<td>0.004</td>
<td>0.40</td>
<td>&lt;0.00050</td>
<td>&lt;0.01</td>
<td></td>
</tr>
<tr>
<td>Nominal</td>
<td>3.2–4.1</td>
<td>58</td>
<td>–</td>
<td>0.02</td>
<td>0.015 max</td>
<td>0.5 max</td>
<td>–</td>
<td>0.4 max</td>
<td></td>
</tr>
<tr>
<td>400 W, 10.58 mm/s</td>
<td>3.59</td>
<td>55.76</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>0.53</td>
<td>–</td>
<td>–</td>
<td></td>
</tr>
</tbody>
</table>
The LP-DED process demonstrates that the values for nickel and chromium decreased from those seen in the supplied powder and nominal values (nickel: 62.34 wt%, 58 wt%, 55.76 wt%; chromium: 21.4 wt%, 21.5 avg. wt%, 20.72 wt%). The element iron decreased from the supplied powder value of 3.4 wt% to 3.19 wt% with a nominal value of 5 wt%. The only concern value was the increase of the carbon element for supplied powder and nominal composition (0.01 wt%, 0.1 wt%), respectively, which increased to 6.71 wt% during the LP-DED process. The vapourisation of chemical elements occurred during MAM in the molten pool at elevated temperatures. The reduction of these chemical elements can be attributed to the vapourisation under laser processing.

The boiling points of these elements under standard pressure for nickel, chromium and iron were 2839°C, 2665°C and 2857°C, respectively [29]. These boiling points were among the lowest in the alloy. The boiling point of carbon was among the highest in the AF sample with a value of 4827°C under standard pressure and among the highest vapourisation temperatures under various pressures (torr) ranging from 10⁻⁵ torr to 1 torr as illustrated in Table 4.

The high boiling point value, LP-DED energy condition, and high vapourisation temperatures could have attributed to the increase in carbon during fabrication. Furthermore, the minimal increase of some of the other elements’ values from the supplied powder during DED process is due to their increased concentration because of the losses of the decreased elements. These vapourisations of chemical elements may affect the mechanical properties due to the change in microstructure and possible deterioration of corrosion. It is important to remember that all elements do not vapourise at the same rate during fabrication processing, and the selective vapourisation of elements often result in an alloy changing composition.

The role of an inert gas inside the building chamber during the LP-DED process is to remove the by-products and air in the process chamber and to protect against oxidation [30]. There are different gas supply options for MAM. This study used argon gas as the inert gas during the fabrication process. The oxygen and nitrogen showed a complete vapourisation in the AF sample compared with the virgin powder chemical composition during the fabrication process. The nominal composition had no oxygen and nitrogen chemical elements. The amount of argon atmosphere inside the building chamber was most likely the cause of the vapourisation of oxygen and nitrogen during the fabrication process. The gas sensor measured the argon atmosphere in the building chamber to be approximately 50–100 ppm during fabrication.

Table 4 shows the vapour pressure data for the selected elements relevant to PBF. An important observation can be made regarding PBF process control. In the 10⁻⁵ torr to 10⁻⁴ torr vacuum range, all elements except aluminium had vapourisation temperatures lower than their respective melting points. In the 10⁻³ torr to 10⁻² torr range, the vapourisation temperatures of most elements exceeded their melting points except for carbon, chromium, manganese and iron. In the 10⁻¹ torr to 1 torr range, the vapourisation temperatures of the majority of the elements also exceeded their melting points except for carbon, chromium and manganese. Therefore, based on the composition element used to fabricate the Inconel 625 sample specimen, the fabrication process should be managed under limited pressure to minimise vapourisation during LP-DED processing [31].
Table 4. Vaporization temperature of selected elements as a function of pressure, adapted from [29,31].

<table>
<thead>
<tr>
<th>Element</th>
<th>Tm</th>
<th>Tb</th>
<th>Vaporization temperatures at various pressures (torr)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10⁻⁴</td>
<td>10⁻³</td>
<td>10⁻²</td>
</tr>
<tr>
<td>Al</td>
<td>659</td>
<td>2 447</td>
<td>882</td>
</tr>
<tr>
<td>C</td>
<td>3 350</td>
<td>4 827</td>
<td>–</td>
</tr>
<tr>
<td>Co</td>
<td>1 495</td>
<td>2 877</td>
<td>1 162</td>
</tr>
<tr>
<td>Cr</td>
<td>1 903</td>
<td>2 665</td>
<td>1 062</td>
</tr>
<tr>
<td>Cu</td>
<td>1 084</td>
<td>2 578</td>
<td>942</td>
</tr>
<tr>
<td>Fe</td>
<td>1 539</td>
<td>2 857</td>
<td>1 107</td>
</tr>
<tr>
<td>Mn</td>
<td>1 244</td>
<td>2 051</td>
<td>697</td>
</tr>
<tr>
<td>Mo</td>
<td>2 577</td>
<td>4 827</td>
<td>1987</td>
</tr>
<tr>
<td>Nb</td>
<td>2 477</td>
<td>4 927</td>
<td>–</td>
</tr>
<tr>
<td>Ni</td>
<td>1 452</td>
<td>2 938</td>
<td>1 142</td>
</tr>
<tr>
<td>P</td>
<td>44.1</td>
<td>280</td>
<td>–</td>
</tr>
<tr>
<td>S</td>
<td>–</td>
<td>444.6</td>
<td>–</td>
</tr>
<tr>
<td>Si</td>
<td>1 415</td>
<td>2 787</td>
<td>1 177</td>
</tr>
<tr>
<td>Ta</td>
<td>3 020</td>
<td>5 457</td>
<td>–</td>
</tr>
<tr>
<td>Ti</td>
<td>1 660</td>
<td>3 287</td>
<td>1 321</td>
</tr>
</tbody>
</table>

Tm = melting temperature; Tb = boiling temperature.

2.3 X-ray diffraction

Bragg’s Law for interplanar spacing data was used to determine the diffraction results of the virgin Inconel powder and the AF Inconel 625 sample specimens. The phases were identified with the Bragg diffraction by matching the calculated peaks until all phases were present. Bragg’s diffraction occurs when the radiation of a wavelength is similar to atomic spacing, which is scattered by atoms of crystalline systems, and results in constructive and destructive interference [2, 11].

The Bragg diffraction analysis was determined based on the resulting wave interference pattern of the crystallographic planes in the crystal lattice [2]. To determine the d-spacing and lattice parameters, the XRD and scan parameters during the analysis were the same for the GA virgin powder and the AF sample. Figure 6a and Figure 6b illustrate the powder and AF sample intensity vs diffraction angle during XRD analysis, respectively. Table 5 lists the crystallographic parameters of the GA virgin powder and the AF sample.
Therefore, according to Bragg’s Equation:

\[ n\lambda = 2d \sin \theta \quad (n = 1, 2, 3, \ldots) \]  

(4)

where \( n \) = positive integer (order of diffraction), \( \lambda \) = incident wavelength, \( d \) = interplanar distance between atoms inside the material, and \( \Theta \) = scattered angle [2].

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**Fig. 6.** XRD pattern of Inconel 625: a) Powder intensity vs diffraction angle; and b) AF sample intensity vs diffraction angle.

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**Table 5.** Crystallographic parameters of GA virgin Inconel 625 powder and AF-DED sample.

<table>
<thead>
<tr>
<th>Powdertype</th>
<th>Inconel 625 samples</th>
<th>Peak position for crystallographic planes</th>
<th>d-spacing for (111) plane [Å]</th>
<th>Lattice parameter [Å]</th>
</tr>
</thead>
<tbody>
<tr>
<td>GA Virgin powder and AF</td>
<td>111 200 220 222</td>
<td>44.45° 50.64° 74.48° 90.46°</td>
<td>2.040</td>
<td>3.536</td>
</tr>
</tbody>
</table>

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The XRD results of the virgin Inconel powders and AF specimens are illustrated in Figure 6a and Figure 6b, respectively. The peaks corresponded to the lattice planes in this superalloy, which was used for phase identification. The powder and AF specimens in Figure 6a and Figure 6b, respectively, illustrate that the outstanding peaks are related to the face-centred-cubic (FCC) lattice \( \gamma \)-nickel phase [32].

The lattice parameters corresponded to the FCC phases, with the strong first peak crystal structure (111) parameter of 3.536 Å, second peak crystal structure (200) and third peak both a parameter of 3.600 and fourth peak (222) crystal structure of 3.760 Å, respectively within the samples. The (111) peak is typically the most intense peak in the XRD pattern and is often used as the reference peak for indexing the crystal structure.

The presence of the (111) peak indicates that the sample has a strong and specific crystal structure along the (111) plane and gives an indication of the orientation of the crystal lattice [11, 32]. The second peak (200) is the second strongest peak and corresponds to the crystallographic plane of FCC. The third peak (220) provides information about the arrangement of atoms in the crystal structure in different directions. The (200) and (220) planes have the same lattice parameters as mentioned, which indicates that these planes have a slight deviation with respect to the reference peak. The peak at (222) also corresponds to the crystallographic plane of FCC. This indicates that the material has a well-defined crystallographic structure in the (222) direction [11, 32]. However, the lattice parameter of...
this peak is notably different from the reference peak at (111). This suggests that there may be distortion or lattice strain in the (222) direction [33, 34]. The peak shift on the 2θ scale for the AF Inconel 625 samples indicate that the lattice parameters increased due to thermal expansion, which resulted in the atomic radius increasing.

The XRD spectra for the powder and AF sample corresponded to a difference in the intensity peaks of virgin powders and AF samples. In Figures 6a and Figure 6b, respectively, the intense peak (200) of the virgin powders was higher than the AF-DED peak (200) and the AF LP-DED peak intensity (220) was higher than the virgin power peak (220). The peak intensity of virgin powder (222) was higher than the AF sample. The peak shifts were caused by the change of crystal lattice shifts to higher angles during the XRD analysis due to compression of the crystal lattice [33]. The variation in peak intensity could reveal useful information about crystallography orientation and developed texture in LP-DED fabricated parts.

The lattice parameters and d-spacing during XRD will give insight and understanding of the precipitation or dissolution of intermetallic and carbides in Inconel 625 without affecting the precipitates form the matrix [34]. Furthermore, the nature of precipitation can be identified from the lattice parameters.

3 Mechanical properties: microhardness test results

The Rockwell microhardness of the AF specimen Inconel 625 and indents were taken 5 mm from either side of each specimen from the first indent centre and two indents 5 mm above and below the centre as illustrated in Figure 7.

![Graph showing Rockwell hardness vs number of indents location](image)

**Fig. 7.** Number of indentation values (location) on samples vs Rockwell hardness.

The microhardness of the first and second indentations for both the parallel and perpendicular Inconel 625 is near the substrate and first few layers, whereas the third and fourth indentations were in the centre layers, and the fifth and sixth indentions were near the last layers. The microhardness of both the parallel and perpendicular samples changed along the building direction variation in the microstructure. The microhardness parallel to the building direction in Figure 7 illustrates that the microhardness is on average higher in the central and last layers. The microhardness perpendicular to the building direction is on
average higher in the first layers and last layers. Furthermore, Figure 7 illustrates that both samples have a wavy profile. The inhomogeneity of the microstructure could have been attributed to the wavy hardness results in both the parallel and perpendicular samples, respectively, which were caused by the cyclic thermal history that varied during processing [19].

The average microhardness of the AF samples parallel and perpendicular to the building direction was 98.8 HRB and 101.6 HRB, respectively. The average hardness for both the parallel and perpendicular samples was similar to the hardness values reported by Gamon et al., Marchese et al., and Wong et al. [34-36]. The microhardness for both parallel and perpendicular samples is slightly lower in the central region of each sample as illustrated in the Figure 7 indentation. Dass and Moridi, Kistler et al., and Shamsaei et al. also reported that the average microhardness in the central region was lower than the first and last layers post the LP-DED process [19], [37-38]. Furthermore, a higher interlayer dwelling time with a higher thermal gradient may also increase hardness [19].

4 Sample defects

Even though MAM fabrication has many advantages compared with conventional or traditional fabrication methods, MAM also has common defects, such as unmelted powders during fabrication, porosity in samples, process cracking, delamination, surface roughness, spatter, and residual stress. Possible defects that may occur during powder production include satellites and internal porosity.

4.1 Defects of feedstock material and as-fabricated sample

Figure 8a illustrates the powder morphology with spherical and deformed particles with satellites obtained with SEM. These surface deformations, namely irregularly shaped particles and satellites, are classified as surface defects that are caused by differences in the solidification rate between the partially smaller and larger molten particle sizes [40]. Research reported by [6-11, 21] showed that GA powder has spherical powder particles and deformed particles with satellites. An internal powder porosity analysis was not conducted, but the section view of GA powders was reported by [45], which showed pores and irregular particles due to particle joining and collision during solidification.

From the above-mentioned sample defects, the AF samples had post-processing spatter, surface defects and gas pores in the sample parallel and perpendicular to the building direction as illustrated in Figure 3a, Figure 3b, Figure 8b, Figure 8c, Figure 8d and Figure 8e, respectively. Spatter cannot be prevented during the fabrication process in L-PBF, DED and conventional laser welding. Spatter behaviour has a correlation with L-PBF and DED, which are complex heat transfer processes that lead to metallurgical defects and degrade mechanical properties [31-33].

Figures 1c and Figures 8b illustrate spatter build-up during the LP-DED process in the x, y and z direction respectively, as well as an edge defect. Spatter has an unfavourable effect during fabrication stability, and the efficiency of the laser influence and diminish the characteristics of the fabricated component sample and may damage the machine [49]. The process mechanism of oxidised spatter has an unfavourable effect on metallic powder recoating and energy absorption during laser powder bed fusion (L-PDF) and DED processes [49]. Furthermore, spatter particles formed during fabrication redeposit into the powder bed during recoating; the voids between the powder and spatter particles may cause part defects [50].
Figure 8b illustrates the blobs and zits on the AF sample that appear when the laser beam switched direction. It creates an extra deposition during the unsuitable dwelling time when the melting pool overlaps. Overlapping occurs at the start and finish position during the fabrication process as overlapping cannot be a seamless process. In addition, edge defects may also occur as either an underlay or overlay on opposite sides of the AF material [51]. This results in an uneven level with the building plane that varies in height from the AF part or component.

There are two types of porosities in MAM, namely intralayer (spherical pores) and interlayer (irregular pores) [19]. Figure 3a and Figure 3b illustrate the LOM images of spherically and irregularly shaped pores, whereas Figure 8c and Figure 8d illustrate the SEM images of spherically shaped pores in the AF samples. The spherically shaped pores in Figure 3b and Figure 3c, and Figure 8c and Figure 8d, respectively, stem from possible powder contamination, voids or melting after layer deposition [45].
Fig. 8. a) Surface morphology and defects of GA virgin Inconel 625 powders; b) Fabrication spatter and development of blobs and zits; c) Spherical gas-shaped pore parallel to the building direction; d) Spherical gas-shaped pore perpendicular to the building direction; and e) Lack of fusion. Figures 8a, 8c, 8d and 8e have different magnifications.
Figure 8c and Figure 8d show that the spherically shaped pores are in random sections, parallel and perpendicular to the AF samples. The spherically shaped porosity transpires when gas bubbles are entrapped in the molten metal bead before solidification or they are caused by a high laser scan speed during the fabrication process [52]. Dass and Moridi also reported that spherically shaped pores occurred in random locations and were observed in regions with lower solidification rates [19]. Irregular shaped pores occur due to insufficient energy intake to melt the LP-DED or LW-DED during the layer-by-layer fabrication process, which may lead to unmelted regions [19].

In Figure 3a, the irregularly shaped pores were between the layers, whereas Figure 8b illustrates that the irregularly shaped pores were between the first couple of layers and near the substrate in the building direction. Spherical pores may cause less damage to the mechanical properties of the MAM parts, whereas irregular pores raise the concentration of stress that may lead to failure [8]. Figure 8e illustrates the lack of fusion: these defects occur when the energy delivered during the fabrication process is insufficient, which causes layers to fuse incompletely, or the new layer and previous layer not to overlap sufficiently [40, 41]. Furthermore, the width of the melting will be small if the laser energy is too low, and insufficient overlap between scanning tracks may occur.

## 5 Conclusion

In summary, the AF LP-DED Inconel 625 sample was fabricated successfully with selected process parameters with virgin Inconel powder to investigate microstructure evolutions, EDX, XRD, material hardness and defects of the virgin Inconel 625 powder and AF sample specimens. Based on the results, the following primary conclusions were drawn from this investigated research:

a. The sample parallel to the building direction had a cellular and columnar grain microstructure. The sample perpendicular to the building direction had a predominately columnar and dendritic-columnar microstructure. The melting pool between the layers affects the grain size, grain structure and orientation due to HAZ.

b. EDX analysis and metallography confirmed the chemical composition and uniform structure of the AF sample, with nickel, chromium and molybdenum as the prominent chemical elements. In addition, the EDX analysis results demonstrated that compared with the supplied virgin powder, the composition of some chemical elements of the AF sample had increased due to the high boiling points and high vaporisation temperatures, whereas some decreased due to the lower boiling points and lower vaporisation temperatures.

c. The phase structure during XRD of the virgin powder and AF sample corresponded to and predominately composed of the $\gamma$-Ni (FCC) phase with a lattice parameter of 3.536 Å for (111).

d. The average microhardness for the AF samples, parallel and perpendicular to the building direction, was 98.8 HRB and 101.6 HRB, respectively. Research reported by Dass and Moridi showed that increasing the substrate thickness of the AF-DED process increases the material hardness and finer microstructure as the thicker substrate has the potential to absorb heat faster during the fabrication process [19]. A hardness study of the DED fabrication process stated that alloy elements selection or post-processing (heat treating or ageing) of MAM components or parts has more control over material hardness than changing process parameters [15].
e. Spatter occurred during the fabrication process on the substrate in the x and y directions. Spatter is an unpreventable fabrication defect caused by the heat transfer process. Spatter degrades a material’s mechanical properties and reduces the quality of fabricated parts or components. Post-processing is essential to remove spatter and enhance surface quality. Mechanical properties may improve and less spatter may occur during the LP-DED fabrication if the process parameters are adjusted.

f. General GA powder defects with surface deformation resulted in irregularly shaped powder particles with satellites caused by different solidification rates during production. The particle size ranged from 20 μm to 50 μm, which included near perfectly spherical and irregularly shaped powder particles. There were general defects in AF samples with spherical and irregular pores. The spherical pores were in random locations in both samples, whereas the irregularly shaped pores were in the first couple of layers.

The results obtained in this research paper demonstrated that LP-DED MAM technology with a low fabrication rate has great potential for prototyping and is suitable for various industrial sectors. However, additional research is required on AF parts to adjust the process parameter variables, including laser beam size, laser power, scanning speed, powder feed rate, and scanning strategy, that affect the mechanical, metallurgical and geometrical properties as well as residual stress.

Understanding the root cause of defects in LP-DED is critical for improving quality and consistency during fabrication. Adjusting these variables may also reduce defects in LP-DED samples in order to achieve optimal results, which will be extremely beneficial to LP-DED MAM processing. Furthermore, research should also be conducted on the mechanical tensile properties at elevated thermal temperatures to improve material characteristics. This additional research is currently planned.

6 Data availability

The data used to support the findings of this study are included in the research paper.

7 Conflicts of interest

The authors wish to declare that there has been no conflict of interest.

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