Microstructural and intergranular corrosion properties of Inconel 625 superalloys fabricated using wire arc additive manufacturing

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Abstract
Inconel 625 superalloy samples were fabricated using wire arc additive manufacturing (WAAM). The phase composition, microstructure, anti-corrosion, and mechanical properties of the Inconel 625 WAAM samples were analyzed. The microstructure of the Inconel 625 WAAM alloy showed good forming quality, no defects, and good metallurgical bonding within the specimens. The metallographic structure exhibited primarily γ-Ni and granular precipitated phases; the average microhardness of the transverse and longitudinal cross-sections of the sample was 243.5 and 243.3 HV0.1, respectively. Yield and tensile strength as well as elongation, decrease in area, and the room-temperature impact values of this alloy were equal to 450 and 736 MPa, 38% and 52%, and 152 J, respectively. The intergranular corrosion test results indicated that the average corrosion rate of the sample is 0.609 mm/year, indicating excellent resistance to intergranular corrosion.

1. Introduction
Additive manufacturing, also called 3D printing or rapid prototyping, uses computer-aided design/computer-aided manufacturing (CAD/CAM) and other technologies to build three-dimensional data models of parts. Subsequently, based on the principle of layered manufacturing and discrete stacking, different raw materials are melted and deposited layer-by-layer gradually composing the desired part. This process describes bottom-up rapid prototyping. Relative to casting, forging, and machining, 3D printing has several advantages, which include the following: the ability to directly convert CAD/CAM data models into solid parts, which significantly decreases the product processing time and human interference during production. Although computer numerical control (CNC) machines can also convert CAD models into actual parts, re-mounting and calibrating parts with complex geometries requires much time. For the processing of some precious metal parts (such as Ti- and Ni-based alloys used in aerospace applications), additive manufacturing has the advantage of low cost. Additive manufacturing can produce small batches of parts with complex geometries that cannot be produced using traditional manufacturing methods. Applications of additive manufacturing in a variety of industries and even our everyday lives are endless. Currently, 3D printing is used in aviation and aerospace, automotive and biomedical industries, education, molding, etc [1–8]. Currently, the heat sources used in additive manufacturing technologies are primarily laser beams, electron beams, plasma arcs, and arcs [9]. The raw materials used for additive manufacturing of metal materials are primarily powders and wires. Due to the cycle and cost limitations of the powder-feeding type, filler wire is often used for the additive manufacturing of large components. Wire arc additive manufacture (WAAM) is a layer-by-layer cladding principle that uses melt inert-gas welding (MIG), tungsten inert gas (TIG) welding, plasma welding power source (PA), and other welding machines as the heat source, through the addition of wires, under the control of a program, according to a three-dimensional digital model from the line-plane-body gradually formed from the advanced digital manufacturing technology of metal parts. Compared with laser and electron beams, the arc has a large heat source and high deposition efficiency. WAAM has become a common focus of research [10–13]. WAAM recently became especially popular in technical fields such as aerospace, marine engineering, and mechanical equipment [14–17].
The Inconel 625 nickel-based alloy is non-magnetic, corrosion- and oxidation-resistant superalloy. Inconel 625 belongs to the Ni-Cr-Mo-Nb-type alloy. Ni and Cr provide the oxidation resistance of the alloy, and Mo can prevent pitting and interstitial corrosion during the alloy usage. Nb improves the alloy welding stability without being sensitized. From low temperatures to 1093 °C, the alloy maintains good yield, tensile, and creep strengths, excellent processability, weldability, and high-temperature corrosion resistance [18–21]. Inconel 625 is a very popular material in aerospace and marine as well as (petro)chemical applications for constructions of engines and gas turbine core components, subsea sensor controllers, ship exhaust pipes, chemical plant hardware, etc [22–25]. However, the traditional processes for manufacturing Inconel 625 workpieces are casting and cutting, which have the advantages of strong applicability and high accuracy of workpiece shapes, but also have disadvantages such as low material utilization, uneven structure, segregation, and porosity. Therefore, researching a new manufacturing process for Inconel 625 parts is of paramount significance.

The research on Inconel 625 additive manufacturing has attracted the interest of scholars [26–34]. Ramenatte et al studied and compared the microstructural and oxidation resistance properties of Inconel 625 samples prepared through traditional forging and laser additive manufacturing, and observed that the oxidation resistance properties of the former were better than those of the latter [35]. Ngueijoa et al studied the difference in microstructural and mechanical properties of forged and additively manufactured Inconel 625 specimens. The results indicated that the microstructural and mechanical properties of the latter were similar to or better than those of forged specimens [36]. Balbaa et al studied the effect of selective laser melting (SLM) parameters on the surface roughness, hardness, and residual stress of Inconel 625. The results indicated that the samples with small surface roughness and dense structure (97%) could be prepared by controlling the scanning and hatch directions [37]. Wang et al studied the microstructural and mechanical properties of WAAM Inconel 625 alloy and observed that the microstructure of the samples primarily contained primary cellular crystals, and the

| Table 1. Chemical composition of the Inconel 625 wire. |
|---------------------------------------------|
| Element | C    | Mn  | Si  | Fe  | Cr  | Nb(Cb)+Ta | Mo  | Ni |
| Wt%      | ≤0.10 | ≤0.5 | ≤0.50 | 20.0–23.0 | 3.15–4.15 | 8.0–10.0 | ≥58.0 |

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Table 2. WAAM parameters used in this work for sample fabrication.

| Diameter (mm) | Voltage (V) | Current (A) | Wire feed speed (m-min⁻¹) | Printing speed (mm-s⁻¹) | Tip to work distance (mm) | Gas flow (L-min⁻¹) | Interpass temperature (°C) | Overlap rate (%) |
|---------------|-------------|-------------|---------------------------|-------------------------|--------------------------|--------------------|---------------------------|-----------------|
| 1.2           | 18.6        | 91          | 4.5                       | 12                      | 10–15                    | 20                 | ≤150                      | 50              |
microhardness and tensile strength of the samples increased slightly with the increase in scanning speed (the deposition direction of each layer was perpendicular to previously deposited layer) [38]. Cardozo et al studied how different forms of Inconel 625 alloys used during plasma transferred arc (PTA) additive manufacturing. Their research results indicated that powder and wire materials had significant effects on the multilayer structure and mechanical properties of the fabricated samples, which was primarily due to the requirement to use different parameters during processing [39]. Hu et al analyzed how solid solution heat treatment affected microstructural and mechanical properties of Inconel 625 samples fabricated using laser additive manufacturing. They observed that at higher temperatures, the microhardness and yield strength of the sample decreased, and the elongation of the sample increased significantly. Still, the tensile strength did not change significantly, and the Laves phase and dislocation density decreased [40].

However, there are relatively few reports on the corrosion resistance of Inconel 625 additive manufactured samples, and corrosion resistance is one of the excellent properties of Inconel 625 nickel-based alloy. Therefore, we studied microstructural and intergranular corrosion properties of the samples fabricated using Inconel 625 as raw material and additive manufactured WAAM techniques. We believe that our results are essential as a theoretical and experimental basis for the widespread application of Inconel 625 alloy as a raw material for additive manufacturing technologies.

2. Experimental procedure

Inconel 625 wire 1.2 mm in diameter, used throughout this work, corresponds to the standard ASME SFA-5.14 ERNiCrMo-3. Its chemical content is presented in table 1. The WAAM system ArcMan-600 is shown in figure 1. The experimental setup was equipped with a TPS4000 cold metal transfer (CMT) advanced power supply. The experimental substrate was a Q235 steel plate (300 × 100 × 10 mm). Single-layer single-pass, single-layer multi-pass, and multilayer multi-pass tests were performed, and the better additive manufacturing process parameters were selected according to the weld bead forming morphology. A Fluke F59 infrared thermometer was used for interlayer temperature measurements. All manufacturing parameters are shown in table 2. High purity (99.999%) Ar was used as a shielding gas.

The sample printed by WAAM in this research was approximately 150 mm × 20 mm × 60 mm. The chemical composition was determined according to ASTM E2594 by an Agilent 51105VDV inductively coupled plasma emission spectrometer, Optima 2100DV inductively coupled plasma emission spectrometer (ICP-OES), and CS800 carbon and sulfur analyzer; the environmental conditions were temperature 24.5 °C, humidity 52.7%. A 100-kN material Sintech 20/G testing machine was implemented for tensile tests performed at 25 °C according to the standard ASTM E8/E8M-16a. The sample impact test was performed at −50 °C, according to ASTM E23-2018, using the ZBC2302-C impact tester. Three different samples were measured for each group. For metallography and scanning electron microscopy (SEM), each sample was cut, ground, polished, and then etched using an aqua regia solution. The microstructure of the metallographic specimens was observed using an Olympus GX71 metallographic microscope. The electron backscatter diffraction (EBSD) (TESCAN MIRA3) analysis was conducted at an accelerating voltage of 20 kV, the specimen tilt angle of 70°, and speed of acquisition was 248.53 Hz. A Quanta650 SEM was used to observe the tensile fracture and corrosion morphologies of the samples after the intergranular corrosion test. A transmission electron microscope (TEM, JEM-2100) was used to observe the crystal structure of the samples, and the acceleration voltage was 200 kV. The microhardness was measured using 100 g force. The hardness tester was VMH-104. During the test, 10 points (at
1 mm equidistant points) were measured at equal intervals in both horizontal and vertical directions of the sample cross-section. The sulfuric acid-iron sulfate intergranular corrosion test was performed according to the requirements of ASTM G28-15. The sample was degreased, measured, and weighed before the test, and it was continuously boiled in iron sulfate-50% sulfuric acid solution for 120 h and removed. After the intergranular corrosion test, the sample was washed and weighed. The corrosion degree was determined by comparing the sample weight change.

3. Results and discussion

3.1. Forming characteristics

Figure 2 shows a sample of the Inconel 625 wall sample printed using WAAM. The figure indicates that the Inconel 625 thin-wall sample printed using the CMT power supply was well-formed, no collapse occurred, and the weld splashing was minimal during the welding process. The excellent forming of the test specimen benefited from the use of CMT power. Innovative CMT technology that partially decouples the arc electrical transients from the filler wire feed rate is based on the MIG/MAG (Metal Active Gas Arc Welding) welding, which digitally coordinates the wire feeding movement and the droplet transition. The droplet short-circuit signal is detected by the welding machine processor, sending the feedback to the wire feeding mechanism and pulling back the welding wire to help the droplet to fall off, so that the droplet transition is in a nearly no-current state. The entire welding process achieves ‘hot-cold-hot’ alternating conversion with up to 70 conversions per second. The welding heat input is significantly decreased, enabling spatter-free, high-quality MIG/MAG welding. During the WAAM process, decreasing the heat input can decrease the heat accumulation in the multilayer and multi-pass surfacing process, prevent collapse, and facilitate the forming control [41-43]. In addition, from the sample cross-section view (metallographic sample), depicted in figure 3, virtually no defects were observed within the sample and the metallurgical bonding was excellent, which further illustrates that Inconel 625 WAAM sample had an excellent forming performance.

3.2. Microstructure

Table 3 presents the chemical composition of the Inconel 625 WAAM samples. The comparison of the chemical composition requirements of the raw wire materials (table 1) indicated that the content of the primary alloying elements Ni, C, Mn, Si, Fe, Cr, Nb(Cb)+Ta, and Mo in the WAAM sample satisfied the requirements of the ASME SFA-5.14 ERNiCrMo-3 standard because the heat input of the CMT power source was low in this study. High purity argon gas was used as a protective gas in the additive manufacturing process, so the alloy element burn-out was relatively small.

Figure 4 demonstrates the optical micrographs of the Inconel 625 WAAM samples at different locations. Figure 4(a) shows the low magnification metallographic structure: the columnar crystal zone is below, the...
remelting zone is in the middle, and the heat-affected zone is above the remelting zone. Figures 4(b) and (c) indicate that the metallographic phases of the columnar crystal zone of the Inconel 625 WAAM sample were $\gamma$-Ni and granular precipitates. The metallurgical structures in the remelted zone were finer $\gamma$-Ni and granular precipitates (figures 4(d), (e)). The comparison of the heat-affected zone structures indicated that the size of the particulate precipitates in the remelted zone increased significantly because (according to the TEM results), the

Table 3. Chemical composition (wt%) of the Inconel 625 sample fabricated using WAAM.

| Element | C    | Mn  | Si   | Fe  | Cr  | Nb(Cb)+Ta | Mo  | Ni    |
|---------|------|-----|------|-----|-----|-----------|-----|-------|
| Weight Percent | 0.023 | 0.01 | 0.053 | 0.376 | 22.96 | 4.01      | 9.46 | 65.34 |
primary component of the precipitate was observed to be NbC. The melting point of NbC is higher at 3500 °C, causing it to be difficult to melt during remelting. NbC grains grew further when the remelting zone solidified, so the particle size was larger. The metallurgical structure of the heat-affected zone of the sample also exhibited γ-Ni and granular precipitated phases (figure 4(d) above and (f)). In addition, the low magnification metallographic photograph in figure 4(a) indicates that no cracks, pores, slag inclusions, unfused and unwelded parts, poor shape and size, and other defects were observed in the sample, indicating that the internal quality of the Inconel 625 WAAM sample was high. Compared with the research results reported in the literature, the grain size of the Inconel 625 sample prepared using the CMT WAAM technology was observed to be smaller than that of Inconel 625 samples prepared using traditional casting technology, which was the primary reason the mechanical properties of the former were better than those of Inconel 625 samples prepared using casting technology [44, 45].

Figure 5 demonstrates EBSD maps of the Inconel 625 WAAM sample. The figures indicate that the image quality map, Euler angle color map, phase map, grain boundary distribution map, inverse pole figure map, inverse pole figures and pole figures of Inconel 625 sample. It can be seen from the high magnification EBSD image that the grain shape of the sample is mainly elongated. The phase composition is mainly nickel phase. It can be seen from the inverse pole figures and pole figures that the crystal orientation is biased towards the (001) crystal orientation.

TEM micrographs and selected area electron diffraction (SAED) patterns of the Inconel 625 WAAM sample are shown in figure 6. there was a granular precipitated phase in the slab-like matrix phase shown in figure 6(a). the corresponding SAED pattern indicated that it was a Ni phase. Figure 6(b) is the TEM image of the intergranular precipitates and the corresponding SAED pattern. the precipitates were irregular blocks of approximately 300 nm. the SAED patterns indicated NbC, which is consistent with the results of Inconel 625 samples prepared by other researchers using additive manufacturing technologies [46, 47]. In addition, other regions of the sample (figure 7), in addition to intergranular precipitates, numerous dislocations were also observed. The appearance of intergranular particle precipitation phases and dislocations is conducive to improving the strength of the sample.

3.3. Microhardness and mechanical properties
The microhardness distribution of the Inconel 625 WAAM sample is shown in figure 8. ten points were measured at equal distances from the top of the sample (2 mm) to the bottom, and from the left (2 mm) to the right, at 1 mm equidistant points. From the microhardness distribution diagram, the microhardness distribution of Inconel 625 WAAM sample in the transverse direction was observed to range from 231 to 258 HV0.1, and the
longitudinal microhardness distribution ranged from 230 to 263 HV$_{0.1}$. The microhardness distribution was relatively uniform. The average microhardness values in the transverse and longitudinal directions were 243.5 and 243.3 HV$_{0.1}$, respectively. The average microhardness values in the longitudinal and transverse directions were almost the same. Minor fluctuations in the microhardness values were caused by the sample microstructure itself.

The cross-sectional morphology of the Inconel 625 WAAM sample (figure 2) indicated that the produced sample had a multilayer and multi-pass deposition structure. The thermal cycling effect of the same layer of weld bead generally included the original columnar crystal, remelting, and heat-affected zones. Different zones exhibited

**Figure 6.** Microstructures of the Inconel 625 sample fabricated using WAAM: (a) matrix and corresponding SAED, (b) intergranular precipitates, and corresponding SAED.

**Figure 7.** TEM images of the Inconel 625 sample fabricated using WAAM: (a) particles + dislocations, (b) dislocations.
different microhardness due to different microstructures (grain size, precipitates, etc). Therefore, the microhardness of the sample section fluctuated.

Mechanical property test results indicated that the samples had excellent mechanical properties: the yield strength was 450 MPa, tensile strength was 736 MPa, elongation was 38%, the percentage reduction of area after fracture was 52%, and the impact value at room temperature was 152 J, which agrees with the results obtained by other researchers [19, 31, 38]. The performance was better than that of the Inconel 625 sample prepared by sand casting [44].

Figure 9 shows the tensile fracture morphology of the Inconel 625 WAAM sample for analysis of the tensile fracture mechanism of the specimen. The low magnification image of the tensile fracture morphology (figure 9(a)) shows that there were no apparent defects in the tensile sample, which was consistent with the experimental results shown in table 4 that the tensile specimen had a high elongation and decrease in area. A high magnification image of the tensile fracture morphology showed numerous dimples in the fracture (see figure 9(b)), indicating that the tensile fracture mechanism of the sample was primarily a ductile fracture, which is consistent with the higher elongation, area decrease, and impact toughness results of the samples shown in table 4.
3.4. Corrosion performance

Table 5 shows the intergranular corrosion results after the 120 h boiling in ferric sulfate-50% sulfuric acid. The sample was measured, and the weight loss calculated (density was $8.44 \text{ g cm}^{-3}$). From the results of intergranular corrosion, the average corrosion rate of the Inconel 625 sample prepared using WAAM was observed to be $0.609 \text{ mm/year}$, indicating that it had excellent resistance to intergranular corrosion. This resistance is better than that reported in the literature for the Inconel 625 alloy with electroslag strip cladding layers [48] and tungsten arc welding joints [49]. The primary reason for this performance may be the use of a CMT power source for the WAAM in this study. The heat input was low, and the prepared Inconel 625 sample had lower intergranular precipitation; thus, the intergranular corrosion resistance was excellent. SEM of the sample after the intergranular corrosion test showed that the sample surface possessed grooves and corrosion pits along the grain boundaries (see figure 10). The corrosion pits were nearly circular. The formation of trenches and corrosion pits was due to the removal of metal ions from the grain boundary corrosion and the high activation energy associated with the grain boundaries, which caused them to corrode with higher chemical activity than adjacent crystals. These are typical intergranular corrosion characteristics. A primary reason for the intergranular corrosion may be due to the difference in the elemental composition between the various zones of the sample (grain interior, grain boundaries, and precipitates). For example, there were NbC precipitates in the Inconel 625 sample prepared using WAAM in this study. The precipitation of NbC caused the formation of Nb-depleted regions around the precipitates, which altered the element composition of the region. In addition, the existence of dislocations in the crystal structure exacerbated the occurrence of intergranular corrosion [50].

![Figure 10. Corrosion morphology of samples after intergranular corrosion test: (a) low magnification, (b) high magnification.](image1)

Table 4. Yield and tensile strengths, elongation, area decrease after fracture, and impact value for the Inconel 625 sample fabricated using WAAM.

| Sample | Yield Strength (MPa) | Tensile Strength (MPa) | Elongation (%) | Percentage decrease in the area after fracture (%) | 25 °C Impact value (J) |
|--------|----------------------|------------------------|----------------|--------------------------------------------------|-----------------------|
| Sample | 450                  | 736                    | 38             | 52                                               | 135, 157, 164         |

Table 5. Intergranular corrosion test results.

| Sample | Length (mm) | Width (mm) | Thickness (mm) | Mass loss (g) | Corrosion rate (mm/year) | Average corrosion rate (mm/year) |
|--------|-------------|------------|----------------|---------------|--------------------------|----------------------------------|
| 1–1    | 30.15       | 19.68      | 3.05           | 0.1021        | 0.592                    | 0.609                            |
| 1–2    | 30.09       | 19.69      | 3.02           | 0.0989        | 0.576                    |                                  |
| 1–3    | 30.12       | 19.67      | 2.73           | 0.1110        | 0.659                    |                                  |
4. Conclusions

Inconel 625 samples were prepared using WAAM. The forming quality of the sample was good, no significant defects were present, and the metallurgical bonding inside the sample was good. The metallographic structure exhibited primarily γ-Ni and granular precipitated phases. The microhardness distribution was relatively uniform. The average microhardness of the transverse section of the sample was 243.5 HV0.1. The average microhardness of the longitudinal section was 243.3 HV0.1. The sample mechanical properties were outstanding: the yield strength was 450 MPa, tensile strength was 736 MPa, the elongation was 38%, the decrease in the area was 52%, and the room temperature impact value was 152 J. The intergranular corrosion test results indicated that the morphology of corrosion was characterized by intergranular grooves and corrosion pits produced by intergranular corrosion. The average corrosion rate of the sample was 0.609 mm/year, which indicated excellent resistance to intergranular corrosion.

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