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Improvement in corrosion resistance of wire arc additive manufactured Inconel 625 alloy through heat treatment

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Abstract

In this study, an Inconel 625 component was fabricated by gas tungsten arc welding-based additive manufacturing and the as-deposited specimens were heat treated at 980 and 1100 °C for 1.0 h, respectively. The effects of heat treatment on the corrosion resistance and microstructure were investigated. Potentiodynamic polarization tests showed that the as-deposited Inconel 625 alloy had disparities in corrosion resistance compared with wrought Inconel 625 alloy. The corrosion resistance deteriorated after heat treatment at 980 °C due to needle-like δ phases provided more sites to pitting initiation. While the corrosion potential (Ecorr) increased by 32%, passivation current density (Icorr) decreased by 52% after heat treatment at 1100 °C, which was comparable with that of wrought Inconel 625 alloy. Detailed microstructural examination demonstrated the recrystallization occurred with the dissolution of Laves and δ phases, weakening of ⟨001⟩ orientation, decrease of low angle grain boundaries and formation of large numbers of stable twin grain boundaries. All the evolution of the crystal and microstructure contributed to the striking corrosion resistance of the 1100 °C heat-treated Inconel 625 alloy.

1. Introduction

Inconel 625 Ni-based alloy has been widely used for components in critical industrial scenarios due to its extraordinary corrosion resistance and mechanical properties, such as in engine and gas turbine core components, underwater sensor controllers, ship exhaust pipes, and chemical plant hardware etc [1–4]. Many Inconel 625 components have complex geometric shapes, which make them costly to manufacture by traditional methods [5]. Additive manufacturing (AM) is a ‘bottom-up’ manufacturing method of material accumulation, which has a high material utilization rate and efficiency and can realize the manufacturing of complex structural components, providing an effective method for the manufacturing of difficult-to-process and valuable components [6]. Moreover, wire arc additive manufacturing (WAAM) has the advantages of low cost and high deposition efficiency for manufacturing large-size components [7]. However, the rapid heating and cooling AM process appear to be detrimental to corrosion resistance. Zhang et al [8] have studied the microstructure and corrosion resistance of Inconel 718 alloy parts fabricated by AM. The results showed that the corrosion resistance of AM Inconel 718 alloy was lower than that of wrought Inconel 718 alloy. There were several reasons for the inferior corrosion resistance of AM materials.

Firstly, the metal solidification process is non-equilibrium, which allows element segregation and leads to precipitation of harmful phases [9, 10]. Wang et al [11] have investigated the microstructure and mechanical properties of AM Inconel 625 alloy fabricated by gas tungsten arc welding and found that there were different amounts of Laves phase precipitated in different positions of the thin wall component. Xu et al [12] have investigated the corrosion resistance of X 65–Inconel 625–clad pipe surfacing joint and found that the segregation of Laves phases in dendrites decreased corrosion resistance. Therefore, it is crucial to control element segregation and precipitates to obtain better corrosion resistance.

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Marchese et al. [13] have studied the microstructure of laser powder Inconel 625 and found that the microstructure of as-deposited material had strong orientation. In the process of WAAM, due to the ‘bottom-up’ deposition mode, the substrate experienced the process of heat dissipation, resulting in a large temperature gradient in the vertical direction, leading to the directional growth of dendrites and thus resulting in a clear surface texture [14, 15]. According previous studies [8, 16], the corrosion resistance of crystal faces with different orientations was considered to be quite different. Other crystal information, such as grain boundary characteristics and dislocation density, also have great influence on corrosion resistance [17].

In conclusion, there are many adverse factors affecting corrosion resistance of as-deposited components due to the rapid heating and cooling characteristics of AM.

Furthermore, Marchese et al. [13] have studied the microstructure of laser powder Inconel 625 and found that the microstructure of as-deposited material had strong orientation. In the process of WAAM, due to the ‘bottom-up’ deposition mode, the substrate experienced the process of heat dissipation, resulting in a large temperature gradient in the vertical direction, leading to the directional growth of dendrites and thus resulting in a clear surface texture [14, 15]. According previous studies [8, 16], the corrosion resistance of crystal faces with different orientations was considered to be quite different. Other crystal information, such as grain boundary characteristics and dislocation density, also have great influence on corrosion resistance [17].

In addition, Mercelis and Kruth [18] have explained the formation mechanism of residual stress in AM materials using a cool-down phase model. Due to layer-by-layer fabrication, each deposition layer undergoes rapid heating and cooling cycles, resulting in the formation of large residual thermal stress in the material. The existence of residual stress in the as-deposited material also has an adverse effect on AM metal corrosion. In conclusion, there are many adverse factors affecting corrosion resistance of as-deposited components due to the rapid heating and cooling characteristics of AM.

When manufacturing large-size parts with multi-layer and multi-channel under large heat input, the residual thermal stress will be larger [19]. Moreover, the dendrite of WAAM Inconel 625 [20] was coarser than that of the laser power AM Inconel 625 [21], and the segregation was more obvious. This may pose a greater threat to the corrosion resistance of WAAM components. Therefore, it is necessary to optimize the corrosion resistance of Inconel 625 alloy after WAAM process. According to the recently investigations [9, 10], heat treatment had significant effect on microstructures of AM Inconel 625 alloy. Hence it can be used as a possible method to improve the corrosion resistance. However, it is not clear yet how the corrosion behavior will be for the heat-treated WAAM Inconel 625 alloy with the unique microstructure.

Therefore, the main purpose of this study was to improve the corrosion resistance of Inconel 625 alloy through heat treatment. WAAM Inconel 625 pieces were heat treated at 980 and 1100 °C for 1.0 h, respectively, followed by a water quench. The corrosion resistance of as-deposited and heat-treated samples were evaluated in terms of electrochemical corrosion. The effects of grain orientation, grain boundary, and precipitation on corrosion resistance were investigated.
2. Experimental procedure

2.1. WAAM process and materials

The Inconel 625 alloy was prepared by gas tungsten arc welding-based WAAM technology. The setup of the WAAM process was shown in figure 1(a). It consisted of a wire feeding device, a welding torch, a DC welding power source (TransTig 5000 Job G/F, Fronius International GmbH) integrated with a welding robot (IRB 2600, ABB Ltd), and an inert gas protective cover connected to an argon gas tank, which was used to isolate the component from the atmosphere during the additive process to reduce metal oxidation at high temperature. Inconel 625 wire was used as filler metal with a diameter of 1.2 mm. The substrate was a Q345A plate with dimensions of 200 × 150 × 20 mm³, which was cleaned with acetone to remove the grease or oil before deposition. The selected process parameters were shown in table 1. After each layer was deposited, the oxide layer on the surface removed with a brush. The as-deposited sample was 285 mm in length, 35 mm in width, and 30 mm in height, and there were twenty layers, each layer had four passes (figure 1(b)). An x-ray fluorescence spectrometer (XRF-1800, Shimadzu Co., Ltd) was used to analyze the as-deposited material chemical composition and the results listed in table 2.

2.2. Heat-treatment

Heat treatments were performed at 980 and 1100 °C for 1.0 h, respectively, and followed by water quench (figure 2). The purpose for heat treatment at 980 °C was to eliminate residual stress and dissolve the Laves phase, which is the recommended intermediate-temperature annealing treatment [2, 22]. Heat treatment at 1100 °C was to achieve a solution annealing temperature for microstructural homogenization (ASTM B443, Grade 2).

2.3. Corrosion resistance and mechanical properties test

Potentiodynamic polarization tests were used to evaluate the corrosion resistance of the as-deposited and heat-treated samples. The experimental results were ensured not to be affected by iron (Fe) dilution in the substrate by...
choosing a sampling position in the middle of the side surface along the building direction (figure 3(a)). Test specimens were prepared by ultrasonic cleaning using ethanol, after mechanical grinding and polishing. The occurrence of crevice corrosion was prevented by sealing the interface between the specimen and epoxy resin with a silica gel sealant, according to Zhang et al [23]. Experiments were performed on a CHI-660E potentiostat (CH Instruments, Inc.) and the test solution was 3.5 wt% NaCl at 25 °C. The scan rate was 60 mV min⁻¹. The specimen embedded in epoxy resin as the working electrode, a platinum grid worked as the counter electrode, and a saturated calomel electrode (SCE) served as the reference electrode. In order to achieve a relatively stabilized open circuit potential, the specimen was immersed in the test solution for 1.0 h before anodic polarization. Repeatability was ensured by conducting all tests in triplicate.

Mechanical properties after heat treatment were confirmed to meet the ASTM B443 standard by performing tensile tests in the scanning direction (y-direction); the schematic picture of a tensile specimen was shown in figure 3(b). Tensile tests were carried out using a computerized MTS tensile testing machine (E45.105, MTS Systems Co., Ltd) at a speed of 0.5 mm·min⁻¹.

2.4. Metallurgical characterizations
The specimens for optical microscopy (OM, Axio Vert.A1, Carl Zeiss Suzhou Co., Ltd) analysis were etched with aqua regia. The specimens for scanning electron microscopy (SEM, JSM-7800F, JEOL Ltd) analysis were
electrolytically etched in a 10% oxalic acid solution at 10 V for 25 s to observe precipitates. The precipitated phase was analyzed by transmission electronic microscope (TEM, JEM-2100, JEOL Ltd) and energy dispersive spectrometer (EDS). Crystallographic information of each sample was examined using electron backscatter diffraction (EBSD). The specimens were electrolytically polished in a 5% HClO₄ + 95% ethanol solution at 30 V for 18 s. All the test surfaces for metallurgical characterizations were parallel to the x-z surface (same as corrosion test sample).

3. Results and analysis

3.1. Corrosion resistance and mechanical properties

Potentiodynamic polarization curves of as-deposited and heat-treated samples were shown in figure 4. Corrosion potential (Ecorr), passivation current density (Icorr), and pitting potential (Epit) were measured from the polarization curves. Lower Ecorr describes higher electrochemical activity and lower corrosion resistance [24]. The Icorr can be used to indicate the kinetics of corrosion process. Lower Icorr means that the corrosion rate is lower [25]. Epit is indicative of the occurrence of pitting corrosion. The electrochemical characteristics obtained from polarization curves clearly showed that the corrosion performance of as-deposited Inconel 625 alloy manufactured by WAAM was much worse than that of wrought Inconel 625 alloy (converted data according to the reference electrode) (table 3). The Epit of the as-deposited and heat-treated samples was basically the same. But after heat treatment at 980 °C, Ecorr decreased by 11% and Icorr increased by 36%, which indicated that the corrosion resistance had decreased. After heat treatment at 1100 °C, Ecorr significantly decreased by 14% and Icorr increased by 20%, which further confirmed the decrease in corrosion resistance.

| Table 3. Corrosion parameters of the polarization curves in 3.5 wt% NaCl solution. |
|--------------------------------|------|-------|------|
| Samples                      | Ecorr (mV) | Icorr (μA cm⁻²) | Epit |
| As-deposited                 | -467     | 0.741     | 654  |
| 980 °C                       | -517     | 1.01      | 656  |
| 1100 °C                      | -316     | 0.354     | 656  |
| Wrought [26]                 | -314     | 0.47      | 600  |

| Table 4. Mechanical properties of the as-deposited and heat-treated WAAM Inconel 625 alloy. |
|--------------------------------|-----------------|-----------------|-----------------|
| Samples                        | Yield stress (MPa) | Ultimate tensile stress (MPa) | Elongation (%) |
| As-deposited                   | 432 ± 13         | 727 ± 13         | 47.5 ± 0.9      |
| 980 °C/1 h                     | 355 ± 16         | 701 ± 5          | 48.9 ± 0.5      |
| 1100 °C/1 h                    | 312 ± 3          | 745 ± 14         | 62 ± 1.2        |
| Wrought [26]                   | 276 (min)        | 690 (min)        | 30              |

* Wrought grade 2, solution annealed at least at 1093 °C.

electrolytically etched in 10% oxalic acid solution at 10 V for 25 s to observe precipitates. The precipitated phase was analyzed by transmission electronic microscope (TEM, JEM-2100, JEOL Ltd) and energy dispersive spectrometer (EDS). Crystallographic information of each sample was examined using electron backscatter diffraction (EBSD). The specimens were electrolytically polished in 5% HClO₄ + 95% ethanol solution at 30 V for 18 s. All the test surfaces for metallurgical characterizations were parallel to the x-z surface (same as corrosion test sample).
increased to $-316$ mV and $I_{corr}$ was the lowest, indicating that the sample surfaces acquired better corrosion resistance after 1100 °C heat treatment, which was comparable with that of wrought Inconel 625 alloy.

Tensile tests carried out on as-deposited and heat-treated specimens. The results were within the requirements of ASTM B443 (figure 5 and table 4).

**Figure 7.** EDS maps of different elements in the as-deposited and heat-treated WAAM Inconel 625 alloy.

**Figure 8.** IPF images of as-deposited and heat-treated WAAM Inconel 625 alloy: (a) as-deposited; (b) 980 °C; (c) 1100 °C.
3.2. Microstructural characterization

The figure 6 showed the OM images of as-deposited and heat-treated WAAM Inconel 625 alloy. The typical dendrites were observed in as-deposited WAAM Inconel 625 alloy (figure 6(a)). The dendrite growth direction was roughly the same but not parallel to the build direction. The dendrites still existed after heat treatment at 980 °C (figure 6(b)). However, as heat treatment temperature increased to 1100 °C, the dendrites disappeared and recrystallization occurred (figure 6(c)).

The present EDS maps revealed that there was obvious element segregation in the interdendritic regions (figure 7). In Nb-bearing superalloys, Nb and Mo with low solution distribution coefficients (k < 1), tend to strongly segregate to liquid during nonequilibrium solidification [27]. Because of rapid solidification in the WAAM process, there was serious Nb and Mo segregation between dendrites. After heat treatment at 1100 °C, element segregation was homogenized concurrent with dendrite disappearance.
The effects of the heat treatment on crystal characteristics were further analyzed by EBSD. With the building direction of the samples parallel to the \( z \)-axis of the sample stage, inverse pole figures (IPF) clearly showed that as-deposited and 980 °C heat-treated WAAM Inconel 625 alloy had strong orientation near the \( \langle 001 \rangle \) direction (figures 8(a), (b)). As \( \langle 001 \rangle \perp \{001\} \), most of the grains of as-deposited and 980 °C heat-treated WAAM Inconel 625 alloy were concluded to show more \( \{001\} \parallel \) test surface (x-z). According to 1100 °C heat-treated WAAM Inconel 625 alloy (figure 8(c)), the orientation of \( \langle 001 \rangle \) direction was found to be weakened due to recrystallization.

According to the degree of misorientation angle (\( \theta \)), grain boundaries can be divided into low angle grain boundaries (LAGBs, \( \theta < 15^\circ \)) and high angle grain boundaries (HAGBs, \( \theta > 15^\circ \)). The grain boundary images showed that there were high proportions of LAGBs distributed at grains interior of as-deposited and 980 °C heat-treated WAAM Inconel 625 alloy (figures 9(a), (b)). The LAGBs almost disappeared and high fraction of twin grain boundaries (TGBs, \( \theta = 60^\circ \)) were observed (figures 9(c), (d)) after 1100 °C heat treatment. TGB is a kind of \( \Sigma 3 \) coincidence site boundaries, with the atoms on both sides of the twin boundary stacked in a mirror symmetry relationship, which reduces the system free energy to the maximum extent [28].

### 3.3. Phase precipitation

SEM images of precipitates in as-deposited and heat-treated WAAM Inconel 625 alloy showed that, in as-deposited WAAM Inconel 625 alloy, there were many irregular bright precipitates in interdendritic regions (figure 10(a)). From results of EDS (table 5), the precipitates were concluded to be Laves phases, which were common in welded and AM Inconel 625 alloy [29, 30]. After heat treatment at 980 °C, Laves phases dissolved and large numbers of needle-like precipitates occurred in interdendrite regions (figure 10(b)). TEM analytical

![Figure 11. TEM images of precipitates in heat-treated WAAM Inconel 625 alloy: (a), (b) \( \delta \) phase and its SAED pattern, (c), (d) MC-type carbide and its SAED pattern.](image)

| Table 5. The chemical compositions (wt.%) of different phase. |
|-------------------|---|---|---|---|
| Element | Laves | \( \delta \) | MC | \( \gamma \)-matrix |
| Ni | 41.13 | 58.22 | 24.11 | 62.25 |
| Cr | 15.96 | 7.30 | 11.13 | 22.22 |
| Nb | 17.84 | 34.48 | 23.61 | 3.95 |
| Mo | 20.83 | — | 4.74 | 9.50 |
| Ti | 0.44 | — | 36.41 | 0.36 |

![Table 5.](image)
results (figures 11(a), (b)) showed that needle-like precipitates, which occur in intermetallic $\delta$ (Ni$_3$Nb) phases, exhibited lattice parameters of $a = 0.51$ nm, $b = 0.42$ nm, and $c = 0.45$ nm with an orthorhombic (DO$_2$) structure. As the heat treatment temperature increased to 1100 °C, $\delta$ and Laves phases dissolved, and a substantial number carbides were observed (figure 10(c)). The chemical composition of the carbide was shown in table 5, which was rich in Nb and Ti. TEM analytical results showed that these carbides consisted of a lattice parameter of 0.43 nm and average size of $\sim 300$ nm with a face-centered cubic (FCC) crystal structure (figures 11(c), (d)), which confirmed the precipitates were MC carbides.

4. Discussion

Potentiodynamic polarization tests showed that corrosion resistance of the as-deposited and 980 °C heat-treated WAAM Inconel 625 alloy was inferior, and it was significantly improved after heat treatment at 1100 °C. Microstructural examination demonstrated that there were obvious differences in crystal characteristics and precipitates between 980 °C and 1100 °C heat treated samples. The influence of microstructures on corrosion resistance after heat treatment will be discussed from the aspects of crystal structure and precipitate characteristics.

4.1. Effects of crystal characteristics on corrosion resistance

The IPF results illustrated that there was obvious (001) orientation in the as-deposited and 980 °C heat-treated WAAM Inconel 625 alloy (figure 8). The (001) texture was considered to promote the formation of porous NiO and inhibit dense Cr$_2$O$_3$ in the passive film, which yielded the material corrosion prone [31]. However, in this investigation, with the weakening of (001) orientation after heat treatment at 1100 °C, it could be supposed that the porous passivation film on the surface had been reduced, which contributed to higher $E_{\text{corr}}$ and lower $I_{\text{corr}}$.

As shown in the grain boundaries images (figure 9), the transformation of grain boundary after heat treatment at 980 °C was not obvious. However, LAGBs decreased and a large number of TGBs appeared due to
recrystallization after heat treatment at 1100 °C. The grain boundary or subgrain boundary can be one of the main reasons for accelerated pitting \[32\]. Therefore, different grain boundary types and grain boundary density have great influence on the corrosion resistance. The statistical results (figure 12) showed that the grain boundary density changed little after different heat treatment processes. Therefore, it could be inferred that the change of grain boundary types contributed to the improvement of corrosion resistance after 1100 °C heat treatment.

Combined with the grain boundaries results (figure 9) and kernel average misorientation (KAM) maps (figure 13), it can be found that higher KAM values distributed at LAGBs in as-deposited and 980 °C heat-treated WAAM Inconel 625 alloy (indicated by arrows). The KAM value is an index of local grain misorientation, which can be used to describe local dislocation densities and local strain levels \[33\]. The higher the KAM value, the larger the dislocation density and the more severe the strain concentration. The high fraction of LAGBs were caused by the presence of substructures with an array of dislocations during the AM rapid solidification process \[19\]. Therefore, the appearance of LAGBs were accompanied by higher dislocation density and strain. In electrochemical corrosion, the anodic reaction tended to happen at high dislocation density region \[34\]. Hence the LAGBs with high dislocation density were prone to initiate pitting. Thus, the high fraction of LAGBs were harmful for pitting resistance of the as-deposited and 980 °C heat-treated Inconel 625 alloy. After heat treatment at 1100 °C, the high distortion energy stored in the strained microstructure, as a driving force of recrystallization \[35\], caused substantial recrystallized twin grains with small distortions. High fraction of TGBs will keep the boundary diffusivity closer to the bulk diffusivity \[17\], which result in the formation of a continuous passive film in the corrosion process and reduce the occurrence of pitting. Therefore, the grain boundary transformation contributed to the improvement of corrosion resistance after heat treatment at 1100 °C.

In addition, the grain boundary density and grain boundary type changed slightly after 980 °C heat treatment. However, the average KAM value slightly decreased from 0.867° to 0.813°, indicating the annihilation of partial dislocations. Theoretically, it was beneficial to corrosion resistance of metal \[16\]. But in fact, the corrosion resistance was deteriorated after 980 °C heat treatment. This meant that other adverse factors played a leading role in the decline of corrosion resistance, such as the precipitates, which will be discussed in the next section.

### 4.2. Effects of precipitation on corrosion resistance

As shown in figure 8, there were plenty of Laves phases in the as-deposited WAAM Inconel 625 alloy, and they dissolved with appearance of δ phase after heat treatment at 980 °C. The SEM morphology of the as-deposited and heat-treated samples after electrochemical corrosion was shown in figure 14. Nucleation sites of pitting corrosion were seen to be preferentially generated at the boundary of Laves phase/matrix interfaces and resulting in microcrack expansion (figure 14(d)). EDS analysis showed that the Ni and Cr contents in Laves phase were lower than those in the matrix, and Mo and Nb contents in Laves were higher than those in the matrix (table 5). Due to differences in chemical composition, the potential between the second phase particles...
and matrix was different, resulting in the formation of micro-galvanic effect, which was one of the main driving forces inducing pitting corrosion [36].

After heat treatment at 980 °C, Laves phase transformed into Nb-rich δ phase. Formation of Nb-rich phase was usually accompanied by decreased Nb concentrations in the surrounding matrix, such that decreased Nb led to the increase of porous NiO passive film and the decrease of Cr2O3 passive film [8], which reduced passivation film stability, as well as creating sensitive locations for pitting. Furthermore, micrographs of corroded surfaces showed that there were more contact interfaces between the needle-like phases and matrix than that between Laves and matrix (figure 14(e)). Consequently, with increased locations of micro-galvanic effects, pitting initiation sites increased, thus the corrosion rate was improved. It was worth noting that the crystallographic characteristics changed slightly after heat treatment at 980 °C, which indicated that the transformation from Laves phase to δ phase may be the main reason for the increase of Icorr and decrease of Ecorr. After 1100 °C heat treatment, only fine microcracks were found around MC carbides (figure 14(f)), while the pitting initiation sites were significantly decreased due to the dissolution of Laves phase, consequently, the Icorr decreased and Ecorr increased.

Briefly, all of the crystal and microstructure characteristics of the 1100 °C heat-treated WAAM Inconel 625 alloy were beneficial to an outstanding corrosion resistance, the recommended heat treatment temperature of WAAM Inconel 625 alloy was 1100 °C.

5. Conclusions

In this study, Inconel 625 samples fabricated by WAAM were heat treated at 980 and 1100 °C for 1.0 h, respectively. The effects of crystal and microstructural features on the corrosion performance were analyzed. The major conclusions were summarized as follows:

1. Due to the special characteristics of WAAM process, as-deposited Inconel 625 alloy had a large number of LAGBs and strong (001) orientation, and there were many Laves phases distributed in chains among dendrites regions. They were detrimental to corrosion resistance.

2. Heat treatment at 980 °C exhibited negligible effect on crystalline structure. Although the dislocation density decreased and the Laves phase between dendrites dissolved, large amounts of δ phases precipitated. There were more interfaces between the δ phase and matrix than those between Laves phase and matrix, such that the Ecorr decreased by 11% and Icorr increased by 36%, indicated the corrosion resistance became worse.

3. After heat treatment at 1100 °C, the Laves and δ phases dissolved, a large number of stable TGBs formed with attendant decreased LAGBs, and the orientation of (001) weakened. Therefore, the corrosion resistance was significantly improved (Ecorr increased by 32%, Icorr decreased by 52%) under the synergistic effects of precipitation dissolution and crystal structural optimization.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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