Influence of heat treatments on microstructure, mechanical properties and corrosion resistance of Inconel 625 overlay cladded using PTIG

Longlong Guo 1, Fei Xiao 2, Fan Wang 2, Wenlan Wei 1, Yutian He 1 and Foshu Luo 1

1 Mechanical Engineering College, Xi’an Shiyou University, Xi’an, 710065, People’s Republic of China
2 Chongqing Pump Industry Co., Ltd, Chongqing, 400033, People’s Republic of China

* Author to whom any correspondence should be addressed.
E-mail: 15108208278@163.com and llguo@xsyu.edu.cn

Keywords: Inconel 625 overlay, heat treatments, microstructure, mechanical properties, corrosion resistance

Abstract
The influence of post-weld heat treatment (PWHT) temperatures on the microstructure, mechanical properties and corrosion resistance of Inconel 625 overlay cladded using pulsed tungsten insert gas (PTIG) was studied. The microstructure observation shows that as welded Inconel 625 overlay exhibits notable difference in grains morphology and size, and some precipitates, such as laves phase and MC phase, are distributed in the interdendritic zone. With an increase in PWHT temperature, equiaxed dendrites transform to columnar dendrites, and the overall difference in the microstructure decreases gradually. Compared with the as welded overlay, the size and number of the precipitates for the overlay heat treated at 650 °C shows very slight change. When the PWHT temperature increases to 750 °C, δ phase precipitates with the dissolving of laves phase. The number of δ phase increases clearly, and its size coarsens obviously with a further increasing PWHT temperature. Tensile tests indicate that there is a slight increase in the yield strength (YS) and ultimate tensile strength (UTS), but a decrease in elongation with the raise of PWHT temperature, which is no greater than 750 °C. Conversely, when the PWHT temperature beyond 750 °C, the YS and UTS decrease, the elongation increases slightly. Corrosion tests in the environment containing H2S and CO2 reveal that the Inconel 625 overlay heat treated at 650 °C exhibits superior corrosion resistance than that of other temperatures. Whereas, with an increase in PWHT temperature, the corrosion resistance degenerates seriously for the formation of δ phase.

1. Introduction
With the increasing demand of energy, many deeper and ultra-deep oil and gas fields which contain high content of H2S/CO2 acid gas are being developed, such as Sichuan oilfield and Chongqing oilfield in China [1]. It is known that the presence of H2S and CO2 results in serious corrosive effect on the metal materials serviced in the process of gas extraction, transportation, and refining in a wet environment [2–4]. Therefore, the equipment, especially the wellhead and tree equipment used in these fields must possess excellent corrosion resistance in addition to satisfied strength and toughness. For the good mechanical properties and corrosion resistance, Inconel 625 has been a good material choice for the oil and gas equipment with high corrosion resistance [5, 6]. The main elements of Inconel 625 are Ni, Cr and Mo. The exist of Cr and Ni provides corrosion resistance against oxidizing environment, while Mo and Ni leads to good non-oxidizing corrosion [7, 8]. Aside from that, high level of Nb enhances the resistance to chloride stress corrosion cracking [9, 10]. For the relative high price and good weldability, Inconel 625 is widely used as weld overlay deposited on the corroded surface using welding process [11–13].

With the action of moving arc heat, the melting, solidification, and re-melting of the weld overlay and substrate leads to local plastic deformation, and serious residual stress after cladding. The high residual stress not
only influences the size and geometry stability of the equipment but also has detrimental effect on the toughness, fatigue strength, creep rupture strength, and resistance to stress induced cracking \[14, 15\]. Hence, it is very essential to perform heat treatment to the weldment. Ban et al \[16\] studied the microstructure of Inconel 625 cladding heat treated at different temperatures with a 24 h hold time, and the corrosion resistance by means of electrochemical tests. Xing et al \[17\] focused on the microstructure evolution of Inconel 625 cladded metal, which is heat treated by various heat treatment processes. Guo et al \[14\] investigated the influence of post weld heat treatment (PWHT) temperatures on the corrosion resistance of Inconel 625 overlay using electrochemical tests. Cortial et al \[18\] studied the effect of PWHT temperatures on the phase transform, elements segregation, mechanical properties, and pitting corrosion resistance of the Inconel 625 cladding welded by tungsten insert gas (TIG). Xu et al \[19\] studied the influence of heat treatments on the microstructure and mechanical properties of Inconel 625 metal deposited using pulsed plasma arc. Liu et al \[20\] focused on the influence of solution treatments on the microstructure and hardness of Inconel 625 overlay cladded using TIG. Masaylo et al \[21\] reported that the Inconel 625 overlay deposited by laser cladding process acquires a structure with extended acicular grains after the heat treated at 950 °C. However, so far only a few articles focused on the influence of PWHT temperatures on the microstructure, mechanical properties, especially the corrosion resistance in the corrosive environment containing CO2 and H2S. Therefore, the purpose of present study is to research the influence of PWHT temperatures on the microstructure, mechanical properties, and corrosion performance of the Inconel 625 overlay deposited by pulsed TIG (PTIG). Particularly, the corrosion resistance of the overlay was tested in a simulated high temperature and high pressure corrosive environment containing H2S and CO2.

2. Experimental

2.1. Materials and cladding

The substrate was AISI 4130 carbon steel plate with sizes of 150 × 150 × 25 mm. The nickel based ERNiCrMo-3 (Inconel 625) was selected as the filler wire, and its diameter was 1.2 mm. The chemical compositions of the substrate and filler wire were listed in table 1. Before cladding, the cladding surface was grinded to the roughness of 0.8 μm, and cleaned with acetone to remove contaminations. In addition, the substrate was preheated 300 °C to decrease the crack initiation. The cladding experiments were performed using a Fronius automatic PTIG system. The preheating current and background current were set at 70 A, 240 A, respectively. Meanwhile, the frequency was set at 5 Hz with 0.3 duty cycle, while the wire feed speed and welding speed were maintained at 2 m min$^{-1}$ and 18 cm min$^{-1}$. Pure argon gas was utilized as the shielding gas with a flow rate of 15 l min$^{-1}$. The length of each weld was about 125 mm and overlap ratio between the adjacent welds was 0.4. As presented in figure 1, the overlay is comprised two weld overlay layers to satisfy the corrosion resistance \[5, 8\].

Five groups of Inconel 625 overlay were cladded using the same process conditions. One was as welded sample, and designated as T1. While the other four groups were heat treated at 650 °C, 750 °C, 850 °C and 950 °C with a two hours hold time, and cooled in air \[14, 16, 22\]. The corresponding samples were marked as T2,

| Table 1. Chemical compositions of the substrate and filler wire (wt%). |
|-----------------|-----|-----|-----|-----|-----|-----|-----|-----|
| Element         | C   | Cr  | Ni  | Ti  | Fe  | Mo  | Al  | Nb  | Others |
| AISI 4130       | 0.29| 0.99| 0.02| 0.006| Bal | 0.175| 0.006| —   | 0.72   |
| Inconel 625     | 0.01| 22.65| 64.24| 0.2  | 0.32| 8.73 | 0.16 | 3.53 | 0.16   |

Figure 1. Macroscopic morphology of Inconel 625 overlay.
T₃, T₄, and T₅, respectively. Then, a series of tests were carried out to assess the effect of PWHT temperatures on the microstructure, mechanical properties, and corrosion performance. The test specimens were extracted from the Inconel 625 overlay with different heat treatments using wire cutting, as presented schematically in figure 2.

2.2. Microstructure characteristics
The surfaces characterized were electrolytically etched in a mixture solution of 12 ml H₃PO₄, 40 ml HNO₃, and 48 ml H₂SO₄ [14]. The etching time was about 15 s and the voltage is 6 V. Then, the microstructure of the Inconel 625 overlay was observed using an optical microscopy (OM) and a scanning electron microscope (SEM) which is equipped with energy dispersive spectrometry (EDS). The compositions and distribution of elements in the specimens were measured by EDS.

2.3. Mechanical properties test
To determine the effect of PWHT temperatures on the mechanical properties of the Inconel 625 overlay, tensile tests were performed with a normal strain rate of 1 mm min⁻¹, in accordance with the standard of ISO 6892-1: 2009 (Metallic Materials-Tensile Testing-Part 1: Method of test at room temperature). The specimens were extracted from the overlay using wire cutting, and the detail dimensions of the tensile specimens were presented in figures 2(b) and (c). After the tensile tests, fracture morphology was observed using the SEM.

2.4. Corrosion test
To evaluate the effect of PWHT temperatures on the corrosion resistance of the Inconel 625 overlay, corrosion tests were performed in a high temperature and high pressure autoclave. The corrosion specimens were cut from the overlay with dimensions of 30 × 15 × 2 mm, as presented in figure 2(a). All specimens were polished by silicon carbide paper up to 2000 grit size to eliminate coarse scratches, cleaned with alcohol, and dried with cold air. Subsequently, the specimens were weighed by an electronic analytical balance with a precision of 0.1 mg. To simulate corrosive environment of a gas field in china, the solution was prepared with deionized water, and the compositions of the solution are listed in table 2. Firstly, the experimental solution was deoxygenated by pure nitrogen. Subsequently, the corrosion test system was applied proposed partial pressure and temperature. The corrosive condition was simulated at a H₂S partial pressures of 3 MPa, CO₂ partial pressures of 4.5 MPa, total pressure of 10 MPa, and testing temperature of 90 °C. After 72 h of corrosion test, the specimens were taken out of the autoclave, washed with inhibitor, and dried in cold air. Subsequently, the specimens were weighted again to calculate the corrosion rate using the following equation [23]:

Figure 2. Schematic of test specimens extracted from the overlay (a) specimens of microstructure and corrosion test, (b) tensile specimens, and (c) dimensions of the tensile specimens.
Where \( CR \) denotes the general corrosion rate, \( mpy \); \( K \) denotes the constant for unit, \( 3.45 \times 10^6 \); \( W \) denotes the weight loss, g; \( A \) denotes the area of corrode surface, cm\(^2\); \( D \) denotes the density of overlay, g/cm\(^3\); \( T \) denotes the duration time of corrosion test, h.

3. Results and discussion

3.1. Microstructure characteristic

The SEM micrograph of the fusion region between the substrate and Inconel 625 overlay is presented in figure 3. It is observed that a good metallurgical bonding is formed between the Inconel 625 overlay and substrate, and there are no welding defects, such as porosity, slag inclusion and crack. For the difference in corrosion resistance, a clear boundary exists between the overlay and substrate. The microstructure evolution with the distance away from the fusion line are plane dendrites, cellular dendrites and columnar dendrites. As shown in figure 4, the content of Fe decreases sharply, while Cr, Nb and Mo increase obviously away from the fusion line. In addition, the content fluctuation of the elements in the overlay is not significantly. Generally, Fe is considered as an index of corrosion resistance \[24\]. Hence, high content of Fe means relative poor corrosion resistance. The content of Fe in the overlay is about 12%–18%, demonstrating that two layers of overlay are necessary to meet the requirement of the corrosion resistance \[8\]. From figure 5, it is found that the chemical compositions in the surface, which used to observe microstructure and measure corrosion resistance, are very similar to that of the Inconel 625 alloy. This indicates that depositing two cladding layers can effectively reduce the dilution caused by the substrate.

Figure 6 illustrates the microstructure of the Inconel 625 overlay heat treated at various temperatures. It is observed that the as welded microstructure exhibits significant difference in grains morphology and size, as presented in figure 6(a). The microstructure at the margin of previous weld is composed of a large number of equiaxed dendrites and seldom coarse columnar dendrites. Contrarily, a large number of dense columnar dendrites are distributed in the margin of later weld. The microstructure of the inner region of the weld is more complicated than that of the overlapping region. Away from the margin of weld, there is an evolution from dense columnar dendrites to coarse columnar dendrites and equiaxed dendrites.

From figures 6(a) to (e), it can be concluded that PWHT temperature has significant effect on the microstructure in morphology and grains size. Compared with the microstructure of as welded overlay, there is no remarkable variation in grains morphology and size of that treated at 650°C, as shown in figure 6(b). The
The microstructure of the overlay treated at 750 °C is mainly composed of columnar dendrites, and there is a transition from equiaxed dendrites to columnar dendrites, as shown in figure 6(c). As presented in figure 6(d), heat treated at 850 °C results in obvious growth of columnar dendrites comparing to the overlay treated at relative low temperatures. In addition, the primary microstructure of the overlay treated at 950 °C is almost coarse columnar dendrite, and its dendrite arm is up to 30 μm, as shown in figure 6(e). It is found that the weld interface is more and more blurred with the increase of PWHT temperature. Hence, it is confirmed that the equiaxed dendrites transform into columnar dendrites, dendrite spacing increase, and the overall difference in the microstructure decreases gradually with the increase of PWHT temperature. Moreover, there are some notable precipitates distributed in the overlay, which heat treated at 850 °C and 950 °C, as shown in figures 6(d) and (e).

Figure 7 shows the precipitates in Inconel 625 overlay treated at different PWHT temperatures. It is observed that there are some irregular shaped precipitates and tetragonal particles distributed in the as welded overlay, as presented in figure 7(a). According to the chemical compositions listed in table 3, the white irregular shaped precipitates consist of a larger number of Mo and Ni, and the tetragonal particles are obvious richer in C and Ni, comparing with the matrix. Considering the morphologies characteristic and elements of the precipitates, it is inferred that white irregular shaped precipitates are laves phase, whereas the tetragonal particles correspond to MC phase [25, 26]. Comparing with the as welded microstructure, there is no obvious change in quantity and size of the phases in the overlay heat treated at 650 °C, as displayed in figure 7(b). It is observed that laves phase seems to dissolve, and that some needle shaped precipitates are distributed in the overlay treated at 750 °C, as shown in figure 7(c). The element compositions of the needle shaped precipitates are also listed in table 3. Combining the orientations and chemical compositions, these needle shaped precipitates are identified as δ [Ni3Nb] phase [17, 27]. It is noted that the δ phase contains a great number of Cr, Ni Fe and Ti, besides Nb and Ni. This is because that Nb can be replaced by Ti and Cr, and Ni can be replaced by Fe and Cr during the formation of δ phase [28, 29]. For the Inconel 625 overlay treated at 850 °C, it can be seen that a substantial number of δ phase is appeared with significant reduction of laves phase, and that the MC phase completely disappear, as shown in figure 7(d). Figure 7(e) illustrates that the laves phase disappears, and that there is some
spindly δ phase located near the plate shaped δ phase. The results indicate that dissolution of δ phase in some directions boost it coarsens in other directions.

3.2. Mechanical properties
The tensile properties of Inconel 625 overlay heat treated at various temperatures are presented in figure 8. The yield strength (YS), ultimate tensile strength (UTS), and elongation of the as welded Inconel 625 overlay are 418 MPa, 704 MPa, and 48.14%, respectively. The Inconel 625 overlay is superior in strength, but slightly close
or inferior in elongation comparing with the Inconel 625 deposited by pulsed plasma arc and cast Inconel 625 [25, 30]. This indicates that the as welded Inconel 625 overlay exhibits good mechanical properties. When the temperature is not larger than 750 °C, the YS and UTS of the Inconel 625 weld overlay increase with the raising in PWHT temperature, while the elongation declines gradually. However, with a further increase in PWHT temperature, the YS strength and UTS decrease, while the elongation increase slightly. It can be concluded that

**Figure 7.** SEM micrographs of precipitates in Inconel 625 overlay heat treated at various temperatures (a) as welded, (b) 650 °C, (c) 750 °C, (d) 850 °C and (e) 950 °C.

**Table 3.** Compositions of phases in Inconel 625 overlay treated at various PWHT temperatures (wt%).

| Phases | C   | Nb | Mo | Ti  | Cr  | Fe  | Ni  |
|--------|-----|----|----|-----|-----|-----|-----|
| Matrix | —   | 3.04 | 8.33 | 0.38 | 22.21 | 2.01 | 64.03 |
| Laves  | —   | 17.28 | 14.41 | 0.58 | 17.55 | 1.37 | 48.81 |
| MC     | 8.88 | 52.05 | 8.91 | 2.69 | 7.74 | 2.73 | 17.01 |
| δ      | —   | 12.31 | 10.60 | 0.59 | 17.72 | 1.44 | 56.80 |
The Inconel 625 overlay heat treated exhibits better UTS, and poorer elongation than the as welded, and YS of the overlay heat treated are all larger than that of as welded overlay, excepting that heat treated at 950 °C.

The fracture morphologies of the Inconel 625 overlay heated treated at various temperatures were investigated by SEM, as shown in figure 9. There are many dimples distributed in the fracture surfaces revealing that the fracture mode of Inconel 625 overlay treated at various PWHT temperature belong to ductile transgranular fracture. The size and deep of dimples decrease with the increase in PWHT temperature, when the PWHT temperature is less than 850 °C. These are consistent with that the ductile of Inconel 625 overlay degenerates with the increase of PWHT temperature. Comparing with the as welded, the dimples of the overlay heat treated at 850 °C and 950 °C are almost distributed along the axis of columnar dendrites, and the depth of dimples decrease dramatically, as shown in figures 9(d) and (e).

The precipitated phase is one of important factors affecting the mechanical properties of the weld overlay. As presented in figure 5, the chemical compositions in the surface located at the secondary cladding layer are very similar to that of the Inconel 625 alloy. The sufficient content of elements such as Nb, Mo, and Cr boost the formation of precipitates. It is observed that there are many precipitates located in the bottom or boundary of the dimples. For the as welded Inconel 625 overlay, the formation of laves phase consume a lot of solution hardening elements such as Nb and Mo, resulting in poorer solution strengthening effect on the matrix [17, 18]. Moreover, laves phase is a brittle intermetallic compound, providing favourable sites for crack initiation and propagation [31]. Several studies reported that there is some fine and dispersed γ″ phase with a size of 10–30 nm precipitated in the Inconel 625 which is heat treated between 650 °C and 750 °C [15, 32]. The γ″ phase is precipitation strengthen phase, and fine γ″ phase is benefit to the YS and UTS of Inconel 625 [32]. Meanwhile, with an increase in PWHT temperature, the dissolution of laves phase provide a larger number of Mo and Nb for the formation of γ″ phase. Hence, the strength of Inconel 625 overlay heat treated at 650 °C and 750 °C shows positive proportion with heat treatment temperatures. On the contrary, the relationship between elongation and the PWHT temperature is negative proportion. Because γ″ phase is metastable phase, it is easy to transform into a stable δ phase when the PWHT temperature up to 750 °C. So, the formation of δ phase leads to a declining in the number of γ″ phase, degenerating the solid solution strengthening and precipitation strengthening [33, 34]. Moreover, the fine and dispersed δ phase is less detrimental to the mechanical properties compared with that of coarse and interconnected [19]. Compared to the fine grains, coarse grains induce more plugged dislocations at grain boundaries, and boost the initiation and propagation of cracks. Combination effect of grains coarsening and phases precipitation reasonably explains that the YS and UTS show negative proportion with the PWHT temperatures, which are higher than 750 °C. The variation of elongation can be attributed for the growth of grains and bad strengthening effect on the matrix.

### 3.3. Corrosion performance

Figure 10 shows the corrosion rate of the Inconel 625 overlay heat treated at different PWHT temperatures in high temperature and high pressure environment containing H₂S/CO₂. It is found that the PWHT temperatures have significant influence on the corrosion resistance of Inconel 625 overlay. The corrosion rate of the as welded Inconel 625 overlay is 0.356 36 mpy. With an increase in PWHT temperatures, the corrosion rate of the
corresponding Inconel 625 overlay heat treated are 0.5219 mpy, 1.49673 mpy, 4.62982 mpy, and 8.2482 mpy, respectively. The results indicate that heat treated at 650 °C is beneficial to improve the corrosion resistance of Inconel 625 overlay, while other PWHT temperatures, such as 750 °C, 850 °C and 950 °C are detrimental to the corrosion resistance. According to the corrosion rate for oil production system listed in the NACE RP 0075 standard, the corrosion rate of the Inconel 625 overlay heat treated at 650 °C is only half of the low corrosion [35]. It means that heat treated at 650 °C is a reasonable heat treatment for enhancing the corrosion resistance, and the corresponding overlay can satisfy the corrosion resistance against the serious corrosive environment.

The micrographs of the corroded surface of Inconel 625 overlay heat treated at different temperatures are shown in figure 11. It is found that there are some disconnected corrosion products distributed at the tested surface of all specimens. The difference of corrosion morphologies between the specimen heated at 650 °C and as welded is not very clear, and there are no obvious corrosion pits distributed at these two surfaces. However, some obvious corrosion pits located at the corroded surface of other specimens. Moreover, the depth and width of the corrosion pit shows an increasing tendency with the increase of PWHT temperatures. It is worth noting that small pits located at the Inconel 625 overlay treated at 950 °C will connect to form large pits, and some precipitates distributed in the corrosion pits. These micrographs of the corroded surfaces are consistent with the corrosion rate.

Figure 9. SEM fractograph of tensile tested Inconel 625 overlay heat treated at different temperatures (a) as welded, (b) 650 °C, (c) 750 °C, (d) 850 °C and (e) 950 °C.
The corrosion mechanism of the Inconel 625 overlay heat treated at different temperatures can be explained by galvanic coupling in which the dendritic matrix acts as the sacrificial anode [14, 24, 36]. For the as welded overlay, the microsegregation of Nb and Mo boost the formation of laves phase and MC phase, causing the depletion of Cr, Nb and Mo at the matrix region, where around the precipitates, and reducing the corrosion resistance of the matrix [37]. During the heat treatments process, corrosion resistant elements, such as Cr, Nb and Mo, diffuse from dendrite cores to interdendritic, providing the conditions for the δ phase precipitates at the interdendritic regions [17]. The formation of δ phase consumes a lot of elements, such as Nb and Ni, also induce the depletion of corrosion resistant elements, and increase the number of corrosion cells. Meanwhile, the diffusion speed of these elements increase significantly with the temperature. Hence, higher PWHT temperature means more serious poor of corrosion resistance elements. In addition, the poor corrosion resistant elements region is vulnerable to initiate corrosion for the more negative potential [14]. Moreover, the stability and consistencies of the corrosion film covering the corrosion resistance elements depleted region is less than that on the matrix dendrites region. Therefore, there is no pits distributed at the tested surface of the as welded overlay and that treated at 650 °C, while some obvious pits located on the surface of overlay treated above 650 °C.

4. Conclusions

In present study, the influence of PWHT temperatures on the microstructure, mechanical properties, and corrosion performance of the Inconel 625 weld overlay deposited using PTIG was investigated. The following conclusions can be obtained:

1. The as welded microstructure shows notable difference in grains morphology and size. With an increase in PWHT temperature, the equiaxed dendrites transform to columnar dendrites, dendrites spacing increases, and the overall difference in the microstructure decreases. The precipitated phases of the as welded are mainly composed of laves phases and MC phases. Compared with the as welded overlay, the variation in phases size and number for the overlay treated at 650 °C is not notable. When the PWHT temperature increases to 750 °C, some needle shaped δ phase precipitates with the dissolving of laves phase. The number of δ phase increases obviously, and its size coarsens obviously with a further increase in PWHT temperature. Particularly, the δ phase coarsens seriously for the overlay treated at 950 °C.

2. When the PWHT temperature is no greater than 750 °C, the YS and UTS increase slightly with the increase of PWHT temperature, whereas the elongation shows a decreasing trend. In additional, when the temperatures beyond 750 °C, the YS and UTS decrease, but the elongation increases slightly. Meanwhile, the fracture mode of the Inconel 625 overlay heat treated at different temperatures are ductile fracture.

3. Reasonable heat treatment is benefit to improve the corrosion resistance of Inconel 625 overlay. The Inconel 625 overlay heat treated at 650 °C exhibits excellent corrosion resistance in the corrosive environment.

![Figure 10. Corrosion rate of Inconel 625 overlay heat treated at different temperatures.](image-url)
containing H₂S and CO₂. However, the corrosion resistance degenerates seriously for the formation of δ phase consuming a large number of Cr and Mo with an increasing in PWHT temperature.

Acknowledgments

This study is financially supported by the Natural Science Basic Research Plan in Shaanxi Province of China (No. 2020JQ-780), the Open Foundation of Chongqing Engineering Technology Research Center for Light Alloy Materials and Processing (No. GCZX202001), and Young Teacher Research Project of Xi’an Shiyou University (No. 0104-134010025).

ORCID iDs

Longlong Guo  https://orcid.org/0000-0001-6280-7211
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