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# Microstructure and Corrosion Resistance of Inconel 625 Overlay Welded by Pulsed TIG Process

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To coordinate the material cost and corrosion resistance of the components served in corrosive environment, Inconel 625 was cladded over the AISI 4130 steel using pulsed tungsten insert gas welding (TIG). The microstructure and corrosion resistance of the Inconel 625 overlay was investigated. The Inconel 625 overlay is mainly composed of  $\gamma$ -Ni matrix and laves precipitate. Increasing layer of the overlay is benefit to reduce the compositions dilution. The content of *Fe* in the second layer overlay is only about 2%. The Nyquist plot of the substrate shows a capacitive loop with a diffusion tail, while that of the Inconel 625 overlay are composed of single capacitive loop. The tested surface near the upper surface of the second layer overlay features the largest charge transfer resistance compared with others. The passivation behavior of the Inconel 625 overlay below the middle surface of the second overlay is more stable than others. The specimen near and below the middle surface of the second overlay is more outstanding comprehensive pitting corrosion resistance than others. Additionally, seriously uniform corrosion occurred at the specimen of the single layer Inconel 625 overlay. There is no pitting corrosion on the specimens located the second layer of Inconel 625 overlay. Those demonstrated that two layers Inconel 625 overlay can efficiency protect the substrate from corrosion.

**Keywords**: Inconel 625 overlay, Microstructure, Corrosion resistance, Potentiodynamic polarization, EIS

# **1. INTRODUCTION**

Inconel 625 is a famous Ni-Cr-Mo nickel-based superalloy, which is primarily strengthened by solid solution hardening effect resulted in Mo and Ni, and also can be strengthened issuing from  $\gamma''$  (*Ni*<sub>3</sub>*Nb*) after a long time in the temperature ranging from 550 °C to 580 °C [1-3]. For the Inconel 625,

the existence of Mo and Ni boosts excellent resistance against non-oxidising corrosion, while Cr and Ni are good for resisting any form of oxidizing corrosion [4,5]. As usually reported, Inconel 625 possesses excellent mechanical properties, outstanding corrosion resistance and satisfied weldability. For these good comprehensive performance, Inconel 625 is widely applied for making the important components in industries, such as aerospace, chemical and nuclear, marine, crude oil and gas industries [5,6]. However, the expensive cost has seriously limited the widely individual application of Inconel 625 in industries [7,8]. Benefiting from the good weldability issued from the high ductility, Inconel 625 can be cladded on the surfaces that suffer the corrode media by fusion welding process to save material cost without sacrificing the corrosion resistance of the components [9,10]. Hence, Inconel 625 has been a suitable material choice for improving the corrosion resistance of the components in modification engineering [11-13].

Most of the relative studies on the microstructure and performance of Inconel 625 weld overlay are mainly focused on exploring and verifying the feasibility of cladding process, such as gas metal arc welding (GMAW), tungsten insert gas welding (TIG), and laser beam welding et al. Dupont [13] researched the solidification behavior of Inconel 625 deposited by GMAW. It was discovered that the solidification is terminated by a  $\gamma$ -Ni/laves eutectic reaction, and that laves phase is enriched in Ni and Mo. Rajkumar et al. [10,14] studied the microstructural evolution and wear behavior of Inconel 625 overlay over AISI 347 pipe using GMAW. It was pointed that higher contact angle and lower dilution were obtained at higher current and higher cladding speed, and the applied load has positive influence on the wear rate. Zareie et al. [15] compared the difference in corrosion behavior of Inconel 625 overlay deposited by TIG cladding and explosive cladding. The results demonstrated that the TIG cladding is more harmful to non-uniform corrosion resistance, while the non-uniform corrosion resistance can be improved by adding fusion layers. Evangeline et al. [9] investigated the microstructure evaluation, wear resistance, and corrosion resistance of the cladding deposited using cold metal arc transfer (CMT) process. The results showed that CMT process, with the suitable process parameters, can be used to build up the components that vulnerable worn out and damage. Dinda et al. [4] analyzed the microstructural evolution and thermal stability of the components welded using laser deposition. Abiove et al. [1,16] analyzed the microstructure and electrochemical corrosion resistance of the Inconel 625 overlay deposited using laser cladding. Mohammad et al. [17] compared the hot corrosion resistance of the Inconel 625 overlay welded using TIG and laser cladding respectively. The results revealed that the hot corrosion resistance of the laser cladding is superior to that of the TIG cladding.

However, so far very scanty studies focused on comparison of the difference in corrosion resistance issued from the different locations of the Inconel 625 overlay. In fact, the corrosion resistance of the different surface which parallel to the cladded surface is different for the various chemical compositions and microstructure resulted in the non-equilibrium solidification of weld pool. Therefore, in present study, the single layer and two layers Inconel 625 overlay were deposited over the AISI 4130 steel using pulsed TIG, and the corrosion resistance of the surface located in the different height of the overlay is mainly compared through electrochemical tests.

## 2. EXPERIMENTAL

#### 2.1 Materials and cladding experiments

The AISI 4130 steel plates with dimensions of  $150 \times 150 \times 25$  mm were utilized as substrate. The mark of filler wire is ERNiCrMo-3, which chemical compositions are equivalent to the Inconel 625 alloy. The diameter of the filler wire is 1.2 mm. The chemical compositions of the substrate and filler wire are listed in Table 1. The deposited surface of the substrate was grinded to roughness of 0.8µm, and cleaned with acetone. Meanwhile, to reduce the cool crack initiation, the substrate was preheated to 300°C before the cladding experiment. A hot-wire pulsed TIG system was utilized to carry out the cladding process. The wire preheating current was 70A, peak current was 240A, frequency was 5Hz with 0.3 duty cycle, and wire feed speed was 2m/min. In addition, argon gas with a flow rate of 15L/min was selected as the shielding gas. To compare the corrosion resistance of the specimens located in various height of the overlay, single layer and two layers Inconel 625 overlay were cladded, as presented in Fig.1.

**Table 1.** Chemical compositions of substrate and filler wire (*wt*.%) [18].

Element	С	Cr	Ni	Ti	Fe	Mo	Al	Nb	Others
Substrate	0.29	0.99	0.02	0.006	Bal	0.175	0.006	-	0.72
Filler wire	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: (a) single layer, and (b) two layers.

## 2.2 Microstructure characteristics

The specimen that sectioned from the single layer weld overlay was labeled as  $T_s$ . As shown in Fig.2, the specimens sectioned from the two layers weld overlay were labeled as  $T_a$ ,  $T_b$ ,  $T_c$ , and  $T_d$ , and the distance between these specimens with fusion surface formed between the first overlay and substrate were 3.5 mm, 4.3 mm, 5.1 mm, 5.9 mm respectively. The horizontal middle surface of the second overlay is located up the specimen  $T_b$ , and below the specimen  $T_c$ . The observed surfaces of the all specimens were prepared basing on standard metallographic procedures. Then, electrolytically etched method with a solution of 12mL H<sub>3</sub>PO<sub>4</sub>, 40mL HNO<sub>3</sub>, and 48mL H<sub>2</sub>SO<sub>4</sub> was used to etch the microstructure characteristics surfaces. The duration time was about 15s, and the voltage is maintained at 6V. A JSM-

6490LV scanning electron microscope (SEM) equipped with energy dispersive spectrometry (EDS) was used to observe the microstructure, and measure the content and distribution of element compositions in the specimens. The chemical compositions of the tested surface were measured using EDS, as listed in Table 2.



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Figure 2. Schematic of the tested specimens cutted from various locations of the two layers Inconel 625 overlay.

**Table 2.** Chemical compositions of the tested specimens of Inconel 625 overlay (*wt.*%)

Specimens	Ni	Cr	Fe	Мо	Nb
$T_s$	57.93	20.66	8.13	8.29	2.67
$T_a$	64.32	23.27	1.92	7.45	2.69
$T_b$	64.51	23.42	1.83	7.47	2.91
$T_c$	64.02	23.01	1.94	7.46	1.97
$T_d$	63.22	22.21	2.01	8.34	3.34

### 2.3 Electrochemical tests

To estimate the corrosion resistance of the specimens, electrochemical impedance spectroscopy and potentiodynamic polarization tests were carried out using an electrochemical testing system (Autolab PGSTAT302N), where counter electrode is a platinum plate, reference electrode is a Ag/AgCl electrode, and the specimens play role in working electrode. Each specimen was jointed with a copper wire by spot welding. Then, the specimen was packaged by epoxy resin, besides the measured surface with dimensions of 10×10mm is uncovered. Moreover, the measured surface was ground with SiC paper and polished with alumina pastes. The tests were performed in a 3.5 wt% NaCl solution prepared with de-ionized water at room temperature, the scanning rate is maintained at 20 mV/min, and the testing potential was scanned over the range of -200 mV to 1500 mV. The alternating potential of EIS testing varied from -5 mV to 5 mV around the open circuit potential with a frequency ranging from 100 kHz to 0.01 Hz. The ZsimpWin software was utilized to fit the EIS data. According to the ASTM standards G5-94 and G61-86, potentiodynamic polarization scan was tested at a 20 mV/min scan rate from the open circuit potential. The SEM was used to observe the morphology and measure chemical compositions of the corroded surface.

#### **3. RESULTS AND DISCUSSION**

#### 3.1 Microstructure

The microstructure of the single layer and two layers Inconel 625 overlay are shown in Fig.3. It can be seen that some white irregular shaped precipitates distributed at the interdendritic regions. Moreover, the precipitates in the single layers Inconel 625 overlay is less than that of the two layer Inconel 625 overlay. The chemical compositions of phases, as listed in Table 3. Taking the chemical compositions and shapes characteristic of phases, it is determined that the matrix is  $\gamma$ -Ni, and the white irregular shaped precipitates is laves phases [13,19]. The laves phase is a  $A_2B$  (A: Ni, Fe, Cr; B: Nb, Mo, Ti) type precipitate [2]. The large number of Nb, Mo, and Ti promote the formation of laves phases. This is the reasonable explanation for that the laves phases in second layer is superior in volume and number to that of the single layer Inconel 625 overlay.



**Figure 3**. Microstructure of the Inconel 625 overlay: (a)  $T_s$ , and (b)  $T_d$ .

Specimen	Phases	Ni	Cr	Fe	Mo	Nb
$T_s$	Matrix	58.76	21.09	8.20	8.77	2.69
$T_s$	Precipitate	45.74	18.92	5.97	14.61	13.43
$T_d$	Matrix	62.92	22.21	2.01	8.33	3.04
$T_d$	Precipitate	42.99	16.31	1.44	11.61	26.68

Table 3. Chemical compositions of the phases distributed in Inconel 625 overlay (*wt*.%).

#### 3.2 Electrochemical impedance spectroscopy

The Nyquist plots of the tested specimens are presented in Fig.4. It is observed that the all Nyquist plots of the Inconel 625 overlay are composed of single capacitive loop, demonstrating that the electrode

reaction is predominated by the electrochemical reaction in the range of measured frequencies [20]. For the Nyquist plot of the substrate, there is a capacitive loop at the high frequencies followed by a diffusion tail, which shows a tilt angle of  $\pi/4$ , at the relative low frequency region. The existence of warburg impedance means that the diffusion process is controlled by the combination of charge transfer and material diffusion, and the control of diffusion exceeds that of the material diffusion [21,22]. Hence, it is concluded that the corrosion mechanism of the Inconel 625 overlay is different from the substrate.



**Figure 4.** Nyquist plots tested in a 3.5 wt% NaCl solution at roomtemperature: (a) specimens cutted from the various locations of Inconel 625 overlay, and (b) substrate.

The equivalent circuits selected to fit the EIS data are demonstrated in Fig.5. In the models,  $R_s$  is solution resistance,  $R_t$  is charge transfer resistance, and  $W_0$  is diffusion impedance. To obtain a good fitting, a constant phase element (CPE) was utilized to take roughness of electrode surface, energy dissipation and other factors into consideration [22,23]. The values of  $R_t$  fitted on the basis of EIS data are presented in Fig.6. It is observed that the  $R_t$  of the substrate is far less than that of Inconel 625 overlay. Generally, the value of  $R_t$  is a positive indicator to evaluate the corrosion resistance [20,24]. Hence, it is inferred that Inconel 625 overlay possesses much better corrosion resistance than the substrate. In addition, there is notable difference in the  $R_t$  values of the Inconel 625 overlay specimens, and the  $R_t$  values follow the sequence:  $T_b > T_a > T_c > T_s > T_d$ . It means that the surface near the top of the second layer of the Inconel 625 overlay features worse corrosion resistance than that of the first layer. Except specimen  $T_d$ , the corrosion resistance of other specimens cut from the second layer is better than that of the first layer. This may be because that increasing the layers of overlay is beneficial to reduce the dilution of the chemical compositions.



Figure 5. Equivalent circuits fitted to the EIS data measured in a 3.5 wt% NaCl solution at roomtemperature: (a) Inconel 625 overlay, and (b) substrate.



**Figure 6.** Charge transfer resistance of substrate and Inconel 625 overlay specimens fitted according to the Nyquist plots.

#### 3.3 Potentiodynamic polarization

The potentiodynamic polarization curves of the specimens are presented in Fig.7. It is observed that the cathodic polarization trend of the specimens is the same. The specimen of substrate shows active dissolution behavior in the anodic polarization region, while the specimens of the Inconel 625 overlay exist passivation stage. Some investigators pointed that the good corrosion resistance of Inconel 625 alloy is mainly due to the high content of Ni, Mo, Nb and Cr, which promotes the formation of passive film on the surface of overlay [22,25,26]. The passive film effectively prevents Cl<sup>-</sup> adsorbed on the surface of the overlay moving to the below of the film to participate in the activation reaction. The passivation behavior of  $T_a$  and  $T_b$  is more obvious and stable than other specimens. The polarization current of the specimen  $T_d$  shows reciprocating various in the horizontal direction. This indicates that the passive film is unstable and susceptible to local damage, and that the newly formed passivation film can recover part of the corrosion resistance [1]. The specimens  $T_c$  and  $T_s$  possess notable repassivation stage after a short period of passivation stage. In the over passivation region of each specimen, the current density increases greatly with the continuous increase of polarization potential, which means that the passive film is destroyed and the corrosion rate is obviously accelerated.



**Figure 7**. Potentiodynamic polarization curves of substrate and Inconel 625 overlay specimens measured in a 3.5 wt% NaCl solution at room temperature.

According to the potentiodynamic polarization curves, the corrosion potential  $(E_{corr})$ , pitting potential  $(E_{pit})$ , and passivation current density  $(I_p)$  were determined to evaluate the corrosion resistance of the specimens, as shown in Fig.8. The magnitude of  $E_{corr}$  reflects the corrosion susceptibility, and the higher value indicates that it is harder to initiate corrosion [1]. The sequence of  $E_{corr}$  is as follow:  $T_b > T_a > T_c > T_d > T_s$ . The  $E_{vit}$  represents the degree of difficulty that local damage forms on the passive film. The high value of  $E_{pit}$  means that permanent damage impossible appear at the passive film under the action of low voltages. The  $E_{pit}$  follow the sequence:  $T_d > T_b > T_a > T_c > T_s$ . The  $I_p$  is considered as an indicator of uniform corrosion resistance. The smaller the  $I_p$  value suggesting that is easier to passive. The formation of passive film at low current density hinders severe loss of material at higher current density [20]. It is found that the value of  $I_p$  follow sequence:  $T_b < T_a < T_c < T_d < T_s$ . Hence, the specimens sectioned from the second weld overlay exhibit better pit corrosion resistance than that of the single overlay. In other words, the pit corrosion resistance of the inconel 625 overlay can be enhanced by adding fusion layers. The similar phenomenon was observed by Rajani et al. [15]. Additionally, the specimen  $T_b$ possesses more outstanding comprehensive pit corrosion resistance than other specimens, which cutted from the second weld overlay. The corrosion difference of the specimens sectioned from the second overlay may be resulted in the difference of microstructure and elements compositions, which induced by complex non-equilibrium solidification.



**Figure 8**. Corrosion parameters determined on the basis of potentiodynamic polarization curves: (a) corrosion potential and pitting potential, and (b) passivation current density.

The morphology of the corroded surface is presented in Fig.9. Some loose corrosion products cover the corroded surface of the specimen  $T_s$ , indicating that the passive film formed at the passive region cannot effectively against corrosion.



**Figure 9.** Morphology of the surface after potentiodynamic polarization testing in a 3.5 wt% NaCl solution: (a)  $T_s$ , (b)  $T_a$ , (c)  $T_b$ , (d)  $T_c$ , and (e)  $T_d$ .

As listed in Table 4, the corrosion products of the specimen  $T_s$  are composed of elements O, Mo, Nb, Cr, Ni and Fe, and the mass fraction of O is higher than 9%. Hence, it is concluded that seriously uniform corrosion occurred in specimen  $T_s$ . The corrosion products of the  $T_a$ ,  $T_b$ ,  $T_c$ , and  $T_d$  are clearly damaged and disconnected around the precipitates, where the matrix is seriously corroded, and the precipitates tend to fall off. From the Table 4, it is found that the corrosion products covering the core dendritic matrix is richer in Mo (21.6 wt%), and O (6 wt%) than that of covering the precipitates. The

large number content of O implies that the core dendrites suffered from oxidation resulting in formation of oxides. Meanwhile, the O of the tested region above the precipitates is only 0.63% and nearly can be ignored, indicating that the tested region did not suffer corrosion attack. The distribution of O in this study is similar to that in the laser cladding of Inconel 625 overlay after potentiodynamic polarisation testing in 3.5 wt% NaCl solution investigated by Abioye et al. [1]. It is confirmed that the corrosion mechanism of the specimens sectioned from the second overlay belong to alvanic coupling, and the dendritic matrix acts as the sacrificial anode. The same conclusion was obtained by Ban et al. [5] and Rajani et al. [15]. The main reason can attitude to the segregation of element Mo, Nb, Cr, and Ni caused by formation of laves phase [5]. Additionally, there is no evidence suggesting that the pitting corrosion occurred on the surface of specimens sectioned from the second overlay. These morphologies are consistent with the characterize that the gradient of the current density against potential is relative low in the transpassive region. The corrosion products of  $T_b$  feature relative complete and dense suggesting the best pitting corrosion resistance. The specimen  $T_c$  shows worst pitting corrosion resistance compared with the specimens sectioned from the second overlay.

**Table 4.** Chemical compositions of the corrosion products covering the specimen after potentiodynamicpolarization testing in a 3.5 wt% NaCl solution (*wt.%*)

Region	Specimen	0	Nb	Mo	Ti	Cr	Fe	Ni
А	$T_s$	9.35	24.44	42.19	1.32	7.00	8.26	7.44
В	$T_s$	9.61	26.12	40.56	1.4	6.89	7.76	7.66
С	$T_b$	6.00	8.88	21.61	0.6	16.74	3.18	42.93
D	$T_b$	0.63	2.19	6.81	/	23.66	1.81	64.80

### **4. CONCLUSIONS**

In this study, the microstructure and chemical compositions of the single and two layers of the Inconel 625 overlay was compared, and the corrosion resistance of the specimens of Inconel 625 overlay was mainly investigated. The following conclusions can be obtained:

(1) The microstructure of the Inconel 625 overlay is mainly composed of  $\gamma$ -Ni matrix and laves precipitate. The dilution of Fe can be effectively decreased by increasing the layer of overlay. The content of the Fe in the single and second layer of the Inconel 625 is about 8.13% and 2.01% respectively.

(2) Unlike the Nyquist plot of the substrate, which shows a capacitive loop followed by a diffusion tail, the Nyquist plots of the Inconel 625 overlay are composed of single capacitive loop. The tested surface near the upper surface of the overlay shows least charge transfer resistance ( $R_t$ ), which is also less than that of the single layer of Inconel 625 overlay. In addition, the tested surface below the middle surface of the second layer features larger  $R_t$  than the surface from other locations.

(3) The passivation behavior of the Inconel 625 specimens located below the middle surface of the second overlay is more obvious and stable than other specimens. The specimen near and below the middle surface of the second overlay possesses more outstanding comprehensive pit corrosion resistance

than other specimens. Therefore, the Inconel 625 overlay can protect the substrate against from corrosion for the formation of passivation films.

(4) Seriously uniform corrosion occurred at the specimen of single layer Inconel 625 overlay. However, there is no evidence of pitting corrosion on the specimens sectioned from the second layer of Inconel 625 overlay. The corrosion mechanism of the specimens sectioned from the second overlay belongs to alvanic coupling, and the dendritic matrix acts as the sacrificial anode.

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