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Transformation mechanism of secondary phase and its effect on intergranular corrosion in laser wire filling welding Ni-based alloy/304 stainless steel

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Abstract: To clarify the transformation mechanism of secondary phase and the mechanism of intergranular corrosion in laser welding Ni-based alloy (Hastelloy C-276)/304 stainless steel with filler wire, the secondary phase was analyzed by electron probe micro-analysis (EPMA) and transmission electron microscopy (TEM). The evaluation of intergranular corrosion resistance of the welded joints was conducted by double-loop electrochemical potentiokinetic reactivation (DL-EPR) method, and at the same time the chemical compositions of the corrosion surface were analyzed by energy-dispersive spectrometry (EDS). The results show that *p* phase has complete coherence relationship with μ phase, and the coherent relationship is described as $[001]_p//[01\bar{1}0]_{\mu}$ and $[430]_p//[0001]_{\mu}$. The μ phase is rapidly transformed from *p* phase, which is the inhomogeneous phase transformation. The transformation of secondary phase will increase the susceptibility to intergranular corrosion. Therefore, the transformation of secondary phase should be avoided in the welding process.

Key words: phase transformation; intergranular corrosion; secondary phase; Ni-based alloy; dissimilar metal laser welding

1 Introduction

Hastelloy C-276 has been extensively used in petrochemical and nuclear power fields because of good corrosion resistance [1–3]. However, it is easy to produce topologically close-packed (TCP) secondary phases (σ phase, p phase, and μ phase) in the hot working process of Hastelloy C [4–6]. SILVA et al [7] reported these three Mo-rich secondary phases in the dissimilar claddings of Inconel alloy 686. The space group of σ phase is P4/mnm, which is tetragonal structure. The space group of p phase is Pbnm with orthorhombic crystal structure. The space group of μ phase is $R\overline{3}m$, which is rhombohedral crystal structure, and μ phase has stacking faults and precipitates isomorphously with A_7B_6 type. On one hand, the precipitation of these Mo-rich secondary phases can reduce the Mo element in the metal matrix, so the corrosion properties of metal are weakened [8,9]. On the other hand, the brittle secondary phases will weaken the mechanical properties of metal [10–12], especially the precipitated μ phase with stacking faults [13].

Welding is a wide hot working process. The study of secondary phases originated from the investigation of CIESLAK et al [14] in Hastelloy C weld metal. They investigated the argon arc weldability of Hastelloy C-4, Hastelloy C-22 and Hastelloy C-276. The precipitation behavior of secondary phases was revealed. In an alloy, a higher

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content of Cr (or Fe) is easy to precipitate σ phase, but higher content of Mo (or W) is easy to precipitate p phase. Besides, μ phase is formed from transformation of p phase. And the influence of secondary phases is also revealed on hot crack sensitivity. However, the non-critical parts are made of stainless steel materials with lower cost and better processability, rather than Hastelloy C due to its higher cost. So, it is very common to weld Hastelloy C and stainless steel [15-17]. According to the work of RAGHAVAN et al [18] in the ternary system of Ni–Cr–Mo, σ phase is difficult to precipitate in the weld because of dilution of stainless steel. Some scholars have investigated the secondary phases in the dissimilar metal weld of stainless steel and Hastelloy C. SHARMA et al [19] reported the secondary phases in the argon arc welding of C-276/A321. ZHOU et al [20] made it clear that there was p phase in the laser lap welding of C-276/304. WU et al [21] reported that both μ phase and p phase precipitated in the laser wire filling welding of C-276/304, and p phase had a better coherent relationship with the weld matrix than μ phase. At the same time, in the hot working of Hastelloy C, μ phase is easy to precipitate from matrix because the precipitation of μ phase has a very wide temperature range (650-1140 °C) and fast rate [22-24]. However, the precipitation mechanism of μ phase is not well clarified in the dissimilar metal weld of stainless steel and C-276, in which μ phase is from the transformation of pphase or from the precipitation of matrix. The phase transformation mechanism between p phase and μ phase is not well expounded.

Moreover, corrosion properties are of great concern to the welds of Ni-based superalloys and stainless steels. There are many articles reporting the corrosion behavior of dissimilar welds of Ni-based superalloys and stainless steels [25,26]. MORTEZAIE and SHAMANIAN [27] studied the relationship between microstructure of weld and corrosion properties in argon arc welding of Inconel 718/310 stainless steel. The results show that the weld metal with Inconel 82 filler has better corrosion resistance because of its higher content of austenite and more homogeneous element distribution. KANGAZIAN et al [28] studied the effects of weld bead layer number, welding process and filler wire type on corrosion behavior of Incoloy 825/SAF 2507 welds. The results show that the weld with Ni-based alloy filler wire has better general corrosion resistance, while the weld with stainless steel filler wire has better pitting corrosion resistance. DERAKHSHI et al [29] studied the corrosion behavior of electron beam welds of Inconel 617/310 stainless steel. Due to the element segregation and precipitated phase, the corrosion resistance of the weld is inferior to that of the base metal. And the increase of welding speed is beneficial to improving the corrosion properties of the weld metal. At the same time, there are some scholars studying the corrosion behavior of C-276 alloy. PATEL et al [30] studied the high temperature corrosion behavior and corrosion mechanism of C-276. LI et al [31] investigated the cavitation corrosion behavior of C-276 and revealed the evolution law of corrosion surface morphology. BAL et al [32,33] reported the mechanism of intergranular corrosion of C-276 weld, and found that the rate of intergranular corrosion was related to the concentration gradient of Mo. The secondary phase will have a great influence on the intergranular corrosion of the weld because of the precipitation along the grain boundary. However, influencing mechanism of secondary phase on intergranular corrosion is not well clarified. All of these are worthy to be further studied and clarified.

Therefore, in this work, the secondary phase produced during laser welding was analyzed and intergranular corrosion resistance of welded joints was evaluated. In addition, the mechanism of intergranular corrosion of welded joints was revealed.

2 Experimental

The thickness of C-276 sheets (base metals) was 0.5 mm and the thickness of 304 sheets (base metals) was 2.0 mm. The welding wire was ERNiCrMo-4 which was special welding wire of the Hastelloy C-276, and the diameter of welding wire was 0.5 mm. The chemical compositions of the two base metals and welding wire are listed in Table 1. In order to facilitate the research, in this work the model of vertical welding with filler wire was established [21]. The average laser power was 250 W, welding speed was 600 mm/min, pulse width was 3 ms, and wire feeding speed was 300 mm/min. After welding, the etching process of the samples was used to examining the C-276 base

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Table 1 Chemical compositions of base materials and welding wire (wt. %)												
Sample	Ni	Fe	Cr	Mo	W	Co	Mn	С	Si	Р	S	V
C-276	Bal.	5.14	16.00	15.58	3.45	1.26	0.53	0.001	0.02	0.006	0.003	0.01
304 stainless steel	8.02	Bal.	18.20	-	-	_	1.12	0.043	0.52	0.036	0.003	_
ERNiCrMo-4	Bal.	5.30	16.00	15.20	3.30	0.11	0.41	0.009	0.03	0.003	0.001	0.01

metal and weld metal (WM) with the solution of 10% oxalic acid (6 V DC supply, time: 3-4 s). Nitromurlatic acid was used to examine the 304 stainless steel (304) base metal. The electron probe micro-analysis (EPMA, JEOL 8530F-Plus) was applied to analyzing the distribution of the secondary phase, and the test equipment was Shimadzu 1600. Meanwhile, the transmission electron microscopy (TEM, Tecnai G220 S-Twin) observation was carried out to analyze the secondary phase. The double-loop electrochemical potentiokinetic reactivation (DL-EPR) method was used to evaluate the intergranular corrosion of the welded joints. The corrosion solution was a mixture of 0.5 mol/L H₂SO₄ and 0.05 mol/L KSCN. The total volume of corrosion solution was 250 mL, and the scanning rate of voltage was 1 mV/s.

3 Results and discussion

3.1 Distribution of secondary phase in weld

The amount of the secondary phase is mainly related to the micro-segregation of Mo element, and the faster cooling rate is beneficial to reducing the micro-segregation of Mo element [20]. Figure 1(a) shows the location of measured points. Figures 1(b-f) show the transverse distribution of secondary phase. The closer the location to the base metals (C-276 and 304) is, the less the amount of secondary phase is. This is due to the faster heat dissipation and the higher dilution rate of the base metals, both of which can reduce the microsegregation of Mo element [20]. In addition, the closer the location to the center of weld is, the more the amount of secondary phase is. However, the largest amount of secondary phase is not at the center of weld (point c in Fig. 1(a)), but at point bor d Fig. 1(a), because solidification rate of the center of the weld is faster than that of point *c* or *e*. Figures 1(g, d, h-j) show the vertical distribution of the secondary phase. On the top zone of excess weld metal, the heat dissipation of the weld is faster, so the amount of secondary phase is less. The amount of secondary phase increases with the

increase of distance from weld surface. However, the closer the location to the bottom of the molten pool is, the faster the heat dissipation is; the faster the cooling rate is, the less the amount of secondary phase is. There is no secondary phase near the bottom of the molten pool. So the amount of secondary phase is the largest at the junction of the excess weld metal zone and the fusion zone of base metal (Point c in Fig. 1(a)). Overall, the most serious precipitation occurs near the center of the junction of the weld reinforcement zone and the fusion zone of base metal. So, this interface was selected as the surface of intergranular corrosion evaluation in the following investigation.

3.2 Transformation path of secondary phase

The size of the secondary phase is very small, $200 \text{ nm}-1 \mu \text{m}$, and the secondary phase is composed of μ phase and p phase [21]. However, the relationship and structure between μ phase and p phase are not clear, so TEM analysis was used to determine the microstructure of secondary phase. Mapping analysis of secondary phase was carried out by TEM-EDS, as shown in Fig. 2. There are some differences between the upper and lower parts of the secondary phase in the distribution of elements. There are more Ni and less Mo in the upper part of the secondary phase. The results of component analysis of secondary phase are shown in Table 2. According to the composition of the secondary phase in similar alloys [34], both p phase and μ phase are Mo-rich phases and μ phase contains more Mo and less Ni than that p phase. Therefore, it is indicated that the upper part of the secondary phase is p phase and the lower part is μ phase. The diffraction pattern of the secondary phase (Fig. 3) further confirms the microstructure of the secondary phase, which is made up of μ phase and p phase. Meanwhile, p phase has complete coherence relation with μ phase. This coherent relationship between p phase and μ phase is described as $[001]_p / [01\overline{1}0]_u$. At the same time the coherent relationship between the secondary phase and weld matrix is $[110]_{p}/[001]_{p}/[0110]_{u}$.



Fig. 1 ETMA images showing distributions of secondary phase: (a) Loaction of measured points; (b–j) Corresponding to points a-i in (a), respectively

Figure 4 shows that p phase has another complete coherent relationship with μ phase, which is described as $[430]_p/[0001]_{\mu}$. And the two coherent relationships are related to $[110]_{\gamma}$ of weld matrix. Figure 5 shows the interface of two secondary phases under the high resolution transmission electron microscopy (HRTEM). There is no obvious boundary between μ phase and pphase, which further reveals that p phase has a complete coherent relationship with μ phase. The $[\overline{3}40]_p$ and $[\overline{1}100]_{\mu}$ are collinear in the coherent plane of two secondary phases, which is the plane of vertical $[430]_p$.

In order to further understand the secondary phase, the HRTEM analysis of the secondary phase

was carried out. The HRTEM image of p phase is shown in Fig. 6. From Fig. 6 the lattice constants of p phase can be calculated to be a=0.905 nm, b=1.714 nm, and c=0.474 nm. Similarly, as shown in Fig. 7, the lattice constants of μ phase are calculated to a=b=0.478 nm, and c=2.625 nm. The misfit degree (δ) between p phase and μ phase is very small ($\delta=(\alpha_{\mu}-\alpha_{p})/\alpha_{\mu}$ [35], $\alpha_{\mu}=0.478$ nm, and $\alpha_{p}=0.474$ nm). Therefore, it is further shown that pphase has complete coherence relation with μ phase. The phase transformation process of weld can be described by Ni–Cr–Mo ternary system as shown in Fig. 8. The top arrow represents the equivalent component of the dendrite core, and the end of the arrow represents the equivalent component between



Fig. 2 TEM image of secondary phase (a) and distributions of major elements around secondary phase (b-f)

 Table 2 Major element compositions of secondary phase

 (wt.%)

Point in Fig. 2(a)	Ni	Mo	Cr	Fe	W
1	29.40	26.63	18.41	18.96	6.60
2	19.92	39.68	17.47	14.84	8.10



Fig. 3 Diffraction pattern showing coherent relationship between secondary phase and weld matrix

the dendrites. The solidification of weld begins with γ phase and ends with $\gamma+p$ phase. With further cooling of the weld, part of *p* phases changes into μ phase because the solidification process of weld is non-equilibrium solidification process. Therefore,



Fig. 4 TEM image showing coherent relationship between p phase and μ phase



Fig. 5 HRTEM image of interface of p phase and μ phase



Fig. 6 HRTEM images of p phase: (a) Plane of vertical $[430]_p$; (b) Plane of vertical $[001]_p$



Fig. 7 HRTEM image of μ phase

the phase transformation process of the weld can be described as $L \rightarrow \gamma + p \rightarrow \gamma + p + \mu$. Even though the cooling rate of pulsed laser welding is fast, the change from p phase to μ phase cannot be prevented. It is not difficult to infer that p phase quite rapidly changes into μ phase. But this phase transformation $(p \rightarrow \mu)$ cannot be martensitic transformation which is very rapid phase transformation [36], because there is a change of composition in the phase transformation. The reason for p phase changing into μ phase may be related to Mo element. With the cooling of the weld metal, the solubility of Mo in p phase and matrix decreases. Therefore, p phase changes into μ phase, which can accommodate more Mo element. The phase transformation from p phase into μ phase is inhomogeneous [37]. And p phase is likely metastable phase.



Fig. 8 Isothermal sections of Ni–Cr–Mo ternary system at different temperatures [18]: (a) 1250 °C; (b) 850 °C

The inhomogeneous nucleation is likely to occur at the interface between p phase and matrix because there are many dislocations and defects. The coherent interface of p phase and μ phase is easy to form because there are coherent relationship between p phase and μ phase as shown by $[001]_p/[01\overline{10}]_{\mu}$ and $[430]_p/[0001]_{\mu}$, so μ phase grows fastest along the plane of vertical $[001]_p$ and $[430]_p$. The substitutional diffusion of Mo element and Ni element makes p phase rapid change into μ phase at the phase interface. And the transformation of p phase $(p \rightarrow \mu)$ is likely to change from the $[340]_p$ direction of the plane of vertical $[430]_p$ because the $[\overline{3}40]_n$ and $[\overline{1}100]_{\mu}$ are collinear. At the same time, TAWANCY [13,22] reported that the precipitation of μ phase from matrix was observed after 1 h of 870 °C exposure and carbides precipitated prior to μ phase from matrix. However, there were not carbides in secondary phase. Therefore, the μ phase is mainly transformed from the *p* phase rather than precipitates from the weld

matrix. That is to say, the secondary phase mainly precipitates in the solidification of the weld and then changes during the cooling process of the weld.

3.3 Influence mechanism of secondary phase for intergranular corrosion

The intergranular corrosion property of the welded joints was evaluated by DL-EPR method. DL-EPR curves of weld and base metals are shown in Fig. 9. The DL-EPR is used to evaluate the intergranular corrosion property index by reactivation rate R, which is the ratio J_r/J_a (J_a is the activation current density peak and J_r is reactivation



Fig. 9 DL-EPR curves of different samples: (a) C-276; (b) WM; (c) 304 base metal

current density peak). Besides, the higher the reactivation rate (R) is, the easier the tendency of the intergranular corrosion is. The reactivation rate (R) of weld metal (WM) is 5.1 and the reactivation rate of 304 base metal is 2.1. However, there is no reactivation current density peak in DL-ERP curves of C-276 base metal because its the reactivation rate is very small. The electrochemical data is the average of three experimental measurements in this work. The weld metal is more sensitive to intergranular corrosion than two base metals.

After DL-EPR corrosion testing, the surface of samples was examined and the results are shown in Fig. 10. The composition of the passivation film of the weld surface was obtained by EDS (65.65 wt.% Mo, 30.58 wt.% O, 3.77 wt.% W). The passivation film is mainly composed of oxide of Mo (small amount of oxide of W), and the Mo/O molar ratio of the oxide is close to 1:3. So, it is indicated that the oxide is MoO₃. In Fig. 11, the mapping analysis of passivation film confirms that the passivation film is mainly related to Mo element, rather than Cr element. Therefore, the electrochemical reaction of Mo in corrosion testing is as follows:

$Mo+3H_2O \rightarrow MoO_3+6H^++6e$

According to poor chromium theory, the intergranular corrosion is usually related to Cr element, but the intergranular corrosion may be related to Mo element in this research. This is because there is only passivation film of Mo element on the weld surface, and the selfpassivation tendency of Fe and Ni is less than that of Mo and Cr. In the DL-EPR corrosion testing, the passive film formed in Mo-poor zone is unstable and easy to be damaged because it is easily dissolved (e.g. reductive dissolution) when the potential moves to the negative direction. Therefore,



Fig. 10 Morphology and chemical composition of passive film on weld surface



Fig. 11 Element mapping of passive film: (a) Mo; (b) O; (c) Fe; (d) W; (e) Cr; (f) Ni



Fig. 12 TEM-EDS line scanning images of secondary phase: (a) p phase; (b) μ phase

the weld metal is further corroded. Figure 12 shows that there is a Mo-poor zone around the secondary phase. And the secondary phase precipitates along the grain boundary, which is Mo-rich zone. The difference of electrochemical performance will be a galvanic interaction between Mo-rich zone and Mo-poor zone. The metal acts as anode in Mo-poor zone and the metal acts as cathode in weld matrix, which eventually lead to the prior intergranular corrosion in Mo-poor zone of weld [38]. There is no secondary phase in C-276 base metal with solution annealing, which is annealing twin structure. So, C-276 base metal is insensitive to intergranular corrosion. However, some carbides, σ phases and δ -ferrite easily precipitate at grain boundaries of solution-annealed 304 stainless steel. Therefore, 304 base metal has certain intergranular corrosion sensitivity. There are many secondary phases in dissimilar metal weld. Therefore, the intergranular corrosion resistance of welded joints is worse than that of both base metals.

In Fig. 12, the width of Mo-poor zone of p phase is 25–30 nm, and the width of Mo-poor zone of μ phase is 55–60 nm. The μ phase can accommodate more Mo element than p phase, so the transformation of p phase $(p\rightarrow\mu)$ will expand the Mo-poor zone. At the same time, there is dislocation pile-up around the secondary phase in Fig. 13, which leads to the prior corrosion around the secondary phase. And there are more dislocations around μ phase than around p phase. The p phase has a better coherent relationship between the weld matrix than μ phase $(p\rightarrow\mu)$ will

increase the susceptibility to intergranular corrosion. That is to say, this phase transformation is detrimental in welding process.



Fig. 13 Bright field TEM image of secondary phase and weld matrix

4 Conclusions

(1) The most serious precipitation of secondary phase occurs near the center of the weld which also serves as the junction of the weld reinforcement zone and the fusion zone of base metal. And the secondary phase mainly precipitates during the solidification of the weld.

(2) The *p* phase has complete coherence relation with μ phase. This coherent relationship is described as $[001]_p//[01\overline{10}]_{\mu}$ and $[430]_p//[0001]_{\mu}$. The μ phase is transformed from the *p* phase. And μ phase grows fastest along the planes of vertical $[001]_p$ and $[430]_p$. Meanwhile, the transformation of *p* phase $(p \rightarrow \mu)$ is likely to change from the $[\overline{3}40]_p$ direction of the plane of vertical $[430]_p$, which is the inhomogeneous phase transformation. And the change from *p* phase to μ phase is quite rapid.

(3) The Mo-poor zone is the cause of intergranular corrosion of weld metal around the secondary phase. The weld metal is more sensitive to intergranular corrosion than two base metals because there are many secondary phases along the grain boundary. The transformation of secondary phase $(p \rightarrow \mu)$ will expand the Mo-poor zone and increase dislocation density, both of which increase the susceptibility to intergranular corrosion. Therefore, the transformation of the secondary phase is detrimental in welding process.

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激光填丝焊接镍基合金/304 不锈钢二次相的 转变机制及其对晶间腐蚀性能的影响

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摘 要:为了阐明激光填丝焊接镍基合金(Hastelloy C-276)/304 二次相的转变机制和晶间腐蚀机制,通过 EPMA 和 TEM 对二次相进行分析;采用双环动电位再活化法(DL-EPR)对焊接接头的晶间腐蚀性能进行评价,并采用 EDS 对焊接接头腐蚀表面进行成分分析。实验结果表明: *p* 相和 *µ* 相为完全共格关系,且其共格关系为[001]_{*p*}//[011]_{*u*} 和[430]_{*p*}//[0001]_{*µ*}。*µ* 相由 *p* 相快速相转变而成,此相变过程为不均匀相变。二次相转变将增大晶间腐蚀敏感性。因此,在焊接过程中应该避免这种相变。

关键词:相变;晶间腐蚀;二次相;镍基合金;异种金属激光焊接

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