Microstructural evolution and liquation cracking in the partially melted zone of deposited ERNiCrFe-13 filler metal subjected to TIG refusion

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Abstract

The microstructure of ERNiCrFe-13 multipass weld metal has been shown to contain Laves/ γ or σ/γ eutectic constituents that can increase susceptibility to solidification and weld metal liquation cracking resulting from the low eutectic reaction temperature. Under poor heat dissipation conditions such as on the edge of large thickness welded components, a partially melted zone (PMZ) may form in the weld metal during multipass welding. The microstructural evolution and liquation cracking susceptibility of this PMZ in ERNiCrFe-13 multipass welds have received little attention. In the present study, a tungsten inert gas (TIG) refusion process is used to simulate a thermal cycle with a long elevated temperature dwell time in order to investigate the microstructural evolution and liquation cracking in the weld metal PMZ. The results show that the eutectic microstructures in the PMZ evolve into three eutectic morphologies after TIG refusion, including long linear chains extending perpendicular to the boundary between the refusion zone and PMZ, skeletal structures, and fine lamellar networks. This evolution contributes to constitutional liquation occurring at the γ /Laves and γ/σ interface. Nb and Mo play a leading role in the constitutional liquation of γ /Laves and γ/σ eutectic microstructures, respectively. Liquation cracking in the PMZ is shown to occur along the linear chain grain boundaries resulting from constitutional liquation.

Keywords ERNiCrFe-13 · TIG refusion · Eutectic microstructure · PMZ · Constitutional liquation, liquation cracking

1 Introduction

Inconel® 690 nickel alloy has been extensively applied in the fabrication of key nuclear power plant equipment, such as the reactor pressure vessel (RPV) and steam generator (SG), owing to its excellent resistance to intergranular corrosion (IGSCC) [1, 2]. Corresponding high-chromium filler metals have also been developed to ensure good IGSCC resistance and ductility dip cracking (DDC) resistance in welded Inconel

690 products. Recently, excellent DDC resistance has been obtained by the development of ERNiCrFe-13 for welding Inconel 690 or joining Inconel 690 to dissimilar metals such as 304 L stainless steel [3] by adding 2.5 wt% Nb and 4 wt% Mo to the previously developed ERNiCrFe-7A filler metal [4]. The high DDC resistance of ERNiCrFe-13 has been demonstrated by the greater than 10% threshold strain obtained during strain to fracture (STF) experiments [5]. Both Nb and Mo show strong segregation tendency during solidification in nickel alloys, which promotes the formation of topologically close packed (TCP) phases such as Laves, μ , P, and σ phases as eutectic reactions [6–9]. It is reasonable to believe that these eutectic microstructures may induce liquation cracking in PMZ. Considerable research has focused on the microstructures and mechanical properties of ERNiCrFe-13 weld [10–13], while the microstructural evolution and liquation cracking susceptibility in PMZ of ERNiCrFe-13 deposited metal have received little attention. PMZ may experience long time during welding thermal cycle under poor heat dissipation conditions. The thermal cycles simulation results by computer

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Table 1 Composition of base metal and deposited metal without dilution (wt%)

	C	Si	Mn	S	P	Nb	Ni	Cr	Fe	Mo	Ti
Q235 base metal	0.15	0.20	0.51	0.020	0.021	–	–	–	Bal.	–	–
Deposited metal without dilution	0.02	0.07	0.92	0.001	0.002	3.30	Bal.	30.35	11.24	3.58	0.21

simulation based on Sysweld™ for narrow groove multipass welds have shown that the elevated temperature retention time above the 1000 °C on nodes below remelt layer on fourth layer is about 4.5 s [14]. For multipass welded components with large thickness, heat can conduct to its surroundings without constraints at the middle of the surface, while heat can only conduct down on the edge at a distance large enough (≥ 35 mm) above base metal surface. Therefore, the heat dissipation condition on the edge was poorer than other positions and PMZ might endure long time for 2–6 s at elevated temperature above the 1000 °C during the next welds welding above it.

The above-discussed issues were addressed in the present study by applying thermal cycling based on a tungsten inert gas (TIG) refusion process without filler wire, and the microstructural evolution and liquation cracking in PMZ of ERNiCrFe-13 deposited metal were investigated. The proposed TIG refusion process represented an extension of the arc heat treatment technique developed to simulate PWHT for investigating the evolution of duplex stainless steel microstructures in a steady state [15]. In this paper, TIG refusion spot welding without feeding wire was developed to simulate the poor heat dissipation condition on the edge of multipass welded components with large thickness and investigate the microstructure evolution behavior and liquation cracking in PMZ.

2 Experimental

Testing sample was obtained by depositing 156 passes (12 passes per layer $\times$ 13 layers) of ERNiCrFe-13 filler metal with a uniform thickness of 25 mm over an area of 100 mm $\times$ 100 mm onto the top surface of a Q235 carbon steel substrate with dimensions of 200 mm $\times$ 200 mm $\times$ 40 mm by a TIG welding process using ERNiCrFe-13 filler wires with diameters of 1.2 mm. The chemical composition of Q235 base metal and deposited metal without dilution with ERNiCrFe-13 filler metal is listed in Table 1. The deposition and proposed TIG

refusion processes were conducted according to the TIG parameters listed in Table 2. As shown in Fig. 1, TIG refusion spot welding (shown in green) without feeding and traveling was conducted in the center of the top surface of deposited metal, which can avoid the effect of dilution from base metal due to 25 mm in thickness, and the red dotted lines represent the top and cross-sectional perimeters of the portion of the sample extracted by grinding for testing. Samples of the as-deposited filler metal were obtained similarly prior to TIG refusion.

The obtained specimens were subjected to polishing and etching with a CrO₃ acid 10% (w/v) aqueous solution at 6 V etching voltage for 10 s to reveal their microstructure. The microstructures were examined by optical microscopy (OM) using an OLYMPUS GX51 optical microscope and by scanning electron microscopy (SEM) using a HELIOS NanoLab 600i scanning electron microscope, which was equipped with an energy dispersive X-ray spectroscopy (EDX) analysis system that was used for semi-quantitative composition analyses to identify the phases. Talos 200 X-type in-situ multifunctional transmission electron microscope (TEM) was used to analyze the morphology and structure of the precipitated phases between dendrites. Differential scanning calorimetry (DSC) measurements were performed in a Netzsch STA 449F3 testing apparatus to determine the phase-transition temperatures of the constituents of the as-deposited metal samples at a heating rate of 10 °C/min from room temperature to a peak temperature of 1400 °C. The solidification path of the as-deposited metal was calculated using the commercial JMatPro-V11.2 software in conjunction with its included Ni superalloy database and the chemical composition listed in Table 1.

3 Results and discussion

Representative microstructures of the as-deposited ERNiCrFe-13 filler metal are presented by the SEM images

Table 2 TIG parameters employed during the filler metal deposition and refusion processes

Process	Current (A)	Voltage (V)	Traveling speed (mm•min ⁻¹)	Feeding speed (mm•min ⁻¹)	Arcing time (s)	Arc ending time (s)
Deposit	220	13.5	120	1200	–	–
Refusion	220	13.5	–	–	4	6

Fig. 1 Schematic illustrating the TIG refusion process and the top (left) and cross-sectional (right) perimeters of the portion of the sample extracted by grinding for testing

in Fig. 2 at various magnifications. As shown in Fig. 2(a), the microstructure included the dendritic γ structural matrix, the γ phase in the segregation regions, and interdendritic precipitates. These regions were selected from the SEM image in Fig. 2(a) for closer examination in Figs. 2(b) and (c), and included MC carbides, Laves, and σ phases. As shown in Fig. 2(b), the MC precipitates were circular particles with diameters of less than 1 μm , and the σ and Laves phases are respectively distributed in the core and along the edges of the coupled microstructures, respectively. The Laves phases possessed script-type and short rod-like morphologies. In addition, Fig. 2(c) possessed a Laves/ γ eutectic microstructure with lamellar morphology.

The results of the EDX analysis for the individual features presented in Fig. 2 are listed in Table 3. The Nb and Mo

contents in the interdendritic region and its various precipitates were generally greater than those within the core of the dendritic γ structural matrix, which was indicative of Nb and Mo segregation. Compared with the chemical composition of the as-deposited metal in Table 1, the MC carbide precipitates were enriched with Nb and Ti, the Laves precipitates are enriched with Nb and Mo, the σ -phase precipitates are enriched with Mo and Cr, and the Laves/ γ eutectic microstructure with the lamellar morphology was enriched with Nb and Mo.

The interdendritic phases identification results by TEM were presented in Figs. 3 and 4. The selected area electron diffraction (SAED) pattern for area as marked by white dotted circle 1 and 2 in Fig. 3 confirmed that the interdendritic phases are tetragonal σ and cubic MC carbide respectively. The

Fig. 2 SEM images illustrating the microstructures of the as-deposited ERNiCrFe-13 filler metal: (a) typical morphology, (b) microstructures of interdendritic precipitates within the area marked as b in (a), (c) Laves/ γ eutectic microstructures within the area marked as c in (a)

Table 3 EDX analysis results (wt%) for the analysis zones of the as-deposited ERNiCrFe-13 metal filler in Fig. 2

Analysis zone	Nb	Mo	Ni	Cr	Fe	Ti
Dendritic γ	1.35 ± 0.52	3.49 ± 0.62	52.63 ± 2.45	30.93 ± 2.63	10.37 ± 2.08	–
γ in segregation zone	4.90 ± 0.85	4.65 ± 0.85	48.00 ± 2.75	29.76 ± 1.78	8.80 ± 1.36	–
MC carbides	33.70 ± 1.46	2.84 ± 0.55	11.97 ± 1.15	9.23 ± 0.98	2.27 ± 0.32	5.21 ± 0.68
Laves	20.46 ± 1.75	7.10 ± 0.91	38.40 ± 3.35	24.87 ± 1.62	6.83 ± 0.76	–
σ	5.04 ± 0.83	18.08 ± 1.65	30.03 ± 1.15	36.45 ± 1.59	7.47 ± 1.32	–
Laves/ γ eutectic	15.70 ± 2.45	6.94 ± 1.40	42.99 ± 3.55	24.95 ± 2.58	6.57 ± 1.41	–

Fig. 3 Identification of σ phase and MC carbide by TEM in bright field and SAED

SAED pattern for area as marked by white dotted circle in Fig. 4 confirmed that the interdendritic phase was Laves. Combined with semi-quantitative results from SEM-EDS in Table 3, the structural formulas of the MC carbide, Laves, and σ precipitates shown in Fig. 2 can be given as $(\text{Nb,Ti})\text{C}$, $(\text{Ni,Cr,Fe})_2(\text{Nb,Mo})$, and (CrMoFe) respectively, which was coincide with microstructure characterization results obtained by TEM for deposited metal with similar composition [16, 17].

The melting and phase transformation points of the as-deposited ERNiCrFe-13 filler metal were measured by DSC to analyze the microstructural evolution of the material in the

PMZ. The obtained DSC heating thermogram was presented in Fig. 5, in which three peaks marked as A, B, and C can be observed. According to the microstructural analysis associated with Fig. 2 and related reports [18–20], we can assign peak A at 1000 °C to melting at the Laves/ γ eutectic microstructure interface, peak B at 1080 °C to melting at the σ/γ eutectic microstructure interface, and peak C in the temperature range of 1280–1350 °C to the melting of the dendritic γ structural matrix. Melting at the MC/ γ eutectic microstructure interface was not observed perhaps owing to the low concentration of MC carbide precipitates.

The solidification behavior of the as-deposited ERNiCrFe-13 filler metal obtained by JMatPro was presented according to the contents of elements in the liquid (L) phase versus decreasing temperature and the solidification path in Figs. 6(a) and (b), respectively. As shown in Fig. 6(a), both Nb and Mo exhibited significant segregation tendency and

Fig. 4 Identification of Laves phase by TEM in dark field and SAED

Fig. 5 DSC curve of the as-deposited ERNiCrFe-13 filler metal during heating

Fig. 6 Solidification behavior of the as-deposited ERNiCrFe-13 filler metal obtained by JMatPro: (a) contents of elements in the liquid phase (L) versus decreasing temperature; (b) solidification path

their concentrations in the L phase gradually increased during solidification, while Cr and Fe exhibited the reverse characteristic at the liquid/solid interface. Here, the concentrations of Cr and Fe in the L phase gradually decreased during solidification, and substantially decreased for temperatures less than 1174 °C during which the Ni content in the L phase increases substantially. The solidification path of the deposited metal proceeds from the L phase as shown in Fig. 6(b). Accordingly, the solid γ matrix phase is first to precipitate from the L phase at 1346 °C. This is followed by three eutectic reaction transformations in turn: $L \rightarrow \gamma + MC$ at 1194 °C, $L \rightarrow \gamma + \sigma$ at 1174 °C, and $L \rightarrow \gamma + \text{Laves}$ at 1162 °C. The increasing Ni, Nb, and Mo contents and decreasing Cr and Fe contents in the L phase for temperatures less than from 1174 °C (Fig. 6(a)) contribute to the precipitation of the Cr, Fe, Nb, and Mo enriched σ and Laves phases. These results were in good agreement with the primary data presented in Figs. 2 and 5, and Table 3.

The microstructure of a representative refusion welding spot cross section was shown by the OM image presented in Fig. 7. Different regions denoted as the refusion zone (RZ), the PMZ, and the heat affected zone (HAZ) exhibited different microstructural characteristics that are observable on the coarse scale of the figure. The SEM images given in Figs. 8(a) and (b) indicated that the microstructure of the RZ consisted of columnar γ -phase dendrites separated by

interdendritic precipitates, while the interdendritic precipitates in the HAZ are relatively small both in size and number. The width of the PMZ was estimated from Fig. 5 to be 0.25 ± 0.05 mm. A comparison of Figs. 2 and 8 indicated that the interdendritic γ eutectic microstructures in the PMZ differed substantially from those observed in the as-deposited metal. Firstly, we note that the grain boundaries run generally perpendicular to RZ-PMZ boundary. Secondly, the high magnification SEM images given as Figs. 8(b) and (c) indicated the presence of interfaces and triple grain junctions between the interdendritic γ phase in the PMZ and Laves, σ , and MC precipitates with three distinct morphologies, including long chain, skeletal, and lamellar.

It can be observed that the eutectic microstructures show significantly different morphology in the PMZ after TIG refusion. The microstructural evolution mechanism in PMZ can be analyzed according to constitutional liquation theory proposed by Pepe and Savage [21] for maraging steel weldments. Here, constitutional liquation is commonly associated with a high heating rate and large concentration gradients of solute elements between eutectic components [22], where the heating of secondary-phase particles to temperatures below the alloy matrix solidus temperature results in localized melting at particle/matrix interfaces and corresponding nearby eutectic reactions. Constitutional liquation at interfaces between the γ -phase matrix and secondary phases such as MC and

Fig. 7 OM image illustrating the microstructures in the refusion zone (RZ), partially melted zone (PMZ), and heat affected zone (HAZ) of a representative ERNiCrFe-13 filler metal refusion welding spot

Fig. 8 SEM images illustrating the microstructures in the PMZ of a representative ERNiCrFe-13 filler metal refusion welding spot: (a) typical morphology, (b) details within the area marked as b in (a), (c) details within the area marked as c in (b)

Laves has been previously reported in nickel alloys [23, 24]. Based on Cr-Nb, Fe-Nb, Cr-Mo and Fe-Mo binary phase diagram, both Laves and σ have a quite high melting point. The peak temperature in PMZ during TIG refusion is lower than the melting point of matrix. The microstructural evolution reason in PMZ was unstable Laves/ γ and σ/γ interface at elevated temperature, which has been supported by DSC results in Fig. 5 and solidification calculation results in Fig. 6(b). The Laves/ γ and σ/γ eutectic microstructures formed as eutectic reactions in the as-deposited metal. Due to Laves and σ with high Nb and Mo contents respectively, the formation of Laves and σ will consume a large number of Nb and Mo atoms in the liquid during eutectic reactions, so the matrix near the particle is not likely to be at the max solubility. There are large concentration gradients of Nb and Mo components at the interface of eutectic components. Therefore, the Laves/ γ and σ/γ eutectic microstructures in the PMZ will melt and solidify during TIG refusion.

To better illustrate the role of chemical composition in microstructural evolution, the effects of Nb and Mo concentrations on the solidification path were analyzed based the isopleth results calculated by JMatPro as presented in Fig. 9. As shown in Fig. 9(a) and (b), both Nb and Mo contents have important effects on solidification path. As indicated by the

vertical dashed lines in the figures, the concentrations of Nb and Mo for the eutectic reactions were 7.3 wt% and 8.5 wt%, respectively. The high concentration of Nb can be expected to play a leading role in constitutional liquation at the Laves/ γ interfaces owing to the relatively low melting point of Laves/ γ eutectic reaction, while the high concentration of Mo can be expected to play a leading role in constitutional liquation at the σ/γ interface owing to the relatively low melting point of σ/γ eutectic reaction. During the rapid heating stage, the Laves and σ phase precipitates decomposed with the release of a large concentration of solute atoms (i.e., Nb and Mo atoms, respectively) and eventually disappear due to the diffusion of the solute atoms into the surrounding γ -phase segregation zone. Constitutional liquation occurs when the composition of the γ -phase reached the eutectic point.

According to the above discussion, it can be concluded that constitutional liquation and solidification occurred successively at the Laves/ γ and σ/γ interfaces of the ERNiCrFe-13 filler metal during the TIG refusion thermal cycle, while the γ -phase matrix retained its dendritic structure throughout. The originally discontinuous and localized pockets of liquid arising from the melting of the interspersed interdendritic secondary phases observed in Figs. 2(a) and (b) readily linked to form the long chains observed in Fig. 8(b) owing to the good

Fig. 9 Calculated isopleth results obtained by JMatPro with respect to (a) Nb concentration and (b) Mo concentration

Fig. 10 SEM images representative of liquidation cracking in the PMZ of a ERNiCrFe-13 filler metal refusion welding spot: (a, b) typical morphologies, (c) high-magnification image

fluidity of the liquid and the long period over which it flowed. The advancement of the liquid/ γ interface progressively decreased the width of the interface, and the Nb and Mo enriched liquid is surrounded within the narrow interdendritic spaces to form long chain γ /MC/Laves eutectic microstructures along the grain boundary. The skeletal γ /MC/Laves microstructures observed in Fig. 8(c) may be attributed to the large-area precipitate/ γ interfaces in the interior of the fine lamellar and skeletal eutectic microstructures of the as-deposited metal shown in Figs. 2(b) and (c). More liquid formed in the interior of the eutectic microstructures than at their edges during heating, and thereby forming Laves-phase skeletal structures with the γ -phase within the skeleton gap. Finally, the densely distributed and large-sized primary precipitates in the as-deposited metal contributed during the cooling process to the solidification of the liquid at triple grain junctions into γ /Laves eutectic microstructures with the fine lamellar morphology shown in Fig. 8(c).

As indicated by the SEM images presented in Fig. 10, the ERNiCrFe-13 filler metal was subject to liquidation cracking in the PMZ of the refusion welding spot. The observed liquidation cracking was nearly always distributed along the eutectic microstructure grain boundaries running perpendicular to the RZ-PMZ boundary, as illustrated in Figs. 10(a) and (b). Fine lamellar γ /Laves microstructures were also observed at the ends of the fractures, as illustrated in Fig. 10(c). Based on past discussions, these liquidation cracks can be attributed to constitutional liquation, which induced shrinkage stress under solidification in the absence of additional liquid flow during cooling. The occurrence of constitutional liquation and the corresponding liquidation cracking observed here has also been demonstrated previously for Ni-based superalloys under large temperature and compositional gradients when the shrinkage stresses involved were sufficiently high [25].

4 Conclusion

1. The γ /Laves and γ / σ eutectic microstructures of the ERNiCrFe-13 metal exhibited constitutional liquation in the PMZ during TIG refusion and solidified upon cooling

as long chain, skeletal, and fine lamellar Laves/ γ /MC eutectic microstructures.

2. Nb and Mo played a leading role in the constitutional liquation of γ /Laves and γ / σ eutectic microstructures respectively.
3. Liquidation cracking occurred along the long linear chain grain boundaries in the PMZ formed by the constitutional liquation at the γ /Laves and γ / σ eutectic microstructure interfaces during heating.

Availability of data and material Not applicable.

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Compliance with ethical standards

Conflicts of interest/competing interests Not applicable.

Code availability Not applicable.

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