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Full Text

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Abstract

SIMP steel welded joints produced by two different welding techniques were exposed to oxygen-saturated liquid lead–bismuth eutectic (LBE) at 550°C for durations of up to 2000h. The results show that, under identical corrosion conditions, the two types of welded joints exhibit only slight variations in the overall thickness of the corrosion layer. In both scenarios, a similar oxide scale architecture develops, characterized by a three-layer structure: an outer layer of Fe3O4, an inner spinel layer of FeCr2O4, an inner oxidation zone mainly composed of Cr2O3 and SiO2. Despite the structural similarity in oxide scale, notable differences in corrosion resistance are

evident between the weld metal and the base metal. This phenomenon is attributed to grain coarsening induced by the heat input during the welding process, as well as the higher concentrations of Cr and Si in the base metal compared to the weld metal. While coarse grains significantly degrade the corrosion resistance of weld metal, the presence of Cr and Si facilitates the formation of a dense protective oxide film that enhances durability. The interplay between these competing factors ultimately results in distinct corrosion behaviors observed between the weld metal and the base metal.

Keywords: LBE; SIMP; Weld Joints; Corrosion.

1 Introduction

Lead-bismuth eutectic (LBE) exhibits excellent physical, chemical, and thermo-hydraulic properties. So it has been regarded as a preferred coolant material for next-generation nuclear energy systems, such as accelerator-driven subcritical systems (ADS) and lead-cooled fast reactors (LFR)[1-3]. Despite its numerous advantages, the compatibility of LBE with structural materials remains one of challenges that constrains the development of ADS and LFR systems[4-6].

The corrosion of structural materials in LBE is primarily governed by two mechanisms: oxidation corrosion and dissolution. Current research indicates that temperature, flow velocity, and dissolved oxygen concentration are the critical parameters influencing corrosion behavior[7-17]. When the oxygen in liquid LBE drops below the critical threshold necessary for the formation of a stable Fe_3O_4 oxide layer, dissolution-dominated corrosion occurs. During this process, alloying elements such as

Ni, Fe, Cr, and Mn continuously migrate from the substrate into the LBE[8,13], leading to significant mass transfer and a subsequent deterioration in the material' s mechanical load-bearing capacity. Conversely, when the dissolved oxygen concentration exceeds the critical threshold for Fe_3O_4 formation, oxygen within the LBE reacts with the structural material to form a protective oxide scale. This layer acts as a diffusion barrier, mitigating further corrosion of the substrate. However, under conditions of elevated temperatures and high oxygen

concentrations, steel components are susceptible to excessive oxidation, leading to the formation of thick oxide layers. The formation of an excessively thick oxide scale poses two primary risks: firstly, it significantly reduces the heat transfer efficiency of reactor thermal components, such as fuel cladding; secondly, the oxide layer is prone to spallation, which increases the risk of flow blockage within the primary coolant circuit. As the operating temperatures of future reactors continue to escalate, the corrosion challenges posed by liquid LBE to structural materials in ADS and LFR systems are expected to become increasingly severe.

The corrosion behavior of materials in LBE is intrinsically linked to their chemical composition, microstructure, and processing history[18–19]. In the fabrication of reactor components, welding stands as the predominant joining technique. During the welding process, the material experiences localized melting and subsequent solidification, resulting in significant compositional and microstructural heterogeneities across the weld metal and the heat-affected zone. These inhomogeneous regions are more susceptible to localized corrosion, including selective dissolution and Liquid Metal

Embrittlement (LME). Under the coupled effects of high temperature, mechanical stress, and neutron irradiation, these corrosion processes may be further exacerbated, potentially leading to catastrophic structural failure. Therefore, studies on the corrosion behavior of welded joints in liquid LBE are essential for elucidating underlying corrosion mechanisms and developing robust life-cycle assessment and prediction methodologies.

Currently, several studies have been conducted worldwide to investigate the corrosion of welded joints in liquid LBE. Chen[20] et al. investigated the influence of post-weld heat treatment on the microstructure of martensitic steels. Their findings demonstrated that post-weld heat treatment can effectively refine the martensitic grain size and promote a more homogeneous grain distribution. Additionally, Mustari[21] et al. observed that the cooling rate during the welding process directly dictates the final morphology; higher cooling rates lead to a finer grain size within the fusion zone. Li[22] et al. investigated the corrosion behavior and underlying mechanisms of ferritic/martensitic (F/M) steel welded joints in liquid LBE. Their research revealed that the degradation of corrosion resistance in welded joints is primarily attributed to the formation of coarse columnar grains. Lei[23] et al. conducted a study on the cavitation erosion of welded joints in liquid LBE at 550°C. Results indicated that under identical experimental conditions, the base metal exhibited superior cavitation erosion resistance compared to the weld metal. Although preliminary investigations have been conducted on the corrosion behavior of welded joints in LBE environments, a systematic and in-depth understanding remains lacking. In particular, the evolutionary patterns of

corrosion behavior and the underlying degradation mechanisms of post-weld materials are not yet fully understood, necessitating further comprehensive research.

Compared with austenitic steels, martensitic steels exhibit superior mechanical strength, and greater resistance to radiation-induced swelling, making them among the most promising candidate materials for future ADS and LFR demonstration facilities. Consequently, the Institute of Modern Physics of the Chinese Academy of Sciences, in collaboration with relevant domestic institutions, has developed SIMP steel—the first novel martensitic steel in China specifically designed for service in LBE environments[24-27]. To evaluate the LBE corrosion performance of SIMP steel welded joints, corrosion tests were conducted under various experimental conditions on joints fabricated using two distinct welding processes: Auto-TIG and Manual-TIG. By systematically characterizing the elemental distribution, microstructure, and corrosion product morphology of the post-test specimens, this study aims to elucidate the corrosion behavior and underlying mechanisms of SIMP steel welded joints associated with different welding techniques.

2 Experiments

2.1 The welding specimens

The SIMP steel welded plates, each with thickness of 15 mm, were fabricated using Manual-TIG and Auto-TIG welding processes, respectively, were supplied by the Shanghai Electric Nuclear Power Group. Table 1 shows the elemental composition of SIMP steel base metal (BM), manual weld metal (MWM) and automatic weld metal

(AWM). The post-weld heat treatment procedure for the joints was conducted as follows:

the specimens were furnace-heated at a rate of 200 °C/h, held at 600 °C for 20 minutes,

then further heated to 720 °C and held for 4 hours. The resulting TIG-welded joints

exhibited excellent formation and structural integrity (Fig. 1).

Fig. 1. SIMP steel joints produced by (a) automatic welding and (b) manual welding. The yellow

bars indicate the sampling orientation and dimensions for the corrosion specimens.

Table 1. Elemental content (%)

	C	Cr	Si	Mn	W	P	S
BM	0.16	11.8	1.5	< 0.1	1.3	0.005	< 0.005
AWM	0.11	9.04	0.497	1.06	1.94	0.009	0.0037
MWM	0.12	8.99	0.5	0.93	2.15	0.003	0.003

The impact properties of the joints fabricated by the two techniques were evaluated.

Fig. 2 presents the average impact toughness value of the weld metal and the

heat-affected zone (HAZ) obtained from both manual and automatic TIG welding.

Statistical analysis of the test results shows that for the manually welded specimens

(Manual-TIG), the impact energy reached 183.9 J in the weld metal and 81.7 J in the

heat affected zone. For the automatically welded specimens (Auto-TIG), the impact

energy was 171.2 J in the weld metal and 76.4 J in the heat affected zone. Furthermore,

tensile tests were performed on the welded joint specimens (Table 2). The results

indicate that the impact and tensile mechanical properties of the joints produced by both welding processes fully meet the requirements stipulated in the relevant National Standards (GB/T 1220-2007, NB/T 47014).

Fig. 2. Average impact properties of the weld metal (1) and heat-affected zone (2) for manual and automatic welded joints.

Table 2. Summary of average tensile properties of manual and automatic welded joints at different temperatures.

Process	Temperature/°C	Yield strength/MPa	0.2% proof strength/MPa	Elongation after fracture/%	Reduction of area/%
Manual-TIG	27	817.7	604	15.3	54.3
	250	625.3	528.5	13.2	52.8
	350	632.3	518	15.8	67.3
	450	568.2	488.8	18.7	76.7
	550	436.3	367.5	23.7	86
Auto-TIG	27	819.7	615.8	17.7	65
	250	667.3	541.3	16.3	70.8
	350	632.3	518	15.8	67.3
	450	568.2	488.8	18.7	76.7
	550	436.3	367.5	23.7	86

To investigate microstructural evolution, Electron Backscatter Diffraction (EBSD) was utilized to characterize the weld metal and base metal of SIMP steel joints. Microstructural morphologies and grain size distributions are illustrated in Fig. 3 and 4. The analysis confirms that the weld metal microstructure consists of lath martensite. Notably, the weld metal grains exhibit significant coarsening relative to the base metal, as evidenced by the grain size statistics.

(a) (b) (c)

Fig. 3. EBSD maps of the weld metal in (a) manual and (b) automatic WM, and (c) the base metal.

(a) (b) (c)

Fig. 4. Grain size distributions of the weld metal in (a) manual and (b) automatic welded joints, and

(c) the base metal.

2.2 LBE Corrosion

The specimens were sectioned along the direction indicated by the yellow bars in Fig. 1 into rectangular coupons with dimensions of 25 mm × 15 mm × 2 mm. The surfaces of these welded-joint specimens were then ground and polished to eliminate surface artifacts. Subsequently, the samples were ultrasonically cleaned in a solution of isopropanol and ethanol to ensure that residual processing scratches would not interfere with the subsequent corrosion behavior. Specimens were subjected to liquid LBE corrosion tests under oxygen-saturated conditions for exposure durations of 100 to 2000 hours. The corrosion experimental apparatus consists of an LBE crucible, a heating furnace, a specimen hoisting system, and a vacuum pump. Temperature-sensing elements are positioned to monitor the coolant temperature in real time. The LBE remains in a static state throughout the test. Based on the oxygen solubility correlation in liquid LBE, given by $\lg C_0 = 1.2 - 3400/T$, the saturated oxygen concentration at 550 °C is determined to be 1.17×10^{-3}%.

2.3 Characterization

The elemental distribution, chemical composition, and surface morphologies of the weld metal and base metal were characterized using Scanning Electron Microscopy (SEM, ApreoS) and Electron Probe Micro Analysis (EPMA, Shimadzu8050G). The grain size distribution across these regions was determined via Electron Backscatter Diffraction (EBSD). Phase identification of the LBE-corroded

welded joint surfaces was performed using X-ray Diffraction (XRD, X' Pert).

Furthermore, the detailed structure and composition of the oxide layers formed

Fig. 6. XRD patterns

Figure 1: Fig. 6. XRD patterns

after 100 hours of exposure were investigated using Transmission Electron Microscopy (TEM) to elucidate the initial stages of the corrosion process.

3 Results

Figs. 5-6 present the XRD patterns of SIMP steel after exposure to LBE for 100, 500, 1000, and 2000 hours. By comparing the results with standard PDF (Powder Diffraction File) cards, it was determined that the corrosion layers on both the weld metal and the base metal are primarily composed of magnetite Fe_3O_4 or $FeCr_2O_4$ spinel. Additionally, traces of Bi were detected within the oxide scale, indicating a minor degree of bismuth penetration into the corrosion products.

Figure labels: Bi; $FeCr_2O_4$; Fe_3O_4.
Axes: Intensity/a.u.; $2\theta/(^\circ)$.
Traces: (a), (b), (c), and (d).

Fig. 5. XRD patterns of corrosion products on manual welded SIMP WM after exposure for (a) 100 h, (b) 500 h, (c) 1000 h, and (d) 2000 h.

Legend: Bi; $FeCr_2O_4$; Fe_3O_4.
Vertical axis: Intensity/a.u.
Horizontal axis: $2\theta/(^\circ)$.
Curves: (a), (b), (c), and (d).

Fig. 6. XRD patterns of corrosion products on automatic welded SIMP WM after exposure for (a) 100 h, (b) 500 h, (c) 1000 h, and (d) 2000 h.

Figures 7 through 10 illustrate the cross-sectional SEM micrographs and the corresponding EDS line scans of the weld metal and base metal of the SIMP after various exposure durations. Using SEM and EDS, the elemental distribution and chemical composition of the oxide scales in both the weld metal and base metal were systematically characterized for each corrosion specimen across all testing intervals. It is clearly observable from the figures that the weld metal and base metal of the SIMP steel, under both manual and automatic welding processes, exhibit a distinct double-layered oxide structure when subjected to identical corrosive conditions [24–34]. These observations are in good agreement with previous studies

regarding corrosion behavior in LBE [35-36].

The SEM results reveal that the thickness of the oxide scales for both manual and automatic welding samples increases progressively with prolonged exposure time. According to the EDX line scan analyses of the weld metal and base metal under different welding processes (indicated by the blue arrows in Fig. 7-

10), the elemental distribution across the oxide layers from the outer surface to the inner substrate can be clearly characterized. The EDX line scan profiles reveal that the outer oxide layer is predominantly composed of Fe and O, with trace amounts of Pb and Bi detected within this region. In contrast, the inner oxide layer including the internal oxidation zone (IOZ), primarily consists of Fe, Cr, Si, and O. Furthermore, comparative analysis indicates that the thickness of each individual sub-layer increases monotonically as a function of exposure time.

Fig. 7. Morphology and EDS analysis of the oxide scales formed on the manual weld welding metal after LBE corrosion for (a) 100 h, (b) 1000 h, and (c) 2000 h.

Fig. 8. Morphology and EDS analysis of the oxide scales formed on the automatic welding weld metal after LBE corrosion for (a) 100 h, (b) 1000 h, and (c) 2000 h.

- **(a)** Matrix; oxide scale thickness: 3.9$±$0.3 μm; scale bar: 10 μm. EDS line scan: Intensity vs Distance (μm); elements: Bi, Cr, Fe, Mn, O, Pb, Si, W.
- **(b)** Matrix; oxide scale thickness: 15.7$±$1.2 μm; scale bar: 10 μm. EDS line scan: Intensity vs Distance (μm); elements: Bi, Cr, Fe, Mn, O, Pb, Si, W.
- **(c)** Oxide scale thickness: 29.3$±$4.1 μm; scale bar: 10 μm. EDS line scan: Intensity vs Distance (μm); elements: Bi, Cr, Fe, Mn, O, Pb, Si, W.

Fig. 9. Morphology and EDS analysis of the oxide scales formed on the manual welding base metal after LBE corrosion for (a) 100 h, (b) 1000 h, and (c) 2000 h.

- **(a)** Matrix; oxide scale thickness: 3.4$±$0.1 μm; scale bar: 10 μm. EDS line scan: Intensity vs Distance (μm); elements: Bi, Cr, Fe, Mn, O, Pb, Si, W.
- **(b)** Matrix; oxide scale thickness: 12.6$±$0.4 μm; scale bar: 10 μm. EDS line scan: Intensity vs Distance (μm); elements: Bi, Cr, Fe, Mn, O, Pb, Si, W.
- **(c)** Matrix; IOZ; oxide scale thickness: 21.9$±$1.9 μm; scale bar: 10 μm. EDS line scan: Intensity vs Distance (μm); elements: Bi, Cr, Fe, Mn, O, Pb, Si, W.

(a) (b) (c)

Fig. 10. Morphology and EDS analysis of the oxide scales formed on the automatic welding base metal after LBE corrosion for (a) 100 h, (b) 1000 h, and (c) 2000 h.

Fig.11 presents the EPMA cross-sectional maps of SIMP steel samples under different welding processes after exposure to oxygen-saturated liquid LBE at 550°C for 500h and 1500h. It is observed that after 1500h of exposure, Cr-rich islands resulting from Cr segregation are evident within the Fe-Cr spinel layer.

Comparative analysis reveals that the number density of these Cr-rich islands within the spinel layer increases significantly with prolonged corrosion duration. Furthermore, these Cr-rich islands exhibit a tendency to align into chain-like configurations as the corrosion time progresses. Concurrently, the penetration of Pb into the outer oxide layer was observed, with the infiltrated Pb exhibiting a degree of enrichment at the interface between the Fe-Cr spinel layer and the magnetite layer.

The total oxide scale thicknesses for the weld metal and base metal of the SIMP steel joints under different welding processes and exposure durations are summarized in

Table 3. These values represent the average of ten equidistant measurements obtained from the SEM cross-sectional images, with the corresponding standard deviations provided. Based on these data, the evolution curves of the oxide scale thickness as a function of corrosion time were plotted in Fig. 12, illustrating the oxidation kinetics for both the weld and base metal regions. Fig. 12 depicts the evolution of the average oxide scale thickness as a function of corrosion time for various welding processes. Combined EDS mapping and line scan analyses reveal the presence of an additional oxide sub-layer at greater depths, primarily composed of Fe, Cr, Si, and O. As clearly evidenced in Fig. 12 the total oxide thickness increases progressively with prolonged exposure duration. Furthermore, Table 3 indicates that under identical corrosive conditions, the discrepancies in oxide scale thickness between corresponding regions of the SIMP steel joints fabricated by different welding processes are negligible.

Nevertheless, as evidenced in Fig. 12, the oxide scales developed on the weld metal are consistently thicker than those formed on the base metal across all samples.

Visible panel labels: 500h-Manual Welding; 500h-Automatic Welding; 1500h-Manual Welding; 1500h-Automatic Welding. Element maps: Fe, Cr, O, Si, Pb, Bi.

Fig. 11. EPMA elemental maps of the oxide scales on SIMP steel samples produced by different

welding processes after exposure to liquid LBE for 500 h and 1500 h.

Table 3. Average thickness of oxide scales formed on different regions of the welded joints after LBE exposure. (MWM) Manual weld metal, (AWM) automatic weld metal, (MBM) manual welding base metal, and (ABM) automatic welding base metal.

Exposure time/h	Samples	Inner layer and IOZ/μm	Outer layer/μm	Total/μm
100h	MWM	2.1$±0.2\|2.6±0.2\|3.9±0.2\|\|\|$	$MBM\|1.9±0.1\|2.0±0.1\|3.4±0.1\|\|\|$	$AWM\|2.2±0.1$

| ABM | 5.8$±0.2|4.2±0.4|8.9±$0.7 |

|–|–|–:|–:|–:| | 1500h | MWM | 9.0$±0.1|9.2±0.2|17.7±0.5|||$MBM$|8.0±0.3|7.3±0.4|14.9±1.7|||AWM|12.0±2.3 |

Fig. 12. Evolution of oxide scale thickness as a function of corrosion time for the weld metal and base metal of SIMP steel produced by different welding processes: mean thickness and (a) Manual weld metal, (b) automatic weld metal, (c) manual welding base metal, and (d) automatic welding base metal.

To further elucidate the liquid LBE corrosion behavior of the SIMP steel welded joints, high-resolution transmission electron microscopy (TEM) was employed. TEM lamellae were extracted using the focused ion beam (FIB) technique to enable

site-specific characterization of the oxide scale' s microstructure and elemental distribution at a finer scale. High-angle annular dark-field (HAADF) imaging was utilized, as it is highly sensitive to atomic number, allowing for a clear distinction between oxygen and the heavier metallic elements. HRTEM analysis reveals the presence of an additional inner oxide zone (IOZ) at greater depths, as depicted in Fig. 13a, 14a, and 15a. From the top to the bottom of the micrographs, the sequence of layers corresponds to the residual LBE, the outer oxide layer, the inner oxide layer, the IOZ, and the steel substrate. EDS mapping results (Fig. 13h-i, 14h-i, and 15h-i) further demonstrate that both the inner oxide layer and the IOZ are predominantly composed of Cr-rich and Si-rich oxides. In contrast, the outer oxide layer consists primarily of magnetite (Fe_3O_4), with no discernible enrichment of Cr or Si observed. Additionally, unoxidized Fe-rich regions were identified in the IOZ. This phenomenon is attributed to the oxygen partial pressure (P_{O2}) gradient, which decreases progressively from the outer surface toward the interior. The localized P_{O2} at these depths falls below the threshold required for Fe oxidation, yet remains sufficient to promote the formation of more thermodynamically stable Cr- and Si-rich oxides.

Figure annotations:
(a) #1; #2; #3; #4; 500 nm.
(b) SAED of #1; Fe_3O_4 [$32\bar{1}$]; 5 nm^{-1}.
(c) SAED of #2; Martensitic, BCC, [311]; 5 nm^{-1}.
(d) HRTEM of #3; 20 nm.
(e) SAED of #4; Martensitic, BCC, [311]; 5 nm^{-1}.
(f) Fe; Mapping.
(g) O.
(h) Cr.
(i) Si.

Fig. 13. STEM characterization of manual weld metal in SIMP steel joint: (a) STEM image, (b) SAED pattern of #1, (c) SAED pattern of #2, (d) HRTEM image of #3, and (e) SAED pattern of #4.

Visible labels in Fig. 14: (a) #1, #2, #3, #4, 500 nm; (b) SAED of #1,

Fe_3O_4 [111], 5 nm^{-1}; (c) SAED of #2, Martensitic, BCC, [311], 5 nm^{-1}; (d) HRTEM of #3, 20 nm; (e) SAED of #4, Martensitic, BCC, [210], 5 nm^{-1}; (f) Fe; (g) O; (h) Cr; (i) Si.

Fig. 14. STEM characterization of the automatic weld metal in the SIMP steel joint: (a) STEM image, (b) SAED pattern of #1, (c) SAED pattern of #2, (d) HRTEM image of #3, and (e) SAED pattern of #4.

High-resolution TEM (HRTEM) was employed for a more in-depth investigation of the inner oxide layer (Fig. 13d, 14d, and 15d). By correlating the elemental distribution of Cr and Si in the HAADF-STEM images with the structural features revealed by HRTEM, it can be confirmed that the oxides within the IOZ are predominantly composed of Cr- and Si-based oxides. This is primarily dictated by the

diminished oxygen partial pressure (P_{O2}) at the oxidation front, which favors the preferential formation of thermodynamically stable Cr and Si oxides, while inhibiting the oxidation of Fe.

Fig. 15. STEM characterization of the base metal in the SIMP steel joint: (a) STEM image, (b) SAED pattern of #1, (c) SAED pattern of #2, (d) HRTEM image of #3, and (e) SAED pattern of #4.

The microstructure and elemental distribution of the various oxide scales were characterized in detail using TEM, with the results presented in Figure 16 and 17. As shown in Figure 16, the inner oxide layer of the manually welded SIMP steel specimen exhibits a distinct porous morphology. By indexing against standard PDF cards, the

primary phase of the inner oxide layer was identified as $FeCr_2O_4$. EDS elemental mapping reveals a pronounced inhomogeneity in the chemical distribution within the IOZ. Specifically, localized Si-rich regions are observed within the IOZ, which are typically encapsulated by Cr-rich layers. Based on these structural and compositional characteristics, it is inferred that the IOZ of the manually welded SIMP steel consists of a composite oxide structure, characterized by SiO_2 cores enveloped by chromium oxides.

HRTEM imaging and the corresponding EDS spectra in Figure 17 further confirm the structural configuration where Cr-oxides encapsulate Si-oxides. Results reveal that the Si-rich regions are distributed in an elongated, lath-like morphology within the IOZ and exhibit a distinct amorphous character, identifying them as amorphous SiO_2.

Fig. 16. TEM and EDS spectra of outer and inner oxide scales for manual welded SIMP steel.

Fig. 17. TEM micrographs and EDS spectra of the IOZ for the manually welded SIMP steel.

Cross-sectional morphologies of the oxide scales formed on the automatically welded SIMP steel after 100 h of exposure to LBE are illustrated in Fig. 18.

EDS elemental mapping clearly delineates the interface between Fe and Cr, which precisely corresponds to the boundary between the outer oxide layer and the inner oxide layer. By indexing against standard PDF cards, the outer oxide layer was confirmed to consist primarily of the magnetite phase (Fe_3O_4). Furthermore, EDS point analysis verified the trans-interface penetration of Pb and Bi elements; these elements are not only present within the outer oxide but have also infiltrated deep into the inner oxide layer. This observation is highly consistent with the elemental line scans presented in Figures 7 and 10.

Fig. 19 illustrates the micromorphological features of the IOZ in the automatically

welded SIMP steel after 100 h of exposure to liquid LBE. It can be observed that the Si-rich oxide regions within the IOZ are encapsulated by Cr-rich oxides. Combined with the high-resolution TEM results, the Si-rich oxides exhibit distinct amorphous characteristics, confirming that the silicon oxides in the IOZ are indeed SiO_2.

Fig. 18. TEM and EDS spectra of the outer and inner oxide scales of automatically welded SIMP steel.

Fig. 19. TEM micrographs and EDS spectra of the internal oxidation zone (IOZ) for the automatically welded SIMP steel.

Fig. 20 presents the morphological characteristics of the oxide scale formed on the SIMP steel base metal after 100 h of immersion in liquid LBE. It is clearly observed that the oxide scale on the base metal surface exhibits a loose and porous structure throughout. EDS analysis reveals that Pb and Bi have infiltrated both the outer and intermediate oxidation layers, indicating that liquid LBE can migrate inward through the pores and defects within the oxide scale.

Fig. 21 illustrates the microstructural characteristics of the IOZ in the SIMP steel base metal after 100 h of exposure to liquid LBE. TEM observations reveal that the Si-rich oxide regions within the IOZ are encapsulated by a continuous network of Cr-rich oxides, representing a typical composite oxide scale structure. Further HRTEM

analysis, indexed against standard PDF cards, confirms that the Cr-rich oxide phase

within the IOZ is Cr_2O_3.

Fig. 20. TEM and EDS spectra of outer and inner oxide scales for SIMP steel base metal.

Fig. 21. TEM micrographs and EDS spectra of the IOZ for the SIMP steel base metal.

Measurement results indicate that the average thickness of the oxide scales

formed on both the manually welded and automatically welded samples is approximately 3.9 μm.

The oxide scales of both materials exhibit a similar triplex structure, consisting of an outer oxide layer, an inner oxide layer, and an IOZ. Distinct interfaces exist between these three layers; notably, the interface between the outer oxide layer and inner oxide layer is considered to coincide with the original specimen surface. EDX analysis of the elemental distribution reveals that the outer oxide layer is enriched in Fe and O, whereas the inner oxide layer and IOZ primarily consist of Fe, Cr, and O. Based on the elemental distribution and diffraction patterns, the phase constitution of each layer was identified: the outer oxide layer primarily consists of Fe_3O_4, while the inner oxide layer is composed of $FeCr_2O_4$ (Fig. 16, 18, and 20). Due to the insufficient oxygen partial pressure (P_{O_2}) at the oxidation front, only the more thermodynamically stable Cr_2O_3 and SiO_2 can form within the internal oxidation zone (Fig. 17, 19, and 21). In the innermost region, which remains unaffected by corrosion, the substrate maintains its original body-centered cubic (BCC) martensitic structure (Fig. 13e, 14e, and 15e).

4 Discussion

4.1 Corrosion Mechanisms in Weld Joints

Based on the aforementioned analysis, it can be inferred that oxidation is the primary corrosion mechanism for both manually and automatically weld metal specimens, as well as base metal specimens. The oxidation kinetics of both weld joints exhibit a near-parabolic dependence, resulting in similar surface oxide morphologies.

The characteristic oxide structure consistently comprises an outer magnetite layer and an inner spinel layer. Our current findings are in good agreement with previous studies[37–39] on ferritic/martensitic (F/M) steels exposed to oxygen-saturated liquid LBE within the temperature range of 350–600 °C. The observed parabolic oxidation kinetics indicate that the oxidation behavior of both the manually and automatically welded joints is primarily governed by the outward diffusion of Fe cations. Based on established oxidation models[40–44], the formation mechanism of the oxide scale can be elucidated as follows: When martensitic steel is exposed to a liquid LBE environment, Fe atoms continuously diffuse toward the liquid LBE/oxide interface and react with the dissolved oxygen in the LBE, leading to the formation of a magnetite (Fe_3O_4) layer. The outward diffusion of Fe results in the accumulation of vacancies within the martensitic steel. As the vacancy concentration rises, these vacancies coalesce to form nanovoids. These nanoscale voids serve as short-circuit diffusion pathways for oxygen, facilitating the reaction between oxygen and alloying elements such as Fe, Cr, and Si. The types of oxides formed within the internal oxidation layer are primarily governed by the local oxygen partial pressure (P_{O_2}). From a thermodynamic perspective, the sequence of oxide formation with increasing

P_{O_2} follows the order of $Cr_2O_3 \rightarrow FeCr_2O_4 \rightarrow Fe_3O_4$.

During the initial stages of corrosion, the extremely low P_{O_2} within the martensitic steel limits oxygen diffusion, leading to the preferential oxidation of Cr to form Cr_2O_3. As the oxidation process progresses and oxygen continues to diffuse inward, P_{O_2} gradually increases, enabling the oxidation of Fe and facilitating the transformation of the initially

formed Cr_2O_3 into the spinel phase ($FeCr_2O_4$).

Beneath the spinel layer, an IOZ develops, a phenomenon attributable to the high concentrations of Cr and Si in SIMP steel. As previously described, oxygen diffuses into the steel matrix, where it reacts to form the spinel layer. However, the oxygen activity (a_o) drops sharply during this process, reaching a critical minimum at the spinel/matrix interface. Given that Cr and Si possess a higher affinity for oxygen than Fe, they undergo selective oxidation even under these low-oxygen conditions, thereby facilitating the formation of the IOZ. As a primary alloying element in SIMP steel, Cr plays a pivotal role in significantly enhancing the material' s corrosion resistance. The cation occupancy of Fe and Cr within the spinel lattice is not static but varies depending on the corrosive environment and film-forming conditions, which subsequently influences the microstructure and growth kinetics of the oxide scale. Adjustments to the Cr content lead to corresponding modifications in the crystalline structure and defect chemistry of the Fe–Cr spinel. As the Cr fraction within the Fe–Cr spinel increases, the lattice become more compact and structurally stable, exerting a more pronounced hindrance on the diffusion of Fe and Cr cations. This diffusion-suppression effect induced by Cr enrichment improves the protective performance of the oxide scale and retards its further thickening. It is noteworthy that the Si-rich oxides are often encapsulated by Cr-rich oxides. Correlation with the EDS mapping results suggests that the Cr-rich oxides predominantly consist of Cr_2O_3, while the Si-rich oxides correspond to SiO_2. These findings indicate that Si facilitates the selective internal oxidation in

SIMP steel: the initially precipitated SiO_2 serves as preferential nucleation sites for Cr_2O_3. Subsequently, the formation of a continuous and dense Cr_2O_3/SiO_2 composite layer effectively blocks the diffusion pathways for Fe and O within the oxide scale, thereby significantly inhibiting further oxidation.

4.2 Grain Size effects on Corrosion Resistance

According to previous studies, the welding process induces significant grain coarsening within the weld metal. The substantial thermal input during welding triggers an increase in grain size, resulting in a grain boundary density in the weld metal that is considerably lower than that of the base metal. This reduction in grain boundary density is the primary cause of the markedly higher corrosion rate observed in the weld metal. Fig. 3 presents the EBSD maps of the manual weld, automatic weld, and the base metal. Combined with the grain size statistics (Fig. 4), it is evident that the weld metal contains a higher

proportion of large-sized grains due to the thermal cycle. Consequently, the corrosion resistance of the base metal is superior to that of the weld zones. Experimental results demonstrate that exposure duration significantly influences the evolutionary pattern of oxide scales. Under identical corrosion conditions, the thickness variations among the oxide scales formed on the weld metal of SIMP steel joints—fabricated via different processes—and the base metal are negligible. All specimens develop a consistent triple-layer structure, comprising Fe_3O_4, $FeCr_2O_4$, and a mixture of Cr_2O_3 and SiO_2 from the surface to the interior[24-32]. This evolutionary pattern is in good agreement with previous reports on the corrosion behavior of

ferritic/martensitic (F/M) steels in liquid LBE. On the other hand, corrosion heterogeneity across different regions of the welded joint is highly pronounced.

Compared with the base metal, the weld metal exhibits significantly coarser grains. The presence of these large grains reduces the total grain-boundary area, which acts as diffusion pathways, thereby accelerating the leaching of alloying elements into the liquid LBE and impairing the overall corrosion resistance of the weld region. This phenomenon underscores the non-negligible impact of heat input during welding on the material' s corrosion resistance. The base metal, retaining its original fine-grained structure, promotes the formation of a more compact oxide scale and thus exhibits superior resistance to LBE corrosion. Consequently, the microstructural characteristics of the weld zone represent a primary factor driving the disparity in corrosion performance between the weld metal and the base metal.

4.3 Elements Effects on Corrosion Resistance

Table 1 presents the elemental composition of the various regions: the Cr content is 11.8 wt% in the base metal, 9.04 wt% in the automatic weld metal, and 8.99 wt% in the manual weld metal. Similarly, the Si content measures 1.5 wt% in the base metal, compared with 0.49 wt% and 0.5 wt% in the automatic weld metal and manual weld metal, respectively. These results indicate that the Cr and Si concentrations in both the automatic weld metal and manual weld metal are significantly lower than those in the SIMP steel base metal. Under high-temperature conditions, silicon facilitates the formation of a continuous SiO_2 film on the metal surface. This layer exhibits extremely

low oxygen permeability, effectively inhibiting the inward diffusion of oxygen atoms.

Furthermore, this SiO_2 layer acts synergistically with chromium to enhance the overall protective performance of the oxide scale[45-48]. Chromium serves as the critical alloying element that imparts "stainless" characteristics to steel, primarily through the formation of a passive layer. Upon reaching a specific concentration threshold within the alloy, chromium spontaneously forms a thin and dense Cr_2O_3 protective film on the surface[49].

The synergistic effect between chromium and silicon facilitates the formation of a dual-layer diffusion barrier, which significantly enhances the oxidation resistance of the material. Furthermore, the average thickness of the internal oxidation layers was determined by taking ten equidistant measurements from Fig.13a, 14a, and 15a. The results indicate thicknesses of 563 ± 23.5 nm for the base metal, 341 ± 19.8 nm for the manual weld, and 363 ± 17.3 nm for the automatic weld. These measurements further corroborate the role of Cr and Si elements in enhancing corrosion resistance. Integrating the statistical results from Fig. 7 to 10, it is evident that the higher concentration of Cr and Si in the base metal facilitates the rapid formation of a protective internal oxidation film. Consequently, the thickness of the inner oxide layer in the base metal exceeds that in the weld zones. Since the inner oxide layer effectively acts as a diffusion barrier for Fe cations, it retards the growth of the outer oxidation layer, resulting in a significantly thinner outer layer in the base metal compared to the weld metal.

5.Conclusions

Based on the aforementioned experimental results and analyses, the following conclusions can be drawn:

(1) Under identical corrosion conditions, the thickness variations of the oxide scales formed on the corresponding regions of SIMP steel welded joints across different processes are insignificant. All specimens developed a characteristic triple-layer structure, consisting of an outer oxide layer, an inner oxide layer, and an internally oxidized zone (IOZ) from the surface to the interior. The corrosion resistance of martensitic steel in liquid LBE is primarily attributed to the inner oxide layer. Further analysis of elemental distributions, EDX line scans, and diffraction patterns confirms that the outer oxide layer is predominantly composed of Fe_3O_4, the inner oxide layer mainly consists of $FeCr_2O_4$, and the IOZ is characterized by the presence of Cr_2O_3 and SiO_2.

(2) Different corrosion rates were observed across various regions of the SIMP steel welded joints. The grain size within the weld metal is significantly larger than that of the base metal, primarily because the thermal input during welding triggers the melting and subsequent recrystallization of the originally fine grains. The resultant grain coarsening reduces the grain boundary density, which shortens the effective diffusion pathways for alloying elements and accelerates their leaching into the liquid LBE. Furthermore, due to the compositional variations between the weld metal and the base metal, the base metal possesses higher concentrations of Cr and Si—elements critical for enhancing corrosion resistance. This elemental disparity further exacerbates the divergence in corrosion performance between the

weld metal and the base metal. Consequently, the overall corrosion resistance of the weld metal is inferior to that of the base metal.

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Conflict of interest

The authors declare no conflict of interest.

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