Local Structural Changes during Martensite Decomposition in Cr-Mo Steel Dissimilar Weldments

Extreme lattice parameter changes in the soft zone led to the presence of a ferrite band, indicating lower hardness

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ABSTRACT

The presence of a soft zone near the weld interface resulted from the martensite decomposition in the 2.25Cr-1Mo heat-affected zone (HAZ) of a Cr-Mo dissimilar joint during postweld heat treatment (PWHT). In this study, ER90S-B9 filler metal was deposited on 2.25Cr-1Mo (T22) base material and various PWHT durations were applied to investigate the changes in the local structures. Microstructural observations revealed the soft zone formation in the T22 HAZ close to the weld interface. During short PWHT durations, lattice parameters were slightly reduced. The T22 HAZ remained as a martensite structure. The transition period from martensite to ferrite was initiated after 2 h of PWHT. Extreme lattice parameter changes in the soft zone resulted from martensite decomposition and led to the presence of a ferrite band, indicating lower hardness. The decomposition of martensite to ferrite was completed after 4 h of PWHT. From the results, an attempt to apply the x-ray diffraction (XRD) technique was insufficient for differentiating the small changes in local structures. Therefore, the extended x-ray absorption fine structure (EXAFS) technique was introduced to give better analyzed data in terms of r-space.

KEYWORDS

- Soft Zone • Martensite Decomposition
- Postweld Heat Treatment (PWHT) • Local Structure
- X-Ray Diffraction (XRD) • Extended X-Ray Absorption Fine Structure (EXAFS) • R-Space

similar joints. Complex metallurgical structures can be observed at the weld interface (Refs. 4, 6, 7). Previous investigations have reported the presence of “soft” and “hard” zones along the weld interface during PWHT. The martensite structure in the heat-affected zone (HAZ) transferred to the ferrite phase resulting in low hardness (Refs. 1, 2, 7–16).

Many in the literature have studied the soft zone formation in dissimilar Cr-Mo weldments by observing metallographic information (Refs. 1, 2, 8, 10, 13–16). However, no investigations determined the microstructural transformation of this zone during PWHT in terms of the local structural changes. Studying the local structural changes of phases could be helpful for identifying the decomposition of martensite to ferrite. Changes in lattice parameters could be performed using x-ray diffraction (XRD), which provided considerable resolution due to its nature in phase-selective technique. For better resolution, extended x-ray absorption fine structure (EXAFS) could be conducted for its element-selective technique. The combination of both techniques could provide a complementary structure view of microstructural transformation (Refs. 17–24).

This paper investigates the local structure changes with different PWHT durations in the soft zone of a dissimilar joint between 2.25Cr-1Mo steel (ASTM SA213 grade T22) and ER90S-B9 filler metal (overmatching to T22 steel) by employing XRD and EXAFS methods. Results obtained from both techniques will be helpful as a baseline for further determination of the microstructural transition mechanism in Cr-Mo steel dissimilar joints.

Experimental Procedures

Materials and Welding Procedure

The material used as the base metal (BM) for the dissimilar weld preparation was 2.25Cr-1Mo steel seamless tube (ASTM SA213 grade T22) in the as-received condition with dimensions 50.8 mm (OD) × 2.7 mm (wall thickness) × 25.0 mm

Introduction

Dissimilar weld joints between different grades of low-alloy ferritic steels are essential for the fabrication and repair of piping or tubing in power plants, especially between low-Cr and high-Cr ferritic steels (Refs. 1–5). Postweld heat treatment (PWHT) must be employed on Cr-Mo steel dis-

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Fig. 1 — Welded specimen preparation: A — Side view; B — front view.

(length). The ER90S-B9 filler metal was fabricated on the cross-section circumferentially for two layers as the B9/T22 weld using the gas tungsten arc welding (GTAW) process, as shown in Fig. 1. The nominal and measured compositions of the base metal and the deposited weld metal (WM) are listed in Table 1. Details of the welding parameters used followed the ASME qualified Welding Procedure Specification (WPS) as shown in Table 2. After welding, the welded joint was cut for PWHT performed at 760°C (conformed to minimum PWHT required by the ASME code) for various time durations (as-welded, 0.5, 1, 2, 4, and 8 h), as shown in Fig. 1.

Metallurgical Preparation and Microhardness

Microstructural examination at the weld interface of the dissimilar joints, especially in the soft zone, was performed using optical microscopy (OM). The welded joints were polished and etched using 10 mL HNO₃ + 20 mL HCl + 30 mL water for 90 s to show the general structure and 5 g FeCl₃ + 5 drops HCl

Fig. 2 — Outline of the dissimilar weldment with region of interest.

+ 100 mL water for 10 s to show the prior austenite grain boundaries, according to the ASTM E407 standard (Ref. 27). The hardness of the base metal and all weldment subzones

Table 1 — Chemical Compositions of the Base Metal and the Deposited Weld Metal

Material	Composition	Elements (wt-%)							
		C	Mn	Si	Ni	Cr	Mo	V	Nb
2.25Cr-1Mo base metal	nominal ⁽¹⁾	0.05–0.15	0.30–0.60	0.50 max	—	1.90–2.60	0.87–1.13	—	—
	measured	⁽³⁾	0.40	0.31	—	2.50	1.12	—	—
ER90S-B9 weld metal	nominal ⁽²⁾	0.07–0.13	1.25	0.15–0.30	1.00	8.00–9.50	0.80–1.10	0.15–0.25	0.02–0.10
	measured ⁽⁴⁾	⁽³⁾	0.68	0.27	0.17	8.04	0.947	0.17	0.03

1. Refer to (Ref. 25)

2. Refer to (Ref. 26)

3. Not determined

4. Measured in the weld metal by SEM/EDS analysis.

Table 2 — Welding Parameters

Configuration	Layer no.	Filler Metal		Polarity	Current (A)	Voltage (V)	Travel Speed (cm/min)
		AWS Class.	Dia (mm)				
B9/T22	1	ER90S-B9	2.4	DCEN	60	11–12	13.3
	2	ER90S-B9	2.4	DCEN	60	11–12	11.6
Preheat Temp.: 200°C min		Interpass Temp.: 300°C max				PWHT Temp.: 760°C

Fig. 3 — OM micrographs at the B9/T22 weld interface after PWHT at 760°C for the following: A — As-welded; B — 1 h; C — 2 h; D — 4 h; E — 8 h. SZ refers to the soft zone, HZ the hard zone, HAZ the heat-affected zone, and WM the weld metal.

was measured using a Vickers microhardness tester with a load of 200 gf and a loading time of 10 s on points equidistant at 250 microns across the weld interface. Figure 2 presents a schematic of the region of interest in the weldment for microstructural observations and microhardness measurements.

XRD Measurement

The XRD analysis was performed on a Bruker D8 Discover diffractometer with Cu K α radiation ($\lambda = 1.54 \text{ \AA}$). This technique was carried out on the T22 HAZ adjacent to the weld interface with different PWHT durations (Fig. 2) to analyze the change in crystal structure of each sample in terms of lattice parameters, diffraction angle, and d-spacing.

EXAFS Technique

To address the oxidation states of the metal atoms and the local environment around these ions at different sample conditions, EXAFS spectra at the Fe K-edge were conducted at beamline 5.2 (XAS) (Refs. 28, 29) (electron energy of 1.2 GeV, bending magnet, beam current 80–150 mA, 1.1 to 1.7×10^{11} photon s $^{-1}$) at the Synchrotron Light Research Institute (SLRI), Nakhon Ratchasima, Thailand. All spectra were measured in the fluorescence mode with four-element silicon drift detector. For the acquisition of all spectra, a Ge (220) double crystal

monochromator with an energy resolution ($\Delta E/E$) of 2×10^{-4} was used to scan the synchrotron x-ray beam. Normalized XAS data were processed and analyzed after background subtraction in the pre-edge and post-edge region using ATHENA software included in an IFEFFIT package (Refs. 30, 31). This method was also conducted in the T22 HAZ where the soft zone occurred, as shown in Fig. 2.

Results and Discussion

Microstructural Evaluation and Microhardness

Figure 3A–E shows optical micrographs along the weld interface of the welded specimens with different PWHT durations. In the as-welded sample (Fig. 3A), the T22 HAZ con-

Fig. 4 — Microhardness profiles of the B9/T22 weldments with various PWHT durations. WM refers to the weld metal, CGHAZ the coarse-grain heat-affected zone, FGHAZ the fine-grain heat-affected zone, ICHAZ the intercritical heat-affected zone, and BM the base metal.

tained martensite (Ref. 3) resulting from phase transformation mechanism during welding (from austenite (γ) to martensite (α') structure). However, after PWHT for 1 h, a soft zone and hard zone occurred at the T22 HAZ and ER90S-B9 weld metal (WM), respectively, across the weld interface — Fig. 3B. The soft zone was composed of a ferrite band while the hard zone contained a precipitate-rich band (Refs. 1, 7, 8, 14). In the soft zone, partial martensite transformed to ferrite (α) structure during PWHT (Ref. 2). The specimen with short PWHT duration would only post an incomplete decomposition of martensite. Longer PWHT durations resulted in a broader soft zone as a ferrite band — Fig. 3C–E. Further decomposition of martensite occurred with the formation of carbide along the grain boundaries of ferrite (product of martensite decomposition) (Refs. 1, 2).

The microstructural examination was in agreement with the microhardness profiles across the weldments, as shown in Fig. 4. From the fusion boundary toward the base metal, the hardness of the T22 HAZ in the as-welded condition was about 340 $HV_{0.2}$ corresponding to the martensite structure. Nevertheless, after PWHT, the lowest hardness (around 130 $HV_{0.2}$) appeared in the T22 HAZ near the weld interface where the soft zone developed (Refs. 1, 2, 8). These results could confirm that the microstructure transformed from the martensite decomposition into ferrite grains. During PWHT, the martensite in the prior austenite grain began to decompose into ferrite structure with carbides at the grain boundaries (Refs. 32–34). As the PWHT times were increased, the larger ferrite grains were observed in the soft zones (Ref. 1).

XRD Analysis

The decomposition of martensite in the HAZ was determined by a change in the lattice parameters in terms of diffraction angle and the d-spacing of dissimilar welded samples with various PWHT times. The d-space or the interplanar distance (d) value was obtained from the XRD measurement based on Bragg Equation ($n\lambda = 2d\sin\theta$) (Refs. 35, 36). According to the equation, the larger diffraction angle, the

Fig. 5 — A — XRD patterns; B — the first diffraction peak of x-ray diffraction at 2θ for T22 HAZ (soft zone) of dissimilar joints with different PWHT durations.

lower d-spacing. Figure 5A shows an overview of the x-ray diffraction pattern in the T22 HAZ (adjacent to the weld interface) of B9/T22 weldments. Figure 5B exhibits the first diffraction peak of x-ray diffraction at 2θ of each welded specimen.

From Fig. 5B, after PWHT for 1 h, the diffraction angle was at 44.565 deg, which can be matched with those of the body-centered cubic (BCC) martensite structure from other research (Refs. 37–41) resulting from the carbon content of this steel lower than 0.6 wt-% (Refs. 42–46). Only martensite was found, although this area was a mixture of martensite and ferrite structure from the microstructural observation. This is due to the fact the magnitude of the ferrite band contained was too small to be detected. The XRD results indicated the diffraction peaks changed with increasing PWHT times. The diffraction peak of the 2-h PWHT specimen shifted considerably to the higher value ($2\theta = 44.638$ deg) compared to the 1-h PWHT sample in which the interplanar distance reduced from 2.03151 to 2.02836 Å as the

Fig. 6 — R-space in the soft zone of B9/T22 dissimilar joints with various PWHT durations.

BCC martensite transformed to BCC ferrite (Refs. 39, 41, 47, 48). With increasing PWHT time, carbon diffused from the iron lattice, which led to partial martensite decomposition in the T22 HAZ close to the weld interface (Ref. 41).

After PWHT for 4 h, the diffraction peak shifted slightly to the left side ($2\theta = 44.628^\circ$) compared to the 2-h PWHT sample and exhibited larger d-space ($d = 2.02881 \text{ \AA}$) resulting from increased martensite decomposition during PWHT and the presence of BCC ferrite phase in the T22 HAZ (soft zone). In addition, the diffraction peak of the 8-h PWHT sample shifted to the right side ($2\theta = 44.642^\circ$) compared to the 4-h PWHT specimen. The crystal structure remained BCC ferrite but with smaller d-space ($d = 2.02821 \text{ \AA}$). This might be due to the absence of interstitial carbon atoms in the iron lattice causing lower distortion of the BCC ferrite structure with longer PWHT durations (Refs. 49, 50).

However, it was difficult to distinguish between martensite and ferrite structures because the difference in lattice parameters (diffraction angle and interplanar distance) was very small (Refs. 39–41, 48, 51). Therefore, XAS measurement using the EXAFS technique was applied to compare data obtained from the XRD investigations.

EXAFS Results

Fourier transformed EXAFS of the Fe K-edge data (in fluorescence mode) was used to determine the change in lattice parameter in terms of the radial distance (interatomic distance; r-space) of the soft zone (T22 HAZ) resulting from the decomposition of martensite. Figure 6 presents EXAFS results of the soft zone in B9/T22 weldments with different PWHT durations.

The T22 HAZ close to the weld interface of the as-welded specimen was composed of a martensite structure. After PWHT for half an hour, the r-space shifted slightly to the left and with longer PWHT (1 h), the r-space also reduced from 2.0862139 to 2.0555343 Å. There was a little martensite decomposition in the T22 HAZ with short-time PWHT.

Nonetheless, when PWHT time was increased from 1 to 2 h, the peak shifted to the higher value from 2.0555343 to 2.1782527 Å, indicating a larger r-space as the BCC marten-

Fig. 7 — The sequence of microstructural transition (martensite → ferrite) in the soft zone of the B9/T22 weldment with different PWHT durations in terms of r-space.

Table 3 — Comparison of the XRD and EXAFS Results in T22 HAZ (soft zone) of Dissimilar Joints

Condition	2θ (XRD)	R-space (EXAFS)
As-welded	N/A	α'
PWHT 0.5 h	N/A	α' (smaller)
PWHT 1 h	α'	α' (smaller)
PWHT 2 h	$\alpha' + \alpha$	$\alpha' + \alpha$ (larger)
PWHT 4 h	α	α (larger)
PWHT 8 h	α	α (smaller)

} Transition Period

site structure transformed to BCC ferrite phase and the BCC ferrite phase became prominent in the soft zone. The microstructural transition resulted from the partial decomposition of martensite during PWHT. With longer PWHT (4 h), the r-space increased slightly compared to the 2-h PWHT. This was due to a complete decomposition of martensite to ferrite. However, the r-space of the 8-h PWHT sample with ferrite phase showed a small decrease (r-space = 2.1475731 Å) compared to the 4-h PWHT specimen, resulting from no carbon trapped in the α -Fe lattice during PWHT.

The EXAFS results presented here were consistent with those from XRD analysis. From Table 3, the T22 HAZ close to the weld interface of the as-welded sample was composed of BCC martensite as a result of phase transformation from austenite to martensite after welding. After short PWHT durations (0.5 and 1 h), the martensite still remained in the T22 HAZ, but with smaller r-space. A slight martensite decomposition occurred during the short PWHT times.

The change in lattice spacing from BCC martensite to BCC ferrite in the T22 HAZ occurred after PWHT for 2 h (transition period), with a large change in R-space resulting from partial martensite decomposition, which led to a mixed structure of martensite and ferrite; however, the ferrite could be distinguished. After PWHT for 4 h, the microstructural transformation was completed with larger R-space due to increased martensite decomposition. Moreover, the 8-h PWHT sample presented a smaller R-space due to lack of carbon occupied in the α -ferrite with the increased PWHT durations. Figure 7 summarizes the sequence of soft zone formation in the dissimilar joints during PWHT.

The results were conformed to microstructural observations in terms of the sequence of transformation, which was not exact in PWHT duration. According to the microstructure,

the ferrite band could be observed from 1-h PWHT. However, XRD and EXAFS analysis could still detect as martensite. This was due to the fact the beam size of EXAFS was larger than the size of the ferrite band presented. Therefore, the signals were acquired from both ferrite soft zone and decomposed martensite. For longer PWHT duration, the size of ferrite band was larger than the beam size. In this case, all the signals were acquired from the decomposed martensite.

Conclusions

Both XRD and EXAFS techniques were performed to investigate martensite decomposition at the weld interface of Cr-Mo dissimilar joints between T22 steel and ER90S-B9 filler metal using the GTAW process. Results were obtained as follows:

- 1) The formation of soft and hard zones was observed in the T22 HAZ and B9 WM adjacent to the weld interface during PWHT in Cr-Mo dissimilar joints.
- 2) The soft zone that occurred in the T22 HAZ was composed of a ferrite phase due to martensite decomposition indicating a lower hardness.
- 3) Both XRD and EXAFS results indicated the change in crystal structure from BCC martensite to BCC ferrite due to martensite decomposition in the soft zone began after PWHT for 2 h. Nonetheless, the usage of EXAFS technique gave a better resolution compared to XRD analysis.
- 4) The transition period of martensite to ferrite in the T22 HAZ near the weld interface (soft zone) occurred after 2-h PWHT, with martensite decomposition and microstructural transformation completed after 4-h PWHT.

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