**Proceedings of the ASME 2020 39th International Conference on Ocean, Offshore and Arctic Engineering
OMAE2020
June 28-July 3, 2020, Fort Lauderdale, FL, USA**

OMAE2020-18017

ON THE EFFECT OF VARIOUS HEAT TREATMENTS ON MICROSTRUCTURE OF AISI 4130 STEEL USED IN SOUR SERVICE PIPES

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ABSTRACT

Nowadays welding is the most common way to connect metal parts and structures. One of the challenges connected to welding it that heat output from the welding alters the microstructure of the metal creating the heat affected zone (HAZ) near the weld. In steel welds HAZ is often harder and more brittle than the base material due to formation of martensite. This might cause hydrogen induced cracking and speed up the fatigue of the weld. To mitigate the martensite formation in the HAZ different heat treatments, like preheat, interpass and PWHT are often applied. However, for 4130 steel, preheat and interpass temperatures are not expected to restrict martensite formation due to materials slow transformation rate. Preheat and interpass temperatures are still important for hydrogen diffusion and reduction of tension in the weld.

This paper investigates the effect of different heat treatments on the microstructure of AISI 4130 steel used in sour service pipes. The welding and sample preparation were performed in accordance with ISO 15156 and ASME B31.3 standards. Two sample sets were produced: one with and one without preheating. The hardness tests of weld profiles were performed in accordance with ISO 15156-2 international standard. Comparison of hardness profiles indicated that preheat had virtually no effect on hardness of the steel in HAZ, although it affected hardness of fusion zone. Preheated samples were further heat treated in a furnace simulating PWHT effect. Three different PWHT condition were tested. The hardness profiles indicated that PWHT led to noticeable changes in steel microstructure. In order to understand those microstructure changes, the heat treatment of the steel during production process was reviewed and microscopic investigations of the weld profiles were performed.

INTRODUCTION

Pipe considered in this paper is part of high-pressure drilling fluid system for deep water drilling. The risk of exposure to hydrogen sulfide containing environment classifies it as a sour service system. That introduces additional requirements to the material hardness which must be accounted for during manufacturing and welding.

Weld defects are one of the main causes of failure in piping systems. Failure of high-pressure drilling fluid system can put a hold on entire drilling operation. Hence, the design and construction of pressure piping in petroleum and chemical processing refineries are extensively regulated by ASME B31.3 code. The latest version of this code is from 2018 [1], while an earlier, 2016 version [2] was applied when the experiments reported in this paper were performed. However, both 2018 [1] and 2016 [2] version prescribe the same PWHT conditions.

Low alloy AISI 4130 steel is a popular material choice for high pressure sour service pipes since this steel displays high strength and is relatively inexpensive compared to stainless or dual phase steels. AISI 4130 grades that are used for high pressure services are mostly stress relieved martensitic steel i.e. their microstructure is mainly tempered martensite. As can be seen for isothermal transformation and continuous cooling diagrams available in for example [3], AISI 4130 forms martensite relatively easily. If not tempered, martensite is very brittle, and its presence can lead to a brittle failure of the material. When AISI 4130 steel is welded, the martensite might easily form in the heat affected zone (HAZ). That is why, ASME code for process piping [1] prescribes compulsory post weld heat treatment (PWHT) of pressure pipes for stress relieving and to temper the martensite ensuring satisfactory fracture toughness and acceptable hardness.

The ASME code provides a range of acceptable PWHT temperatures for a range of materials based on base metal grouping in ASME BPVC IX, see Table 331.1.1 in ASME B31.3 [1] or [2]. This paper aims to determine what would be the optimal PWHT condition for the supplied AISI 4130 within the allowable range.

MATERIAL

Samples are made of 2" (OD 66,33 mm.), Sch. XXS (wt 11,07 mm.), quenched and tempered seamless A519 – grade 4130 pipe. This is a low alloyed martensitic steel with carbon, manganese, chromium, and molybdenum as main alloying elements. The material certificate states that the supplied steel contains 0.310% carbon C, 0.240% silicon Si, 0.54% manganese Mn, 0.006% phosphorus P, 0.001% sulfur S, 0.93% chromium Cr and 0.17% molybdenum Mo by weight. Energy Dispersive X-Ray Spectroscopy (EDS) has been performed to verify that the chemical composition of the supplied material corresponds to its material certificate. EDS is not as accurate as chemical analysis and the result should be interpreted with caution. Table 1 shows the comparison between measured and stated of alloy elements; note that Table 1 gives no measured value for wt% of carbon C, since in EDS this value is likely to be inaccurate due to contamination of the samples or vacuum chamber with organic matter. Given the relative error of EDS analysis, it can be concluded that the AISI 4130 chemical composition corresponds to the material certificate.

	C	Si	Mo	Cr	Mn
AISI4130	wt%	wt%	wt%	wt%	wt%
Certificate	0.310	0.24	0.17	0.93	0.54
EDS and error	-	0,2 ±0.09	0.1 ±0.06	0.9 ±0.2	0.3 ±0.2

Table 1. AISI4130 material certificate vs. EDS measurements

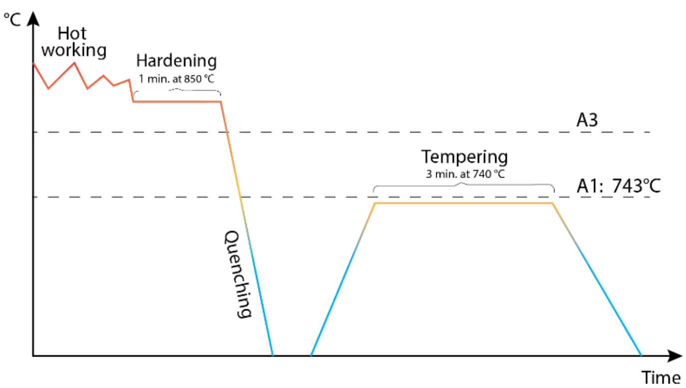


Figure 1. Heat treatment of supplied AISI4130 according to material certificate.

The material certificate states that the supplied steel has yield strength of 645 MPa, the tensile strength of 745 MPa and 32% elongation at fracture. The material certificate also describes the heat treatment as following: hardening at temperature 850°C with 1-minute holding time, cooling in water, tempering at temperature 740°C with 3 minutes holding time, cooling in air. The heat treatment during production is illustrated graphically in Figure 1. The indicated lower critical temperature A1 was calculated based on the chemical composition using the following empirical formula available in e.g. [4]

$$A_1(in\ ℃) = 727 - 10.7(\%Mn) - 16.9(\%Ni) + 16.9(\%Cr) + 29.1(\%Si) \qquad (1)$$

Discussion regarding accuracy of formula (1) and exact A1 temperature of AISI 4130 is outside the scope of this paper.

SAMPLE PREPARATION

Two 150 mm (5.90 inches) long sample pipes were used, Figure 2. Each sample was cut in two and joint together again using shielded metal arc welding (SMAW). Butt weld end preparation was done in accordance with ISO 9692-1 standard [5]: grove angle is 60°, root gap is 3 mm and root face is 2 mm. ASME B31.3 standard [2] dictates a mandatory preheating, thus one of the samples was preheated to temperature of 120° C prior to welding. The second pipe sample was not preheated, allowing to compare the samples and see how preheating effects the weld.

Figure 2. As-received pipe, welding preparation and sample cutting

Choice of the electrode was made in accordance with the strength and composition requirements. The filler material's trade name is OK 48.08 and the classification is E 46 5 NI B 32 H5. According to the supplied datasheet, the welding electrode has the following composition 0.06% carbon C, 0.4% silicon Si, 1.2% manganese Mn and 0.8% nickel Ni by weight. The typical mechanical properties of weld metals are yield strength of 540 MPa, tensile strength of 600 MPa and 26% elongation at fracture.

See Figure 3 for welding parameters and sequence. Infrared thermometer was used to make sure that interpass temperature stays inside following range: 150°C - 250 °C, as in accordance with Norsok M-601 [6].

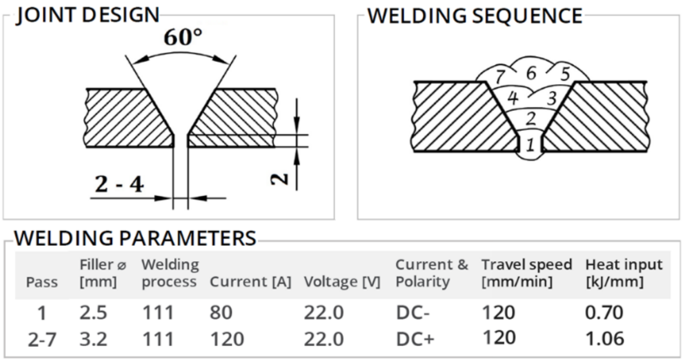


Figure 3. Welding parameters and sequence, welding position H-L045, J-L045, stringer bead technique

Sample	preheat	PWTH	Holding time
	° C	° C	minutes
1	na	na	na
2	na	na	na
3	na	na	na
4	na	na	na
1,1	120	na	na
2,1	120	650	20
3,1	120	650	120
4,1	120	705	20

Table 2. The list of hardness samples for testing

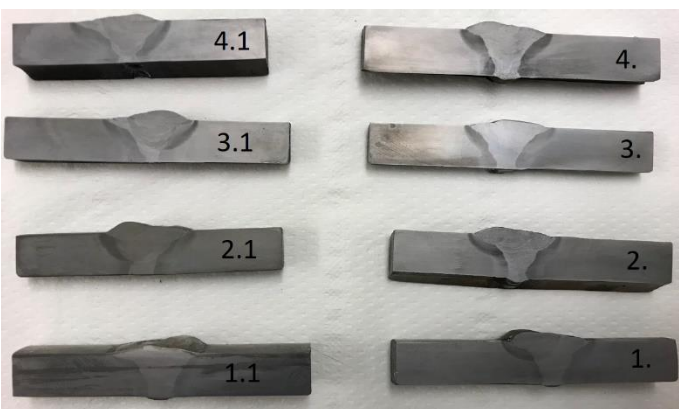
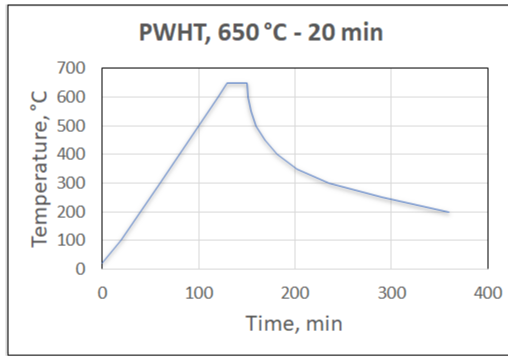


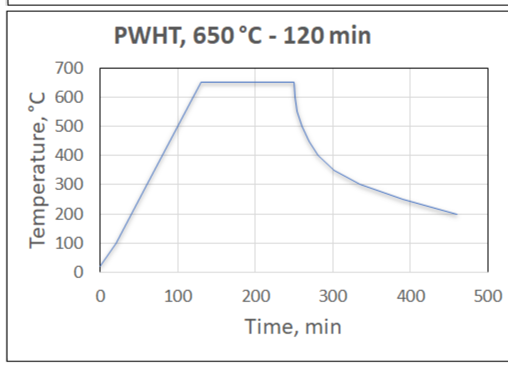
Figure 4. Etched samples for hardening testing

The hardness test samples were cut out using a band saw as illustrated in Figure 2. The samples were prepared in accordance with ISO 6507 standard [7]. Milling ensured samples surface flatness and parallelism, which was confirmed with digital leveling equipment to be less than 0,005mm. Polishing was done in following steps: P320, P800, P1200, P2400, ensuring roughness of less than 0,05 µm. For etching, nitric acid (Nital) was used.

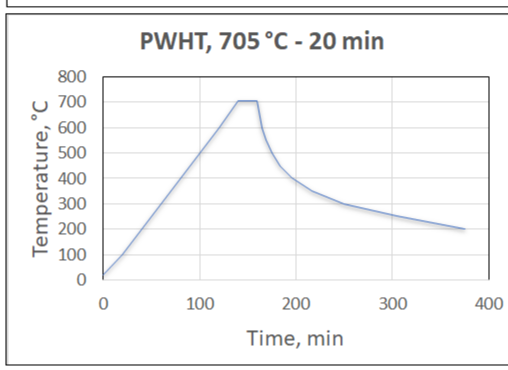
It total four hardness test samples were produced out of each welded pipe. Table 2 gives an overview of the samples. The samples that were subjected to preheat were also subjected to post weld heat treatment in the furnace. One sample was kept untreated for the reference, while three others were subjected to three different hear treatments, as indicated in Table 2. A programable box-type furnace was used. The heating rate was set to 250° C/hour, while the cooling was performed by simply turning off the furnace and letting the samples to cool with the furnace. Figure 5 illustrates the heat treatment profiles.



650 °C - 20 min	
temp. °C	time, min.
20	0
100	20
300	60
600	120
650	130
650	150
600	151
550	154
500	160
450	169
400	181
350	202
300	235
250	290
200	360



650 °C - 120 min	
temp. °C	time, min.
20	0
100	20
300	60
600	120
650	130
650	250
600	251
550	254
500	260
450	269
400	281
350	302
300	335
250	390
200	460



705 °C - 20 min	
temp. °C	time, min.
20	0
100	20
300	60
600	120
705	140
705	160
600	165
550	169
500	175
450	184
400	196
350	217
300	250
250	305
200	375

Figure 5. PWHT temperature profiles

TESTING EQUIPMENT

Vickers HV10 hardness tests of the welded samples were performed in accordance with international standard ISO 15156-2 [8]. Testing was done using ZHU250CL testing machine from Indentec. The picture of the hardness testing machine and the tested specimen is shown in Figure 6.

Microstructure of the samples was investigated using the scanning electron microscope (SEM) and energy dispersive spectroscopy equipment, Jeol JSM-7200F. The JSM-7200F incorporates multiple types of electrode detectors. For this analysis, pictures were taken using the lower electron detector (LED) with 10 kV acceleration voltage.

Figure 6. Hardness testing and a sample with indentation marks

RESULTS HARDNESS MEASTURMENTS

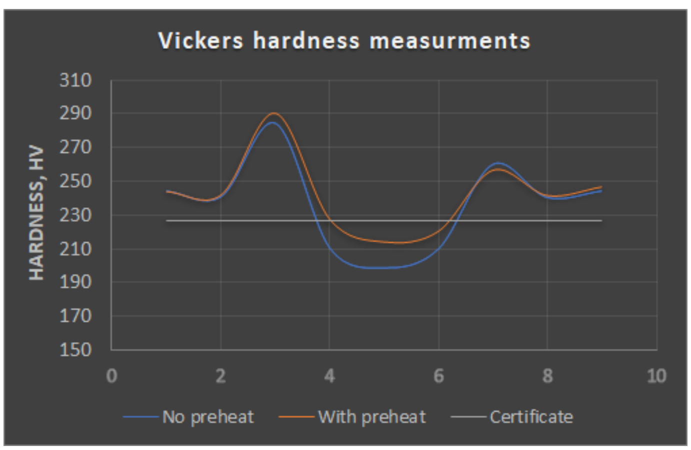


Figure 7. Effect of preheat on the hardness, average over all samples

Figure 7 illustrates comparison of average hardness for preheated and not preheated samples. A grey line in Figure 7 indicates the base material hardness stated in the material certificate. As can be seen from the figure the hardness is nearly identical in base material and HAZ, while some difference in hardness is observed for the fill material. No observable difference in hardness profile indicates that preheat has no significant effect on microstructure in HAZ at given condition. Note that preheat is usually required of other reasons, such as mitigation of residual stresses in the root string due to material contraction during cooling.

Table 3 illustrates the comparison of hardness values before and after PWHT while Figure 8 gives a graphical representation of the hardness profile for the sample 4,1. The maximum allowable hardness value after PWHT is HV 250 according to ISO 15156-2 [8]. As can be seen from Table 3, PWTH at 650°C for 20 minutes does not allow to achieve the desired reduction of hardness in HAZ. After PWTH at 650°C for 120 minutes, the desired hardness is achieved nearly everywhere except one point, which is 253 HV. That is just 3 HV or 0,3 HRC above the limit, which is considered acceptable in accordance with permitted individual HRC reading exceeding of 2 HRC [8]. The heat treatment to 705°C for 20 minutes causes more severe hardness reduction; not only it causes hardness of HAZ to drop significantly below the level of base metal, but the hardness of base metal itself appears to be reduced, which might indicate weakening of the material. This hardness drop can be seen in Figure 8.

Sample 2.1 Preheat 120 ° C PWHT 650 °C for 20 min									
Before PWHT					After PWHT				
Base	HAZ	Weld	HAZ	Base	Base	HAZ	Weld	HAZ	Base
244	336	229 234	274	246	245	271	239 238	273	249
	349		273			277		242	
245	294	218	253	245	246	245	213	234	246
	230		235			229		221	
240	232	220 210	226	231	244	231	212 212	229	227
Sample 3.1 Preheat 120 ° C PWHT 650 °C for 120 min									
Before PWHT					After PWHT				
Base	HAZ	Weld	HAZ	Base	Base	HAZ	Weld	HAZ	Base
247	324	222 211	290	244	243	249	215 206	253	245
	333		274			245		250	
243	281	203	267	247	243	220	187	226	243
	270		234			226		222	
235	227	223 219	227	244	238	212	210 216	227	243
Sample 4.1 Preheat 120 ° C PWHT 705 °C for 20 min									
Before PWHT					After PWHT				
Base	HAZ	Weld	HAZ	Base	Base	HAZ	Weld	HAZ	Base
250	345	234 213	270	245	230	235	216 202	248	236
	345		287			230		213	
244	307	201	271	248	232	217	184	216	232
	245		230			209		208	
236	233	238 237	237	240	226	202	194 195	209	231

Table 3. Hardness measurements before and after PWHT

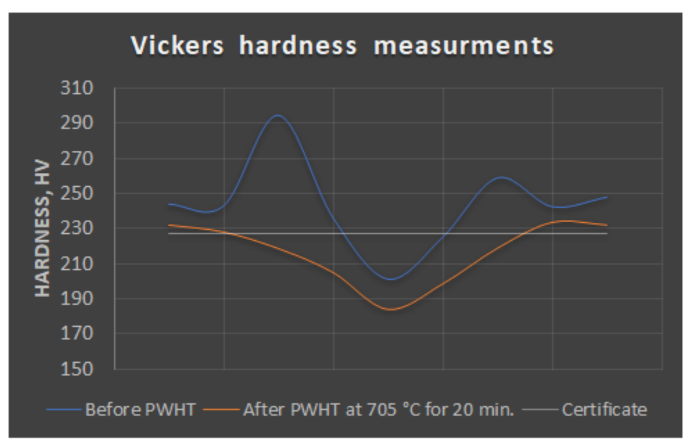


Figure 8. Hardness profiles for Sample 4,1

MICROSTRUCTURE

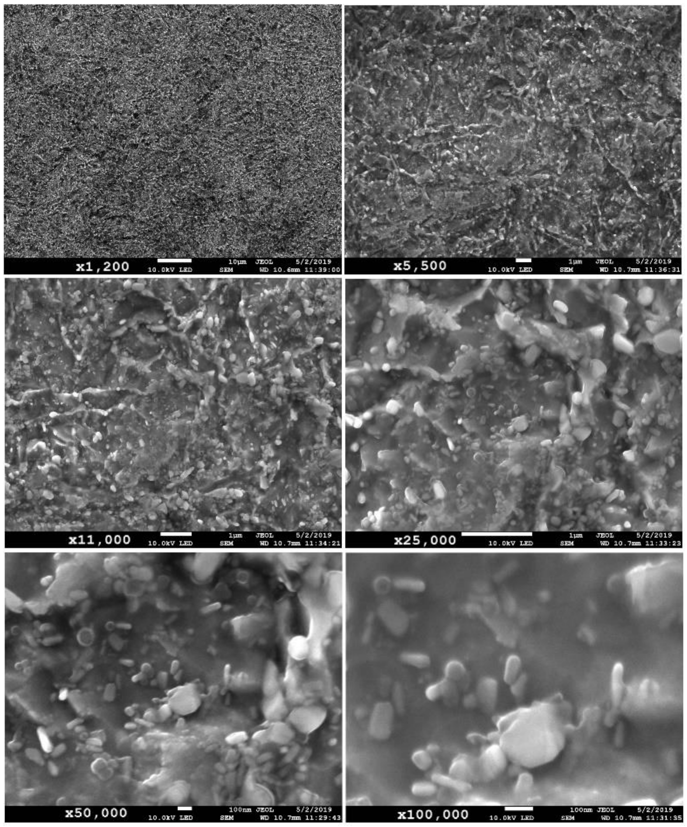


Figure 9. SEM images of AISI 4130 steel, Sample 3,1 base material

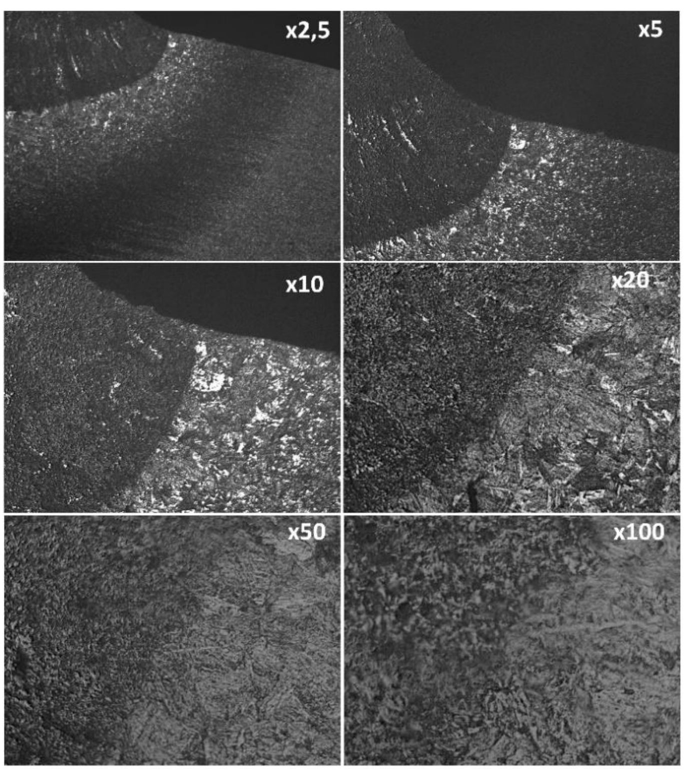


Figure 10. Optical microscope images of fusion line in Sample 3,1

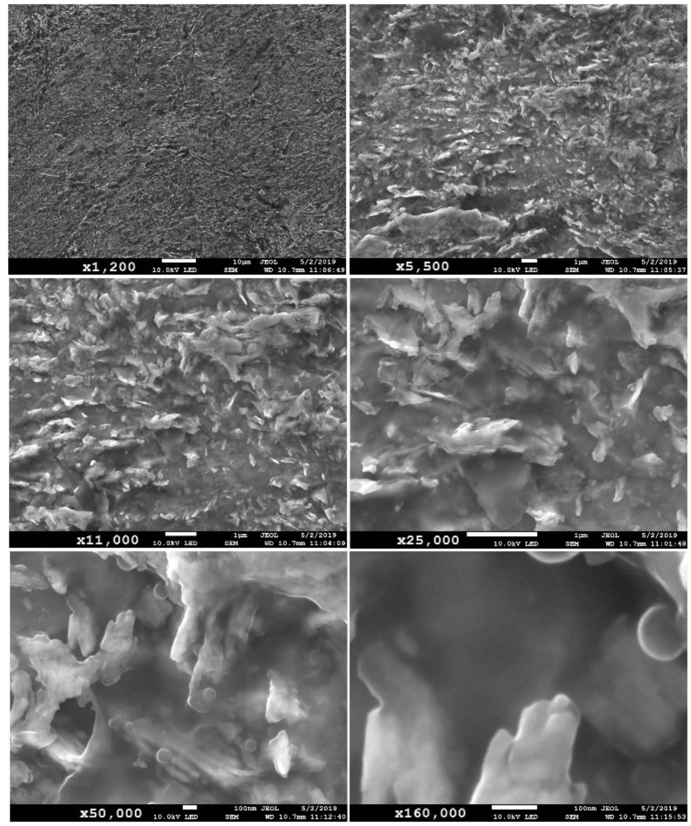


Figure 11. SEM images of Sample 3,1 HAZ before PWHT

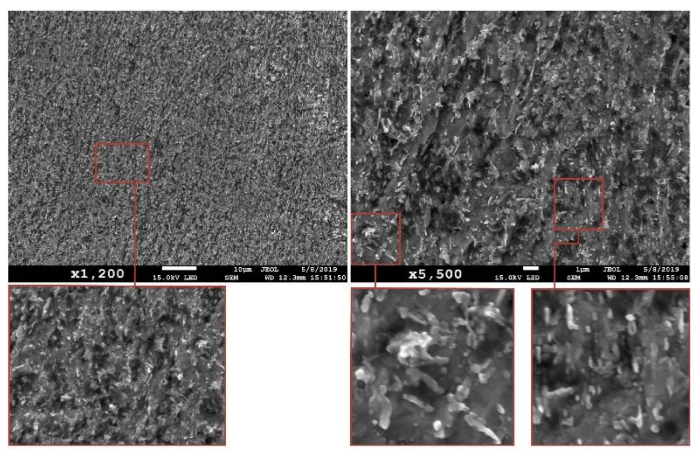


Figure 12. SEM images of Sample 3,1 HAZ after PWHT.

Based on heat treatment during manufacturing, the tested AISI 4130 steel is expected to consist mainly of tempered martensite. The base material microstructure is shown in Figure 9, which indicates that the microstructure is mainly tempered martensite with inclusions of carbides.

The highest percentage of untempered martensite is expected to form close to the fusion line of the last weld pass, where HAZ is not tempered by other welding passes. After etching, the fusion line and HAZ regions becomes visible. Figure

10 illustrates optical metallograph images of fusion line at the last welding pass in Sample 3, while Figure 11 provides high resolution SEM images of HAZ of the same pass. Here we can see needle like structures typical for untempered martensite. Figure 12 illustrates same HAZ area after PWHT at 650 °C for 120 minutes, showing the results of the tempering.

DISCUSSIONS AND CONCLUSIONS

The aim of the presented study was to suggest the optimal PWHT conditions for quenched and tempered AISI 4130 steel pipes. The prescribed PWHT temperature range for these pipes has been reduced from 704-746°C [9] down to 650-705°C [1][2]. Thus, there has been a tendency to reduce the maximum allowable PWTH temperatures, increasing the safety margin between PWHT and A1 temperatures. The experimental result in this paper fully support this tendency. It was found that the PWHT at higher temperatures leads to undesirably high hardness reduction not only in HAZ but also in the base metall. Since hardness is correlated to the material strength, reduction of hardness in base metal implies weakening of the material.

PWHT at 650°C and 705°C were tested, which are the lowest and the highest allowable PWHT temperatures respectively [2]. The tests showed that tempering of martensite was effectively achieved with PWHT at 650°C. The longer holding time gives greater-reduction of hardness in HAZ, while the base material stays unaffected. The PWHT at 705°C leads to extensive loss of hardness at 20 minutes soaking time, which is short, considering that the minimum holding time for PWHT is 15 min [1].

We can hereby conclude that it is beneficial to heat treat the investigated AISI 4130 pipes at the lowest allowable PWHT temperature of 650°C, which allows for even heat distribution in the material and keeps the temperature safely below lower critical temperature A1. Two hour holding time seems to be ideal. It does not over soften the HAZ and does not seem to have any impact on the base material. This holding time qualifies for range of thicknesses up to 50 mm [1]. Use of higher PWHT temperatures is not recommended as it might reduce the strength of the welded connection and thus compromise the quality of the weld.

ACNOWLEGMENTS

This article is based on the experimental findings of the second author's master thesis performed under the supervision of the first author at the University of Agder, Norway [10]. The authors would like to express their deep gratitude to the university personnel who assisted with the experimental work. In particular, the authors would like to thank chief engineer Cecilie Ødegård who trained the second author in operating the Vickers hardness testing machine and heat treatment furnaces, senior engineer Naureen Akhtar responsible for the SEM-microscope, who performed the microscopic investigation and PhD-fellow Gerrit Ralf Surup who assisted with microscopic investigation and EDS analysis. The authors would also like to thank the MHWirth for provided resources and support during the master thesis project.

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