RESEARCH PAPER

Effect of tempering time on the mechanical properties of P91 flux cored wire weld metal

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Abstract ASME Grade 91 is one of the main materials for heavy-wall pipes in thermal power plants. Matching flux cored wires for welding P91 have already been available for several years and become more and more popular as flux cored arc welding (FCAW) offers several technical and economic advantages. To achieve high toughness of the weld metal at ambient temperature, temperature and/or time of post-weld heat treatment (PWHT) can be increased. As the temperature has to be kept below the transformation temperature A_{c1} , the range of suitable temperature is very small. In this contribution, the effect of increased tempering time on the mechanical properties at ambient temperature and elevated temperatures is investigated and the influence on long-term properties is discussed. Longer PWHT increases toughness of all-weld metal. MatCalc simulations and scanning electron microscopy (SEM) investigations indicate a coarsening effect of precipitates without detrimental effect on long-term creep properties.

Keywords (IIW Thesaurus) Creep resisting materials . Cored filler wire . GMAwelding . Mechanical properties

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1 Introduction

Flux Cored Wires for welding P91 have already been available for several years. A rutile slag system provides good outof-position weldability and high productivity. However, heat input and layer sequence have to be set accurately to meet the requirements of the most common specifications [[1](#page-8-0)]. Originally, the chemical composition of all-weld metal was based on the standards AWS A5.29 and EN ISO 17634. Ni content was set comparatively high within the requirements Ni \leq 1.0 wt% and Mn+Ni \leq 1.5 wt% to increase toughness at ambient temperature.

1.1 Influence of Mn+Ni content on A_{c1} temperature

Figure [1](#page-1-0) shows the influence of Mn+Ni content on measured A_{c1} temperature of P91 flux cored wire weld metal according to ASTM A1033-10. These measurements correspond with published investigations that the Mn+Ni content has a very strong effect on A_{c1} temperature in P91 steel grade [\[2](#page-8-0)].

With Mn+Ni, slightly below 1.5 wt% following the requirements of AWS A5.29 the A_{c1} temperature is 770 °C. However, the risk of exceeding A_{c1} could not completely be eliminated during post-weld heat treatment (PWHT) at 760 ± 15 °C according to AWS A5.29. In this case, fresh martensite is formed after PWHT, which is detrimental for toughness. A PWHT temperature above A_{c1} either causes the formation of fresh martensite or soft ferrite, depending on the Mn+Ni content [[3\]](#page-8-0).

A slight reduction of Mn+Ni to 1.3 wt% increased A_{c1} to almost 780 °C, and further restriction of Mn+Ni to a value of 1.0 wt% ensures that the PWHT temperature is clearly below A_{c1} and no transformation to austenite leading to fresh martensite occurs.

Fig. 1 Influence of Mn+Ni content on A_{c1} temperature

Although the maximum for Mn+Ni in AWS A5.36/ A5.36M:2012 is limited to 1.4 wt%, an additional limitation to 1.0 wt% found its way into customer requirements and necessitated the modification of the flux cored wire.

1.2 Influence of Ni on mechanical properties

Ni was found to be detrimental for long-term creep properties in P91 base material. A remarkable drop in creep rupture strength in long-term tests was attributed to a higher Ni content promoting coarsening of precipitates and nucleation of Zphase by consuming MX particles [\[4](#page-8-0)]. Therefore, the Mn content was kept constant and the Ni content was reduced to fulfil the requirement of Mn+Ni \leq 1.0 wt%.

Previous investigations showed that the reduction of Ni had no significant influence on yield strength and tensile strength, neither at ambient temperature nor at elevated temperature [[5\]](#page-8-0). However, it caused a drop in impact energy at ambient temperature of about 10 J. For PWHT of 760 °C/4 h, for example, the average value decreased from 61 to 54 J.

A classification of the flux cored wire as E91T1-M21PY-B91 according to AWS A5.36 requires 27 J at ambient temperature. This can be fulfilled without any difficulty even with lower Ni content. However, EN ISO 17634-A requires 47 J in all-weld metal, which is challenging.

1.3 Influence of PWHT on mechanical properties

The mechanical properties of all-weld metal, especially impact energy at ambient temperature, are strongly depending on tempering temperature and time. With increase of PWHT, temperature and duration hardness and tensile strength decrease and ductility and impact toughness increase [[6](#page-8-0)]. For tungsten inert gas (TIG) welding, this effect is only observed up to 4 h [\[7\]](#page-8-0).

Figure 2 shows the influence of PWHT on mechanical properties of flux cored wire all-weld metal acc. EN 15792-1 welded with shielding gas $Ar + 18\%$ CO₂.

Therefore, impact energy can either be increased by increasing temperature and/or time of PWHT. As Mn+Ni ≤1.0 wt% leads to A_{c1} of about 800 °C (see Fig. 1), it would be possible to increase the PWHT temperature to 770 or even 780 °C without the risk of exceeding A_{c1} . However, PWHT temperature is often limited by the tempering temperature of the base material. So, increasing the time is the remaining possibility to increase impact energy at ambient temperature.

In this contribution, the effect of tempering time on mechanical properties at ambient temperatures and at elevated temperatures is investigated.

2 Experimental procedure

All-weld metal tests according to EN ISO 15792-1 (see Fig. [3](#page-2-0)) were performed with a P91 flux cored wire. Table [1](#page-2-0) shows the nominal chemical composition of all-weld metal with shielding gas Ar + 18% $CO₂$ $CO₂$ $CO₂$. Welding parameters are given in Table 2.

To investigate the influence of tempering time on mechanical properties, impact tests at ambient temperature according to EN ISO 9016, tensile tests at ambient temperature according to EN ISO 6892-1 and hot tensile tests at 500 and 600 °C according to EN ISO 6892-2 were performed after PWHT at

Fig. 2 Influence of PWHT on mechanical properties of all-weld metal at ambient temperature

Fig. 3 Setup for all-weld metal samples acc. EN ISO 15792-1

Table 1 Nominal chemical composition of all-weld metal (typical values in wt%)

			C Mn Si Cr Mo Ni V Nb N	
			0.10 0.7 0.2 9.0 1.0 0.2 0.2 0.04 0.04	

Table 2 Welding parameters for all-weld metal samples acc. EN ISO 15792-1

Position	PA		
Shielding gas	$Ar + 18\%$ CO ₂ , 16 $1/min$		
Wire feed rate	13 m/min		
Voltage	28 V		
Current	$250 - 270$ A		
Preheating	200 °C		
Interpass temperature	260 °C		
Welding speed	30 cm/min		
Variation of PWHT	760 °C/1 h 760 °C/4 h 760 °C/12 h		

760 °C for 1, 4 and 12 h respectively. The position of samples is shown in Fig. 4.

Additionally, light optical microscopy (LOM), scanning electron microscopy (SEM), HV10 hardness measurements in the cap layer and an HV1 hardness mapping in the centre of the weldment were performed in as-welded condition and after PWHT.

The thermokinetic software package MatCalc™ was used for calculating the evolution of precipitates in allweld metal with a chemical composition according to Table 1 during multipass welding, PWHT and service at 600 °C. To simulate the temperature effect of multipass welding, a temperature history with additional peak temperatures of 1300, 1100 and 750 °C respectively; an interpass temperature of 260 °C and a $t_{8/5}$ of 20 s for every cycle was applied.

3 Results and discussion

3.1 Effect of tempering time on impact energy

Figure [5](#page-3-0) shows the results of impact tests of all-weld metal at ambient temperature. A considerable increase of impact energy can be observed with increased time.

After tempering of only 1 h, the mean value of 30 J meets the requirement of 27 J according to AWS A5.36. However, to meet the requirement of 47 J according to EN ISO 17634-A, a longer time at 760 °C is necessary. With PWHT at 760 \degree C/4 h, more than 50 J could be achieved, and with increasing PWHT to 760 \degree C/12 h, impact energy could be raised to 70 J.

To meet the requirements of the common standard, a PWHT of 760 °C/4 h is recommended. Elongated PWHT of 12 h describes the effect of multiple PWHT or tempering of thicker components.

3.2 Effect of tempering time on tensile properties

Figures [6](#page-3-0), [7,](#page-3-0) and [8](#page-3-0) compare the results of tensile tests at ambient temperature and hot tensile tests at 500 and 600 °C after PWHT at 760 °C for 4 and 12 h respectively.

Yield strength and tensile strength slightly decreased at ambient temperature as well as at elevated temperatures with longer time. However, elongation increased.

Fig. 4 Position of samples (*left*: impact samples; *right*: tensile samples)

Fig. 5 Impact energy of all-weld metal

3.3 Effect of tempering time on hardness and microstructure

Figure 9 shows the microstructure in as-welded condition where the layer sequence can be clearly seen. The position of the hardness measurement in the cap layer is marked red, and the position of the hardness mapping in the centre of the weld metal is marked green.

The results of hardness measurements in the cap layer in as-welded condition and after PWHT at 760 °C for 1, 4 and 12 h respectively, are listed in Table 3. Hardness decreases considerably from almost 400 HV10 in aswelded condition to 236 HV10 after PWHT at 760 °C

Fig. 8 Tensile properties at 600 °C

Fig. 9 Microstructure and position of hardness measurements

for 1 h. Longer tempering times cause a further moderate decrease to 220 HV10 after 4 h and 210 HV10 after 12 h respectively.

Table [4](#page-4-0) shows the results of the HV1 hardness mapping in the centre of the weld metal. Hardness varies in as-welded condition from 317 to 471 HV1. After PWHT at 760 °C, it decreases to 209-275 HV1 after 1 h, 188–255 HV1 after 4 h and 173–243 HV1 after 12 h respectively.

A sequence of areas with lower and higher hardness, which represents reheated areas and pure weld metal, can be identified especially in as-welded condition. Hardness decreases in reheated areas from about 400–470 to about

Table 3 Hardness (HV10) in the cap layer in as-welded condition and after PWHT

				As-welded $760 \text{ °C}/1 \text{ h}$ $760 \text{ °C}/4 \text{ h}$ $760 \text{ °C}/12 \text{ h}$
HV ₁₀ (mean value)	394	236	220	210
Deviation	4.6	3.2	2.7	2.3

Table 4 Hardness (HV1) in the centre in as-welded condition and after PWHT

320 to 400 HV1. After PWHT, this effect declines and the differences in hardness are lower. The highest measured value is 275 HV1 after tempering for 1 h, decreasing to 255 HV1 after 4 h and 243 HV1 after 12 h.

Fig. 10 LOM (left) and SEM (right) in as-welded condition

Fig. 11 LOM (left) and SEM (right) after PWHT 760 °C/1 h

Figures 10, 11, 12, and [13](#page-6-0) show the microstructure in LOM and SEM in as-welded condition and after PWHT. All samples reveal martensitic microstructure. SEM

investigations do not show any precipitates in aswelded condition. After PWHT, Cr carbides are visible in the tempered martensite at grain boundaries and lath

Fig. 12 LOM (left) and SEM (right) after PWHT 760 °C/4 h

Fig. 13 LOM (left) and SEM (right) after PWHT 760 °C/12 h

boundaries. The position of EDX measurement is marked in Fig. 13. With increased time, the precipitates coarsen up to about 500 nm.

Coarsening of precipitates resulting in a decrease of number density is also described for shielded metal arc welding (SMAW) with PWHT at 760 $^{\circ}$ C for up to 100 h [\[8\]](#page-8-0).

Fig. 14 Evolution of precipitates with PWHT at 1 (dashed line) and 4 h (solid line)

3.4 MatCalc simulations

The evolution of precipitates during welding, PWHT at 760 °C for 1, 4 and 12 h respectively, and service at 600 °C for 100,000 h has been calculated. Figure [14](#page-6-0) shows the evolution of temperature (first row), phase fraction in wt% (second row), mean radius in nanometre (third row) and number density of the main precipitates in per cubic metre (fourth row) up to 100,000 h with PWHT at 760 °C for 1 (dashed lines) and 4 h (solid lines).

There is no significant effect on the long-term stability of the precipitates between a PWHT of 4 and of 1 h at 760 °C. M_7C_3 are slightly longer and stable with shorter PWHT. However, they dissolve after approximately 2000 h. MX particles precipitate a little bit earlier and with a slightly higher number density with a PWHT of 4 h. However, after 100,000 h, number density and mean radius are almost equal. NbC, which precipitate in austenite, are slightly longer and stable with shorter PWHT. However, they vanish after several hundred hours when VN precipitates in the martensite.

Figure 15 compares the results of 4 (solid lines) and 12 h (dashed lines) in the same way.

Increasing PWHT at 760 °C to 12 h accelerates the transformation of M_7C_3 carbides to M_2C_6 . VN are slightly coarser with reduced number density. However, they converge with longer running times.

SEM investigations revealed a coarsening of precipitates after extended PWHT. This corresponds to the MatCalc simulations. However, these effects seem to diminish with longer running times. Therefore, more detailed investigations and experimental validation are necessary to investigate the influence on long-term properties of all-weld metal.

For base material P91, tempering at 770 °C for up to 10 h resulted in coarsening of precipitates, reduced number density and lower creep strength at short testing times but showed similar values after 60,000 h [\[9\]](#page-8-0).

4 Summary

The Ni content of the P91 flux cored wire was reduced to meet the requirements of Mn+Ni ≤ 1.0 wt%, which caused a

Fig. 15 Evolution of precipitates with PWHT at 4 (solid line) and 12 h (dashed line)

reduction of impact energy from about 60 to 50 J, with a PWHT of 760 °C/4 h.

With PWHT at 760 °C, the tempering time shows a strong influence on impact energy at ambient temperature.

In order to increase toughness, time of PWHT can be increased. However, yield strength and tensile strength and hardness slightly decrease.

Simulation of microstructural evolution indicates a coarsening effect of tempering time on precipitates. However, after longer running times, mean radius and number density of precipitates converge again. This corresponds to investigations of P91 base material creep rupture strength tempered at 770 \degree C for up to 10 h.

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