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Microstructural and Mechanical Characterization of 9Cr-1Mo-1W Weld Metal

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ABSTRACT

This paper presents microstructural and mechanical characterization of E911 weld metal. The types and transformations of the phases and microstructures of all-weld metal have been investigated through scanning electron microscopy, optical microscope, x-ray diffraction analysis. The elemental analysis and mechanical tests of the weld metal was determined by x-ray fluorescence, hardness measurements, and tensile tests respectively. This study will contribute the understanding of microstructural progression, microstructure characterization and mechanical properties of E911 steel after the post-weld heat treatment (PWHT).

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INTRODUCTION

A number of studies have been conducted to reduce the use of fossil fuels and gas emissions in power generation industry (thermal and nuclear power plant) [1]. In recent years new material technology has emerged depending on this quest [2]. Cr-Mo steels are widely used at thermal power plant constructions as piping, combustion chamber, and tubing. They are known as heat resistant materials due to high creep strength, as well as their low thermal expansion and high thermal conductivity [3]. They have significantly reduced the thickness of structures thanks to its high strength; it has significantly reduced the thickness of the structures, which in turn reduces weight and the cost of production. However, the lifetimes of these steels are reduced owing to the excess oxidation that occurs when they are exposed to high temperatures for a long time [4]. Therefore, during the few decades, Cr-Mo steels have been developed by alloying with elements such as W, Ni, Nb, V, Ti as a result of extensive studies carried out in some countries with the participation of various project partners In Europe, E911 steel which includes 9% Cr, 1% Mo and 1-2% W was developed by European Creep Collaborative Committee (ECCC) [5]. E911 steel has martensitic microstructure under air cooling after normalizing. Therefore it is used as tempered condition following normalizing.

The work of Wang H. et al. examined microstructural changes and properties of 9Cr-1Mo metals treated at high temperature for long periods and after post-weld heat treatment [6]. Barnes A. and Abson D. investigate possible increase toughness in weld metals W-including. Consequently, they observed that toughness of W-including weld deposit superior to the W-free weld deposits [7]. In the literature, there are a lot of works on P91 and P92 steels and their weld metal [8-16]. However, there are limited studies on characterization of microstructure and mechanical properties of E911 steel and its weld metal [17-18]. Therefore, this paper aims to presents microstructural and mechanical characterization of E911 weld metal.

EXPERIMENTAL PROCEDURE

In this study, all weld metal was produced by stick electrodes (SMAW technic). Stick electrodes were fabricated in Gedik Welding Company in Turkey. The chemical composition of E911 weld metal was determined by Rigaku ZSX Primus-II XRD devi-

ce and it can be seen in Table 1. The base metal size is 20x170x300 mm and welding parameters are given in Table 2. The schematic view of welding producing E911 weld metal shown in Fig. 1. Low carbon steel was also used to support as a backing plate (made of steel) during all welding. Heat input during welding was calculated according to Equation1.

$$\text{Current} * \text{Volt} * 60 * \text{Factor} / \text{cm/min.} / 10.000 = \text{kJ/mm} \quad (1)$$

The all weld metal was heat treated at 760 °C for 4 hours for post weld heat treatment (PWHT) as illustrated in Fig. 2 to AWS 5.5. The metallographic specimen was ground to 2000 mesh and polished with 3 µm diamond paste to observe micro structure of E911 weld metal. Then specimen was etched using 2.5 gr picric acid, 2.5 ml HCl, 100 ml ethanol (Picral) [19]. Nikon MA 100 model optical and Zeiss Evo/LS10 model scanning electron microscopes (SEM) with EDX were used for microstructure analysis. SEM was also used for fracture surface analysis. Thermal analysis (DSC) was carried out to determine critical transformation temperatures (A_1, A_3, M_f, M_s, and T_{curie}).

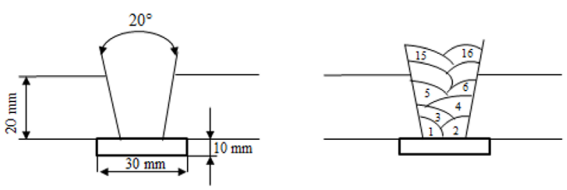

Figure 1. Schematic view of producing E911 weld metal

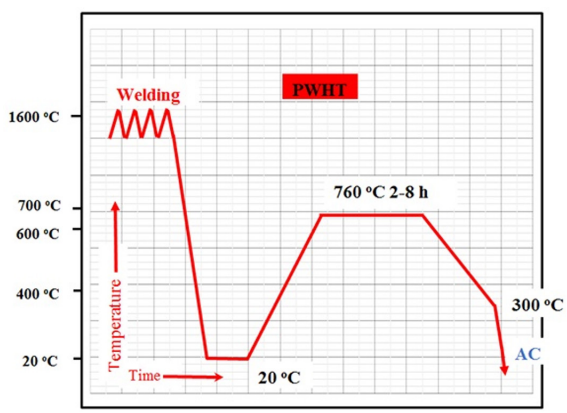

Figure 2. Schematic view of the post weld heat treatment (PWHT) process

Brinell and micro Vickers hardness tests were used to measure hardness of the weld metal by Bulut Digirock-Rbov and Microbul-1000 D models hardness tester devices respectively. Tensile test was performed at room temperature by Instron 8801 model with 100 kN tester device. Tensile test samples were taken from weld metal to AWS 5.5 specification as showed in Fig. 3 and the samples were prepared according to the ASTM E8/8M-08 standard [20].

Table 2. Welding parameters.

Diameter of covered electrode (mm)	*3.20*
Current (A)	*135*
Arc voltage (V)	*33.5*
Welding speed (mm/min)	*125*
Factor Value for SMAW (EN 1011-1)	*0.8*
Welding position (EN 6947)	*PA*
Heat input (kJ/mm)	*1.78*
Pass pieces	*25*
Preheat and inter pass temperature (°C)	*150 , 200*

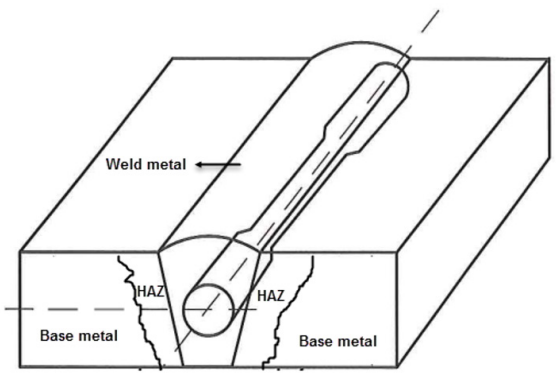

Figure 3. Schematic view of tensile test samples taken from all weld metal

RESULTS AND DISCUSSION

Microstructure Characterization

In this study it was observed that the microstructure of E-911 steel consisted of tempered martensitic with columnar structure. In the literature; it was reported that typical martensitic structure was observed in the form of columnar [21]. In SEM examinations, $Cr_{23}C$, Mo_2C, W_2C and VC were detected inside grain and grain boundary. L. Cipolla et. al. reported that E911 base and weld metal consist of similarity carbides [22].

In the microstructure examination, delta ferrite phase was observed as seen in Fig. 4. Higher amounts of ferrite-forming elements in the structure lead to an increase in chromium equivalent. This situation encourages the formation of delta ferrite in the microstructure.

Chemical composition of E911 weld metal was given

Table 1. Chemical composition of 9Cr-1Mo-1W steel (wt. (%)).

Element	*Si*	*Mn*	*Ni*	*Cr*	*S*	*Mo*	*Co*	*V*	*W*	*Fe*	*C*
	0.22	*0.53*	*0.43*	*8.74*	*0.005*	*0.96*	*0.01*	*0.20*	*0.98*	*Balanced*	*0.07*

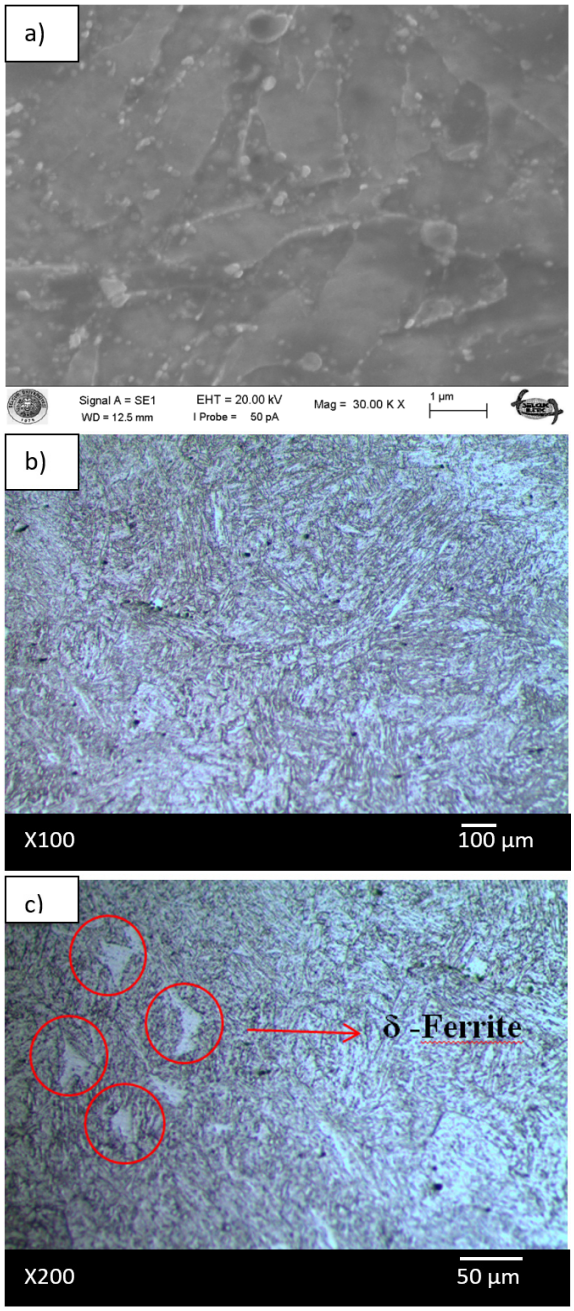

Figure 4. Microstructure of E911 weld metal (a) SEM analysis, low (b) and, high (c) magnification optical microscopy

Table 2 and Creq was calculated as 11.97 (wt %) to Equation 2. When Creq is higher than 9.0 (wt %) delta ferrite can form, which is detrimental effect for toughness and creep strength [23].

$$Creq = (\%Cr) + 6(\%Si) + 4(\%Mo) + 11(\%V) + 5(\%Nb) + 1.5(\%W) + 8(\%Ti) + 12(\%Al) - 4(\%Ni) - 2(\%Co) - 2(\%Mn) - (\%Cu) - 40(\%C) - 30(\%N) \quad (2)$$

Mapping analyses were shown in Fig. 5. Also, line analyze shown in Fig. 6 (b) can be seen as green arrow in Fig. 6 (a). As it can be seen the figures, $Cr_{23}C_6$, Mo_2C, W_2C and VC carbides precipitated along the tempered martensitic lath structure and inside of the grains. In the line analyze shown in Fig. 6 as green arrow, white precipitates can be identified as $M_{23}C_6$ carbides including Fe and Cr (Table 3).

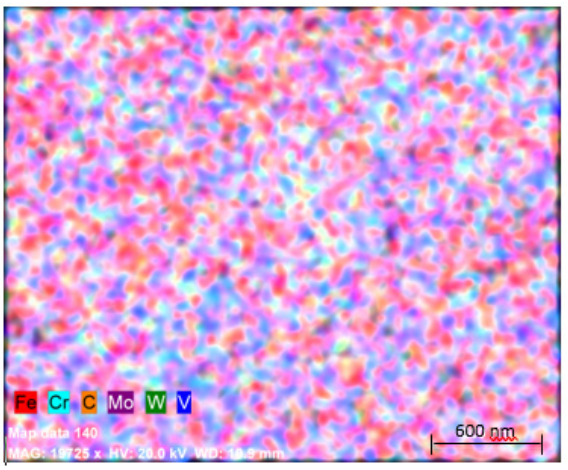

Figure 5. Mapping analyses of E911 weld metal

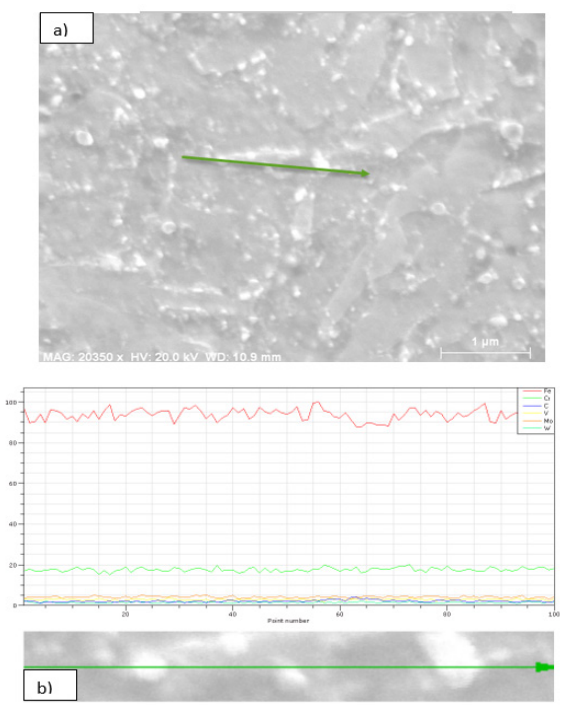

Figure 6. Line analyze from the weld metal (a) line direction (b) high magnification for line

Table 3. Element analysis of the line in Figure 5(b).

Element	*Norm. C (wt %)*
Carbon	*1.40*
Vanadium	*0.29*
Molybdenum	*0.42*
Tungsten	*2.91*
Chromium	*8.92*
Iron	*85.06*
Total	*100.00*

Thermal analyzing (DSC)

According to examined of thermal analysis, in the heating A_{c1}, A_{c3} and Tcurie temperatures are 830, 890, 750 °C respectively and in the cooling Mf temperature is 650 °C, Ms temperature is 690 °C. G. Cumino et. al. in their study evaluated the transformation temperatures A_{c1} and A_{c3} 841, 948 °C respectively [24]. Fig. 7 showed that transformation temperature.

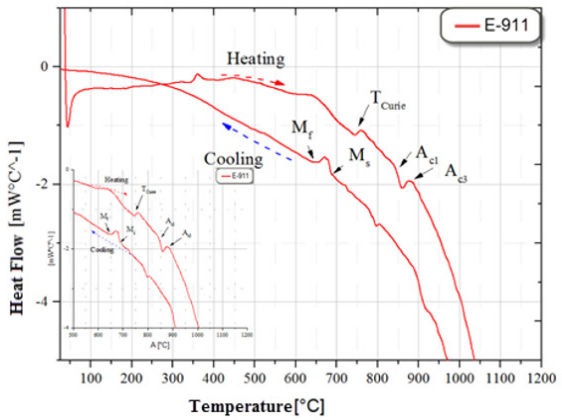

Figure 7. DSC curve of E911 weld metal (20 °C/min- 40mg)

When X-ray analysis was examined, carbides (Cr_7C_3, VC, and $Cr_{23}C_6$) were detected in the microstructure as shown in Fig. 8.

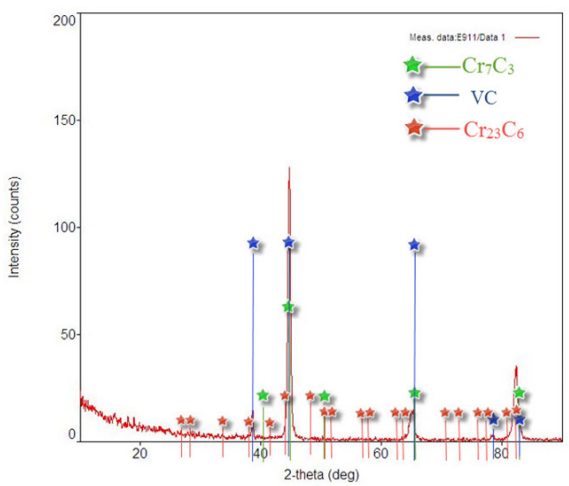

Figure 8. X-ray diffraction analysis of E911 weld metal

Mechanical Properties

The Brinell hardness test was carried out at room temperature and under a load of 187.5 kg. The average Brinell hardness is 230 HB. The micro hardness tests were applied for 20 seconds under 2 kg load. The average micro hardness of δ-ferrite zone in microstructure was determined as 165 HV (~ 164 HB).

Yield strength, tensile strength and elongation were determined as 550 MPa, 712 MPa, and % 18 respectively according to tensile test results at room temperature. Experimental values are reported in Table 4. The studies in the literature [22, 24] and ASTM standard [25] were examined; it was observed that similar mechanical properties were obtained.

Table 4. All-weld metal test results.

Test Temp. °C	*Heat Treatment Temp. °C*	*Yield Str. N/mm2*	*Tensile Str. N/mm2*	*Elongation %*
+20	*760 °C 4 h*	*550*	*712*	*18*

The fracture surfaces of E911 weld metal were given as low and high magnification. In the low magnification as shown in Fig. 9 (a), typical cup and cone fracture zone was observed. In the high magnification dimples can be seen in Fig. 9 (b). Both of the fracture surfaces show E911 weld metal produced in Gedik Welding Company, ductile fracture occurred during tensile test.

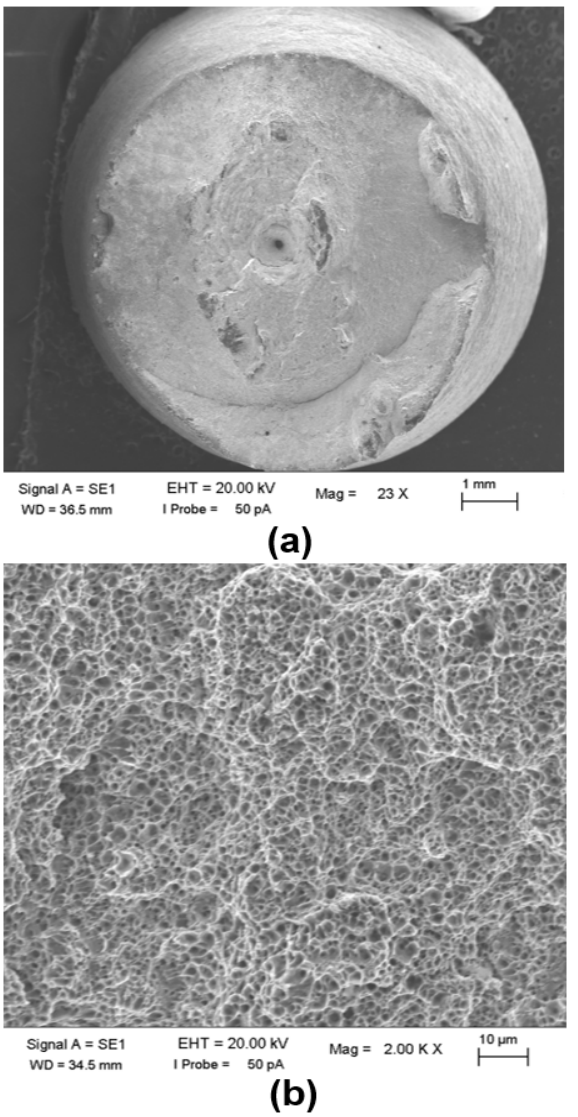

Figure 9. Low (a) - and high (b) magnification SEM fractography of fractured tensile specimens of E911 weld metal

CONCLUSION

In this study, E911 all weld metal was produced with stick electrodes (SMAW). The all weld metal was characterized as microstructure and mechanical properties. Conclusions extracted from the study are listed as below:

a- Microstructure: tempered martensitic was observed into lath boundaries, prior austenite grain boundaries, and delta ferrite phases were observed. Laves phases were not observed.

b- DSC analyses showed that A1 transition temperature is 830°C for the weld metal.

c- After PWHT, VC, $Cr_{23}C_6$ and Cr_7C_3 precipitates were detected by XRD analyse.

d- Mechanical properties of the weld metal are acceptable level according to specifications.

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