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# Influence of Coincidence Site Lattice Boundary on Creep Resistance of P91 Steel Weldments

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# Abstract

The grain boundary structure is usually described by the coincidence site lattice (CSL) model based on the misorientation of adjoining crystals. Therefore, the objective of the present investigation is to seek the correlation between CSL fraction and creep resistance of modified 9Cr–1Mo steel (P91) with and without boron addition. Results showed that CSL fraction increases with increase in heat treatment temperature and this increase is more prominent in boron containing modified 9Cr–1Mo steel. Creep test results show the increase in creep rupture life with increase in CSL fraction for both the base metals; but this increase is more in boron containing steel than the boron free steel. This improvement is attributed to the stability of CSL boundaries in the material. In spite of the increase in CSL boundaries with normalizing heat treatment temperature, boron free material shows less creep rupture life in its weldment than the boron containing steel weldment.

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Keywords:

# 1. Introduction

9Cr based steels are well known for their superior thermo-physical properties, high temperature mechanical strength, weldability and oxidation resistance and hence are used extensively in fossil power plants operating in the temperature range of 500–600°C. Industrial significance and contemporary development of these steels are also reviewed by Ellis [1] and Francis et al.[2] Microstructure of these steels in normalized and tempered (N&T) condition consists of tempered lath martensite where precipitates decorate different boundaries. Prior austenite grain consists of packet, block and lath boundaries. To achieve good creep resistance, the stability of these

boundaries is required. The stability depends on boundary energy and it should be as low as possible so that the kinetics of recovery remains slow. In this regard, importance of low-energy  $\Sigma$  3 boundary in engineering materials is noteworthy in improving the surface and mechanical properties.[3–5] In lath martensitic steel, this boundary is predicted by the classical transformation models of Kurdjumove–Sach (K–S) and Nishiyama–Wassermann (N–W). Increase in CSL boundary fraction that can be achieved through the incorporation of thermo mechanical and annealing processes is phenomenal in materials with FCC crystal structure as compared to those with BCC structure.[6–9]

Recently, it has been observed that the addition of 100 ppm boron with controlled amount of nitrogen in modified 9Cr–1Mo steel (P91) increased its creep rupture life.[10] The increase is more for steel subjected to normalizing at high temperature. Interesting observations were made when the creep tests were carried out for the weldments, which were prepared from different heat treated base materials. The increase in creep rupture life for boron containing P91 (P91B) cross weld specimen was observed which is in line with that observed for its base metal but creep rupture life decreased for P91 weldment with decrease in stress. Such an improvement and decreased in creep rupture life for P91B and P91 weldments respectively, is correlated with the stability and instability of microstructure in the ICHAZ of respective weldments.[10] For ease in discussion, hereafter, boron free modified 9Cr–1Mo steel is designated as P91 and boron containing steel is designated as P91B in this paper.

Increase in prior austenite grain size with increase in normalizing temperature is well reported. Creep strength of coarse grained material is higher (normalized at high temperature) than the fine grained (normalized at low temperature) single phase materials like stainless steel. [11] However, unlike in austenitic stainless steels, correlation between mechanical properties and prior austenite grains size is not straightforward in martensitic steel. There are attempts to correlate the yield strength with effective grain size of material in martensitic class steels, as the microstructure of this steel consists of different boundaries like packet, block and lath as stated earlier. Precipitates are observed on these boundaries which provide strengthening as well as stress concentration. Cavities are reported at the triple points of creep tested specimens. Therefore, one would expect that the spacing between these boundaries, rather than the prior austenite grain size, would be the most important microstructural parameter in this class of material.

In addition to this, base metal microstructure undergoes refinement in the heat affected zone (HAZ) of the weldment causing variations in prior austenite grain size and these microstructures are termed as coarse grained (CG), fine grained (FG) and inter critical (IC) heat affected zone (HAZ) based on peak temperature experienced. Influence of these microstructural variations on the performance of weld joint is well reported. Creep strength of ICHAZ is lower than that of the base metal and weld metal. This is due to the formation of microstructure that is inherently softer than the rest of the weld joint during welding and subsequent post weld heat treatment (PWHT). Therefore, improvement in creep strength of ICHAZ is of paramount significance. Boron addition improves creep strength of ICHAZ. Hence, the objective of the present works is study of (i) influence of normalizing temperature on boundary character distribution and on creep rate and, (ii) effect of prior microstructure on creep deformation behavior of weldment in particularly creep behavior of ICHAZ.

## 2. Experimental Procedure

MMAW

P91 and P91B steels were normalized in a muffle furnace at 1050, 1100 and 1150°C for 1h followed by tempering at 760°C for 3h. In order to understand the effect of prior austenite grain size on the performance of weld joints, respective heat treated materials were used for preparation of weld joints. Manual Metal Arc Welding (MMAW) is employed for welding and, welding parameters and heat input used for welding are given in Table 1. All weld joints were subjected to post weld heat treatment (PWHT) at 760°C for 3h.

Welding process	Current (A)	Voltage (V)	Heat input (kJ/mm)

100

Table 1: Welding parameters used for welding	Table 1: Welding parameters used for w	elding
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25

1.0

For microstructural characterization the base metals and weld joints, specimens were extracted from the respective materials and were metallographically polished till 0.25  $\mu$ m in diamond slurry. Polished specimens were etched using Villella's reagent and the etched specimens were observed under optical and scanning electron (SE) microscopes. For electron back scattered diffraction (EBSD) studies, specimens were mechanically polished at a low rotation speed of 100 rpm during each stage to minimize the mechanical deformation. Subsequently, they were electrochemically polished at 10 V for 10–15 s in a solution of 20 ml of perchloric acid and 80 ml of methanol maintained at sub–zero temperature. EBSD measurements of the specimens were conducted using SEM equipped with an EBSD–system operating at an accelerating voltage of 40 kV with the sample tilted at 70° with respect to horizontal axis. For EBSD analysis step size of 0.5  $\mu$ m was used. Two structure files of bcc ( $\alpha$ -Fe) and fcc ( $\gamma$ -Fe, austenite) were used to generate EBSD results.

# 3. Results and Discussion

#### 3.1 Chemistry and Microstructure

Chemical composition (in wt.%) of P91 steel is C: 0.1, Cr: 9.5, Mo: 1.0, Si: 0.48, Mn: 0.39, V: 0.25, Nb: 0.1, S: 0.009, P: 0.021, Ni: 0.14, Al: 0.024, N: 0.0065, Fe: bal.), and P91B is C: 0.1, Cr: 8.5, Mo: 1.04, Si: 0.4, Mn: 0.3, V: 0.09, S: 0.002, P: 0.005, Ni: 0.02, Al: 0.03 Ti: 0.03, N: 0.0021, B: 0.01, Fe: bal.). A comparison of chemical composition between both the steels shows that B containing material has very low nitrogen. Though nitrogen content is specified in the range of 500 to 650 ppm in P91 grade steel, it was reduced intentionally in P91B steel to avoid the formation boron nitride. Boron nitride is reported to deteriorate mechanical properties in high nitrogen containing 9Cr based steel.[12]

It is commonly known that the prior austenite grain size increases with increase in normalizing temperature and the rate of increase in grain size depends on the presence of second phase in the material.[13] Variation in prior austenite grain size with normalizing temperature is shown in Figure 1. From the plot, it is evident that the prior austenite grain size for P91B steel is higher than P91 steel and a significant increase in grain size occur at normalizing temperature of 1150°C. As nitrogen content is low in P91B steel, the volume fraction of MX types of precipitates is less in P91B steel than in P91 steel. To verify this, the phase evolution in both the steels is studied using MetCalc software and results are shown in Figure 2 and 3, respectively. Thermodynamic database fe– data6.tdb was used to determine the effect of boron in the steel. The Y–axis represents the phase fraction in logarithmic scale from 0.001% to 100%. Figures also show volume fraction of different probable precipitates formed at different temperatures. From these two figures, it is clearly evident that volume fraction of MX type of precipitates that are stable at higher temperature is less in P91B steel. This in turn brings down the pinning resistance for grain boundaries and is the reason for observation of bigger prior austenite grain in P91B steel than in P91 steel, heat treated at the same temperature.

## 3.2 Grain Boundary Character Distribution

Grain boundary character distribution (GBCD) is closely related to the prior austenite grain size of the material.[14] Inverse pole figure (IPF) maps obtained for both P91 & P91B steels (normalized at  $1150^{\circ}$ C / 1h and tempered at  $760^{\circ}$ ) / 3h) are shown in Figure 4(a,b) and it is evident that the block size is larger in P91B than in P91. Similar observation was made in other heat treated conditions (1050 and 1100°C). A block consists of laths with similar orientation. Therefore, region of same color on the IPF map indicate similar orientation, defined as a block. CSL boundary fraction estimated for different N&T steels are given in Figure 5.



Figure 1 Variations in prior austenite grain size with normalizing temperature



Figure 2 Phase fraction diagram for P91 steel calculated using thermodynamic database fe\_steel.tdb

In the present study, grain boundaries with  $\Sigma \leq 29$  were classified as CSL boundaries due to their lower energy, the others being classified as random boundaries due to high energy. From this figure, it is seen that the fraction of CSL boundary is higher in P91B steel as compared to P91. Grain boundary misorientaion analysis shows number fraction of HAGBs increases with increase in normalizing temperature / prior austenite grain size in P91B. On the other hand increase in LAGBs with increase in prior austenite grain size / normalizing temperature is observed in P91 and details are given in a separate paper. [15] Lath boundary is low angle boundary whereas block, packet and prior austenite grain boundaries are high angle boundaries. At higher normalizing temperature, MX type of precipitates dissolve and these alloying elements goes into the solution which in turn increases tetragonality. Maki et al. [16] have shown that the character of lath martensite changes with the carbon content, and Greninger and Troiano [17] have observed a change in the morphological appearance of plate martensite in Fe–C alloys. Detailed study is required to understand these variations in CSL fraction evolution in these steels as the composition of both steels are similar except for boron and nitrogen.



Figure 3: Phase fraction diagram for P91B steel calculated using thermodynamic database fe-data6.tdb



Figure 4 IPF maps (a) P91 and (b) P91B steels normalized and tempered at 1150°C/1h and 760°C/3h, respectively

# 3.3 Creep Deformation

Typical creep curves of P91 and P91B steels (normalized at 1050°C and tempered at 760°C3h) tested at 600°C and 100MPa are shown in Figure 6. It shows improvement in creep resistance for boron containing steel compared to boron free steel. Variation of minimum creep rate with CSL fraction is plotted for P91B steel and this is shown in Figure 7, which clearly reveals decrease in creep rate with increase in CSL fraction and increase in creep life (Figure 7 and 8). CSL boundaries are low energy boundary and these boundaries are related to transformation

induced twin. Increase in CSL fraction with increase in normalizing temperature and concurrent increase in creep lives for P91 steel is less and it has not been shown here. Improvement in creep rupture life for P91B weld joint compared to that of the P91 weldment was also observed. CSL fraction estimation was carried out across the weldments prior to the creep test and found that it remains similar to that in base metal in P91B specimen but marginal decrease in CSL fraction was observed in ICHAZ of P91 weldment. In order to understand CSL evolution in the post creep tested specimens, further studies are in progress. Bright field TEM images (Figure 9(a) and (b)) of inter critically simulated specimens (875°C/5min) shows presence of lath martensite in P91B steel and substructure in P91 steel and it is reason for lower creep rupture life for P91 steel. Table 2 shows summaries of creep test results of base metal, weldment and simulated heat affected zone (heat treated at 875°C for 5 minutes). From the table it is clearly evident that boron addition and normalizing heat treatment at higher temperature improved creep resistance of P91 steel and its weldment significantly. This is attributed to CSL fraction. Yardley et al.[14] also reported increase in CSL fraction for 10–11Cr based ferritic steel with addition of W and Co.



Figure 5 Variation of CSL fraction with prior austenite grain





Figure 8: Creep curves of P91B steel normalized at 1050, 1100 and 1150°C tested at 650C and 100 MPa stress.

Table 2: Rupture lives (h) of P91 and P91B base metal, weldment and its simulated heat affected zone; creep tests was done at 600°C.

Stress		P91		P91B		
	Base Metal			Base metal		
	1050°C	1100°C	1150°C	1050°C	1100°C	1150°C
80	18784		22000	10080#		
100	8618		15278	12960#		
120	2733		8912	17280		9440#
	Weld joint			Weld joint		
80	10859		6499	12960		13000#
100	49113		4211	11059		13100#
120	1004		2665	2817		7641
	ICHAZ			ICHAZ		
80	5635	236				
100	147	1350		10311		
120	425	5173		3414		

# Tests are in progress; specimens not yet ruptured



Figure 9 Bright field TEM image of (a) P91 and (b) P91B specimen after  $875^{\circ}C / 5$  min heat treatment and simulated PWHT at  $760^{\circ}C / 3$  h; base metal was normalized at  $1150^{\circ}C$  for 1h followed by tempering at  $760^{\circ}C$  for 3h.

# 4. Conclusion

From the above results and discussion following conclusions can be drawn:

- (i) Fraction of CSL boundary is more in P91B steel than P91 steel.
- (ii) Volume fraction of MX type of precipitates is less in P91B steel than P91 steel.
- (iii) Fraction of CSL boundary remains similar to that of base metal in the P91B weldment but it decreases marginally in the ICHAZ of P91 weldment.
- (iv) Creep rupture life of P91 and P91B base metals increased with increase in CSL fraction but increase in creep rupture life of P91B is significant compared to P91 weldment.
- (v) Creep rupture life of P91 steel is lower than P91B steel

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