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Effect of Boron on Creep Behaviour of Inter-critically Annealed Modified 9Cr-1Mo Steel

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Abstract

Two different heats of modified 9Cr-1Mo steel, one without boron (P91) and the other with controlled addition of boron (P91B) and very low nitrogen were used for the present study. Microstructure of P91 steel, annealed at 875°C (inter-critical region) consisted of very fine prior austenite grains without lath martensite. Grain size increased with increase in heat treatment temperature and showed coarse prior austenite grains with clearly defined lath after 900°C heat treatment. In contrast, the microstructures of P91B steel subjected to identical heat treatment did not vary much with heat treatment temperature, prior austenite grain size remaining similar to that observed in the as received material. Significant improvement in the creep properties of P91B steel compared to that of P91 steel subjected to inter-critical temperature is explained based on the difference in microstructures observed in these two steels after the heat treatment.

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Keywords: Modified 9Cr-1Mo steel; boron; inter-critical annealing; sub-structure

1. Introduction

Creep rupture life of weldment of creep resistant ferritic steels is lower than that of the base metal from which the weldment is made. Lower rupture life of the weldment is due to presence of soft zone in the inter-critical heat affected zone (ICHAZ) of weldment which is sandwiched between base metal and CGHAZ and thus acts as a metallurgical notch. Das et al.[1] reported improvement in creep rupture life of boron added modified 9Cr-1Mo steel weldment (P91B). Improvement in rupture life is attributed to significantly different microstructure observed in the ICHAZ of this steel from those observed in the ICHAZ of other ferritic steels. In the ICHAZ of this steel clearly defined lath martensite was reported; in contrast sub-grain or sub-structure, without a clear lath structure is reported in the ICHAZ of P91 steel without boron. In order to understand the effect of microstructrual differences on creep rupture life of their respective weldment, creep test of inter-critically heat treated P91 and P91B steels were carried out.

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2. Experimental procedures

Two different heats of modified 9Cr-1Mo steel, one without boron, designated as P91 (chemical composition (wt.%): 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 the other with controlled addition of boron with very low nitrogen, designated as P91B (chemical composition (wt.%): 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.) were used for the present study. In order to know the transformation temperatures, Differential Scanning Calorimetry (DSC) study was carried. Based on this, specimens prepared from as- received material, which is in the normalized and tempered (N&T) condition, were heat treated for five minutes at various temperature in the range of 850 to 1175°C to simulate the different HAZ microstructures in weldments of these steels. Heat treatment carried out at 875°C corresponds to inter-critical annealing. Subsequently, these heat treated specimens were subjected to simulated PWHT at 760°C for 3h. Specimens after these heat treatments were subjected to microstructural examination as well as hardness measurements. Creep specimens with 6.0 mm gauge diameter and 50.0 mm gauge length were machined out from the specimens subjected to inter-critical annealing and creep tests were carried out at 600°C for two stress levels of 100 and 120 MPa.

3. Results

3.1. Microstructure and phase transformation

Microstructure of both steels consists of tempered lath martensite, where lath, packet and prior austenite grain boundaries are decorated with precipitates. Precipitates size are smaller in P91B than P91 steel. Results of DSC studies carried out at a heating rate of 3°C / minute indicate transformation temperatures are higher for P91B than for P91. Effect of boron on transformation temperature is shown in Figure 1. Continuous oscillation on the DSC output after A_{c3} transformation temperature, followed by an endothermic reaction at 960°C in P91B is evident from the figure. This indicates, below this temperature, dissolution of precipitate is not complete in boron containing steel, which is an indication of the high stability of the precipitates in this steel.

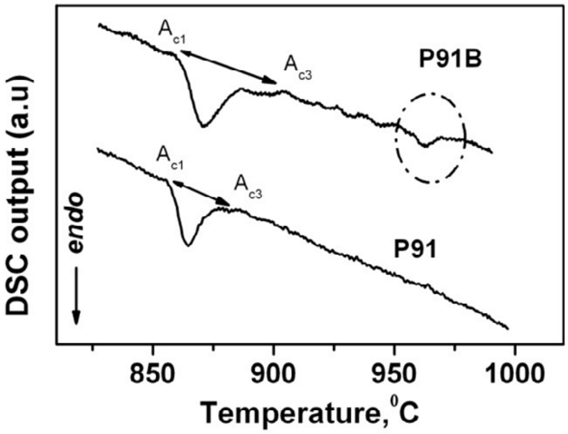

Fig. 1. The effect of boron addition on the α→γ transformation in P91 steel.

3.2. Microstructures of heat treated specimens

Optical photomicrographs of P91 and P91B specimens simulated at 1175, 900 and 875°C are shown in Figure 2(a-f). Increase in prior austenite grain size in P91 with increase in simulation temperature is evident from Figure 2(a-c). On the other hand, comparable prior austenite grain size in P91B with increase in peak simulation temperature was evident from Figure 2(d-f). Prior austenite grain size of P91 steel increased with increase in the heat treatment temperature from 850 to 1175°C as observed in other ferritic steels.[1-4] In this steel prior austenite grain size in the N&T condition is 19 μm. It changed to 2-10 μm, 10-20 and 30-50 μm for heat treatments carried out at 875, 900-1000 range and 1175°C respectively. These temperatures and grain size ranges corresponds to ICHAZ, Fine Grained HAZ (FGHAZ) and CGHAZ of a steel weldments. Martensitic lath structure was present only in the specimens subjected to 900°C and 1175°C heat treatments. On the other hand, the average prior austenite grain size of P91B steel did not vary with heat treatment temperature; it remained more or less unchanged at 28 μm, same as that of the N&T steel. Further, lath martensitic structures with precipitates decorating the lath boundaries are also present in all the specimens after the simulated heat treatment, irrespective of the temperature of the initial heat treatment. SEM image of P91 and P91B specimens after 875ºC heat treatment and subsequent 3h simulated post weld heat treatment (PWHT) at 760ºC are shown in Figure 3(a,b). Figures show well defined sub-structure / sub-grain in P91 specimen and lath martensite in P91B. As the temperature 875ºC falls between Ac_1 and Ac_3 temperatures of these steels, microstructure produced in the steel after this heat treatment corresponds to that of ICHAZ in the steels.

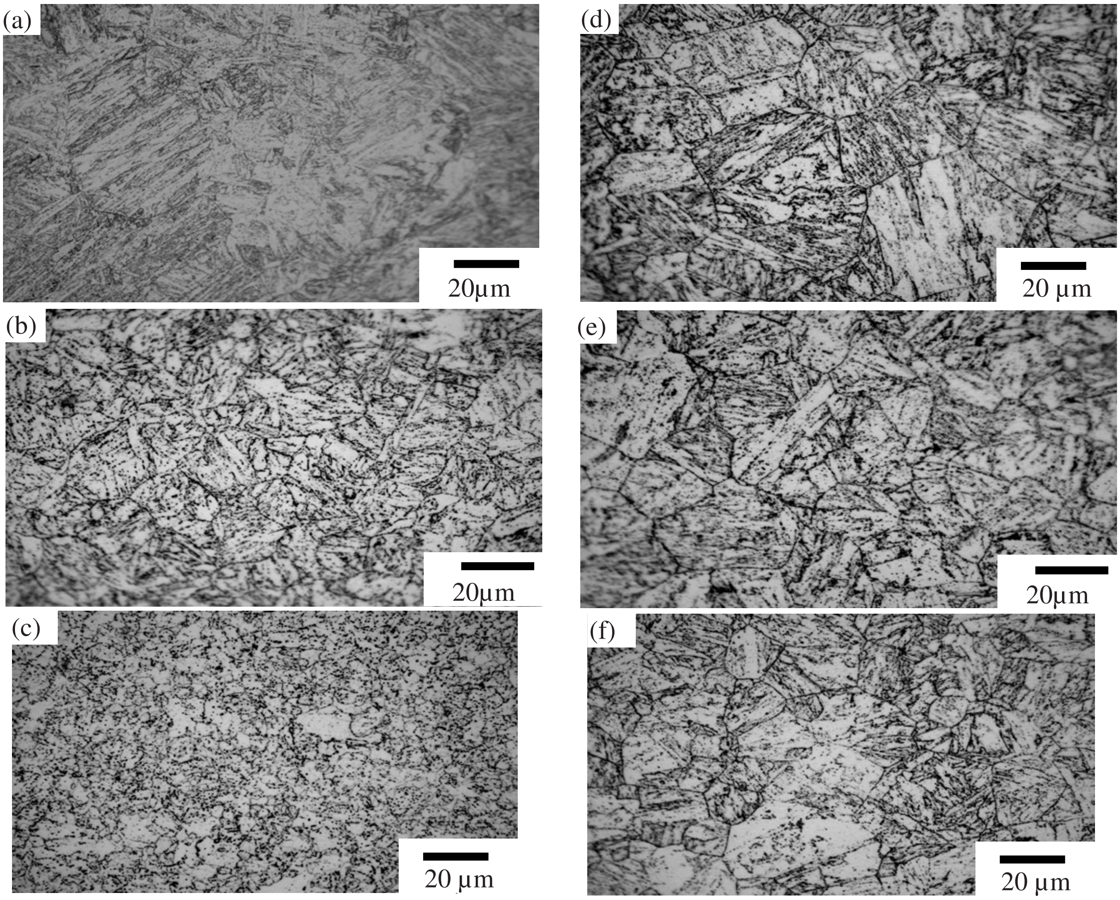

Fig. 2. Photomicrographs of P91 specimens after different peak simulation heat treatment and simulated PWHT at 760°C/3h (a) 1175C (b) 900°C and (c) 875°C; photomicrographs of P91B specimens heat treated at (d) 1175C (e) 900°C and (f) 875°C and followed by simulated PWHT at 760°C/3h.

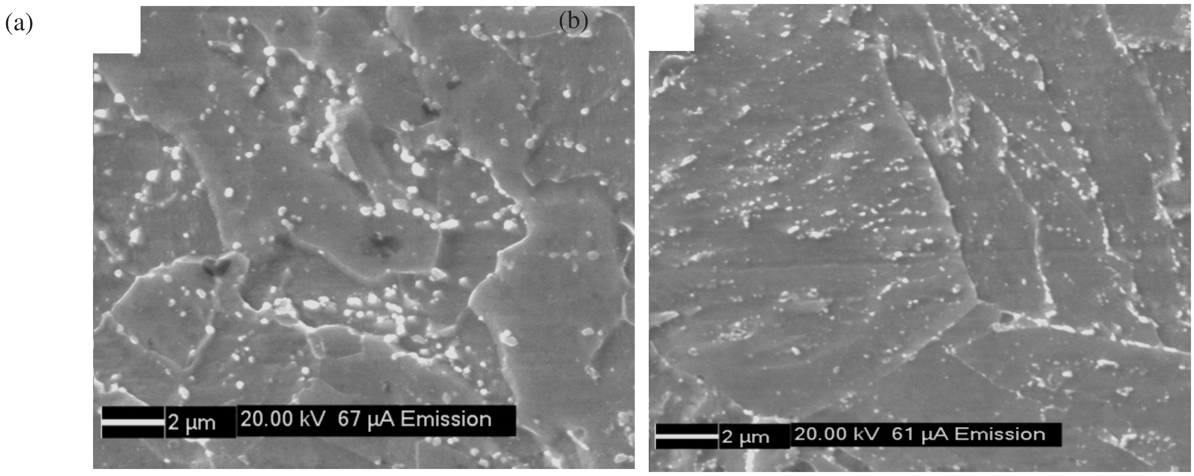

Fig. 3. SEM images of (a) P91 and (b) P91B steels heat treated at 875°C / 5min followed by tempering at 760°C / 3h.

3.3. Hardness

Hardness profile for P91 and P91B specimens after various initial heat treatment at various temperatures for 5 minutes and the subsequent simulated PWHT are plotted in Figures 4a and 4b as a function of temperature. As simulated specimens of P91 and P91B are designated as P91_As_sim and P91B_As_sim respectively, whereas as simulated followed by simulated PWHT specimens are designated as P91_temp and P91B_temp respectively. A clear dip in hardness of the specimen subjected to 875º heat treatment is observed for P91 steel; but only a negligible reduction in hardness is observed for P91B steel for identical treatment.

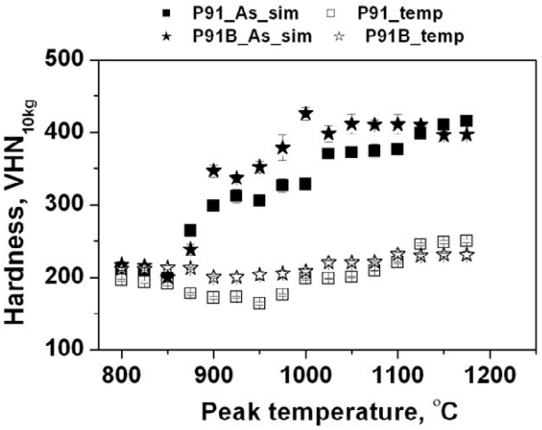

Fig. 4. Hardness vs. peak simulation temperature.

3.4. Creep studies

Results of creep tests for intercritically heat treated P91 and P91B steel specimens carried out 600°C and applied stress of 100 and 120 MPa are shown in Figure 5(a). Creep strain rate vs. time curves for the same tests are shown in Figure 5(b). The creep rupture life of P91 was 425 h at applied stress of 120 MPa. Under similar test conditions creep rupture life of P91B was found to be 3414 h. Similarly, at applied stress of 100 MPa the rupture life for P91B specimens was found to be 10102 h which was significantly higher than for P91 specimens (1474 h). Creep rate in transient state was found to be marginally lower for P91B than P91. Similarly, substantial decrease in creep rate was observed in the steady state region. From Figure 5(a) and (b), it is clear that creep rupture life of P91B is increased compared to P91 steel.

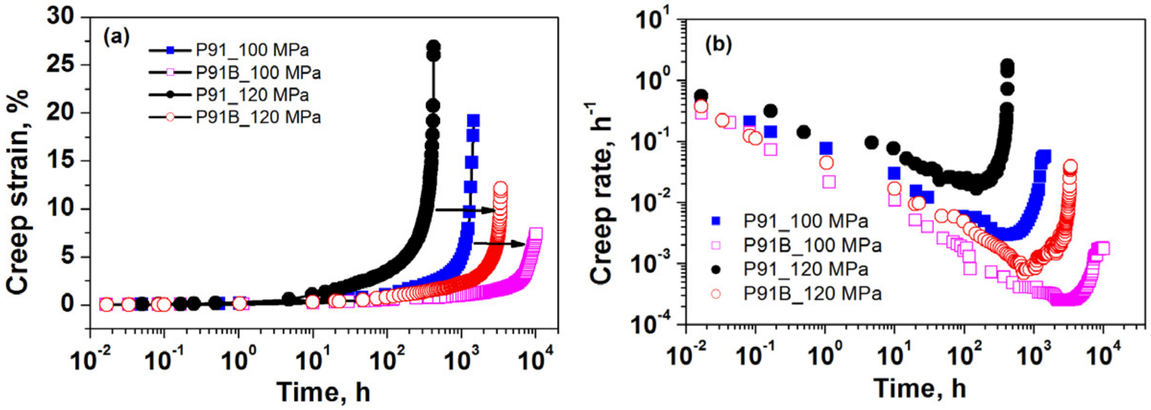

Fig. 5(a) Creep curves for P91 and P91B specimens and (b) Variation of creep rate with time for ICHAZ simulated specimens of P91 and P91B tested at 600°C and applied stress of 120MPa.

4. Discussion

Results presented above confirmed that the prior austenite grain size increased in P91 with increase in peak simulation temperature, whereas, it remained unchanged in P91B under similar heat treatment conditions. The microstructural variation observed after the heat treatments in the temperature range of 800 to 1175°C is similar to those in HAZs of P91 and P91B weldments.[2-4] The increase in prior austenite grain size in P91 can be correlated with random nucleation of austenite during reverse transformation at prior austenite grain boundaries as well as at lath, block and packet boundaries and its subsequent growth in absence of strong pinning effect from the precipitates as they either dissolve or coarsen during the heat treatment. Precipitates control the pinning force and hence grain growth, whereas stability of lath boundaries determines morphology of austenite formed during heating as well as that of lath martensite formed during cooling.[1,5] Formation of less pronounced martensite / sub-structure in P91 specimens, simulated at 875°C is due to formation of low carbon martensite. Presence of boron stabilizes $M_{23}C_6$ precipitate, hence acicular austenite during reversion and martensitic transformation during subsequent cooling is pronounced in P91B. Hardness dip in the ICHAZ of P91 steel is in agreement with sub-structure / sub-grain formation.[1-4] Uniform hardness of P91B irrespective of the heat treatment temperature is in accordance with unifrom grain size and lath structure observed for all the heat treated specimens of this steel. [1] These results also agree with those reported for HAZ of the welds made for thisl steel. Presence of lath martensite as well as delay in formation of sub-structure during creep test in boron containing steel cause in 9 fold increase in the rupture life, Figure 5(a,b) of the inter-critically heat treated P91B steel compared to that of the P91 steel. The onset of acceleration in creep rate was slowed down in P91B resulting in increase in transient creep region which was stretched to longer times. The longer duration of transient creep region was due to the stability of the microstructure in P91B provided by the presence of boron in the precipitates which stabilised the lath boundaries. Microstructural observation of creep tested specimen, from gauge portion revealed sub-structure growth in P91 steel accompanied by coarsening of precipitates and increase in inter-particle distance. Development of sub-structure in P91B is minimum, accompanied by presence of fine precipitates and low inter-particle distance. In presence of boron the progressive local coalescence of two adjacent lath boundaries is difficult, as a result laths do not coarsen in P91B simulated specimens resulting in the increase in creep life.

5. Summary

Prior austenite grain size increased with increase in heat treatment temperature in the range of 850-1175°C for P91 steel and prior grain size remained same as that of N&T condition in P91B steel. Inter-critical heat treatment destroyed the lath structure of P91 steel and subsequent PWHT caused coarsening of the precipitate resulting formation of sub-structures. In contrast lath structure was retained in P91B steel for the same heat treatment conditions and precipitate size remained more or less unchanged in this steel. Retention of hardness in the P91B steel subjected to inter-critical heat treatment and better creep strength of this steel than P91 steel after this heat treatment is attributed to these microstructural differences observed between these two steels.

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