Abstract

The study systematically varied the concentrations of B (0-100 ppm) and N (20-500 ppm) in modified 9Cr-1Mo steel base metals to understand the combined effect of B and N addition on the microstructural stability and the creep resistance (650 °C, 120 MPa). All the five steels hot rolled at 1050°C, followed by normalization (1100°C/1050°C/1000°C-1 h) and tempering (760°C- 1 h) (HR+N+T) before conducting the tests. Auger Electron Spectroscopic (AES) analysis reveals the enrichment of B within the $M_{23}C_6$ precipitates at the vicinity of prior-austenite grain boundaries in B added steels both in normalized and tempered specimens and also in creep tested specimens. The 70 ppm B steel with 108 ppm N showed the best creep resistance (rupture time as high as 1536 h and minimum creep-rate as low as 1×10^{-5} / h), followed by 90 ppm B, 90 ppm N steel. Too low B content (25 ppm or less) or too high N content (500 ppm) affected precipitate stability and consequently the creep resistance of the base metal steels. AES, Atom probe tomography (APT) and Transmission electron microscopy (TEM) combinedly shows the proof for B distribution in $M_{23}C_6$ precipitates and prior austenite grain boundaries, its role in reducing the coarsening kinetics and thereby improving the creep strength is established and quantified. Additionally, the necessity to have a small quantity of nitrogen in the steel to take advantage of strengthening by carbo-nitrides along with boron-carbides is also proven from the characterization performed on selected different steels after creep tests, one with 500ppm N with no intentional addition of B (rupture life-44 h for creep test at 650°C/120MPa), another with 70 ppm B and 108ppm N (rupture life-770 h) and the third steel with 100 ppm of B and 20 ppm of N (rupture life-282 h). To comprehend the combined impact of B and N concentrations on modified 9Cr-1Mo steel's heat affected zone (HAZ) creep resistance, the same steels after heat treatments, subjected to two types of weld thermal cycles (WTC), namely coarse-grained HAZ simulation and inter-critical HAZ simulation with peak temperatures of 1175°C and 850°C, respectively. Prior to and after creep testing (at 650°C, 120 MPa stress), the microstructures and precipitates in the WTC simulated samples were studied using optical and electron microscopy, electron backscattered diffraction, and auger electron spectroscopy techniques. As expected, simulated HAZs of high B-containing steels (70-100 ppm B) showed superior creep resistance compared to low B steels (0-25 ppm B) due to the B stabilizing effect of $M_{23}C_6$ precipitates (primarily $(Fe,Cr)_{23}(B,C)_6$) though the rupture time for the simulated HAZs are lower than that of the base metals tested at the same test temperatures and stress levels. An interesting finding is that, despite the fact that 70 ppm B steel (with 108 ppm N) demonstrated

the best creep resistance in normalised and tempered condition, whereas 100 ppm B steel (with just 20 ppm N) obtained the best creep resistance of simulated HAZ. Large difference in the rupture lives between simulated ICHAZ and simulated CGHAZ specimens reveals the effect of triaxility in reducing the rupture lives in simulated CGHAZ specimens. Since the simulated CGHAZ contains all the sub-HAZ's, this study is applicable to actual weld joints. Charpy impact and tensile tests conducted on the heat-treated (HR+N+T) modified 9Cr-1Mo steels, to check the relative effect of B and N concentrations and austenitization temperature. Charpy impact test shows that the ductile-brittle transition temperature (DBTT) of all the B added steel base metal decreases with increase in austenitization temperature, where the 100 ppm B steel offers the lowest DBTT (-85°C). Similarly, strength increases with the increase in austenitization temperature (1100°C) with a slight drop in ductility. The influence of precipitates on microstructure and mechanical properties is explained considering the B enrichment at the precipitates and the thermodynamic stability of the precipitates. The 100 ppm B steel (containing the maximum B and minimum N), normalized from 1100°C austenitization, shows the best combination of tensile and Charpy impact properties, owing to the effective dissolution of coarse $M_{23}C_6$ and MX precipitates during the normalization treatment and formation of fine B-rich $(Fe,Cr)_{23}(B,C)_6$ precipitates during the subsequent tempering.

In summary, both B and N have some advantageous effects, out of that B segregates at the PAG boundaries and also enrichment of B in $M_{23}C_6$ precipitates significantly improves the precipitate stability and thereby the creep strength. Whereas N forms different MX nitride precipitates with V and Nb, which possess higher stability than MX carbide precipitates and thereby can contribute to the creep strength. However, optimising B and N is an important factor so that their beneficial effects can be utilised by avoiding the harmful BN. In this thesis, it is shown that, when B and N are on the higher side (70-100 ppm B and 90-110 ppm N), then the highest precipitate stability is achieved, which results in the best creep resistance in the base steel. However, high precipitate stability may not be required when there is a weld thermal cycle (WTC) since the pre-existing precipitates (in base steel) do not dissolve and then new precipitates cannot form during post-weld heat treatment (PWHT). Here high B with low N content is beneficial (100 ppm B and 20 ppm N) since the MX precipitates mostly dissolve during the WTC and fine precipitates are formed after PWHT which provides better creep resistance.