

Investigation of Thermal Creep Behavior and Microstructural Degradation in modified 9Cr-2W steel

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1. Introduction

Next-generation nuclear systems, including sodium-cooled fast reactors, are designed to maximize thermal efficiency and safety. The reactor core operates in extreme environments with high temperatures up to 600°C and severe fast-neutron irradiation exceeding 200 dpa [1,2]. Therefore, fuel claddings and structural materials must secure excellent creep resistance and microstructural stability. HT9, a conventional representative 12% Cr ferritic/martensitic steel, exhibits excellent resistance to neutron-induced swelling [3,4]. However, it has shown distinct limitations in high-temperature strength. To overcome these limitations, the modified FC92 steels were newly developed by optimizing the boron (B) and nitrogen (N) contents based on proven 9Cr-2W steels [5,6]. B segregates at grain boundaries to suppress the coarsening of precipitates [7]. N forms fine MX carbonitrides within the matrix to impede dislocation motion [8]. However, the complex mechanisms by which these elements affect matrix solute solubility and dynamic dislocation recovery under long-term high-temperature exposure remain unclear. Therefore, this study correlates the results of thermal creep tests conducted for up to 20,000-h with scanning electron microscope (SEM) and transmission electron microscope (TEM) analyses. Through this, we aim to systematically elucidate the fundamental effects of B and N additions on the long-term creep behavior and microstructural degradation mechanisms of FC92 steels.

2. Methods and Results

The investigated FC92 steels were cast into 2-ton ingots through vacuum induction melting and electroslag remelting processes. They were formed into precision cladding tubes with an outer diameter of 7.4 mm and a wall thickness of 0.5 mm through homogenization, hot rolling, pilgering, and cold drawing. According to the alloy design, the variant containing 130 ppm B and 200 ppm N was defined as FC92B. The variant with 40 ppm boron and 800 ppm nitrogen was defined as FC92N. Details of the fabrication process were reported in previous studies [9]. To simulate a multiaxial stress state, internally pressurized specimens were fabricated by arc-welding

end caps to both tube ends and injecting Ar gas. Thermal creep tests were conducted at 650 °C under a hoop stress of 32 MPa for up to 20,000-h. Diametral changes were precisely measured at 1,000-h intervals using a laser measurement system. The tubes were scanned at 5 mm intervals along the axial direction. Specimens were also rotated at 90° intervals for repeated measurements to ensure data reliability. After the creep tests, cross-sections from regions with the maximum effective strain were extracted for SEM and TEM analyses.

2.1 Thermal creep behavior

As shown in Fig. 1, analysis of the long-term thermal creep behavior showed that both FC92B and FC92N exhibited highly stable secondary steady-state creep without premature rupture for 20,000-h. At 20,000-h, FC92N and FC92B recorded strains of 0.73% and 0.25%, respectively. In particular, the steady-state creep rate of FC92B between 12,000 and 20,000-h was evaluated at $3.1 \times 10^{-9} \text{s}^{-1}$ only about one-third of the rates for FC92N ($1.0 \times 10^{-8} \text{s}^{-1}$). This clearly demonstrates that controlling trace elements decisively alters long-term high-temperature creep resistance.

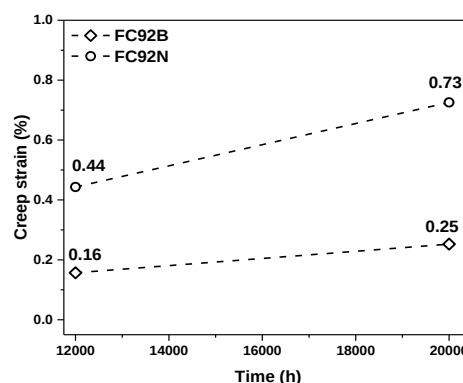


Fig 1. Thermal creep strain curve HT9 and FC92 steels under 32MPa at 650 °C for 20,000-h

2.2 Microstructural Stability and Dynamic Recovery

Crystallographic orientation stability and dynamic recovery behavior were observed through EBSD analysis in Fig. 2 and 3. The sizes of prior austenite grains and martensite packets remained largely unchanged even after long-term creep. However, the differences were found in matrix lattice curvature collapse and dislocation annihilation behavior. In FC92B, high-angle boundaries (HABs) were highly stabilized due to the segregation of B at grain boundaries. Consequently, about 82% of the initial geometrically necessary dislocation (GND) density was firmly preserved after 20,000-h. Conversely, FC92N experienced active boundary migration and rapid dislocation annihilation during creep exposure. Its residual GND density dropped sharply to 48% of the initial level. This quantitatively proves that severe dynamic recovery and material softening occurred in the FC92N.

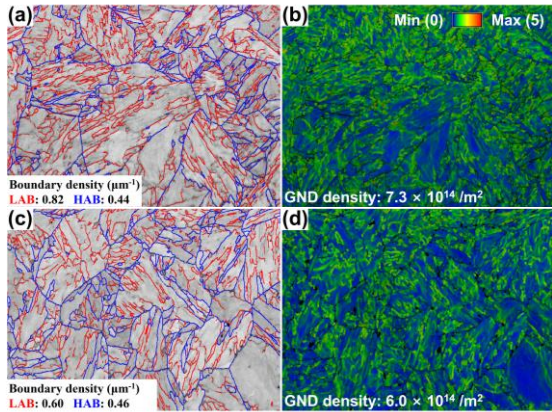


Fig 2. EBSD misorientation and KAM maps of FC92B (a, b) initial state and (c, d) after 20,000 h of creep: (a, c) misorientation maps and (b, d) KAM maps.

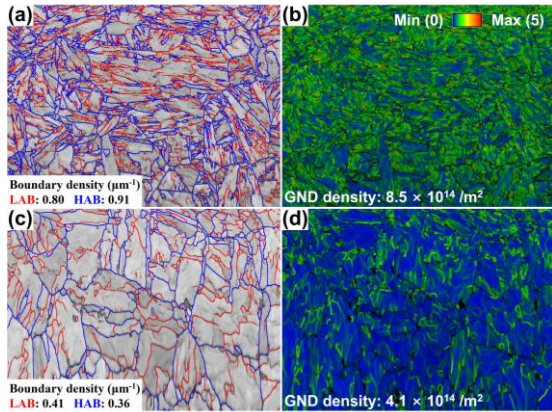


Fig 3. EBSD misorientation and KAM maps of FC92N (a, b) initial state and (c, d) after 20,000 h of creep: (a, c) misorientation maps and (b, d) KAM maps.

2.2 Precipitate Evolution and Dislocation Interactions

As shown in Fig. 4, Secondary phase evolution and dislocation-precipitate interactions were identified using scanning transmission electron microscope (STEM) and high-resolution TEM. Literature reports that B atoms

substitute carbon sites within $M_{23}C_6$ carbides [5]. In FC92B, B migrated and segregated to grain boundaries, effectively retarding precipitate coarsening as well as the growth and migration of grain and lath boundaries. This suppression also delayed the heterogeneous nucleation and growth of adjacent coarse Laves phases. Consequently, the rapid depletion of major solid-solution strengthening elements like tungsten and molybdenum was prevented, maintaining the critical resolved shear stress (CRSS) of the matrix at a high level over an extended period. These stable precipitates exerted strong Zener pinning pressure on the martensitic lath boundaries, strictly limiting lath width expansion to within a factor of 2.5. Fine MX particles within the matrix also fully preserved a dislocation knitting structure to continuously impede dislocation glide.

In contrast, for the FC92N alloy, prior studies on tensile tests after high-temperature aging reported that N addition yields higher initial strength, but results in significant strength degradation after long-term aging [10]. Under the long-term creep conditions in this study, the fine MX carbonitrides within the matrix also exhibited limitations in restraining dislocations. Mobile dislocations readily bypassed the MX carbonitrides, leading to a loss of the dislocation pinning effect. Simultaneously, the premature coarsening of $M_{23}C_6$ carbides and the aggressive formation of coarse Laves phases were accelerated. This promoted the depletion of tungsten and chromium in the matrix, severely degrading the matrix CRSS. Consequently, lath boundary migration was activated, causing the lath width to coarsen by more than a factor of 3.6 compared to its initial state. The unpinned dislocations rapidly migrated toward the boundaries and accumulated into dislocation walls, rearranging into subgrain structures. This resulted in severe microstructural softening and degraded creep resistance during ultra-long-term high-temperature exposure.

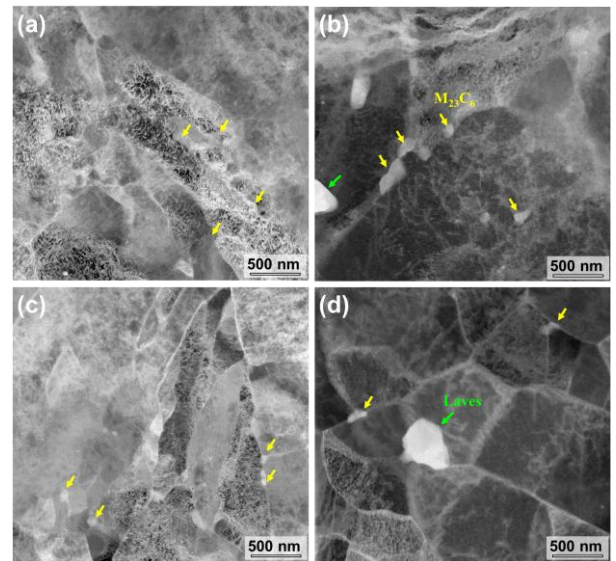


Fig 4. STEM micrographs of (a, b) FC92B and (c, d) FC92N: (a, c) initial state and (b, d) after 20,000 h of creep testing.

3. Conclusions

This study successfully correlated rigorous electron microscopy characterizations with thermal creep tests conducted at 650 °C and 32 MPa. Through this, the critical mechanisms governing the long-term lifespan of FC92 steels modified with B and N were derived. The FC92B alloy effectively suppressed precipitate coarsening via the grain boundary segregation of B. This fundamentally prevented the premature depletion of solid-solution strengthening elements in the matrix. Consequently, it retained approximately 82% of its initial dislocation density after 20,000-h, demonstrating superior long-term creep resistance. In contrast, the N-modified FC92N alloy suffered from the loss of dislocation pinning capability by MX carbonitrides and solute depletion induced by the coarsening of secondary phases. Dislocation annihilation and subgrain formation were accelerated, leading to the premature degradation of long-term creep durability. In conclusion, stabilizing grain boundaries through trace B control is essential for designing nuclear structural materials exposed to extreme high-temperature environments. This approach was confirmed to be a far more effective and robust metallurgical strategy for securing long-term reliability than intragranular precipitation strengthening based on N

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