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Effects of pre-creep on dislocation and tensile property of Cr-Ni steel

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Abstract

Pre-creep experiments were performed on chromium-nickel (Cr-Ni) stainless steel in the early stage of creep. The temperature was held for 500–2000 h under high-temperature load conditions (873 K and 150 MPa), and various analysis methods, including optical microscopy, electron backscatter diffraction (EBSD), transmission electron microscopy (TEM), and X-ray diffraction, were used to determine the pattern of dislocation evolution under different temperature-holding times. The results showed that the slip bands intersected at the initial stage of pre-creep, and a quadrilateral network structure was formed by the dislocation pinning. As the temperature-holding time increased, the dislocation network began to climb to form dislocation walls, dislocation cells, and other substructures. At 2000 h, the grain boundaries widened considerably; creep holes were found at the grain boundaries; and dislocation pairs with oscillating contrast occurred, which indicates nitrogen diffusion. The yield and tensile strengths of the Cr-Ni steel samples subjected to pre-creep at holding times of above 1000 h decreased after they were subjected to room-temperature tensile tests.

1. Introduction

Chromium-nickel (Cr-Ni) stainless steel (316LN) is the main structural material of nuclear power pipelines. It exhibits good high-temperature performance, corrosion resistance, wear resistance, and long service life. However, the long-term exposure of nuclear power pipelines to chemically corrosive environments leads to a decline in the comprehensive performances of the materials, which is not conducive to the safe operation of nuclear power plants [1, 2]. For example, such exposure can result in long-term creep.

The relationship between the stress and steady-state creep rate satisfies Norton’s power law ($\dot{\varepsilon} = A \sigma^n$) [3], and different stress indices ($n$) correspond to different creep mechanisms. Ganesan et al. [4] showed that the creep stress index of 316LN with 0.14% nitrogen was 6.9. The nitrogen content of the material selected in this study was approximately 0.16%, and the stress index should be slightly lower than 6.9. The main creep mechanism was dislocation creep, and the diffusion of solute atomic nitrogen affected the dislocation movement.

The steady-state creep rate of 316LN at 650 °C and found that as the N content increased from 0.07% to 0.22%, the power-law exponent decreased from 13.6 to 5.5. [4]. This occurred because the dislocations generated during the intermediate-temperature creep process at a low nitrogen content were rearranged in the form of subgrains, and the tendency of 316LN to form subgrains decreased with an increase in the nitrogen content. Sundararaman et al. [5] studied the pattern of Cr$_2$N precipitation under temperatures of 750 °C and 850 °C and holding times of 25–100 h and found that over time, the precipitation sequence of the Cr$_2$N was as follows: uniformly distributed chromium-nitrogen complexes $\rightarrow$ localized and disordered face-centered cubic Cr-N clusters $\rightarrow$ intragranular type-A Cr$_2$N hexagonal close-packed (hcp) precipitates ($a = 2.748 \, \text{Å}, c = 4.438 \, \text{Å}$) $\rightarrow$ intragranular type-B Cr$_2$N hcp precipitates ($a = 4.76 \, \text{Å}, c = 4.438 \, \text{Å}$) $\rightarrow$ cellular precipitation of Cr$_2$N. The precipitation of A-type Cr$_2$N occurred when the temperatures were held at 750 °C and 850 °C for 25 h.
During the steady-state creep process, the internal dislocation structure of the material undergoes complex changes. The dislocation density will increase during the initial deformation, forming dislocation tangles and then a cell structure, and this type of dislocation at the cell wall is orderly arranged to form subgrain boundaries [6]. Blum [7] found that the parallel dislocations at the center of the creep process reacted with the dislocation loops of the octahedral slip system. When the orientation of the Burgers vector participating in the reaction was at a specific angle, a plane hexagonal dislocation network formed. Nakazawa et al [8] found that when a material is subjected to stresses below the yield strength for a long period of time, solid solution atoms and dislocations were prone to thermal activation and could drive dislocation slip when a force was applied; moreover, macroscopic creep occurred after long-term accumulation. Therefore, during the pre-creep process, microscopic changes, mainly dislocation generation and decomposition, are present within the grains [9–12]. However, these dislocations will be activated to different degrees and move in different ways under different pre-creep processing conditions [13–15]. In addition, some studies have reported that the pre-creep failure deformation often has a certain relationship with the sliding and migration of the grain boundaries [16]. Due to the accumulation of the grain boundary sliding, macro-creep fracture occurs. However, changes in the internal dislocations in the first stage of the steady-state creep have not been reported.

In the present study—to further clarify the relationship between the dislocations and precipitation phases with the pre-creep failure of 316LN—the critical stress and temperature were used as pre-creep test parameters. The effects of nitrogen on the dislocation slip and the evolution mechanism of the dislocation network under critical conditions were investigated for 316LN. The tensile properties were tested, and the effect of the changes of the dislocations on the creep failure was revealed.

### 2. Materials and methods

Chromium-nickel stainless steel (316LN) for nuclear power pipelines was selected as the research object. The alloy elemental composition is presented in table 1. The experimental sample (figure 1) was prepared from 316LN. The sample was further subjected to a creep process for 500–2000 h at a critical temperature of 873 K and critical stress of 150 MPa (table 2). Optical microscopy and scanning electron microscopy—electron backscatter diffraction/energy-dispersive X-ray spectroscopy were used to determine the sample grain size, changes in the grain boundary angles, and element distribution patterns. Electron backscatter diffraction (EBSD) measurements were performed along the plane perpendicular to the transverse direction of the sheet using a field-emission scanning electron microscope (Zeiss Ultra 55, Carl Zeiss Microscopy, Jena, Germany). The operating voltage was 15 kV. The EBSD data were evaluated using orientation imaging microscopy (OIM, HKL–Channel 5) software [17]. To clarify the
evolution mechanism of the dislocations in the pre-creep process, transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) were used to analyze the micro-changes of the dislocations. The tensile properties of 316LN were tested at room temperature, and fracture analysis was performed via scanning electron microscopy.

3. Results and discussion

3.1. Changes in grain and grain boundary orientation difference

When the deformation temperature is greater than the critical temperature for recrystallization, the grain size of Cr-Ni stainless steel will increase with increasing temperature-holding time and heating temperature [18]. In
this study, the grain structures of the original sample, i.e., before pre-creep (figure 2(a)), and those of the samples subjected to pre-creep at 873 K/5000 h, 873 K/1000 h, 873 K/1500 h, and 873 K/2000 h (figures 2(b), (c)) were compared. The grains did not grow because the deformation magnitude of the sample did not change significantly during the pre-creep process, preventing the strain energy absorbed by the matrix from providing the driving force for grain growth. Therefore, the grain size effect can be ignored in the study of the dislocation slip pattern.

The changes in the grain boundary angles of the alloy at 500–2000 h were measured by EBSD, and the results are illustrated in figure 3. Considering that the grain boundary structures did not change, the changes in the grain boundary angles were mainly due to the formation of subgrain boundaries through dislocations rearrangement. Therefore, the distribution trends of the grain-boundary orientation difference at different
times were essentially the same. The peak value appeared at the grain boundaries with a small angle of $0^\circ - 5^\circ$, and the peak value of the twin boundaries appeared at the grain boundaries with a large angle of $60^\circ$. The difference was that the peaks in the small-angle distribution areas gradually decreased with time. This occurred because the dislocation slips could annihilate each other or because the subgrains could merge and grow due to the prolonged temperature-holding time, causing small-angle subgrain boundaries to disappear.

3.2. Formation mechanism of dislocation with oscillating contrast

Owing to the long temperature-holding time, the gradient of the solute atom diffusion at the grain boundary changed, and the original distribution of the atoms changed, causing the atoms to redistribute during the sub-diffusion. Some of the solute Cr and N atoms could produce a certain amount of segregation, but it is uncertain whether the Cr and N atoms formed precipitate phases\cite{19}. The thermodynamic correlation by Grujiicic et al\cite{20} showed that there is more than an 80% probability of nitrogen atoms being surrounded by three or more chromium atoms in Wagner’s cell model. In the solid-state Cr-Ni stainless steel, nitrogen was randomly distributed in the matrix. During the pre-creep experiment, the nitrogen atoms exhibited a distinct micro-migration tendency. Due to the high affinity between nitrogen and chromium, Cr$_2$N clusters formed during the diffusion process (Shankar et al., 1999). The TEM observations of the grain boundaries did not show precipitates of the Cr$_2$N phase, because after the steel was held at 873 K for 2000 h, the diffusion and migration of nitrogen and chromium were not sufficient to form Cr$_2$N precipitates larger than 10 nm (Patricia et al., 2008). The segregation of nitrogen and chromium led to the formation of nitrogen-depleted regions and an uneven distribution of nitrogen. Although atomic clusters were difficult to detect, the combination of nitrogen atoms and dislocations could produce an oscillating contrast because the nitrogen atoms were blocked and interacted when they encountered dislocations during migration\cite{17}. Dislocation pairs with oscillating contrast were found in the pre-creep samples heated at 873 K for 500–2000 h (figure 4), which further demonstrates that nitrogen underwent significant migration and diffusion during the two temperature-holding processes. Narrow dislocation pairs were found in the samples at 1000 and 2000 h (figures 4(c), (d)), and this has been attributed to

![Figure 5. Evolution of the dislocation network after pre-creep: (a) 873 K/150 MPa/500 h; (b) 873 K/150 MPa/1000 h; (c) 873 K/150 MPa/1500 h; (d) 873 K/150 MPa/2000 h.](image-url)
the weakening of the interactions between nitrogen and the dislocations (Shankar et al., 1999). Consequently, only short-range ordered chromium-nitrogen clusters formed, but Cr₂N did not precipitate.

3.3. Evolution pattern of dislocation networks

In the early stage of pre-creep, the two dislocation slip bands moved along both the b₁ and b₂ directions (figure 5(a)) and intersected each other. The dislocations were fixed to form a relatively regular quadrangular structure. It has been proposed [20] that this type of network can hinder the dislocation movement and delay the creep occurrence. With the prolongation of the pre-creep temperature-holding time, the dislocations tended to move continuously under the combined action of the stress and high-temperature activation energy, and the dislocation network gradually became smaller until it was damaged. With the prolongation of the holding time, the dislocation climb formed a dislocation wall (figure 5(b)). The black dots near the dislocation wall were observed via HRTEM, and no precipitation phases were present. Thus, the dislocations intersected each other and had a certain pinning effect, which led to dislocation climb under the continuous high-temperature load. As shown in figure 5(c), the dislocation climb formed a large number of closed dislocation cell structures. As the temperature-holding time continued to increase, the dislocation cells gradually grew and were distributed in a wavy pattern in a specific direction (figure 5(d)). Importantly, during the pre-creep process, with a temperature-holding time of 2000 h as an example (figure 6), a dislocation source formed due to dislocation accumulation at the grain boundaries. This resulted in the widening of the grain boundaries, and dense creep holes formed on them. The formation of these creep holes mainly depended on the continuous slip of dislocations on the grain boundaries.
3.4. Analysis of tensile properties and microstructure of pre-creep Cr-Ni alloy at room temperature

The dislocation network structure could delay the creep failure. As the dislocation network gradually transformed into dislocation cells (or subgrains) with a relatively low dislocation density and few creep holes, the strength of the Cr-Ni steel gradually decreased. As shown in figure 7, the strength of the pre-creep sample with a temperature-holding time of 1000 h slowly increased. This indicates that although many slips did not occur during the pre-creep phase to generate high-density dislocations, a small number of slips caused the number of dislocations to increase, and the formation of mutual pinning of the dislocation networks hindered the movement of dislocations, which led to an increase in the strength of the sample. With increasing temperature-holding time, the hardness and yield strength gradually decreased, and the corresponding elongation rate gradually decreased.

The TEM results of the sample cross section that was subjected to a room-temperature tensile test after the 500 h pre-creep process showed the appearance of deformed twins, as confirmed by the diffraction patterns displayed in figure 8(a). The deformation mechanism of the sample was dominated by twins and slip deformation, and the appearance of deformed twins also indicated an increase in the yield strength. As shown in figure 8(b), when the temperature of the creep sample was held for 1000 h before the tensile test, the probability of dislocation slip increased, and the slip bands moving in different directions intersected. As shown in figure 8(c), the slip bands that intersected each other in different directions gradually formed sub-structures, such as dislocation cells, by dislocation climbing, leading to a reduction in the dislocation density. Therefore, the deformation mode after the room-temperature tensile tests was mainly the elongation of the crystal grains. The grain boundary slip with dislocations is also evident in figure 8(d).

The tensile fracture mainly reflected the fracture mode of the sample. Under the holding time of 500 h, because the pre-creep time was the shortest, the fractures and dimples of the sample were densely distributed after the tensile tests (figure 9(a)), showing typical ductile fracture characteristics. The tensile fracture mode did not change in the 500 h creep sample, and the fracture mode in the 1000 h creep sample (figure 9(b)) is similar to

![Figure 8. Transmission electron microscopy profiles of pre-creep specimens after tensile tests at room temperature: (a) 873 K/150 MPa/500 h; (b) 873 K/150 MPa/1000 h; (c) 873 K/150 MPa/1500 h; (d) 873 K/150 MPa/2000 h.](image-url)
that in figure 8(a). However, when the 1500 h (figure 9(c)) and 2000 h (figure 9(d)) creep samples were subjected to tensile tests, dense irregular holes appeared, which were caused by creep failure. At this time, the fracture mode was affected by creep, and a mixed structure of dimples and irregular holes appeared, accelerating the failure of the Cr-Ni steel [21–26].

4. Conclusions

(1) The grain boundary migration and the formation of holes in the grain boundaries due to the dislocation slip also marked the beginning of pre-creep failure. It is predicted that a higher temperature and greater applied stress would lead to more severe grain boundary widening and more substantial creep failure.

(2) Diffused nitrogen was present in the form of atomic clusters, and no distinct precipitation phase formed. However, the occurrence of dislocation pairs with oscillating contrast indicated the occurrence of nitrogen diffusion.

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