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Characterization of mechanical properties for creep-fatigued ferritic heat-resisting steel by nano-indentation

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Abstract

Creep-fatigue test was conducted for a ferritic heat-resisting steel that contained 12mass%chromium and 2mass% tungsten. The creep-fatigue fracture originated from prior austenite grain boundaries. Subgrains neighboring the prior austenite grain boundaries became coarse during creep-fatigue testing. Nano-indentation tests were performed on the coarse subgrains neighboring grain boundaries and finer subgrains inner grains. As the results, the nano scale-hardness of the coarse subgrains were markedly lower than those of the finer subgrains inner grains. Therefore, it is suggested that the coarse subgrains neighboring grain boundaries play an important role of creep-fatigue fracture mechanism.

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Keywords: ferritic chromium-tungsten steel, tempered martensite, subgrain, grain boundary, creep-fatige, nano-indentation

1. Introduction

High chromium (Cr) ferritic heat-resisting steels have high creep rupture strength and smaller thermal expansion coefficients than austenitic stainless steels. Therefore, they have been developed for use in Ultra Super Critical (USC) power plants. Fossil fuel power plants are operated under daily start-up and shut down cycles, and so thermal stresses are induced on the structural components. The environmental and thermal conditions of USC plants, under high steam pressure at 873K, 923K, are very severe to the steels. High temperature fatigue properties, as well as creep strength, are important factors for strength design of the components and for safety.

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We have evaluated the creep failure and fatigue properties of 9-12mass%Cr - 2-3mass%W ferritic heat-resistant steel and have found that failure occurs in the grains in steels with a relatively long creep fatigue life and at the prior austenite (γ) grain boundaries in steels with a short creep fatigue life [1,2]. A quantitative evaluation of the distribution of precipitates, blocks and subgrains performed after the creep fatigue failure of steels with a short creep fatigue life has shown that the promotion of the densification of boundary precipitates and the local coarsening of subgrains adjacent to the grain boundaries assists the creep fatigue failure that occurs at the grain boundaries [3]. As a result, the local change in the microstructure during creep fatigue has been shown to be closely related to creep fatigue failure. The mechanical properties associated with the change in the microstructure have not been quantitatively evaluated. Nanoindentation is known as a technique that can evaluate the mechanical properties associated with nano-scale structural factors. In this study, we have measured the hardness distribution in blocks before creep fatigue testing and the hardness distribution in coarse subgrains adjacent to the prior γ grain boundaries formed after failure. A quantitative evaluation of the local change in the mechanical properties has shown that the non-uniformity of hardness associated with the microstructure is closely related to the mechanism of creep fatigue failure.

2.**Experimental procedures**

The specimens used were made of 12Cr-2W steel (0.14C-0.26Si-0.65Mn-0.74Cu-0.4Ni-11.03Cr-1.95W-0.29Mo-0.2V-0.07Nb; mass%) and were cut from thick wall tubes (ASTM-SA355-P122) that had been fabricated into main steam tubes for a fossil fuel-fired power plant. They were heated at 1323K for 3.6 ks, normalized by cooling in air, and then annealed at 1053K for 21.6 ks. The size of the prior γ grains was 150-200 μm. The high-temperature tensile properties of the specimens were measured at 923K (0.2% yield strength: 229 MPa, tensile strength: 285 MPa, elongation: 34%, reduction in area: 88%). Figure 1 shows the previously reported creep fatigue properties [1]. In the figure, the specimens used in this study are represented as 12Cr-2W (pipe). The total strain range for a trapezoidal wave was 1.0% and the strain hold-time was 10.8 ks. The initial strain rate was 5×10^{-4}/s. The number of creep fatigue cycles to failure under these conditions was 309 (a total time of 3349 ks). The failure occurred at the prior γ grain boundaries.

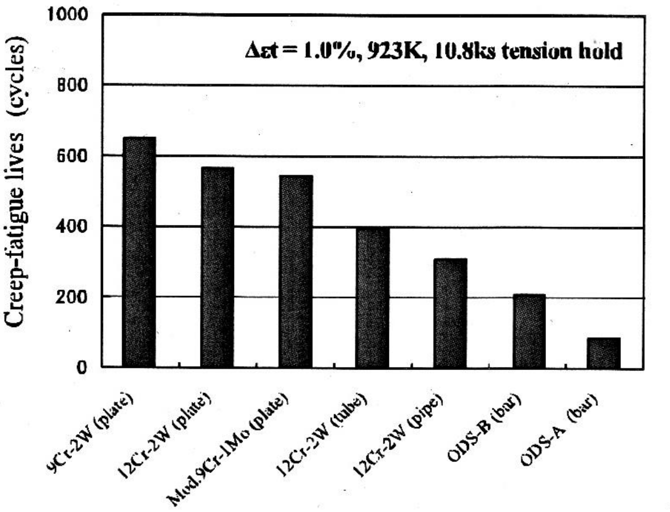

Fig. 1. Creep—fatigue lives tested in trapezoidal strain wave shape with 10.8 ks hold time at tension side at 923K, for various heat-resisting steels used in the previous study[1]. The material used in this study is shown as "12Cr-2W (Pipe)" in this figure.

Pieces 5 mm long, 7 mm wide and 1 mm thick were cut in a longitudinal direction from the parallel section of the pre-creep fatigue test tubes and the creep-failed tubes, and were used as specimens for structure observation and nanoindentation testing. The surfaces of the specimens to be observed were ground with 1 μm diamond and polished to a mirror finish, and then subjected to a chemical mechanical polishing (CMP) [3] process.

A nanoindentation tester is based on an atomic force microscope [4]. Its X-Y scan range is 9 μm × 9 μm. A pyramid indenter with an apex angle of 60° was used. In the nanoindentation test, the specimens were loaded at a rate of 30 μN/s, held at a maximum load of 866 μN for 5 seconds, and then unloaded at the same rate. For the pre-creep test specimens, the indenter was pushed approximately into the center of a relatively coarse block. For the creep-failed specimens, the indenter was pushed into the center of a coarse subgrain near a grain boundary.

3. Results and discussion

3.1. Structure Observation

Figures 2 and 3 show backscattered electron images of the CMP surface before the creep-fatigue test and after creep-fatigue test failure, respectively. Subgrains can be determined from the difference in contrast resulting from the difference in misorientation. The contrast was insufficiently high before the creep test and was high after creep failure. Previously reported TEM images [3] suggest that this is because the dislocation density was high before the creep test and the recovery of dislocations was promoted after the creep failure. After the failure, the strip-shaped subgrains were coarsened in the width direction, and some nearly equiaxed subgrains were observed. Measurements

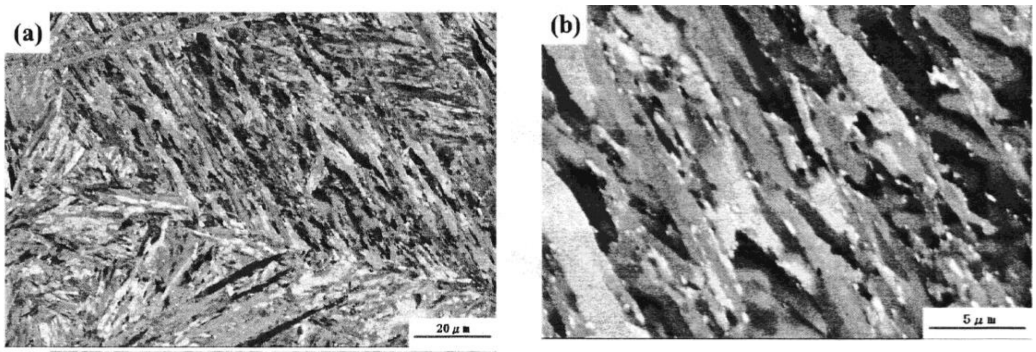

Fig. 2. Low-and high-magnification FE-backscat-tered electron images of the chemical mechanically polished surface before the creep-fatigue testing in (a)and(b),respectively. Subgrains represented by black and white contrast are clearly distinguished.

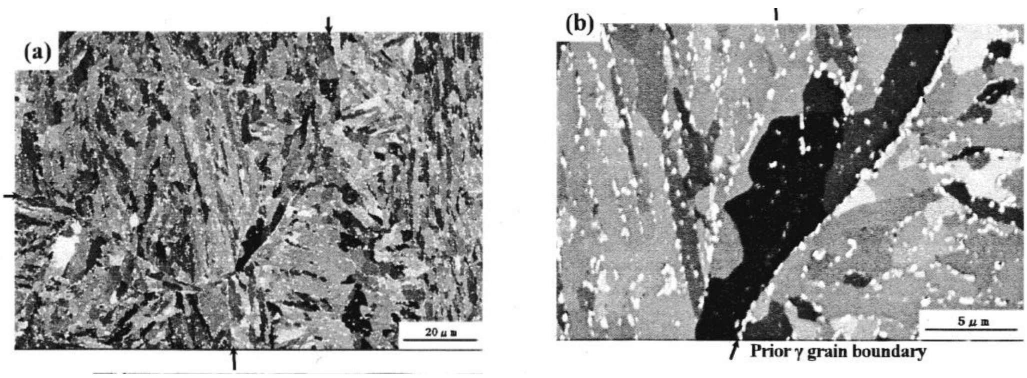

Fig. 3. Low-and high-magnification FE-backscat-tered electron images of the chemical mechanically polished surface after the creep-fatigue testing in (a) and(b) ,respectively. Subgrains and prior γ grain boundaries are clearly distinguished.

of the distribution of the subgrain width (the diameter of equiaxed subgrains or the length of oval and elliptical subgrains in the short-axis direction) taken before the creep test and after the failure show that the average subgrain width was 0.54 μm before the test and 1.16 μm after the failure. The subgrain width after the creep failure was about twice that before the test. The variation in the distribution increased after the creep failure, and coarse subgrains larger than 2 μm in width (for example, the average width of the blocks) were additionally formed. As shown by the backscattered electron images of the CMP surface after the failure, both the contrast of the subgrain structure and the precipitate phase were high due to the high density of the grain boundary precipitates, and the location of the prior γ grain boundaries was clearly visible. Coarse subgrains were often observed near the prior γ grain boundaries.

3.2. Nanoindentation Test

Figure 4(a) is the load-indentation (F-h) curve obtained in the nanoindentation test performed in a block before the creep fatigue test. As the load increased, the indentation increased continuously and reached a maximum value of 373 nm. Figure 4(b) is the F-h curve inside a subgrain obtained from the specimens after the creep fatigue test. A discontinuous (pop-in) phenomenon was observed at F=100 μN. This pop-in phenomenon is known to occur in metal single crystals and annealed mild steels with a sufficiently low dislocation density. The TEM observation described in the previous report [3] suggests that a significant rearrangement and recovery of dislocations in the subgrain occurred due to creep fatigue in the steel used in this study, which has a martensitic structure.

Our research group has proposed a metal single crystal-based method for converting nanoindentation test results to Vickers hardness [5]. The F-h curves in Figures 4 were converted by this conversion method to Vickers hardness and are shown in Figure 5. Figure 5 shows the converted data obtained for three blocks before the test and three subgrains after the failure. In the figure, the white circles indicate data points before the creep test, and the black circles indicate data points after the failure. The hardness of the blocks before the creep fatigue test was approximately 200 and that of the coarse subgrains after the failure was 150. This shows that the decrease in the strength of the coarsened subgrains near the prior γ grain boundaries is closely related to failure in 12Cr-2W steel with a short creep fatigue life, where failure occurs at the prior austenite (γ) grain boundaries.

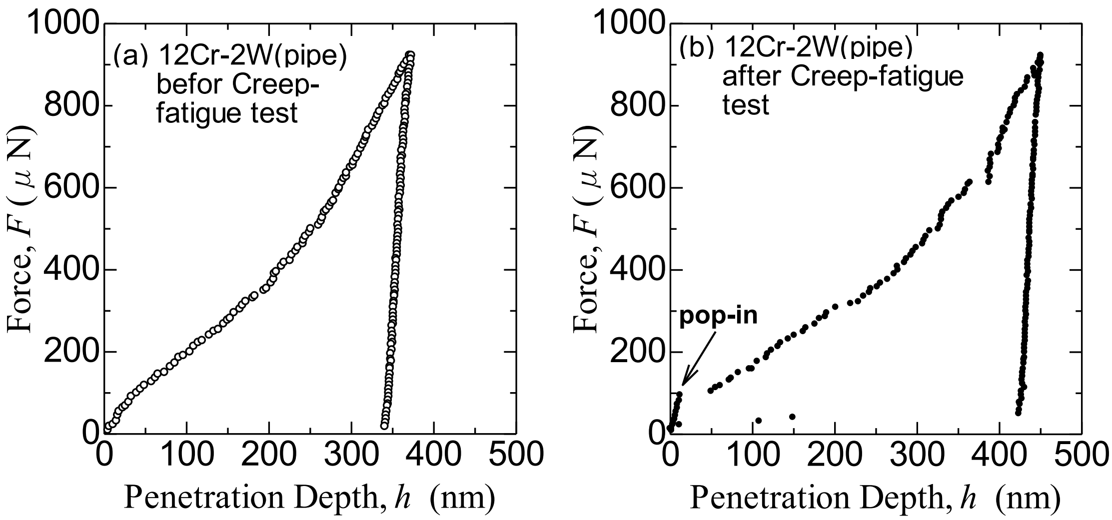

Fig. 4. F-h curves.
(a) Nanoindentation test was performed in a block before the creep fatigue test.
(b) Nanoindentatin test was performed in large subgrain after the creep fatigue test.

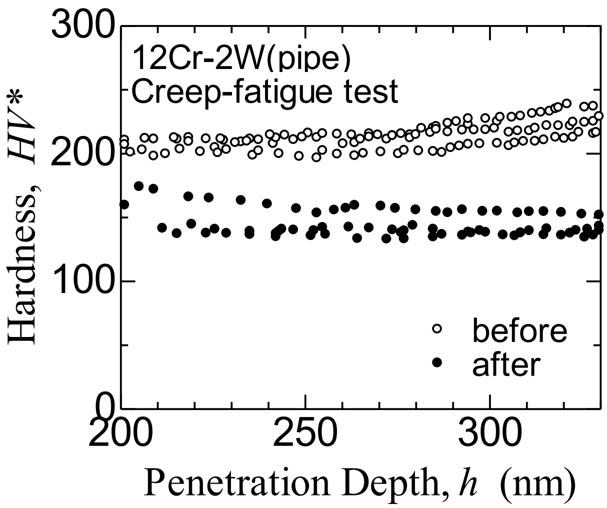

Fig. 5. Estimated Vickers hardness.

4. Summary

Nanoindentation analyses were performed for the creep-fatigued 12Cr-2W steel. As the results, the hardness in the coarse subgrains neighboring to prior austenite grain boundaries is markedly decreased by recovery of dislocation. Therefore, it is suggested that decreasing of hardness in the coarse subgrains is correlated with the grain boundary fracture mechanism.

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