

Comparative Study on Creep Behavior of Two HR3C Heat-Resistant Steels Based on Stress Relaxation (Postprint)

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Abstract

Using the stress relaxation method, the high-temperature creep deformation behavior of as-received and aged specimens of two types of HR3C heat-resistant steel was studied, and their microstructural characteristics were analyzed. The results show that although the chemical compositions of the two types of HR3C heat-resistant steel are similar, their creep behavior exhibits significant differences. Under the same conditions, the creep rates of both the as-received and aged states of the coarse-grained HR3C heat-resistant steel are lower than those of the fine-grained HR3C heat-resistant steel, indicating higher creep resistance. After high-temperature aging treatment, the creep resistance of both types of HR3C heat-resistant steel decreases significantly. For the fine-grained HR3C steel, the reductions in stress exponent (n) and apparent creep activation energy (Q) after high-temperature aging are more pronounced, indicating that the stability of creep resistance of the fine-grained HR3C heat-resistant steel is lower than that of the coarse-grained HR3C heat-resistant steel.

Full Text

Creep Behavior of Two HR3C Heat-Resistant Steels Based on Stress Relaxation Tests

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Abstract

Rupture life is a critical property for materials used in high-temperature applications, typically obtained through creep rupture testing. Since both creep and stress relaxation represent primary high-temperature deformation behaviors, researchers have sought to establish interrelationships that would allow one behavior to be derived from the other. Recently, stress relaxation testing has gained attention as an alternative to conventional creep rupture testing for studying creep behavior and predicting rupture life. The stress relaxation method offers several advantages including shorter testing times, reduced material damage, and richer information content.

This work employs stress relaxation testing to analyze the creep deformation behavior of two HR3C heat-resistant steels with different grain sizes, examining both as-received and aged specimens to assess microstructural effects. Despite similar chemical compositions, the two steels exhibited significantly different creep behaviors. The coarse-grained HR3C steel demonstrated lower creep rates and higher creep resistance than the fine-grained variant in both conditions. Long-term aging degraded the microstructure and reduced creep resistance in both materials, with the fine-grained steel showing a more pronounced decrease in stress exponent (n) and apparent activation energy (Q), indicating less stable creep resistance compared to its coarse-grained counterpart.

KEY WORDS HR3C heat-resistant steel, stress relaxation, creep

Introduction

Creep rupture life represents one of the most critical high-temperature properties of engineering materials. Conventionally, creep deformation behavior under high-temperature conditions is studied through creep testing, which measures deformation evolution under constant load over durations ranging from tens to hundreds of thousands of hours [2]. In contrast, stress relaxation testing examines the relationship between applied stress and time under constant strain, offering a relatively short-term variable-stress experiment [3]. Due to its advantages of short duration, minimal material consumption, and rich creep information, stress relaxation testing has proven particularly effective for comparing creep behaviors between different microstructures, especially when material availability is limited or testing stresses are low [4].

The stress relaxation behavior of a material is intimately related to its creep behavior, enabling derivation of creep data and high-temperature properties from stress relaxation measurements [5,6]. This approach has increasingly attracted researcher attention [7-12]. HR3C steel is a novel dispersion-strengthened austenitic heat-resistant steel with high Cr and Ni content, widely employed in high-temperature zones of ultra-supercritical boiler units in China [13]. Consequently, its high-temperature performance has garnered significant interest.

Although HR3C steel has standardized chemical composition specifications (e.g., ASME Code Case 2115-1), microstructural variations exist between products from different manufacturers. Furthermore, the internal microstructure evolves continuously during service, making microstructural evolution and its impact on high-temperature performance critical concerns for industry and researchers [14-16].

This study investigates and compares the microstructural characteristics of two HR3C steels with different grain sizes, analyzes their microstructural evolution after long-term aging, and evaluates the effects of these microstructural changes on high-temperature properties using stress relaxation testing. The creep deformation behavior, creep stress exponent (n), and apparent activation energy (Q) are derived from the experimental data.

Experimental Materials and Methods

The experimental materials consisted of two ultra-supercritical HR3C heat-resistant steels with chemical compositions listed in Table 1. Both compositions complied with ASME Code Case 2115-1 standards, showing minimal differences in major elements. Figure 1 [Figure 1: see original paper] presents optical micrographs of the as-received microstructures. The steel with finer grains (Figure 1a) is designated F-HR3C, while the relatively coarse-grained steel (Figure 1b) is designated C-HR3C. Neither micrograph showed clear, continuous grain boundaries, attributed to sparse precipitates at grain boundaries in the as-received condition and resulting weak etching contrast.

To simulate long-term service microstructural evolution, as-received specimens were aged at 750 °C for 3000 h. Test specimens were machined according to GB/T 2039-1997 standards (5 mm diameter, 25 mm gauge length). Stress relaxation tests were conducted at 700, 750, and 800 °C with temperature control within ± 1 °C. The testing procedure followed GB/T 10120-1996: specimens were tensile-loaded at 0.002 mm/s to 0.4% plastic strain, then held constant for 20 h. A grating micrometer sensor ensured deformation control precision better than ± 0.0002 mm during relaxation.

Microstructural observation was performed using a MEF-3 optical microscope (OM). Stress relaxation data processing was conducted using Origin software.

When specimens reach predetermined strain (ϵ_t), the internal elastic strain (ϵ_e) gradually transforms into plastic strain (ϵ_p), causing continuous stress reduction according to the model [16,17]:

$$\epsilon_e + \epsilon_p = \epsilon_t = C$$

where C is a constant. Differentiating equation (1) with respect to time t yields the general stress relaxation equation:

$$\dot{\epsilon}_p = -\dot{\epsilon}_e = -\frac{1}{E} \frac{d\sigma}{dt}$$

where $\dot{\epsilon}_p$ is the plastic deformation rate (i.e., creep rate), $\dot{\epsilon}_e$ is the elastic deformation rate, $d\sigma/dt$ is the applied stress change rate, and E is the specimen's elastic modulus. Equation (2) converts stress-time relaxation data into creep rate-stress relationships.

2.1 Microstructural Evolution

Figure 2 [Figure 2: see original paper] shows optical micrographs of both HR3C steels after aging at 750 °C for 3000 h. Compared with the as-received condition, aged microstructures exhibit increased second-phase precipitation and more distinct grain boundaries. Literature [18] indicates that the solution-treated HR3C microstructure contains primarily simple tetragonal NbCrN precipitates (0.2-0.5 μm) within grains, with dislocation structures but no significant precipitation at grain boundaries. After 750 °C aging for 500 h, needle-like M₂₃C₆ precipitates (20 nm wide, 0.2-1 μm long) dominate within grains, accompanied by fine NbCrN and minor Nb(C,N) phases, while grain boundaries show extensive M₂₃C₆ precipitation and minor δ phase.

With extended aging, M₂₃C₆ and NbCrN precipitates gradually coarsen, albeit slowly. When aging exceeds 2000 h, 0.5 μm M₂₃C₆ precipitates form in the matrix and blocky δ phase appears at grain boundaries [19]. These abundant second-phase particles at both grain interiors and boundaries alter the HR3C microstructure and consequently affect high-temperature properties. The extensive grain boundary precipitation reduces corrosion resistance, enabling clear observation of grain boundaries in aged specimens. Grain size measurements using Image Pro Plus software revealed no significant change during aging: F-HR3C remained at approximately 17 μm , while C-HR3C measured about 40 μm .

2.2 Relationship Between Stress Relaxation and Creep

Figure 3 [Figure 3: see original paper] presents stress relaxation results for both steels in as-received and aged conditions. For F-HR3C (Figure 3a), residual

applied stress is inversely proportional to test temperature, decreasing with increasing temperature because higher temperatures facilitate thermal activation processes and accelerate dislocation motion, enhancing relaxation [20]. At identical temperatures, as-received and aged specimens show similar initial stresses but different relaxation magnitudes: aged specimens exhibit greater relaxation and lower residual stresses compared to as-received specimens. C-HR3C (Figure 3b) shows similar trends, with residual stress decreasing at higher temperatures and aged specimens relaxing more than as-received specimens.

Using equation (2), the stress-time data from Figure 3 were converted to creep rate-stress relationships, shown in Figure 4 [Figure 4: see original paper]. For F-HR3C (Figure 4a), creep rate-stress relationships are essentially linear, with creep rates increasing with temperature and decreasing with residual applied stress. The difference between as-received and aged specimens is small initially but becomes more pronounced during later relaxation stages as stress and creep rate decrease. C-HR3C (Figure 4b) exhibits similar linear creep rate-stress relationships, with rates proportional to temperature and residual stress, and diverging between conditions as stress decreases.

Comparing Figures 4a and 4b reveals subtle differences: during later relaxation stages, the creep rate difference between as-received and aged C-HR3C is smaller than that for F-HR3C. Figure 5 [Figure 5: see original paper] directly compares creep rate-stress relationships for both steels under identical conditions. Both as-received (Figure 5a) and aged (Figure 5b) conditions show that F-HR3C exhibits higher creep rates than C-HR3C under the same experimental conditions. The rate difference is small initially but widens as residual stress decreases with time. Additionally, as-received specimens show greater scatter in creep rate-stress curves than aged specimens, attributed to poorer microstructural homogeneity in the initial state, whereas long-term aging produces more uniform microstructures and smoother curves.

2.3 Creep Stress Exponent and Apparent Activation Energy

Most materials follow a power-law creep constitutive relationship where creep rate is a function of stress and temperature:

$$\dot{\epsilon} = A_1 \sigma^n \exp\left(-\frac{Q}{RT}\right)$$

where $\dot{\epsilon}$ is the steady-state creep rate, A_1 is a material- and temperature-dependent constant, n is the stress exponent, R is the gas constant, T is absolute temperature, and Q is the apparent activation energy for creep.

The linear creep rate-stress relationships in Figure 5 allow determination of stress exponent n from single relaxation curves at constant temperature. Results are plotted in Figure 6 [Figure 6: see original paper]. The as-received

F-HR3C shows a stress exponent of 7.1 ± 0.2 , decreasing to 5.4 ± 0.4 after aging. In contrast, as-received C-HR3C exhibits the highest stress exponent at 8.0 ± 0.7 , which decreases to 7.4 ± 0.3 after aging. The change magnitude is smaller for C-HR3C than for F-HR3C.

Generally, pure metals have $n \approx 3-5$, while the HR3C steels show higher values (5.0-8.7) due to dispersion strengthening, where fine second-phase precipitates dominate creep deformation resistance. The as-received HR3C contains primarily fine NbCrN precipitates, yielding higher n values. Aging introduces numerous M_2C , Nb(C,N), and δ phases, particularly at grain boundaries, coarsening the microstructure and degrading creep resistance, thereby reducing n . The more significant n reduction in F-HR3C reflects more severe microstructural coarsening.

The apparent activation energy Q characterizes the thermal activation process of deformation, representing the energy barrier for physical-chemical processes. Q is typically obtained from $\ln \dot{\epsilon}$ vs. T^{-1} relationships, shown in Figure 7 [Figure 7: see original paper]. Both steels show high Q values in the as-received condition that decrease after long-term aging. The reduction is more substantial for F-HR3C (199 kJ/mol) than for C-HR3C (142 kJ/mol). Combined with stress exponent analysis, these results indicate that C-HR3C possesses better stability of high-temperature creep resistance under identical service conditions.

Rearranging equation (3) by moving the exponential term yields an Arrhenius-type normalized equation where the right side becomes stress-dependent only:

$$\dot{\epsilon} \exp\left(\frac{Q}{RT}\right) = A_1 \sigma^n = P(\sigma)$$

Using equation (4), relaxation curves at different temperatures can be normalized onto a single data band to determine Q , as shown in Figure 8 [Figure 8: see original paper]. The Q values obtained from this method closely match those from $\ln \dot{\epsilon}$ vs. T^{-1} analysis. Combined results show as-received C-HR3C has $Q = (538 \pm 12)$ kJ/mol, decreasing to (405 ± 4) kJ/mol after aging, while F-HR3C has $Q = (547 \pm 9)$ kJ/mol, decreasing to (352 ± 5) kJ/mol. The change in Q for F-HR3C significantly exceeds that for C-HR3C.

Reported values for iron self-diffusion in 15Cr20Ni steel and lattice diffusion in 25Cr20Ni steel are (308 ± 9) kJ/mol and 270 kJ/mol, respectively [23,24]. The higher Q values (347-556 kJ/mol) for HR3C steels reflect dispersion strengthening by second-phase precipitates that enhances creep resistance. Normalization also effectively broadens the creep rate data range by approximately two orders of magnitude at low stresses through temperature compensation.

Discussion

The stress relaxation results and derived parameters (n and Q) correlate directly with HR3C microstructure. As a dispersion-strengthened steel, dislocation motion past precipitates governs creep rate. The as-received HR3C contains fine, dispersed NbCrN within grains and dislocation structures at grain boundaries without significant precipitation, providing strong boundary resistance to creep and resulting in slower relaxation, lower creep rates, and higher n and Q values.

Long-term aging precipitates abundant M₂₃C₆, NbCrN, and δ phases, particularly at grain boundaries, coarsening the microstructure and reducing boundary inhibition of creep [25]. Consequently, aged HR3C shows increased creep rates and decreased n and Q , indicating degraded high-temperature creep resistance.

C-HR3C exhibits superior creep resistance and stability because: (1) its larger grain size means less grain boundary area per unit volume, resulting in fewer boundary precipitates after aging and thus less degradation of creep resistance –this is the primary factor; and (2) F-HR3C contains more numerous larger second-phase precipitates in the matrix. Despite similar compositions, the two steels show distinct stress relaxation behaviors, with F-HR3C exhibiting higher creep rates and poorer creep resistance stability.

Conclusions

1. Two HR3C heat-resistant steels with similar as-received compositions but different grain sizes were investigated. Stress relaxation testing revealed that coarse-grained HR3C steel possesses better creep resistance with lower creep rates, while fine-grained HR3C steel shows higher creep rates and poorer resistance. The coarse-grained steel exhibited a stress exponent of 8.0 ± 0.7 and apparent activation energy of (538 ± 12) kJ/mol, compared to 7.1 ± 0.2 and (547 ± 9) kJ/mol for the fine-grained steel.
2. After long-term aging, both steels maintained similar grain sizes but developed extensive second-phase precipitation within grains and at boundaries, reducing both stress exponent and apparent activation energy. The coarse-grained steel showed values of 7.4 ± 0.3 and (405 ± 4) kJ/mol, while the fine-grained steel showed 5.4 ± 0.4 and (352 ± 5) kJ/mol. The coarse-grained HR3C exhibited smaller changes in these parameters, demonstrating more stable creep resistance, whereas the fine-grained variant showed larger variations and less stable creep resistance.

References

- [1] Sha J J, Park J S, Hinoki T, Kohyama A. Mech Mater, 2007; 39: 175
- [2] Zhao J. Statistical Analysis and Reliability Prediction on the Creep Rupture

- Life of Heat Resistant Steel. Beijing: Science Press, 2011:8
- [3] Dotsenko V I. Phys Stat Sol, 1979; 93B: 13
 - [4] Woodford D A. JSME International Journal, 2002; 45A: 98
 - [5] Ek C G, Hagström B, Kubát J, Rigdahl M. Rheol Acta, 1986; 25: 534
 - [6] Woodford D A, Wereszczak A A, Bakker W T. J Eng Gas Turbines Power, 2000; 122: 206
 - [7] Holm Altenbach, Konstantin Naumenko. Int J Mod Phys, 2008; 22B: 5413
 - [8] Chandler H D. Mater Sci Eng, 2010; A527: 6219
 - [9] Beddoes J. J Strain Anal Eng Des, 2011; 46: 416
 - [10] Zhan L H, Yang L. J Plastic Eng, 2013; 20: 126
 - [11] Guo J Q, Xuan F Z, Wang Z D, Tu S D. Proc Chin Soc Electrical Eng, 2009; 29: 92
 - [12] Raghavender R G, Gupta O P, Pradhan B. Int J Pres Ves Pip, 2011; 88:65
 - [13] Li T J, Liu F G, Fan C X, Yao B Y. Hot Work Technol, 2010; 39: 43
 - [14] Liu J W, Luo C P, Xiao X L, Chen H X. Acta Metall Sin, 2002; 38: 127
 - [15] Iseda A, Okada H, Semba H, Igarashi M. Energy Materials, 2007; 2: 199
 - [16] Guo J Q, Xuan F Z, Wang Z D, Tu S D. Nucl Power Eng, 2009; 30:9
 - [17] Zhu Z, Zhang L W, Gu S D. Chin J Nonferrous Met, 2012; 22: 1063
 - [18] Fang Y Y, Zhao J, Li X N. Acta Metall Sin, 2010; 46: 844
 - [19] Fang Y Y. Master Thesis, Dalian University of Technology, 2010
 - [20] Tan J, Li C, Sun C, Ying S H, Lian S S, Kan X W, Feng K Q. Acta Metall Sin, 2009; 45: 173
 - [21] Yan W Z, Gao H S, Yue Z F. Rare Met Mater Eng, 2013; 42: 1250
 - [22] Zhang J S. High Temperature Deformation and Fracture of Materials. Beijing: Science Press, 2007: 56
 - [23] Rothman S J, Nowicki L J, Murch G E. J Phys , 1980; 10F: 383
 - [24] Ruano O A, Wadsworth J, Sherby O D. J Mater Sci, 1985; 20: 3735
 - [25] Kong Q P, Dai Y. Mater Sci Prog, 1988; 2: 1

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