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Effect of rare earth alloying on creep rupture of economical 21Cr-11Ni-N heat-resistant austenitic steel at 650 °C

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Abstract: The effect of rare earth (RE) on creep rupture of economical 21Cr-11Ni-N heat-resistant austenitic steel was investigated at 650 °C under different stress levels. It was found that RE could increase the time to creep rupture, especially at long-term creep duration. The logarithm of the time to creep rupture ($\lg t_r$) was a linear function of the applied stress (σ). RE addition was favorable to generating a high fraction of low-coincidence site lattice (CSL) boundaries which was a possible cause for improving the creep rupture resistance. The fracture surface of RE-added steel exhibited less intergranular cracks suggesting the alteration on the nature of grain boundaries due to the presence of RE. RE addition changed the morphology of the intergranular chromium carbides from continuous network shape to fragmentary distribution which was another cause for longer creep duration. These results strongly suggested that the effect of RE alloying played a crucial role in improving the creep rupture resistance.

Keywords: heat-resistant austenitic steel; rare earths; alloying; creep rupture

Heat-resistant austenitic steels are commonly used in the power generation industry at temperatures higher than 650 ºC and stresses of 50 MPa or more[1]. As always, the alloy design concepts for the material are obtaining better creep rupture resistance and lowering the material cost[2]. The type 21Cr-11Ni-N steel is just a cost effective lean alloy on the basis of the design concepts. Although this novel alloy contains just 11% Ni, it can offer the potential for heat resistant properties competitive with some heat resistant alloys, such as 309S (13% Ni), 310S (20% Ni) stainless steels and even 330 (35% Ni) nickel alloy[3], but at much lower cost. Substantial improvement on heat resistant properties of this alloy is made by precise control of micro alloy additions, especially the rare earths elements (RE, such as Ce, Y) combined with N.

Being a strong austenite stabilizer, N will change stacking fault energy and hence the dislocation structure will also be changed, leading to changes in creep resistance. Effects of N have been looked into in several recent publications[2,4,5]. RE, as the active element, has also been found to improve creep cavitation resistance in austenitic steels, particularly by co-addition with B[2,4,6]. Ce is found highly effective in removing O and S in 347H austenitic steel, forming Ce_2O_2S, such that more B may segregate to cavities[4]. RE is also believed to tie up the S segregated at grain boundaries (GBs), preventing S-induced embrittlement[3,7]. The above mentioned studies on the RE effect have mainly focused on modification of inclusions and deep purifying. Actually, RE can show a subtle role as an alloy element under high purification metallurgical condition, particularly segregation effects at the GB or/and the interface[3,8,9]. On the other hand, generally, since the GBs of heat resistant alloys become weak as temperature increases, appropriate methods should be employed to strengthen the GBs at high temperatures[2]. Presumably, RE shows some sort of GB strengthening effects in RE doped 21Cr-11Ni-N steel. However, the details why the micro-alloyed RE improves the creep rupture properties are still unclear. The aim of this work thus was set to highlight the effects of RE alloying on creep rupture of the novel 21Cr-11Ni-N steel, especially from a viewpoint of modification of GB. Also, more fundamental data on heat resistance of the austenitic steel can be obtained.

1 Experimental

The nominal compositions of the test 21Cr-11Ni-N steel are as follows (wt.%): 0.089% C, 20.88% Cr, 11.07% Ni, 1.38% Si, 0.76% Mn, 0.17% N, 0.0015% S, 0.02% P, and Fe in balance. In order to eliminate or at least minimize the difference on other main compositions

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except RE in tested steels, a furnace of molten steel was poured into two crucibles for solidification. One of them, designated as HRSS1, was added with the RE alloys (RE>99 wt.%, rich in Ce) during pouring, the other one was designated as HRSS0 without RE. The total RE content (RE_t) in HRSS1 was about 0.05%, and the RE solution content (RE_{sol}) obtained by ICP optical emission spectrometer was only 0.0063%. The two cast billets were reheated to 1200 ºC for 60 min, hot rolled from 50 to 12 mm. The materials then underwent solution heat treatment at 1050 ºC for 20 min, and final water quenching.

Specimens for creep rupture test were taken from the plates perpendicularly to the rolling direction. The cylindrical specimens with 5 mm gauge diameter and 25 mm gauge length were prepared in accordance with the National Standard of the PR China, GB/T2039-2012. Creep rupture test were carried out at 650 ºC/300 MPa, 250, 200, 150, 100 MPa. Accordingly, the thermal aging test at 650 ºC was carried out for corresponding microstructure evaluation.

The initial microstructure was analyzed through electron backscattering diffraction (EBSD) at the acceleration voltage of 20 kV and the raw data were post-processed using TSL OIM software. The fracture surfaces and the detailed microstructurical characteristics of the ruptured specimens were examined using scanning electron microscopy (SEM) combined with energy dispersive spectroscopy (EDS) and electron probe micro-analyser (EPMA). Some detailed analysis on the microstructure was examined using transmission electron microscopy (TEM) and selected area electron diffraction (SAED).

2 Results and discussion

2.1 Analysis of creep-rupture properties

The results of the creep tests at 650 ºC in the steels studied are summarized in Table 1. It can be seen that the time to rupture of both tested steels increases as stress decreases. Clearly, the creep rupture life of HRSS1 is longer than that of HRSS0 at the same stress level. Moreover, the longer the creep duration (namely, the lower the stress level), the bigger the difference of rupture life gap, for example, RE addition doubles the time to rupture at 100 MPa. This means that the discrepancy of material deterioration in the two tested steel may appear at a low stress level and a long time.

Based on numerous creep rupture data of several heat resistant steels, Manabu et al.[10] developed a new precise relation among the time to creep rupture, the stress and the temperature through assuming the velocity of dislocation being controlled by a thermal activated process and the equation can be expressed as:

$$\sigma = \frac{Q}{V} - \frac{2.3RT}{V}(\lg t_r - \lg t_{r0}) \quad (1)$$

where t_r is rupture life, Q is the activation energy, V is the activation volume which is a proportional factor to the stress, T is the temperature, σ is applied stress, R is the gas constant, t_{r0} can be set as constant and the value of ($-\lg t_{r0}$) is approximately equal to Larson-Miller constant (about 20)[10]. It is clear that the logarithm of the time to creep rupture, $\lg t_r$, is a linear function of the applied stress, σ. According to the Eq. (1), the linear relation between σ and $\lg t_r$ can be obtained, as shown in Fig. 1. The value of slope of the fitting line, $-d\sigma/d\lg t_r$ ($=2.3RT/V$), decreases with RE addition, indicating a less accumulation of creep damage with creep exposure in the RE-added steel leading to higher creep strength. By regression calculation, the materials constants and quantitative equation are listed in Table 2. It can be seen that the Q and V, which are strongly affected by deformation mechanism and/or microstructure[10], in HRSS1 are higher than that in HRSS0. This suggests that the average dislocation velocity in the RE-added steel is slow, leading to reduced creep rates, namely the RE addition can retard the creep rupture. Corresponding microstructural mechanism will be discussed afterward.

2.2 Possible mechanism of beneficial effects of alloyed RE

2.2.1 Analysis of initial microstructure

It is known that the initial grain size may cause sig-

Table 1 Rupture life (h) of the tested steels at different stresses

Steels	Applied stress/MPa				
	300	250	200	150	100
HRSS1	54	120	323	1337	6893
HRSS0	49	95	267	905	3463
Δt_r	5	25	56	432	3430

Table 2 Material constants and quantitative equation of the tested steels

Steel	Material constants		Quantitative equation
	V/(mm^3/mol)	Q/(kJ/mol)	
HRSS1	190	438	$\sigma=-93.03\lg t_r+447.19$
HRSS0	168	430	$\sigma=-105.29\lg t_r+465.12$

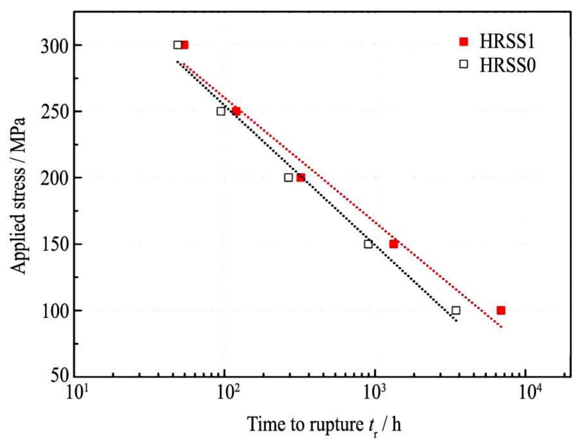

Fig. 1 Linear relation between σ and $\lg t_r$