Cold Working Effect on Creep Strength of AISI 347 Stainless Steel

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Abstract

The effect of 5, 10 and 15% cold working on creep strength of AISI 347 stainless steel was investigated at 650 and 700 °C, with initially applied stresses of 196, 157 and 127 MPa. The relationship between minimum creep rate, applied stress and temperature was derived from the best fitting of experimental points. The stress exponent, n , varied from 4.0 to 5.7, and the activation energies ranged from 450 to 496 kJ/mol. The effect of cold working on creep rates, rupture times and ductilities were also studied. At 196 and 157 MPa, the 10% cold worked samples showed the lowest values both in ductility and in creep rate, and accordingly the rupture time was the maximum. The influence of cold working levels on strength and rupture behaviour was related to ageing processes and to dynamic restoration.

Some preliminary observations were performed by TEM; no particular differences were revealed in the microstructure of the specimen crept at 650 °C under various stresses or after different prestrain.

Introduction

The need for higher working temperatures in power plants is behind the development of alloys with improved performances in corrosion and creep resistance. The stabilized stainless steels AISI 321, 347 and 348 are candidates for structural components in superheater (Sh) and reheater (Rh) banks, owing to their high temperature stability. The construction of Rh and Sh banks imposes the bending of the tubes, introducing high levels of cold work that could condition the high temperature properties of structural materials.

In stabilized stainless steels, the creep strength is affected by the balance of Nb and Cr, which at low temperatures is responsible for ageing hardening. The precipitation of fine Nb carbides on grain boundaries during stationary creep obstructs the grain sliding and extends the rupture time /1,2/. In creep of 25Cr-25Ni-Nb stabilized stainless steel, a large reduction of minimum creep rate and ductility was attributed to high dislocation density and cavitation, respectively, produced by 25% cold work /3/.

After ageing the AISI 347 stainless steel, 30% cold

worked (CW) specimens exhibited limited increases in microhardness, whereas more severe CW values produced softening due to recovery /4/.

The aim of this research is to investigate the effect of different cold work levels on creep behaviour of the niobium stabilized stainless steel AISI 347.

Materials and Experimental

Constant temperature and constant load creep rupture experiments were done on specimens machined from tubes of the following dimensions: 51 mm outer diameter, 7.2 mm wall thickness. The composition of the alloy was as follows (wt%): 17.3 Cr, 10.4 Ni, 0.048 C, 0.52 Nb and <0.01 Ta.

The creep specimens were cylindrical and their gauge length was 50 mm, with a 5 mm diameter. Creep tests were performed on as received as well as on pre-deformed specimens. The pre-straining was performed on a tensile machine at room temperature to 5, 10 and 15% deformation. The creep tests were carried out at 650 and 700 °C under stresses of 127, 157 and 196 MPa. Temperatures varied within ± 3 °C of the test values; strain measurements were performed using LVDT (linear variable differential transformers). The linearity error in elongation measurements was < 0.2%.

Results

Figs. 1a-1d show various creep curves obtained at different temperatures under different applied stresses; the data marks represent experimental results, while the curves were fitted by using the θ projection concept /5/ modified with the addition of the $\epsilon_{\text{cut off}}$ method /6/. The $\epsilon_{\text{cut off}}$ method takes into account the reduction of sample diameter due to cavitation and necking by erasing the last point in the fitting experimental data. The strain corresponding to $t = 0.8t_{\text{rupt}}$, where t_{rupt} is the rupture time, was arbitrarily assumed as ϵ_{co} . The last part of the creep curves is not shown in Fig. 1, owing to different tertiary behaviour typical of stainless steels in constant stress and constant load experiments /7/.

The proposed equation is:

Fig. 1: Effect of cold work on creep curves at different test conditions: a) 196 MPa, 700 °C; b) 196 MPa, 650 °C; c) 157 MPa, 650 °C; d) 127 MPa, 650 °C.

(♦) as received; (■) c.w.5%; (▲) c.w.10%; (●) c.w.15%.

$$\epsilon = \theta_1(1 - e^{-\theta_2 t}) + \theta_3(e^{\theta_4 t} - 1) + \epsilon_0 \quad (1)$$

where θ_i ($i = 1, 4$) depends on test conditions through the following equations:

$$\ln(\theta_i) = a_i + b_i T + c_i \sigma + d_i \sigma T$$

$$i = 1, 4$$

where T is the temperature and σ is the applied stress. The minimum creep rate ϵ_m , (Fig. 2) was calculated by deriving Eq. 1 and substituting the time t_m , transition time from primary to tertiary stages, defined as follows:

Fig. 2: Variation of MCR with the amount of cold work for different test conditions: 700 °C - 196 MPa (●); 650 °C - 196 MPa (○); 650 °C - 157 MPa (Δ); 650 °C - 127 MPa (◇).

$$t_m = \left(\frac{1}{\theta_2 + \theta_4} \right) \ln \left(\frac{\theta_1 \theta_2^2}{\theta_3 \theta_4^2} \right) \quad (3)$$

All creep curves in Figs. 1a-1c are typical three-stage creep curves, with relatively short primary and secondary regions. Table 1 lists the values of the minimum creep rate (MCR) and t_m for the various creep experiments. In general, pre-straining lowers the minimum creep rate and increases creep life. The lowest MCR and longest t_r were obtained for the 10% pre-strained specimens, except for the very low load experiments. It is to be noted that 10% prestraining extends creep life by an order of magnitude compared to that of annealed specimens. The apparent activation energy for creep, Q , and the stress sensitivity parameter, n , were calculated by means of the following equation:

$$\dot{\epsilon}_m = A \sigma^n e^{\left(\frac{-Q}{RT} \right)} \quad (4)$$

where A is material constant and R has the usual meaning.

The n values at 650 °C were 5.7, 4.7 and 4 for 5, 10 and 15% CW, respectively, and the activation energy ranges from 496 to 450 kJ/mol. These results agree very well with data reported in the literature [8].

Microstructure

Preliminary observation performed by TEM showed a regular three-dimensional network of dislocations and a high number of fine NbC particles. A rather rough estimation of dislocation densities is summarized in Table 2; a tendency for dislocation density to increase with cold work level can be observed, even if no dependence on creep load occurs. The homogeneity of particle densities increases with increasing cold deformation.

Three principal types of particles were revealed in the crept specimens:

- Type 1: coarse intergranular particles (Fig. 3)
- Type 2: coarse intragranular particles having an

Table 1
Effect of c.w. and test conditions
on MCR and time t_m

c.w. %	T °C	σ MPa	$\dot{\epsilon}_m$ %/h	t_m h
0	650	196	.01	6
5	650	196	.0019	270
5	650	196	.0023	305
10	650	196	.00078	289
15	650	196	.00095	228
15	650	196	.00091	203
5	650	157	.00039	508
10	650	157	.00020	312
15	650	157	.00040	341
5	650	127	.00017	111
10	650	127	.00016	120
15	650	127	.00013	93
5	700	196	.0425	10
10	700	196	.0216	27
15	700	196	.0236	28

Table 2
Estimation of dislocation densities (10^{14} m^{-2})
in specimens crept at 650 °C at
various stresses after different cold work

c.w. %	127 MPa	157 MPa	196 MPa
0	-	-	3.2
5	3.0	2.5	3.5
10	4.8	-	4.0
15	4.8	3.2	5.6

Fig. 3: Coarse intercrystalline precipitates observed by TEM in 15% c.w. specimen crept at 650 °C and 157 MPa.

Fig. 4: Coarse equiaxial intracrystalline precipitates observed by TEM in 5% c.w. specimen crept at 650 °C and 127 MPa.

Fig. 5: Fine precipitates observed by TEM in 5% c.w. specimen crept at 650 °C and 127 MPa.

Fig. 6: Fine precipitates observed by TEM in 10% c.w. specimen crept at 650 °C and 196 MPa.

equiaxial shape (Fig. 4)

- Type 3: very fine particles precipitating mainly on dislocations (Figs. 5 and 6)

While particles of types 1 and 3 are present in all specimens, type 2 particles occur predominantly in specimens having time to rupture longer than 500 h.

Discussion

The fact that n and Q fit very well the reported values for stainless steels in literature indicates that the basic creep deformation mechanism in this steel is not different from that of the others [8]. On the other hand, slight plastic deformation by pulling at room temperature has a significant influence mainly on the minimum creep rate. It is suggested that the significant improvement of creep behaviour due to cold work is to be attributed to the precipitation of niobium carbides during creep.

Fig. 7 shows the plot for the Monkman-Grant relationship. These results show that prior cold work does not produce considerable damage or cavity nucleation, and pre-straining does not introduce qualitative changes in creep deformation mechanisms, even if, according to Ahn et al. [9], these conclusions can change drastically with the variation in testing procedures.

During creep at elevated temperatures, carbides precipitate and cause pinning of dislocations [10-12]; the pinning not only increases creep strength, but also stabilizes the dislocation network. Higher dislocation densities in pre-deformed specimens present better conditions for particle nucleation. Microstructure observations reveal a regular three-dimensional network of dislocations and a high number of particles precipitated on the dislocations. Both dislocation and particle densities increase with the increasing cold work level before creep test. On the other hand, prolonged holding at high temperatures results in carbide growth and dislocation recovery. Cold work promotes both nucleation and growth of carbides. Experimental results of decreasing stress exponent with increased particle density (Table 4) contradict Senior's ideas based on threshold stress caused by an irrecoverable dislocation network pinned by fine

Fig. 7: Monkman-Grant relation: symbols represent different level of cold work: (♦) c.w.0%; (■) c.w.5%; (▲) c.w.10%; (●) c.w.15%.

Table 3
NbC particle sizes (nm) in specimens crept at 650 °C under various stresses after different cold work

c.w. %	127 MPa	157 MPa	196 MPa
0	-	-	9.0
5	8.5	6.0	8.7
10	8.0	-	8.5
15	7.0	5.5	-

Table 4
NbC particle densities (10^{21} m^{-3}) in specimens crept at 650 °C and at various stresses after various pre-straining

c.w %	127 MPa	157 MPa	196 MPa
0	-	-	2.0
5	1.4	1.8	2.2
10	1.9	-	2.5
15	2.2	2.5	-

particles /13,14/. In Fig. 2, it is possible to observe that differences in creep behaviour tend to reduce at lower stresses.

It seems that an optimum amount of cold work exists which results in the most stably pinned dislocation network and the highest creep strength (corresponding to the lowest MCR, Fig. 2). This amount of pre-deformation is temperature and stress dependent, and 10% cold work is the optimum one at 650 °C.

Conclusions

The results of the interpolation of the experimental data and the microstructural study can be summarized as follows:

- Creep characteristics of cold worked AISI 347 are similar to those typical for other stainless steels. The Monkman-Grant equation is valid and not influenced by pre-straining level; creep strength is controlled by creep strain processes, and no important microstructural damage occurs after cold working.
- Cold deformation does not alter significantly creep constants n and Q , even if at high stresses (196 and 157 MPa); the maximum strengthening effect can be observed in 10% pre-strained specimens; further pre-straining produces an increase in minimum creep rate.
- Difference in creep strength between "stabilized" and cold worked conditions are less important at lower stresses used in industrial applications. The influence of pre-straining on creep strength seems to be non-linear at high stresses.

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