Tensile strength and creep behaviour of austenitic stainless steel type 18Cr - 12Ni with niobium additions at 700°C

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Abstract. The effect of niobium additions up to 2.36 wt% on the creep behavior of a series of seven extra low carbon 18Cr-12Ni austenitic stainless steels at 700°C has been investigated. Grain size and hardness measurements, hot tensile tests and constant stress creep tests from 90 to 180 MPa were carried out for each alloy, in the solution treated condition at 1050, 1200 and 1300°C followed by quench in water. The mechanical behavior at high temperature was related to the amount of NbC precipitation occurring during the tests. Solid solution and intermetallic compound effects were also considered. Creep data analysis was done to determine the parameters of the creep power-law equation \( \dot{\varepsilon} = A \sigma^n \) and the Monkman-Grant relation \( \dot{\varepsilon} t^n = K \). Niobium-carbide precipitation in these steels reduces the secondary stage dependence of strain rate with applied stress, resulting in \( n \)-values which indicate the possibility of operation of various creep mechanisms. The creep strength during the secondary stage is primarily controlled by the amount of NbC available for precipitation. However, the rupture times increase progressively with niobium content, as the amount of undissolved carbide particles in grain boundaries and the Laves phase precipitation increase.

1. Introduction
Niobium can be added to austenitic stainless steel as to stabilize carbon in austenite, reducing the formation of chromium carbides at the grain boundaries during high temperature service. Thus it is possible to avoid the losses in corrosion resistance, and the tensile strength can be improved by the solid solution strengthening effect of niobium in the austenitic matrix. Creep rupture life can be improved by the precipitation of niobium carbides on dislocations. This effect is generally followed by some ductility loss, since deformation is concentrated at or near the grain boundaries by the high strength of the matrix. Depending on solution treatment conditions and on chemical composition of the steel, formation of intermetallic compounds, like \( \sigma \)-phase and Fe\(_2\)Nb, may occur [1,2,3]. In this work it was considered that niobium carbide solubility in austenite can be described by the relation: Log (Nb.C) = 3.3 – 7200/T, proposed by Minami and Kimura [4,5], and that the composition of niobium carbide is next to Nb(CN), whose stoichiometric relation is Nb/C\(_{eq}\) = 7.74, where C\(_{eq}\) = (C + 6/7N). Thus, it was possible to estimate the quantities presented in Figure 1. The mechanical properties were evaluated by tensile tests and constant stress creep tests at the temperature of 700°C. Creep data analysis was made by determining the parameters of the creep power-law.
equation $\dot{\varepsilon} = A\sigma^n$ and the Monkman-Grant [6] relation $\dot{\varepsilon}.t_r^m = K$, where $\dot{\varepsilon}$ is the minimum creep rate, $\sigma$ is the applied stress and $t_r$ is the rupture time and $A$, $n$, $m$ and $K$ are constants to be determined.

2. Experimental

Seven alloys were considered in this research with chemical composition indicated in Table 1. They were prepared by vacuum melting and submitted to hot rolling to produce plates with about 10 mm thickness. The material was solution-treated at the temperatures of 1050, 1200 and 1300°C, for 1 hour in argon atmosphere followed by quench in water. Hilliard grain size and Vickers hardness measurements, hot tensile and constant stress creep tests were carried out at 700°C. Screw-ended specimens with diameter / gage length ratio of 4 mm / 20 mm and 5mm / 50 mm were used for the tensile and creep tests, respectively. The applied stresses for creep ranged from 90 to 180 MPa, producing rupture times varying from about 0.1 to 5000 h.

Table 1. Chemical compositions of the steels

| Alloy | C (%) | Si (%) | Mn (%) | Cr (%) | Ni (%) | Nb (%) | Nb/Ceq |
|-------|-------|--------|--------|--------|--------|--------|--------|
| A1    | 0.020 | 1.02   | 1.16   | 18.24  | 11.00  | zero   | 0      |
| A2    | 0.014 | 0.71   | 0.91   | 17.80  | 11.80  | 0.22   | 11.6   |
| A3    | 0.020 | 1.00   | 1.13   | 18.80  | 11.20  | 0.52   | 16.8   |
| A4    | 0.026 | 0.73   | 0.97   | 18.13  | 12.84  | 0.91   | 24.6   |
| A5    | 0.020 | 1.02   | 1.21   | 18.20  | 11.10  | 1.14   | 32.6   |
| A6    | 0.016 | 0.92   | 1.04   | 17.80  | 13.20  | 1.78   | 55.6   |
| A7    | 0.020 | 1.09   | 1.11   | 17.99  | 13.20  | 2.36   | 63.8   |

3. Results and discussion

As shown by Figure 2, hardness increases with niobium content, what could be attributed to the solid solution hardening effect caused by niobium in the austenitic matrix. However, the hardness of steels with high amounts of niobium is more sensible to solution treatment temperature than the hardness of steels with lower additions, in opposition to what happens with grain size. Therefore, the lower hardness observed for steels solution-treated at 1300°C cannot be justified only by grain size, since grain size presents only a light dependence on the solution treatment temperature for steels with high niobium contents, where hardness varies in a more noticeable way. On the other hand, steels with low niobium contents show great variation in grain size, and practically stable hardness. Increases in hardness for a certain temperature of solution treatment temperature also cannot be explained only by the quantities of niobium in solid solution, because this would imply greater hardness for steels solution-treated at higher temperatures, in opposition to what has been observed. It is possible, therefore, that the presence of undissolved carbides and undissolved intermetallic particles of Fe$_2$Nb (Laves phase), is the main factor controlling the hardness of these alloys in the solution-treated condition, as already suggested by other authors [7-10].
Figure 3 shows that, for the steels with low niobium additions, the ultimate tensile stress is lower when the solution-treatment is made at 1300 °C, but, as the amount of niobium increases, this situation is reverted. For yield stress this trend does not repeat. Tensile strength seems to result from several effects: the amount of niobium dissolved in the austenitic matrix, the amount of NbC available for precipitation and the amounts of undissolved Fe2Nb and NbC particles. Comparing Figure 3 to Figure 1, yield stress seems to depend specially on the presence of undissolved particles and on the amount of niobium dissolved in the matrix. Carbide precipitation is strongly accelerated by plastic deformation and, therefore, might have greater influence on the ultimate tensile stress than on the yield stress. It was also observed that the level of deformation at maximum load dropped from 25% for sample A1 (0% Nb) to 15% for sample A7 (2.36% Nb) and that, in general, solution-treated alloys at 1300 °C presented the lowest values of final elongation. Denham and Silcock [9] had even observed that the presence of undissolved Fe2Nb increases the total elongation at 700 °C, but they also pointed out that maximum loads were obtained at too low deformations levels, and great part of subsequent deformation was attributed to the formation of cracks at grain boundaries.

Figure 4 shows the trend of increase in creep rupture time with increase of niobium content. The lowest strain rates at secondary creep stage are obtained for Nb/Ceq relations next to 20, matching with the maximum niobium carbide availability for precipitation indicated in Figure 1. Figure 5 presents the values of the parameters $n$ and $m$ for the equations $\dot{\varepsilon} = A \sigma^n$ and $\dot{\varepsilon}_R = A' \sigma^m$, respectively, as function of Nb/Ceq relation. For alloys with low Nb/Ceq ratio, the values of $n$ decreases as the solution-treatment temperature decreases. Increasing the Nb/Ceq ratio means obtaining smaller and smaller grain sizes, less and less dependent on solution-treatment temperature. At the same time, the possibility of niobium carbide precipitation increases, reaching its maximum level around the Nb/Ceq ratio of A4 alloy, and turning down again for greater Nb additions (See Figure 1). From a certain level of addition, creep process is supposed to be controlled by dislocation climb over precipitated particles, reducing the dependence of strain rate on applied stress. Thus, the minimum $n$-values obtained for the alloys with more niobium carbide available for precipitation can be explained, as well as the increase of $n$-values with the decrease of solution-treatment temperatures for the alloys with high addition of niobium. The fact that the $n$-exponent decreases, tending to 1, as the precipitation level increases, may indicate more and more participation of mechanisms which involve diffusion creep in controlling secondary stage strain rates.

A trend of decrease in the values of the $m$-exponent in Figure 5 can be observed for Nb/Ceq rates that promote niobium carbide precipitation, namely, the A3, A4 and A5 alloys. Low $m$-values means that very different rupture times can be obtained with small variations of secondary stage creep rates. Decreasing $m$-values suggest some loss in ductility and major contribution of secondary stage to the
total rupture time. The greatest rupture times obtained in this work correspond to the alloys with highest Nb additions, but, according to the adopted solubility relation, these alloys do not present the highest amounts of carbide available for precipitation during the test. In fact, these long rupture times were obtained due to a major participation of tertiary stage in the creep test, because of the presence of undissolved Fe$_2$Nb and NbC particles at the grain boundaries. These particles are well known to help preventing cracks at the grain boundaries from propagating, resulting in an apparent increase in total creep ductility [1,9].

![Figure 5](image.png)

4. Conclusions
The reduction in grain size and the increase in the amounts of niobium dissolved in the matrix contribute to the increase in hardness of the studied steels. However, the main hardness-controlling factor is the presence of undissolved NbC and Laves phase particles, which overcome the effects of grain size and niobium into solution.

The lowest secondary stage creep rates occur for the alloys which present higher amounts of niobium carbide available for precipitation during creep test. The greatest rupture times are related to the alloys which presented higher amounts of undissolved phases after the solution treatments.

The presence of undissolved Fe$_2$Nb and NbC particles makes crack propagation at grain boundaries more difficult, resulting in an increase in rupture time and apparent ductility.

Niobium additions with distinct Nb/Ceq relations and the use of different solution temperatures resulted in a large variations in the $n$ values, in the range from about 1 to 12, which indicate the possibility of operation of various creep mechanism in these steels.

The lower $n$ values were obtained for the conditions where the amounts of niobium carbide available for precipitation are higher, which indicates predominance of the mechanism of diffusion creep, where $n = 1$. A trend of decrease in the values of the $m$-exponent can be observed for Nb/Ceq rates that promote niobium carbide precipitation, which suggest some loss in ductility and major contribution of secondary stage to the total rupture time.

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