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CREEP BEHAVIOR OF TYPE 310 STAINLESS STEEL. PART 1: PARAMETERS FROM THE NORTON, ARRHENIUS AND MONKMAN-GRANT RELATIONS[1]

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Abstract

Creep behavior of AISI 310 stainless steel was studied by constant stress tests in the range from 70 to 375 MPa, at 700ºC. The variation of the initial strain rates, secondary creep rates and rupture time with stress were observed in tests carried out without interruption until the end of tertiary stage. For the secondary stage creep rates, Norton relation was verified with an exponent **n**=4.5 from 70 to about 130 MPa and **n**=7.1 from about 130 to 250 MPa. The initial strain rates were also verified to follow Norton relation with two **n** exponents having values similar to the secondary stage. Some creep tests were also carried out at other temperature levels between 600 and 700ºC. Monkman-Grant relation was verified to hold, with **m** very close to 1. In another set of experiments, activation energy values were determined by small temperature changes (±10ºC) in 3 steps above and below 700ºC, during secondary stage at various stress levels from 70 to 210 MPa. The average apparent activation energy value in this stress range was determined to be $\mathbf{Q_c}$=307+47 kJ/mol. The values of all these creep parameters agree well with those found for other versions of austenitic stainless steels of the 300 series. Small stress variations were also performed at strain levels above and below the establishment of minimum creep rate (secondary creep stage) for a test at 150 MPa, 700°C. A trend for increase in **n** and decrease in **A** values with creep strain around secondary creep stage was observed.

Keywords: Type 310 stainless steel; Creep behavior; Stress/temperature changes; Creep parameters.

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1 INTRODUCTION

For a great number of metallic systems the creep rate in secondary stage at high temperatures can be described by an expression of type:

$$\dot{\varepsilon}_s = A.\, \sigma^n .\, \exp(-\, Q_c \,/\, RT) \qquad (1)$$

where **A** and **n** are constants depending on the material and test conditions, **σ** is the applied stress, Q_c the apparent activation energy for creep, **R** the universal gas constant, and **T** the absolute temperature.[1,2] The coefficient **A** includes the elastic modulus of the material and when its dependence on temperature is considered, in the case of pure metals and solid solution alloys, the value obtained for Q_c = Q_{sd} = self diffusion activation energy of the base metal.[3] In these cases, the stress exponent **n** exhibits values varying from 3 to 5.[1,2]

For complex alloys, however, values of **n** are generally much greater than 5. For precipitation hardened steels and superalloys, for instance, values varying from 5 to 10 have been obtained, and for dispersion hardened alloys values higher than 10 up to 40 have been reported.[1,2]

In certain cases the stress exponent may assume two or three distinct values at different stress ranges, or it can be show dependence with temperature. On the other hand, measurements of creep activation energy in complex alloys have revealed values of Q_c much higher than Q_{sd} , being, in various situations, also stress dependent.[1,2]

It has been demonstrated that the high values of the stress exponent and activation energy can be understood when consideration is made for the effect of retard exerted by second phase particle in the process of recovery during creep. It has been also generally accepted that deformation at high temperatures does not occur under the effect of the total applied stress, but under the effect of an effective stress given by the difference between the applied stress and a certain level of internal stress related to the micro-structural state of the material and the substructure generated by the deformation process during creep.[1]

This article describes results involving basically measurements of the stress exponent **n**, activation energy Q_c, and other creep parameters, for stainless steel AISI 310, basically at the temperature of 700°C, as part of a general program for the study of creep behavior in this steel, considering the concepts involving the participation internal stress in the process. In the two subsequent articles (Part 2 and Part 3) of this work, this question is focused more specifically.

A preliminary article on the present subject was already published some time ago by the same authors.[4] A set of new data were added to the original results and a more extensive analysis is presented on the subject in the present article.

2 MATERIALS AND METHODS

The steel was manufactured by a company in Brazil and was supplied in the form of hot rolled bars, with ½" diameter, with the following composition: 24.0 Cr – 21.1 Ni – 0.11Si – 1.60 Mn – 0.17 C (% in weight). The material was solution treated at 1050°C, for 1 h, quenched in oil, after which exhibited a grain structure with size of the order of 210 μm. Before the creep tests, a set of hot tensile tests was carried out using an INSTRON Universal Machine model 8802.

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The tensile tests were conducted under constant crosshead speed, at several levels in the range from 0.001 to 20 mm/min. Creep tests were carried out in conventional *constant stress* creep machines employing the Andrade-Chalmers profile attached to the load lever. The testpieces used for both kind of tests were similar in shape and dimensions: they were of cylindrical shape, having shoulders with M9x1,25 thread, with gauge-length **Lo** = 50 mm and gauge-diameter **do** = 5 mm. Temperature measurements were taken with cromel-alumel thermocouples and temperature stability during the test was maintained within the level of $\pm$ 2^{o}C by the use of PID controllers. For the strain measurements during creep, high temperature extensometry with LVDTs was employed connected to a type x-t chart recorder. With this arrangement, resolution for the strain readings was of the order of $2x10^{-5}$. Since the creep test were conducted under constant stress, all the data of deformation, deformation rate and stress mentioned in this article correspond to the true values of these parameters.

A series of continuous creep tests until rupture was carried out at 700^{o}C at the following applied stresses: 70 – 90 – 130 – 140 – 165 – 180 – 210 – 250 – 300 – 344 and 375 MPa. Three other creep tests until rupture were also carried out at the temperature levels: 600 ºC, 625 ºC and 670ºC, with 210 MPa.

Another set of tests was carried out with small temperature changes during secondary creep stage, also at 700^{o}C, to determine the apparent creep activation energy of the material. In these cases the following applied stress levels were used: 70 – 90 – 105 – 120 – 165 – 180 – 210. The specimens were deformed until middle of secondary stage, being subjected thereafter to incremental temperature changes of +10^{o}C until 720^{o}C, returning to 700^{o}C, followed by decremental temperature changes of -10^{o}C until 680^{o}C, and returning again to 700^{o}C. In each of these events, care was taken to wait for the settling down of a clear constant creep strain rate after the temperature changes. The time necessary to distinguish this new secondary stage after the changes varied a lot, from about 10 minutes in the tests at 210 MPa, for instance, to about 2 to 4 h in the tests at 70 MPa.

Finally, one creep test was carried out with small stress changes in three points around the region where secondary stage happens. The temperature was also 700^{o}C, and the applied stress σ = 150 MPa. Several changes in this stress level, most of them incremental (of about 2 to 5% of 150 MPa), were applied when creep strain attained the readings: ε = 11, 15 and 20%.

3 RESULTS

Figure 1a shows the graphs of Stress vs. Strain from the hot tensile tests for 8 different crosshead speeds varying in the range from 0.001 to 20 mm/min, which correspond to nominal strain rates varying from $3.3x10^{-7}$ s^{-1} to $6.7x10^{-3}$ s^{-1}. The dependence of the strength of the material with strain rate in the hot tensile tests is remarkable.

Figures 1b and 1c show the set of continuous creep curves conducted until rupture. The longest test (at 70 MPa) presented a rupture time of about 1500 h and the shortest test (at 375 MPa) a rupture time of about 4.5 min. It is interesting to notice that for this kind of steel (austenitic stainless) the secondary stage happens at high levels of creep strain, at times of the order of half (or more) of the rupture time in each test, with a lower participation of tertiary creep stage. This behavior is rather different from what happens with low alloy ferritic steels which show reduced secondary creep stage followed pronounced tertiary creep stage.[1]

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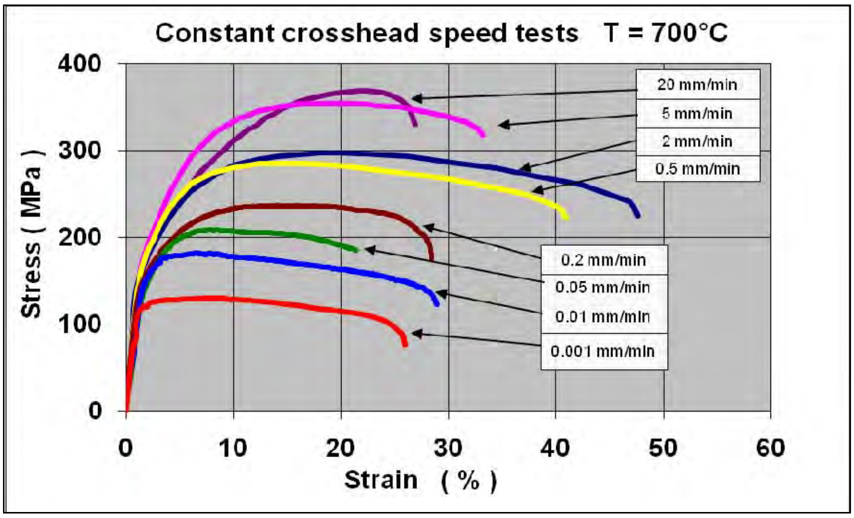
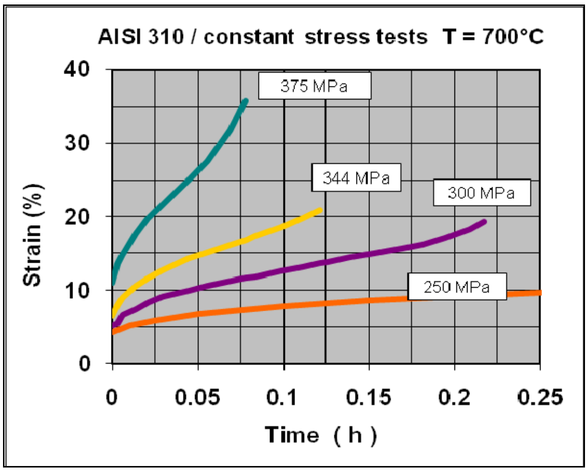
Figure presents 2a the typical curve of loss of creep strength with rupture time derived from the creep test data. Superimposed in the graph are the data from the hot tensile tests which were converted to creep data using a recent criterion of equivalence between both kind of tests proposed by Bueno.(5) According to this criterion: the UTS, the nominal strain rate and the time to reach UTS in a hot tensile test corresponds respectivelly to the applied stress, the minimum creep rate and the rupture time in a creep test. More details about this methodology of analysis and its succesful use to join hot tensile with creep data for analysis of high temperature behaviour of different kinds of metallic materials have been presented recently.(6-9) Since the creep tests in this study were carried out at constant stress, the hot tensile data representing the UTS and nominal strain rate were taken as the true values of these parameters, to make the equivalence. It can be observed that the compatibility of both kind of results in Figure 2a is notorious.



a)



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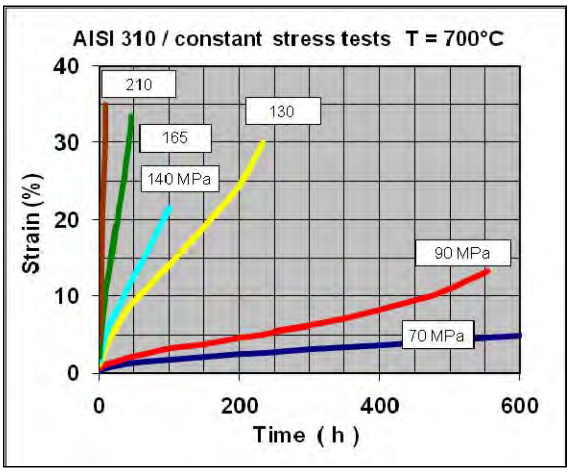

c)

Figure 1 – a) Tensile test curves at 700ºC for different crosshead speeds; b) and c) Creep test curves at 700ºC for different applied stress levels.

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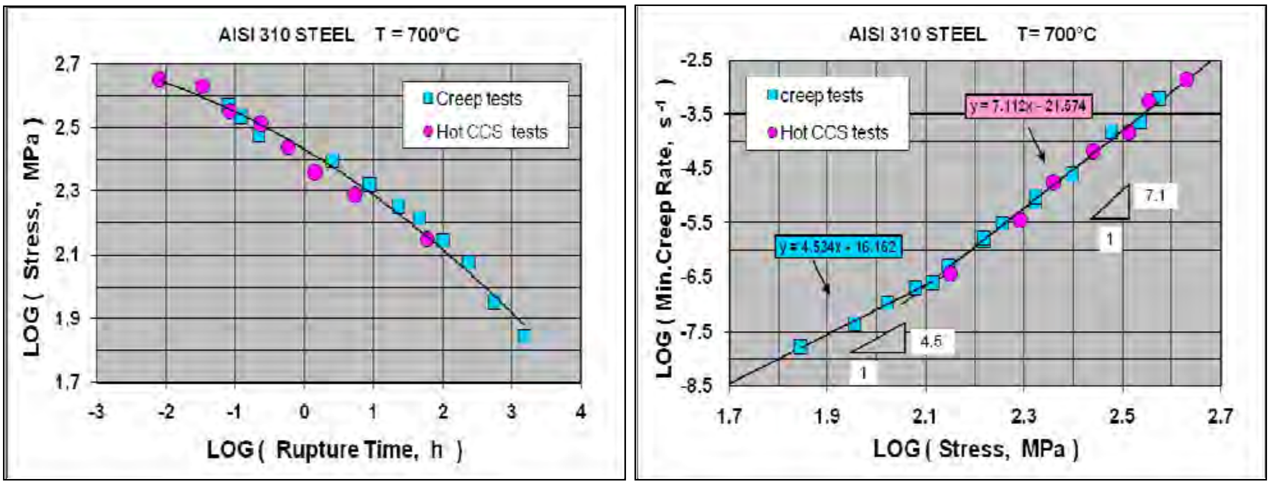

a) b)

Figure 2 – a) Loss of creep strength with rupture time at 700ºC. Hot tensile data (equivalent to creep) are plotted together for comparison; b) Variation of Minimum Creep Rate with Stress at 700ºC, showing inflection with two Norton exponents. Hot tensile data (equivalent to creep) are plotted together for comparison.

Figure 2b shows the variation of Minimum Creep Rate (representing the secondary creep rate) with Stress both for the creep test data and for the equivalent hot tensile data. The compatibility between both kind of results is also remarkable.

It is important to observe, in Figure 2b, that the creep data can be expressed by straight lines, according to Norton law:

$$\dot{\varepsilon}_s = A \cdot \sigma^n \qquad (2)$$

however, with an inflection point at the stress of about 130 MPa. For stresses lower 130 MPa, the stress exponent **n** = 4.5 and for stresses higher than 130 MPa, **n** = 7.1.

Figure 3a shows the variation of Minimum Creep Rate with Inverse of Temperature for the tests carried out at 210 MPa. This kind of plot is usually adopted to verify the validity of the Arrhenus relation:

$$\dot{\varepsilon}_s = B.\exp(-\,Q_c\,/\,RT) \qquad (3)$$

With this kind of data a value of apparent creep activation energy can be derived and value was calculated as Q_c = 330 kJ/mol, for the iso-stress condition of 210 MPa.

Figure 3b presents the variation of Minimum Creep Rate with the Rupture Time, from the creep tests conducted until rupture. Superimposed in this figure are the equivalent data from the hot tensile tests. The creep data in this figure can be very well expressed by a straight line, in the form predicted by the Monkman-Grant relation:

$$\dot{\varepsilon}_s \times t_r^{\,m} = K \qquad (4)$$

with **m** very near to 1. The compatibility of the hot tensile data with the creep data in Figure 3b can be considered satisfactory, since it is well known that creep data in Monkman-Grant plots present a rather high degree of scatter.

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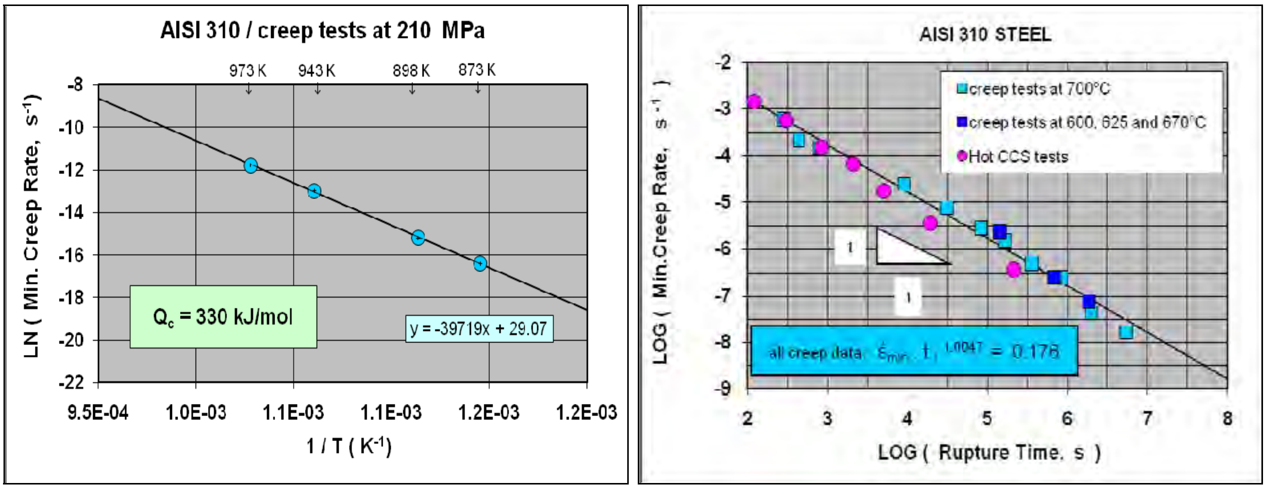

a) b)

Figure 3 – a) Variation of Minimum Creep Rate with Inverse Temperature for creep tests at 210 MPa; b) Variation of Minimum Creep Rate with Rupture Time for all creep tests from 600ºC to 700ºC. Hot tensile data (equivalent to creep) are plotted together for comparison.

In addition to the calculation of Minimum Creep Rates for the continuous tests until rupture, at 700^{o}C, also values for the Initial Creep Rates in each test were determined. In this case, to clarify the results, an extra creep test was carried out at 700^{o}C with σ = 40 MPa, exclusively to generate a value of Initial Creep Rate at this lower stress level. The test was not taken until secondary stage, and let alone to rupture (which should occur for a duration of about 1 year). Figure 4 presents the variation of the Initial Creep Rate and of the Minimum Creep Rate with Stress, for comparison with each other. It is interesting to notice that the Initial Creep Rate exhibits the same kind of inflection that was noticed with the secondary creep rate data, at a stress level of about 130 MPa, as shown in Figure 2b. For stress lower than 130 MPa the stress exponent **n** = 3.9 and for stresses higher than 130 MPa, **n** = 6.3.

Figure 5a depicts the results of the variation of Minimum Creep Rate with Inverse Temperature from the tests with small variations in temperature during the secondary creep stage, for seven stress levels as explained in section 2. This kind of Arrhenius plot with such data provides determination of a value of $\mathbf{Q_c}$ with a single specimen at the applied stress chosen for test. The straight lines fitted to the each set of data are reasonably parallel to each other and the average apparent creep activation energy for the whole set of data was calculated as $\mathbf{Q_c}$ = 337 + 39 kJ/mol.

It is interesting no notice, in Figure 5b, the good compatibility between the activation energy data for 210 MPa using different specimens (shown in Figure 3a) with the data from the single specimen (shown in Figure 5a). The value of activation energy at 210 MPa considering the nine points in Figure 5b becomes a little higher, namely $\mathbf{Q_c}$ = 345 kJ/mol. Considering the degree of scatter that is normally associated with this kind of result, all the three values mentioned for $\mathbf{Q_c}$ in this work (330 – 337 – 345 kJ/mol) can be considered to be very close to each other.

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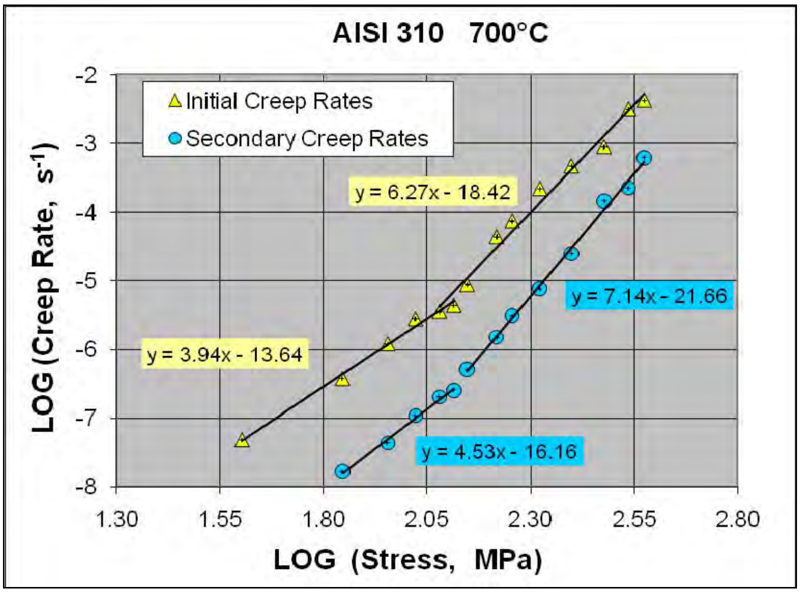
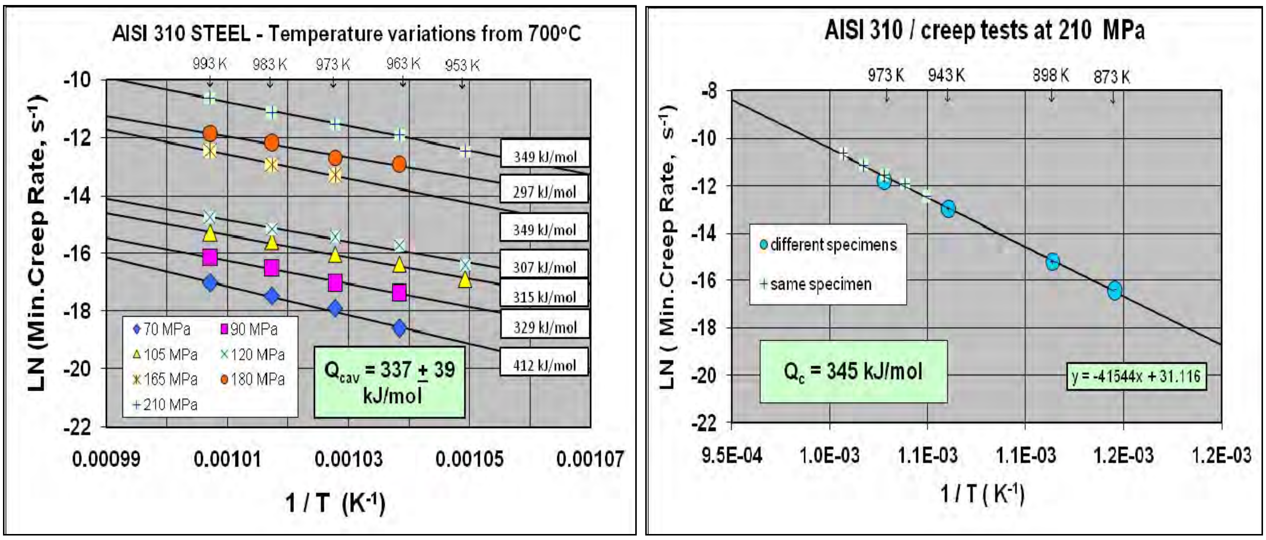


Figure 4 – Variation of Minimum Creep Rate and Initial Creep Rates with Stress , showing inflection at **σ** ≈ 130 MPa with two Norton exponents in both cases.

Figure 6a illustrates the possibility of variation of Minimum Creep Rate with Stress from the same data taken from creep tests with small temperature changes during secondary creep stage, at different stress levels, shown in Figure 5a. Although the data are not specifically of stress variation in each specimen, a cross-plot of the data is possible, providing Figure 6a. It is clearly observed that for all the five temperature levels there is indication for the presence of two different Norton exponents, with an inflection point, also in this case, around 130 MPa, so that for stresses lower than 130 MPa the values are of the order of **n** ≈ 4.8 and for stresses higher than 130 MPa **n** ≈ 7.0.



a) b)

Figure 5 – a) Variation of Minimum Creep Rate with Inverse Temperature for small variations in temperature during the secondary creep stage; b) Comparison of the variation of Minimum Creep Rate with Inverse Temperature for creep tests using different specimens (Figure 3) and same specimen (Figure 5a), at 210 MPa.

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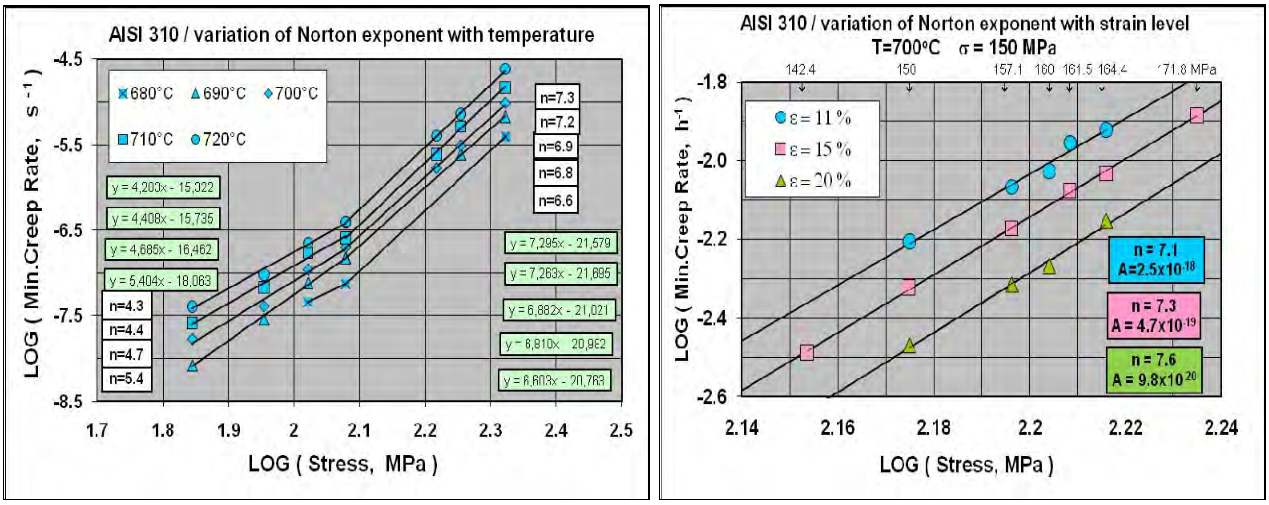

a) b)

Figure 6– a) Variation of Minimum Creep Rate with Stress from creep tests with small temperature variations during secondary creep stage, at different stress levels; b) Variation of Minimum Creep Rate with Stress for small stress variations at different strain levels in the region around secondary creep at 150MPa and 700ºC.

Figure 6b shows results relative to the creep test with small stress variation at three different strain levels around the secondary creep stage, for a stress of 150 MPa, at 700^{o}C. The three sets of data can be well expressed by straight lines, producing Norton exponents **n**=7.1 - 7.3 - 7.6 and **A**=2.5x10^{-18} - 4.7x10^{-19} - 9.8x10^{-20}, corresponding respectively to the strain levels **ε** = 11 - 15 - 20%. This data seems to give an indication that the stress exponent **n** increases and the parameter **A** decreases with increase in creep strain in the so called *secondary creep region*.

4 DISCUSSION

The pattern of data shown in Figures 2a, 2b and 3b demonstrates that the results from the creep tests carried out until rupture are very consistent. Whenever Monkman-Grant is verified to hold with an exponent **m** ≈ 1 (i.e. $\dot{\varepsilon}_s \times t_r = C$, the original form of the relation proposed by the authors), rupture time can be expressed by the relation:[(9)]

$$t_r = B.\ \sigma^{-n}.\ \exp\left(Q_c / RT\right) \qquad (5)$$

For tests at constant temperature, if **B** remains constant, the following relation should also be valid: $t_r = B'.\ \sigma^{-n}$. A careful analysis of Figure 2b shows also that the points can be divided in two regions: one for low stresses (σ < 130 MPa) where **n** ≈ 3.8 and another for high stresses (σ > 130 MPa) where n ≈ 7.8, as shown in Figure 7 where the data have been plotted according Equation 5.

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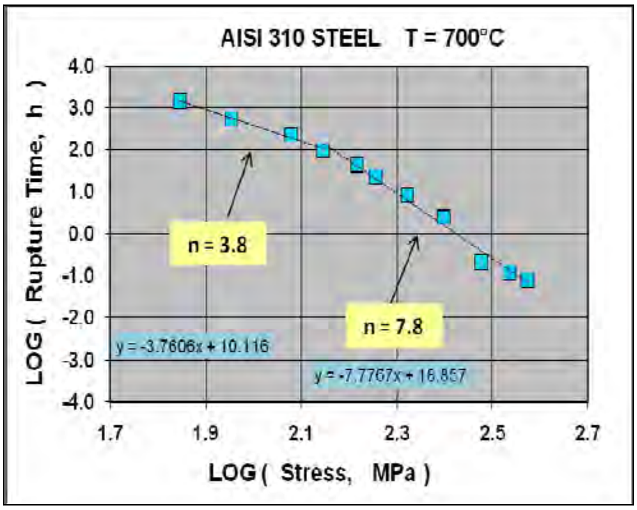

Figure 7 – Variation of rupture time with stress.

In Figure 2b, the value of the stress exponent **n** = 4.5, in the low stress range (σ < 130 MPa), is close to the values reported for creep in pure metals or solid solution alloys.[1,2] The last three points in the high stress range (σ > 300 MPa) presented a surprising result (tests carried out at 300 – 344 – 375 MPa), since according to the deformation mechanisms maps for AISI 304 and 316, it was expected that these three data would enter the region of exponential creep, with a change in mechanism with control from dislocation climb to dislocation glide, as reported by Frost and Ashby.[11] A great inflection was expected to occur from 300 MPa to 320 MPa also for AISI 310 steel, at 700°C. In fact, these last three points are well aligned with the rest of the points in the interval from 130 MPa to 250 MPa. The value of **n** = 7.1 agrees well with values reported for AISI 304 and 316,[1,2,11] in the same temperature and an equivalent stress range.

The transition of the Norton exponent from 4.5 to 7.1 occurred at a creep strain rate level compatible with the prevision of Sherby and Burke[3] who suggested that the transition from potential do exponential creep should happened when:

$$\dot{\boldsymbol{\varepsilon}}_{\mathbf{s}} \,/\, \mathbf{D} = 10^9 \text{ cm}^2 \qquad (6)$$

where **D** = self diffusion coefficient of the base metal. Calculation with the present results gave $\dot{\boldsymbol{\varepsilon}}_{\mathbf{s}} / \mathbf{D}$ = 0.7x10^9 cm^2, that can be considered satisfactory. However, the present data show that after surpassing this region (where σ = 130 MPa), the creep strain rates continue to be well represented by a potential relation with stress, up to the highest stress level applied in this work (375 MPa, where $t_r \approx$ 4.5 min). Many years ago, Garofalo et al.[9] reported a similar transition for AISI 316, at 700°C, where **n** changes from 4.1 to 8.3 at a stress level of about 120 MPa.

It is interesting to mention that the yield stress $\sigma_{0.05}$ at 700°C for the present material occurs for a stress level of about 125 MPa that is near the transition level of 130 MPa observed in many plots mentioned before. A more detailed consideration on this subject will be presented in Part 2 of this work.

According to Figure 8 (prepared with the data mentioned in Figure 5), the values of activation energy remains approximately constant in the stress range from 90 to 210 MPa,. A slight trend for increase in shown for lower stress of 70 MPa, what could be normally expected [1,2]. Since only one point represents this region, it was

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preferred to consider an average value $Q_{c\ av}$ = 337 kJ/mol for the whole interval from 70 to 210 MPa, although an average value somewhat lower, $Q_{c\ av}$ = 324 kJ/mol, would be valid from 90 to 210 MPa.

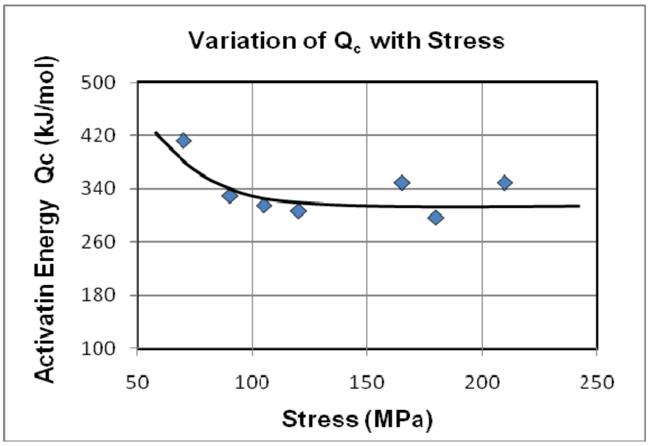

Figure 8 – Variation of the apparent creep activation energy with stress.

The values of Q_c from Figure 5a and also those derived from the data in Figure 5b (345 kJ/mol), are considerably higher than the activation energy of self-diffusion for γ-iron, which is about Q_{sd} = 280 kJ/mol, as reported in literature.(11)

According to Evans and Wilshire(1) the high values found for Q_c in complex alloys can be rationalized by corrections in its calculation, taking into consideration the dependence of the parameters **A** and **n** with stress and temperature, respectively. The dependence usually observed for Q_c with temperature and for **n** with stress results in fact from the behavior of internal stresses which are developed during creep, and which are consequently critically dependent on the micro-structural state and deformation substructure developed in the material.

According to Barret et al.(12) a correction in Q_c can be applied taking in consideration the dependence of the elastic modulus **E** of the material with temperature, by the expression:

$$Q_c' = Q_c - n[\,(RT^2 / E).\,(dE/dT)] \qquad (7)$$

where Q_c' represents the values of activation energy without correction.
When these calculations are performed, new values of Q_c are obtained, as shown below in Table 1:

Table 1 – Values of Q_c corrected by the effect of temperature on elastic modulus

MPa	70	90	105	120	165	180	210
Q_c'	412	329	315	307	349	297	349
Q_c (corr)	404	286	272	276	331	283	297

The average value for the apparent activation energy drops now to $Q_{c\ av}$ = 307 ± 47 kJ/mol in the interval from 70 – 210 MPa, or to $Q_{c\ av}$ = 291 ± 22 kJ/mol in the interval from 90 – 210 MPa. This last figure is now much closer to the value of Q_{sd} = 280 kJ/mol mentioned in literature for γ-iron.

The data represented in Figure 6a is very significant, since it confirms that an inflection in power-law relation ($\dot{\varepsilon}_s = A.\sigma^n$) occurs for this material at 700°C, and temperatures around. Also significant is the behavior exhibited by the variation of

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Initial Creep Strain Rates with Stress, shown in Figure 4, where the same kind of inflection is observed. According to Evans and Wilshire[1] these observation of a sequence of two or three **n** values can be rationalized to a single stress exponent of lower value (of the order of 3 to 4) when the levels of internal stress (or friction stress, σ_o) are considered in the analysis of the data. An experimental technique have been suggested by these authors[1] , based on progressive decremental stress changes performed during creep, usually in secondary stage, for determination of σ_o values. The results of an investigation of this kind, for AISI 310 steel, will be presented in Part 2 of this work.

Furthermore, the data represented in Figure 6b, is also significant, since it points out to the fact that the Norton exponents **n** and the parameter **A** may vary with the strain level during a creep test. This is clearly connected with the fact that internal stresses are also varying during a creep test. The results of an investigation on this subject, also for the AISI 310 steel, will be presented in Part 3 of this work.

More recently, however, Wilshire[13] points out, that measurements of **n** and $\mathbf{Q_c}$ do not provide a reliable indication of the dominant creep processes and have not lead to reliable predictive capabilities, so confidence must still be placed on empirical parametric methods, in order to obtain long-term stress-rupture estimates. The author[13,14] suggests other approaches, such as θ-methodology, that seek to quantify the shapes of creep curves and the variation they undergo with changing test conditions.

5 CONCLUSIONS

The following conclusions can be drawn from this work involving constant stress creep tests until rupture on AISI 310 steel, carried out at 700^{o}C, in the range from 70 to 375 MPa:

- The value of the stress exponent of the secondary creep rate exhibits two distinct values. For stresses σ < 130 MPa, n = 4.5 and for stresses σ > 130 MPa, n = 7.1.
- Tests of very short duration, with stresses varying from 300 < σ < 375 MPa did not indicate a transition to the region of exponential creep.
- The transition of the Norton exponent from 4.5 to 7.1 occurred at a creep strain rate level compatible with the prediction of Sherby and Burke[3], according to the relation: $\dot{\boldsymbol{\varepsilon}}_{\mathbf{s}}\mathbf{/D}$ = 10^9 cm^2.
- The initial creep rates also can be expressed by potential relations with stress, also with two exponents: n = 3.9 for σ < 130 MPa and n = 6.3 for σ > 130 MPa.
- The region of transition around 130 MPa is close to the yield stress ($\sigma_{0.05}$) of the material at 700^{o}C.

The creep tests carried out with small temperature variation around 700^{o}C, in the range from 70 to 210 MPa, presented the following results:

- Measurements of apparent creep activation energies, corrected by the effect of temperature on the elastic modulus, indicated the average value $\mathbf{Q_{c\ av}}$ = 307 $\pm$ 47 kJ/mol, that is somewhat higher than the self-diffusion activation energy of iron in austenite ($\mathbf{Q_{sd}}\approx$ 280 kJ/mol). In the interval from 90–210 MPa, $\mathbf{Q_{c\ av}}$ = 291 $\pm$ 22 kJ/mol which is closer to $\mathbf{Q_{sd}}$.
- Cross-plots of the data of secondary creep rate with temperature variation confirmed the existence of a transition with two different stress exponents at the

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stress level of 130 MPa, with approximately the same values obtained with the continuous creep tests until rupture.

The creep tests carried out with small stress variation in three different levels of strain around secondary stage (11 – 15 – 20%), with 150 MPa, at 700°C have shown that:

- The stress exponent **n** increases and the parameter **A** decreases as level of creep deformation increases around secondary stage.

The following conclusions can be taken from the hot tensile tests carried out at 700°C using different crosshead speeds in the range from 0.001 to 20 mm/mm, corresponding to nominal strain rates varying from: $3.3\times10^{-7}\ s^{-1}$ to $6.7\times10^{-3}\ s^{-1}$:

- The AISI 310 shows remarkable dependence of the Ultimate Tensile Stress with the strain rate employed in the tensile tests.
- Conversion of the hot tensile data to the format of creep data (using a Criterion of Equivalence proposed to correlate results from both kind of tests), produced good association with the creep results generated in this work, when analyzed according to plots of Log(σ) vs. Log($\mathbf{t_r}$), Log($\dot{\boldsymbol{\varepsilon}}_{\mathbf{s}}$) vs 1/T and Log($\dot{\boldsymbol{\varepsilon}}_{\mathbf{s}}$) vs. Log($\mathbf{t_r}$).

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