THE OPTIMIZATION OF INVESTIGATED SUPERPLASTIC CHARACTERISTICS OF AN AUSTENITIC Fe-Mn-Al STEEL

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Abstract. This work aims to study different processing routes, which rationally made some improvement on superplasticity characterization on Fe-Mn-Al steel, since this austenitic alloy, through results of previous work showed some possibility of exploring and improve the potential use of these materials on superplastic regime. It was at temperatures higher than 700°C, and with these results it was possible to make appreciable improvements in such results obtained in first work. The material was prepared by different thermo mechanical processing routes, as to obtain sheets with thickness around 1 mm having a fine grain equiaxial dual phase austenitic/ferritic structure with grain size around 3 µm . The material was submitted to both tensile and creep test on a temperature range from 600 to 1000°C and strain-rates range from 10^{-4} to 1 s^{-1}. Values of maximum elongation observed at rupture (ε_r) could be obtained from the tensile and creep tests. The m (strain rate sensitivity exponent) parameter could be determined in both cases.The results from both procedures, i.e. tensile and creep tests were compared showing good agreement with each other. The largest values of ε_r above 600% associated to the largest m value around 0.54 were observed in case of tensile test at temperature 800°C for strain rate in the range from 10^{-4} to $10^{-3}s^{-1}$, and in case of creep test with constant stress in range from 20 to 50 MPa.

Keywords**: Fe-Mn-Al steel, hot tensile test, strain-rate sensitivity, superplasticity**

1. INTRODUCTION

The most important superplastic characteristics is associated to high elongations that occur in region II, of a usually three-stage relationship in the steady-state strain rate ($\dot{\varepsilon}$) dependence of the applied stress (σ), with progressive drop in superplastic characteristics in both regions I and III, according to Langdon [(1)]. There are, among some used test methods, two kinds of mechanical tests that explore structural superplasticity of metals as: a) tensile tests on tension machines with constant crosshead speed (or constant strain rate) where the flow stresses measured as function of strain rates are related by: $\sigma = C\,\dot{\varepsilon}^{\,m}$, here C is a constant including temperature dependence, and m is the strain rate sensitivity exponent ($m = d\mathrm{Log}\sigma/d\mathrm{Log}\dot{\varepsilon}$); and b) tensile tests on creep machines with constant load (or constant stress), here the strain rates are measured as function of the imposed stresses, being related by: $\dot{\varepsilon} = A\,\sigma^{\,n}$, here $n = 1/m$ with m as strain rate sensitivity and n as stress exponent. The values of m, generally considered to produce superplastic behavior are obtained in range from 0.35 to 0.8.

The austenitic steels of Fe-Mn-Al alloy may exhibit good combination of properties like mechanical strength, ductility, corrosion/oxidation resistance and lower density, being considered as alternative materials to the Fe-Ni-Cr stainless steels in some applications. The mechanical behaviour of these alloys at high temperatures, however, remains little explored, with some information existing in the literature on creep and very little on superplastic properties, for instance. There was apparently, few there other than presented by Sordi & Bueno [(2)] and Zhang et al [(3)] on creep studies.

The previous study of superplasticity on a Fe-Mn-Al austenitic steel, to our knowledge were first presented by Toscano [(4)] showing some possibility of exploring the potential use of such materials in this regime for temperatures higher than 700°C, with chemical composition (wt %) of Fe-32Mn-11Al-1.5Si-1.0C and a fully austenitic structure submitted to high hot-rolling reductions obtaining sheets with 1mm thickness. Isothermal tensile tests carried out at a fixed crosshead speed Vc = 0.5 mm/min showed a drop in yield and tensile strength of the material in range from 800 to 1000K, here was suggested to occur a very fine grain size structure in the austenite grain boundaries from 913K. This study was followed by a work of Guanabara & Bueno [(5)], here for the first time the occurrence of such behaviour was systematically conducted due to characterize superplasticity. The hot tensile tests were carried out on Instron machine with constant crosshead speed in range from 600 to 1000°C involving initial strain-rates from 8.3 x10^{-5} to 8.3 x 10^{-2} s^{-1}, the initial set of results indicated only a modest superplastic performance, with the maximum elongation of about 320% at 850°C/8.3x$10^{-5}$$s^{-1}$ Guanabara [(6)] , rather below the result reported by Toscano [(4)] of about 500% at 800°C/8.3x$10^{-4}$$s^{-1}$, so enabling this present work performed with the same range of temperature, which rationally made some improvement on previous results.

So the material was prepared, in this present work, by different thermo mechanical processing routes to obtain sheets with ~ 1 mm thickness having a fine grain equiaxial structure with grain size around 3 µm and dual phase austenitic / ferritic. Thus the maximum elongation at rupture (ε_r) values could be determined from the tensile and creep tests, and m values (strain rate sensitivity exponent) could be improved from results obtained in both cases, Guanabara e Bueno [(7)].

2. EXPERIMENTAL PROCEDURES

The material was prepared in the form of ingots weighting about 3.5 kg with approximately 50 x 50 x 220 mm each. The chemical composition (wt %) was determined as: Fe– 24.5Mn– 6.5Al– 1.5Si– 1.1C– 0.009P– 0.016S. The ingot was first submitted to solution heat-treatment at 1050°C for 24 hours, followed by quenching in oil. So the material was final annealed at temperature 850°C for 1 hour. This was the better condition of previous chosen more favorable annealing and grain growth characteristic treatments, at temperatures 800°C, 850°C and 900°C for 1 hour each, due to obtain a more favorable superplastic characteristic. Then grinding operation was used to square all the faces before sectioning the sample in two slabs with about 25 x 50 x 200 mm each. The material hardness at this condition was 286 HV_{30}. The slabs were subjected to three series of cold rolling steps of low deformation followed by heat treatments of 1050°C during 1 hour. The accumulated deformation levels after each cold rolling stage corresponded to about 25, 50 and 75% reduction in thickness. After the last solution treatment the sample was cold rolled continuously until its final shape of a stripe with 1mm final thickness, so completing the thermo mechanical processing routes.

Tensile samples were machined from the stripes in the rolling direction, having a nominal gauge length Lo = 10 mm and gauge width w = 3.0 mm. Tensile tests were carried mainly at 600°C, 700°C, 800°C, 900°C, and 1000°C with at least four crosshead speed levels, namely: V_c = 0.05, 0.5, 5 and 50 mm/min, corresponding to initial strain rates of: $8.3x10^{-5}$, $8.3x10^{-4}$, $8.3x10^{-3}$ and $8.3x10^{-2}$ s^{-1} respectively. The hot tensile tests were carried out on a universal Instron machine model 5500R with a tubular electric resistance furnace. The temperature stability during all tests was about ± 1°C, maintained by P.I.D controllers.The variation in sensitivity of stress with strain rate was observed using *distinct specimens* for each combination of crosshead speed with temperature, as well as *single specimens* subjected to several crosshead speed changes during a certain temperature level.

Creep sample, machined in rolling direction with rectangular shape and fixing role at edge, were similar as such used in tensile test. Creep tests were carried out initially at constant load on a temperature range from 600 to 1000°C and strain-rates range from 10^{-4} to 1 s^{-1}. Each chose stress and weight corresponding were verified with a VEB dynamometer of 400kgf using norm ASTM E-139 (1990). The adopted applied stress was chosen using the peak stress, as associated criterion obtained of tensile experiment in Instron machine, performed at same temperature with respective V_C (crosshead speed), in a wide range of rupture time as load test increase [(8)]. The systematic creep tests were carried out on a MF-1000/STM constant load creep machine with a tubular electric resistance furnace. The steady state strain rate, in this case is recorded for the imposed stress and data, which values are logarithmically plotted as $\acute{\varepsilon}$ x σ (strain rate against stress).

3. RESULTS AND DISCUSSION

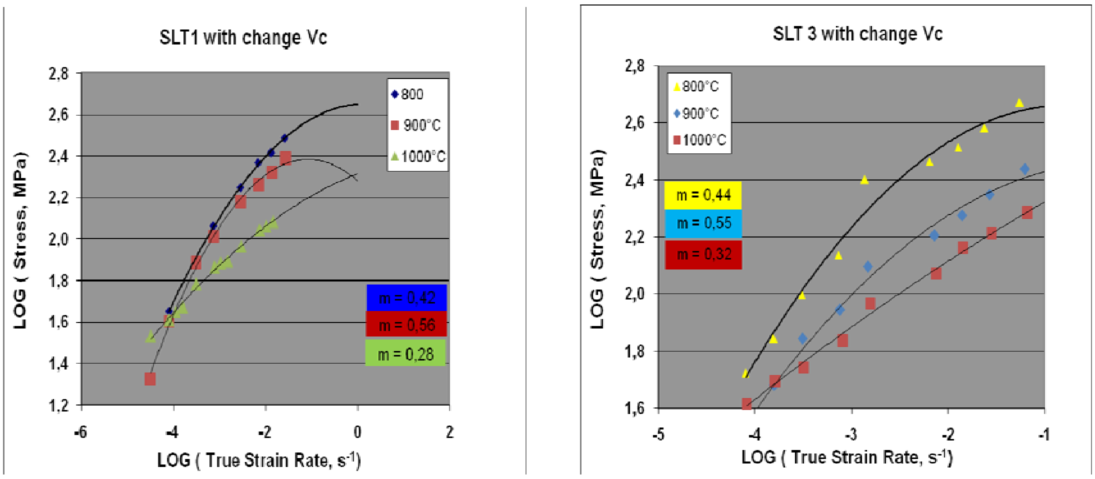
The obtained results of this second set of experimental work were a sequence of research studies of austenitic Fe-Mn-Al steel at several temperatures; hence better treatment conditions of lower hardness value, after data analysis, were chosen to subsequent series of thermo-mechanical treatment. These materials were performed at 3 SLT conditions of annealing treatments (SLT1, SLT2 and SLT3) at temperatures respectively: 800°C, 850°C and 900°C for 60 minute each. The m values are mainly used as estimated parameter to characterize superplastic behavior, because only elongation is not sufficient to obtain such results. These results were obtained both tensile with constant $\acute{\varepsilon}$ (strain rate) and creep with constant σ (stress) experimental tests techniques. It's because through n parameter one could compare and confirm, by the relationship n = 1/m, such m values obtained in same condition as through tensile test [(8)].

The flow stress was measured as function of strain rate related by the expression [(1)]. $\sigma = C\, \acute{\varepsilon}^{m}$, here C is constant including temperature dependence, and m is strain rate sensitivity exponent obtained through $m = dLog\sigma / dLog\acute{\varepsilon}$.

So the experimental set of this work were performed with tensile test of distinct specimens strained until rupture under different combinations of chosen crosshead speed and temperature, on hot tensile test cases, due to determine and confirm m value among other parameters, the samples finally were pulled until rupture under different combinations of crosshead speed from 0.01 to 20 mm/min. at temperature 800°C.

There was interest in carrying out some systematic creep test in the early stage at temperature 900°C on applied stress in range from 14 to 85 MPa, followed by systematic creep test carried out on applied stress in same range, at temperature of 600°C, 700°C; 800°C and 850°C. The creep test data from specimens of austenitic steel Fe-Mn-Al (SLT2) with different stress were used to plot a logarithmically Arrhenius relation Ln (initial $\acute{\varepsilon}$) x 1/ T The n value (stress exponent) is so far the most important parameter for creep testing of characterization superplastic behavior. It's because through this n parameter one could compare and confirm, through relationship n = 1/m, such m values obtained considering the same condition of the tensile test [(9)].

Figure 1 shows a typical true stress with strain rate curve of Fe-Mn-Al tensile tested sample with change V_C and different temperatures: (a) of (SLT1) and (b) (SLT3). Such figures show curves of tensile tested samples with change V_C at temperatures 800°C, 900°C and 1000°C with respective average m values evolution for each annealing and temperature.



(a) (b)

Figure 1. True stress with strain rate curve of an austenitic Fe-Mn-Al steel tested sample tensile tested with change V_C at temperature 800°C, 900°C and 1000°C and respective average m value of condition: (a) (SLT1) and (b) (SLT3).

Figure 2 shows a comparison of results between true stress (σ) with strain rate (έ) curve of hot tensile sample tested with different V_C and temperature of an austenitic steel Fe-Mn-Al thermo-mechanical treated at SLT2 condition. Such curves show strain rate sensitivity (m) values evolution for each annealing and temperature.

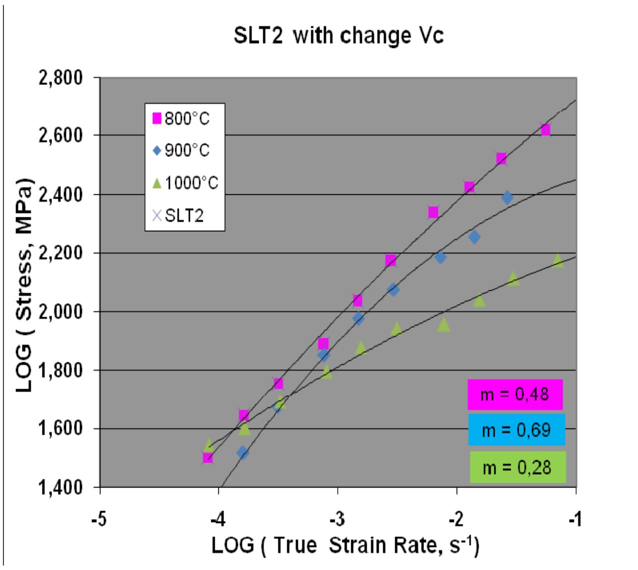
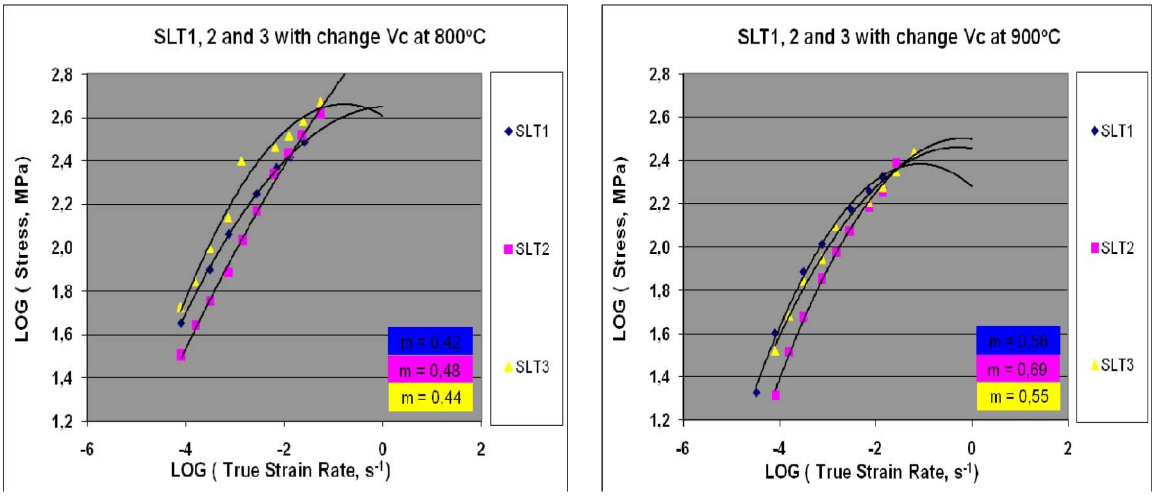


Figure 2. Comparison between true stress versus strain rate curve of hot tensile tested sample with different V_C showing m value evolution for each annealing treatment and experimental temperature.

Figure 3 show comparison of hot tensile test result of Fe-Mn-Al steel sample with change V_C for annealing treated SLT1, SLT2 and SLT3, with temperature range from 800°C to 900°C, which presents better potential of superplastic behavior for this material. These figures let see also both flow evaluation trend and respective m value for each SLT´s material curve.



(a) (b)

Figure 3. Comparison of hot tensile test result with change Vc for 3 different annealing treatments SLT1, SLT2 and SLT3 of austenitic Fe-Mn-Al steel samples with respective m values at temperatures: (a) 800°C; (b) 900°C.

Figure 4 shows example of typical hot tensile test result at true stress versus strain curve, with different V_C in range from 0.01 to 200 mm/min., for steel Fe-Mn-Al (SLT2) at temperature 800°C. This Figure shows trend of flow material and u.t.s. with each V_C values curve, at room temperature with very high yield and tensile strengths, as reported by Toscano [(2)] in cold rolled condition. As temperature increases strength becomes more and more $\acute{\varepsilon}$ sensitive. It also shows material flow stress and very high u.t.s. at room temperature, as temperature growth and becomes more sensible to ($\acute{\varepsilon}$). Its observe trend to the left of curve with decreasing stress values indicating upon m predominance and strain hardening exponent (n`) decreasing influence as consequence. Tensile tested sample appearance of different elongation until rupture (ε_r) with constant V_C curve, at several crosshead speed and temperature 800°C, also observe better result > 600 % (around 660 %) with V_C = 0.5 mm / min.(practically double value) if compared with that, without annealing treatment sample obtained at first work[(1)]. The more pronounced result was initial strain rate values of $\acute{\varepsilon} = 2.47 \times 10^{-4}\ s^{-1}$.

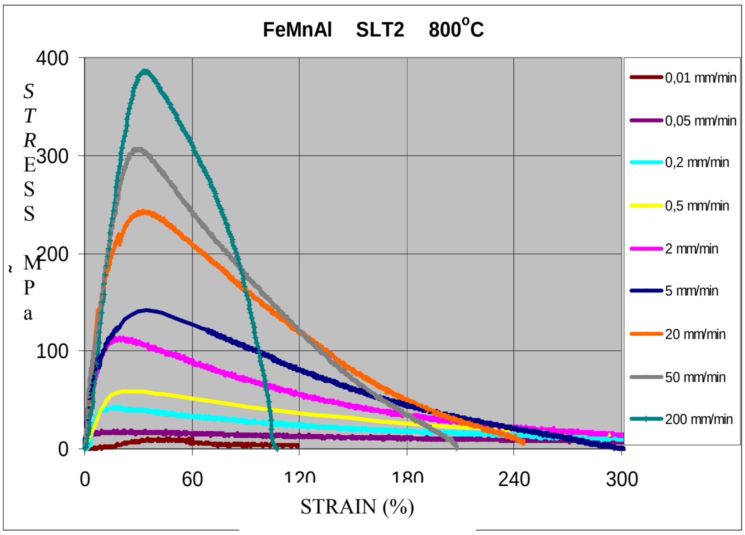


Figure 4. True typical Stress versus Strain curves for austenitic Fe-Mn-Al steel at SLT2 condition, at same temperature 800°C and different V_C (crosshead speeds).

Figure 5 show strains with time curve of creep tested Fe-Mn-Al sample SLT1 and SLT2 with constant load in the stress range from 14 to 85 MPa at test temperature 900°C. Figures 5(a) and 5(b) show creep test with constant load performed at 5 stress level prior adopted values, kept of initial selected stress analyzed at temperature 900°C. Such discretion used to associate u.t.s. (hot tensile test x $\acute{\varepsilon}$) with couple maximum stress / deformation of m (the most estimated characteristic) values.

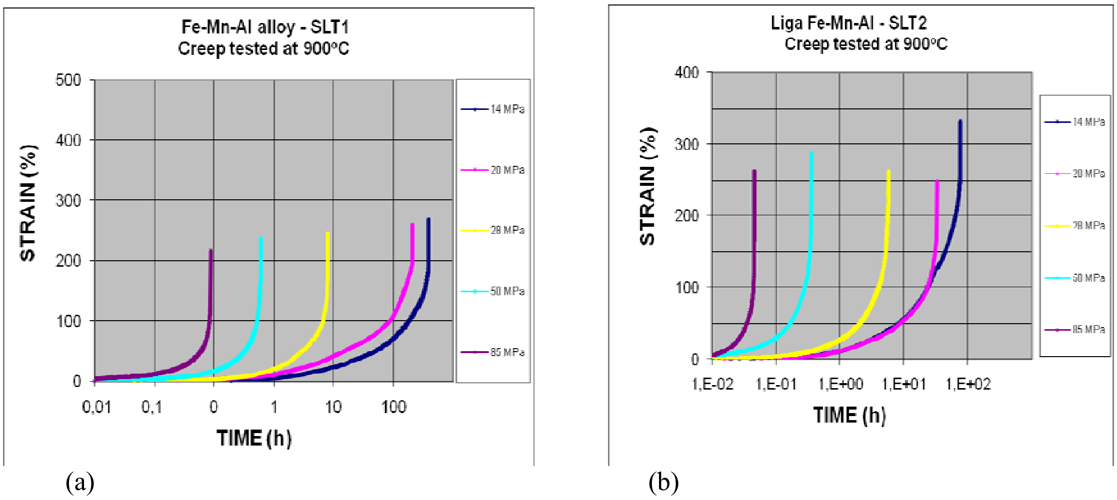


Figure 5. Curve of Strain with Time of creep tested sample with constant load in a range from 14 MPa to 85 MPa at temperature 900°C here: (a) SLT1; (b) SLT2 condition.

Graphs as: Norton; Monkman-Grant and Decreasing Creep strength with rupture Time were derived from these figures, due to compare n value (stress exponent) trend performed at those stress range and experimental temperature of 900°C.

Figure 6 shows comparative graphs of SLT1; SLT2 and SLT3 Fe-Mn-Al steel from creep tested sample with constant load in stress range from 14 to 85 MPa at test temperature 900°C as: Norton; Monkman-Grant and Decreasing creep rate strength with time. The values could be initially obtained without correction of n values at several temperatures, as Figure 6(a) shows in Norton graph for 900°C.

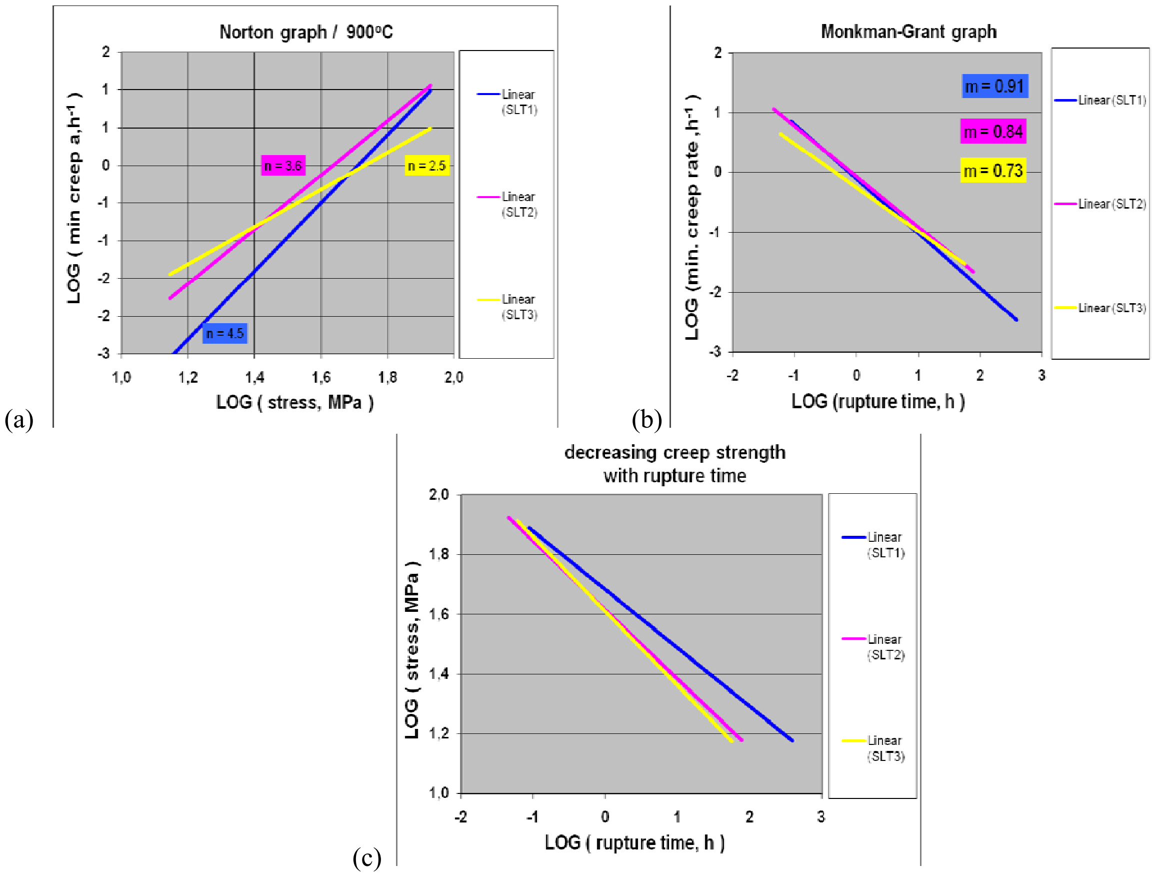


Figure 6. Comparative graph of SLT1; SLT2 and SLT3 Fe-Mn-Al steel from creep tested sample with constant load in stress range from 14 to 85 MPa in such graph as: (a) Norton; (b) Monkman-Grant and (c) Decreasing creep rate strength with rupture time.

The austenitic steel Fe-Mn-Al (SLT2) samples were creep tested with constant load at temperatures 700°C and 800°C, , in terms of n values (stress exponent) from Log (mín. nominal creep rate, h^{-1}) x Log (nominal stress, MPa), keeping just used stress, with material on 3 SLT´s condition, i.e. SLT1, SLT2 and SLT3 respectively. The experiments were performed at different temperatures of minimal creep rate values such as: nominal; true and initial creep rate.

This comparative of n values used to form a graph shows grouped n (stress exponent) data results, at temperatures 700°C, 800°C and 900°C. Such data letting more precise trend analysis of minimum rate variation and so n (stress exponent).They were composed with minimum nominal rate; true rate and rate initial curves. The n values (found in 3 curves at temperature 800°C) are closed to 2, to minimum nominal and initial rate with $n \approx 2$. Minimum corrected rate increases n (stress exponent) value to $n = 2.5$ at same temperature. Three rates show very different values at temperature 900°C. It happens with meaningful increase of minimum true rate related to nominal one.

Such comparative was followed by Arrhenius graph, verifying the influence of creep rate behavior in different stress with several tested temperature also with stress level related temperature and n exponent. It´s plotted at Ln (initial rate, h^{-1}) x 1 / T (K^{-1}) condition with a detailed survey of initial and minimal rates indicating σ (stress) and Q_c (creep activation energy) associated to each curve [7].

4. CONCLUDING REMARKS

This work represents a second assessment of superplastic properties in this material improving first results. Instead of taking the material for the tensile tests directly in the cold worked condition, other annealing treatments were explored to produce a stable fine grained structure before the tests, so using new thermo-mechanical processing routes. The samples thus obtained were performed not only with hot tensile tests, but complemented by new series of creep tests, to define the better combination of temperature and strain rate, which showed better conditions of superplastic behaviour. There were performed more studies involving micro structural observation on the deformed regions of the specimens, necessary to confirm and understand the superplastic effects in this material.

The new thermo-mechanical processing routes coupled with tensile tests (*with constant strain rate*) and creep tests (*with constant stress*) allowed improving the values of parameters of characterization.

The Fe-Mn-Al steel selected for this study, if compared to the superplastic performance of previous results [5] (first work) showed a very better performance with higher deformation. A tensile test performed at constant $\acute{\varepsilon} = 2.47 \times 10^{-4}\ s^{-1}$ produced a maximum elongation until rupture of $\boldsymbol{\varepsilon_r}$ = 750% and creep test with constant σ = 30 MPa with maximum elongation of $\boldsymbol{\varepsilon_f}$ = 737 % (without rupture of the specimen), data sensibly better than those results obtained in first work [1].

The result from both procedure, i.e. tensile and creep test showed good agreement to each other. The comparison between tensile with constant $\acute{\varepsilon}$ and creep with constant stress tests results at temperature 800°C/V_C based upon tensile test after correction (Log (σ_{max}, MPa) x Log($d\varepsilon/dt, s^{-1}$)), comprise perfect symmetry between m and n exponents, since $n = 1 / m$.

Both experimental tensile and creep tests allowed achieving better parameter m (strain rate sensitivity) values, at region of m maximum, thus confirming superplastic behavior of this austenitic steel Fe-Mn-Al alloy.

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7. RESPONSIBILITY NOTICE

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