ASSESSING THE SUPERPLASTIC BEHAVIOR OF A Fe-Mn-Al AUSTENITIC STAINLESS STEEL [1]

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Abstract

The superplastic behaviour of Fe-Ni-Cr duplex stainless steel system has been the subject of several investigations in the last two decades, with superplastic forming applications already existing in practice. A rare example of study of superplasticity on a Fe-Mn-Al steel was presented by Toscano (1983) showing some possibility of exploring the potential use of such materials in this regime for temperatures higher than 700°C. The present work was programmed to explore the occurrence of this behaviour in a similar steel with systematic hot tensile tests carried out in the range from 600 to 1000°C and strain-rates varying from 8×10^{-5} to 8×10^{-2} s^{-1}. The variation in sensitivity of stress with strain rate ($\sigma = C \dot{\varepsilon}^m$) was observed using distinct specimens pulled until rupture under different combinations of crosshead speed and temperature, as well as single specimens subjected to a sequence of crosshead speed changes during the hot tensile test. At 800 and 900°C the maximum values of **m** (**m** = $\partial \text{Log}\sigma / \partial \text{Log}\, \dot{\varepsilon}$) were found to be about 0.57 and 0.66 respectively, which confirms the material susceptibility to superplastic behaviour. The maximum elongation values were observed to stay around 300% obtained with the lowest strain rate level of 8×10^{-5} s^{-1} at 850°C.

Key words: Fe-Mn-Al steel; Hot tensile test; strain-rate sensitivity; Superplasticity.

AVALIAÇÃO DO COMPORTAMENTO SUPERPLÁSTICO DE UMA AÇO INOXIDÁVEL AUSTENÍTICO Fe-Mn-Al

Abstract

O comportamento superplástico de aços inoxidáveis duplex do sistema Fe-Cr-Ni tem sido focalizado por vários investigadores nas últimas duas décadas, havendo atualmente várias aplicações desses materiais em processos de conformação no regime de superplasticidade, com relatos de alongamento atingindo valores da ordem de 1500%. Com aços do sistema Fe-Mn-Al, no entanto, esses tipos de estudo são praticamente inexistentes. Um raro exemplo é o trabalho apresentado por Toscano (1983) que mostra alguma possibilidade do potencial de uso desses materiais em regime superplástico em temperaturas acima de 700°C. O presente trabalho foi programado para se investigar a ocorrência deste comportamento em um aço desse tipo com testes sistemáticos de tração a quente na faixa de 600 to 1000°C e taxas de deformação entre 8×10^{-5} e 8×10^{-2} s^{-1}. A variação na sensibilidade da tensão com a taxa de deformação ($\sigma = C \dot{\varepsilon}^m$) foi observada usando-se amostras distintas tracionadas até a ruptura sob diferentes combinações de velocidade da travessa e temperatura, e também usando-se uma mesma amostra sujeita a uma seqüência de mudanças na velocidade de tração durante o ensaio em uma certa temperatura. A 800 e 900°C os valores máximos de m (m = $\partial \text{Log}\sigma / \partial \text{Log}\, \dot{\varepsilon}$) foram de 0,57 e 0,66 respectivamente, que confirma a susceptibilidade do material ao comportamento superplástico. Os valores de alongamento máximo situaram-se em, no entanto, torno de apenas 300% obtidos na menor taxa de deformação empregada, ou seja 8×10^{-5} s^{-1}, a 850°C.

Palavras-chave: Aço Fe-Mn-Al; Ensaios de tração a quente; Sensibilidade à taxa de deformação; Superplasticidade.

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1 INTRODUCTION

It is well known that superplastic materials exhibit usually a three-stage relationship in the steady-state strain rate ($\dot{\varepsilon}$) dependence of the applied stress (σ). These three ranges are named regions I, II and III, respectively. The most important superplastic characteristics associated to high elongations occur in region II, with progressive drop in superplastic characteristics in both regions I and III, as shown schematically in Figs. 1a and 1b, according to Langdon.(1) There are two kinds of mechanical tests to explore the superplastic properties of metals: a) tensile tests on constant crosshead speed (or constant strain rate) machines where the flow stresses are measured as function of the strain rates and are related by the expression: $\sigma = C\,\dot{\varepsilon}^{m}$, where C is a constant including the temperature dependence, and **m** is the strain rate sensitivity exponent (**m** = $d\text{Log}\sigma/d\text{Log}\dot{\varepsilon}$); b) tensile tests on creep machines at constant load (or constant stress), where the strain rates are measured as function of the imposed stresses, being related by the expression: $\dot{\varepsilon} = A\,\sigma^{n}$, with **n** = 1/**m**. Values of **m** ranging from 0.35 to 0.8 are generally considered to produce superplastic behaviour.

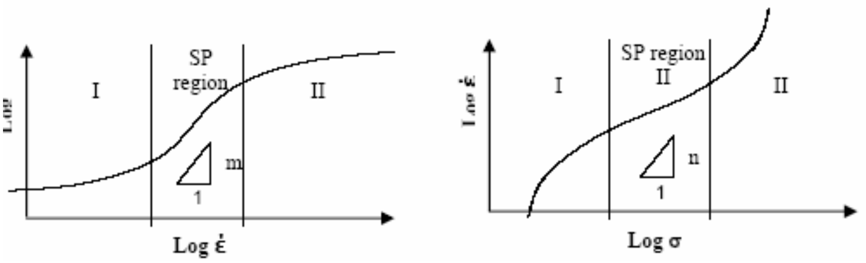

Figure 1. Illustration of the two different procedures used to plot the mechanical data of superplastic materials: **a)** stress vs strain rate; **b)** strain rate vs stress.

The Fe-Mn-Al stainless steels 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. Several studies have been reported since the 1960s involving the characterization of their conventional properties at room temperature. The mechanical behaviour of these alloys at high temperatures, however, remains largely unexplored, with very little information existing in the literature on creep and superplastic properties, for instance. On the other hand the superplastic behaviour of Fe-Ni-Cr duplex stainless steel system has been the subject of several investigations in the last two decades, with superplastic forming applications already existing in the aerospace industry. To our knowledge the only study of superplasticity on a Fe-Mn-Al austenitic steel was presented by Toscano(2) showing some possibility of exploring the potential use of such materials in this regime for temperatures higher than 700°C. The steel had the following chemical composition (wt%) Fe-32Mn-11Al-1.5Si-1.0C with a fully austenitic structure being submitted to high reductions by hot-rolling to obtain sheets with 1mm thickness which were finished by 40% cold-rolling. Isothermal tensile tests carried out at a fixed crosshead speed Vc = 0.5 mm/min have shown a drop in the yield and tensile strength of the material from 800 to 1000K , followed by an increase in hardness in the same temperature range, due to the precipitation of the β-manganese phase, as shown in Figures 2a and 2b, respectively. During the hot tensile tests, from 793K, incipient precipitation of the β-manganese phase was suggested to occur in the austenite grain boundaries, which promotes a very a fine grain size structure from 913K. The appearance of superplastic behaviour in Fe-Mn-Al steel is explained by the movement of the rigid particles of β-phase in the "non-Newtonian fluid" of the matrix. After this work, apparently, there are no other investigations reported in literature concerning superplastic behaviour of Fe-Mn-Al stainless steels.

The present work was programmed to explore the occurrence of this behaviour in this type of steel with systematic hot tensile tests carried out on a constant crosshead speed machine in the range from 600 to 1000°C involving initial strain-rates from 8.3×10^{-5} to 8.3×10^{-2} s^{-1}.

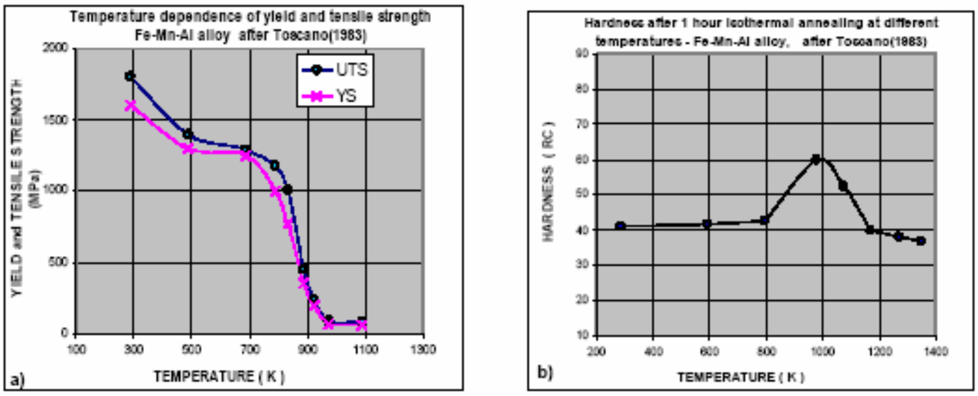

Figure 2. Fe-Mn-Al steel data: **a)** Temperature dependence of Yield and Tensile Strength at different temperatures; **b)** Variation in Hardness after 1 hour isothermal annealing at various temperatures followed by cooling under vacuum. Adapted from Toscano.[2]

2 EXPERIMENTAL PROCEDURES

The material was prepared in the form of ingots weighing 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. 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. Hardness of the material 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. Figure 3 illustrates the sequence of shape changes at each stage in the preparation of the material.

Tensile samples were machined from the stripes in the rolling direction, as shown in Figure 4a, having a nominal gauge length Lo = 10 mm and gauge width w = 3.0 mm, similar to the samples used by Toscano.[2] 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: 8.3×10^{-5}, 8.3×10^{-4} , 8.3×10^{-3} and 8.3×10^{-2} s^{-1}, respectively. The hot tensile tests were carried out on a universal Instron machine model 5500R with a tubular electric resistance furnace. A previous work reported by Cintho et al.[3] with a similar Fe-Mn-Al steel rolled by the same procedure can be used to estimate the grain size of the material, depending on the time and temperature level used in annealing treatments, as illustrated in Figure 4b.

The temperature stability during all tests was about ± 1°C, maintained by P.I.D controllers. There was an interest in carrying out some extra tests: a) at room

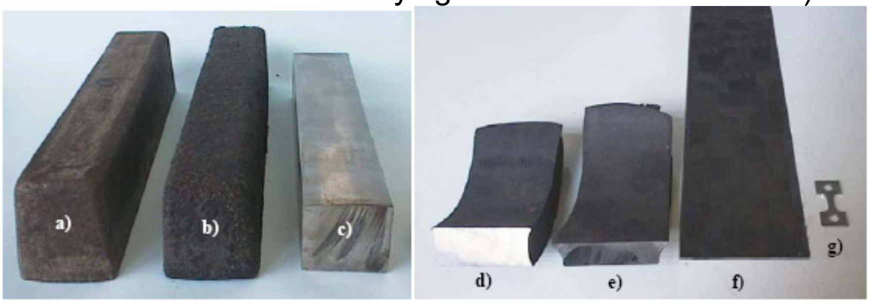

Figure 3. a) Ingots of Fe-Mn-Al steel as cast; **b)** Ingot after solution treatment at 1050°C, during 24 h, quenched in oil; **c)** After grinding operation prior to rolling; **d)** and **e)** slab after different rolling stages; **f)** final shape as stripe with about 1mm thickness; **g)** tensile specimen machined in the rolling direction.

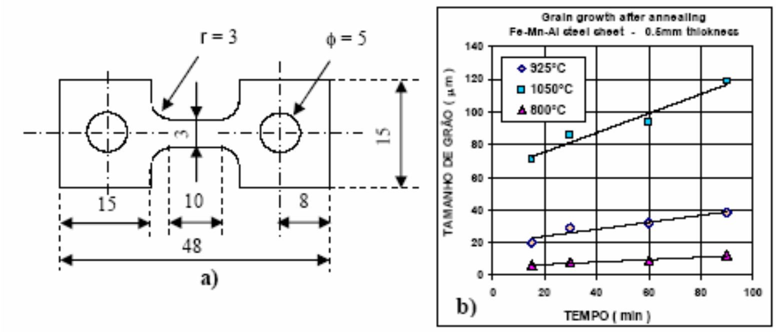

Figure 4. a) Specimen for the hot tensile tests; **b)** Variation of grain size with temperature and time during annealing for Fe-Mn-Al steel sheet with 0.5 mm thickness, according to Cintho et al. [3]

temperature (25°C), 220°C, 420°C, 560°C and 580°C at V_c = 0.5mm/min (the same crosshead speed used by Toscano[2] in his tests); b) four others at 750°C, 825°C, 850°C, 875°C, and 1050°C at 0.05mm/min; c) 850°C and 1000°C at 0.01mm/min; d) 850°C at 0.005mm/min.

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.

Thin sheets of Fe-Mn-Al steel were also produced some years ago by Cintho et al.,[3] using similar alternating sequences of cold rolling/1050°C heat treatments for processing the material. The steel used had the following chemical composition: 21.4Mn-7.9Al-0.64Si-0.44C, and sheets with 0.5mm thickness were manufactured by a final cold rolling pass of about 80% reduction in thickness. The material was heat treated at 925°C for 30min and exhibited a fully austenitic structure with 30μm average grain size and 170 HV_{15} hardness. Figure 4b shows the data of previous annealing treatments on cold rolled samples of the sheet made by Cintho et al[3] illustrating the grain size variation with annealing time at 800°C, 925°C and 1050°C, which were used to select the micro-structural condition of the material. This lower C version of the material was selected intentionally as the objective of their work was the deep drawing performance assessment at room temperature.

3 RESULTS AND DISCUSSION

Figure 5a show examples of typical curves of the hot tensile tests at the same crosshead speeds and different temperatures, and Figure 5b curves at the same temperature and different crosshead speeds. At room temperature the material shows very high yield and tensile strengths in the cold rolled condition, as reported by Toscano.[2] As the temperature increases the strength of the material becomes more and more strain rate sensitive.

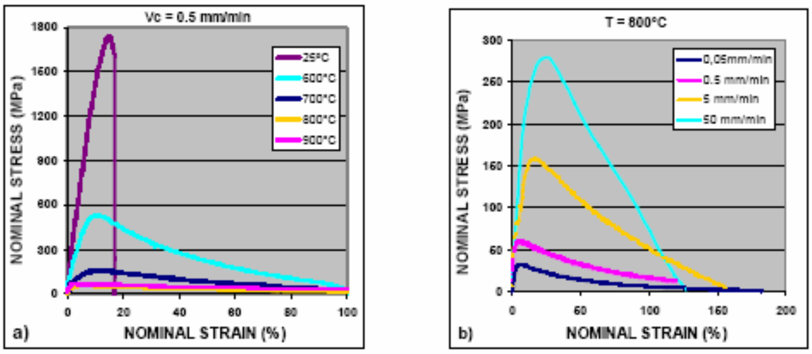

Figure 5. Typical Stress versus Strain curves for the Fe-Mn-Al steel used in this work: **a)** at the same crosshead speed and different temperatures; **b)** at the same temperature and different crosshead speeds.

Figure 6a presents the variation of the tensile strength with temperature at the different crosshead speeds. The pattern of the lines fitted through the point in this figure was envisaged by considering other results which will presented and discussed later in this article. Figure 6b shows a comparison between results of this work with those by Toscano[2] obtained at the crosshead speed V_c = 0.5mm/min, indicating good agreement of the data.

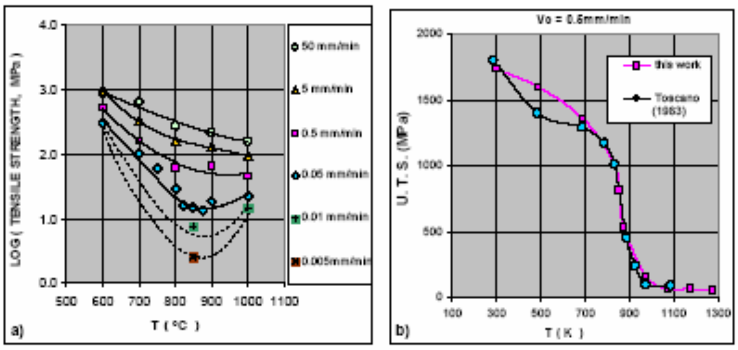

Figure 6. a) Variation of Tensile Strength with Temperature for different crosshead speeds; **b)** Comparison between results of this work with data by Toscano (2) at the crosshead speed V_c = 0.5mm/min.

Figures 7a, 7b, 7c, 7d and 7e show the sensitivity of true stress with true strain rate at the nominal stress peaks, using different specimens for each crosshead speed at 600°C, 700°C, 800°, 900°C and 1000°C, respectively. Although only four different values of strain rate were explored in most cases, the data shows approximately the typical sigmoidal trend mentioned in literature for superplastic materials. The maximum values of **m** were estimated in each case. At 800°C and 900°C the strain rate sensitivity reach maximum values of **m** = 0.49, decreasing to **m**=0.31 at 1000°C. Figure 7f shows a comparison between the sensitivity of stress with strain rate at the five temperature levels using distinct specimens.

Figure 8 presents a typical nominal stress versus nominal strain curve obtained with a single specimen submitted to changes in crosshead speed after reaching the nominal peak stresses. Figures 9a, 9b, 9c, 9d and 9f show the sensitivity of true stress with true strain rate at 600°C, 700°C, 800°C, 900°C and 1000°C, respectively, using a single specimens in each case. The curves also show the sigmoidal trend similar to the Figs. 7a, 7b, 7c, 7d and 7e respectively. The maximum value of **m** could better estimated due to the higher number of points obtained in these kind of experiments. The maximum strain rate sensitivity exponent is observed to increase from about 0.28 to 0.66 as temperature increases from 600 to 900°C, respectively. At 1000°C the maximum sensitivity exponent decreases to 0.42. Figure 9f shows a comparison between the sensitivity of stress with strain rate at the five temperature levels using single specimen in each case.

Figures 10a and 10b show a comparison between the sensitivity of true stress with true strain rate, using single specimens and distinct specimens at 800 and 900°C, respectively, demonstrating good agreement between the results from the two kinds of testing techniques.

Figures 10c and 10d show the variation of the strain rate sensitivity exponent with strain rate using single specimens at 800 and 900°C, with the estimated maximum **m** values of 0.57 and 0.66, respectively. The probable regions of superplastic regime are schematically indicated in each case, assuming a certain pattern of symmetry for the data, as usually reported by various authors (Langdon[1], Maehara[4], Bae and Ghosh[5]).

Figure 10e shows a comparison between the variation of the strain rate sensitivity exponent with strain rate at 800 and 900°C, respectively. The curve at 900°C exhibits a maximum m-value higher than the curve at 800°C, as expected.
However, the curve at 900°C is clearly situated at the left hand side of the 800°C curve, contrary to the normal trend expected for the effect of temperature on such data (Bae and Ghosh[5]). Normally, the increase in grain size displaces the curve to the left and lower its maximum m-value. Figure 10f presents the variation of the strain rate sensitivity exponent with temperature from 600°C to 1000°C, indicating the existence of a maximum around 900°C. Figure 12b agrees notably well with Figure 6a, which also shows evidence of greater strain rate sensitivity between 800°C and 900°C.
Figure 11 illustrates the appearance of some tensile specimens with different elongations after rupture at various temperatures and the crosshead speed V_c= 0.05 mm/min, where the superplastic effect was more prominent. The highest elongation value obtained in the present work was about 320% for the specimen tested at 850°C at $8x10^{-5}$ s^{-1} (V_c= 0.05mm/min). Although the values of the strain rate sensitivity exponent are relatively high (0.57 to 0.66) in the range from 800 to 900°C indicating possibility of superplastic behaviour, the values of final elongation were a little below the expectation, considering estimated values of about 500% at800°C at $8x10^{-4}$ s^{-1} from the work of Toscano.[2] Studies on cold rolled commercial duplex Ni-Cr stainless steels reveal that elongations higher than 700% may be obtained at temperatures above 950°C (Zhang et al[6]) or even above 1500% depending on the steel composition and thermomechanical processing (Maehara[4], Sagradi et al[7]).

Figure 12 shows the variation of hardness and microstructure of the Fe-Mn-Al alloy observed on specimen shoulders after tensile testing at various temperatures, using V_c=0.5mm/min, which correspond to about 25 min under test at each temperature. A remarkable increase in hardness is noticed from about 400°C which reaches a maximum between 600 and 700°C. From 700°C there is continuous decrease in hardness up to 1000°C (the maximum tensile test temperature for V_c=0.5mm/min). These data show reasonable agreement with similar data obtained

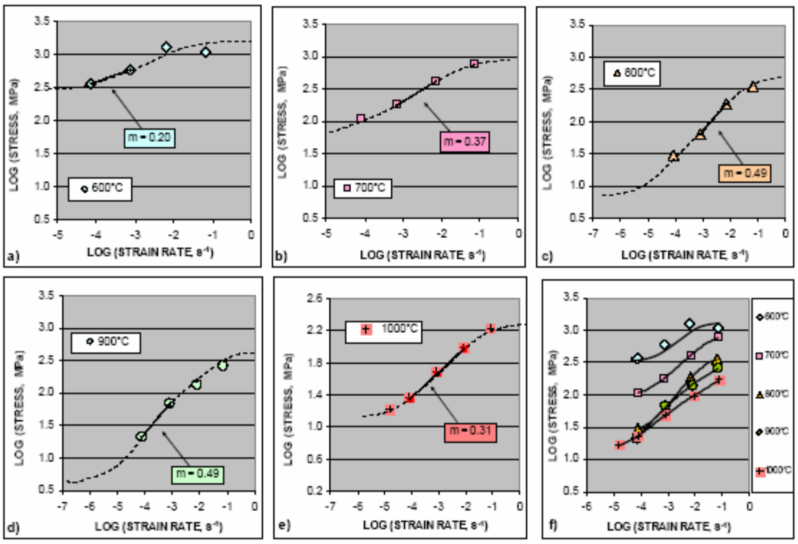

Figure 7. Sensitivity of true stress with true strain rate at the nominal stress peaks, using different specimens for each crosshead speed at : **a)** 600°C; **b)** 700°C ; **c)** 800°C ; **d)** 900°C; **e)** 1000°C; **f)** comparison between the sensitivity of stress with strain rate using distinct specimens at different temperatures and strain rates.

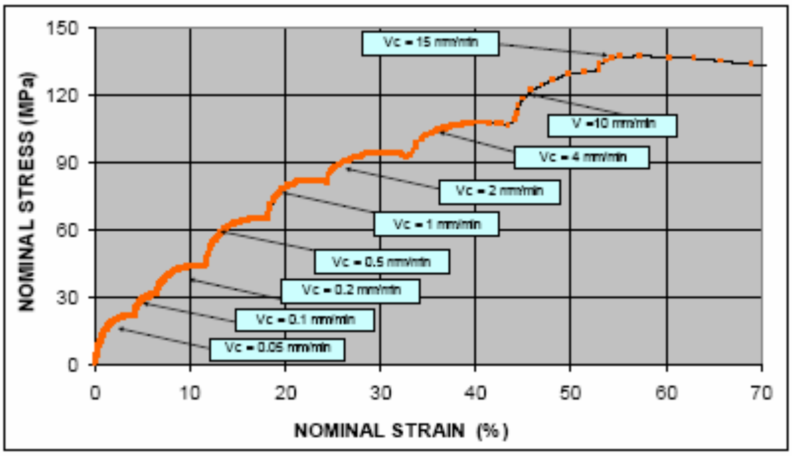

Figura 8. Typical nominal Stress versus Strain curve with changes in crosshead speed after reaching the peak stress. T = 800°C.

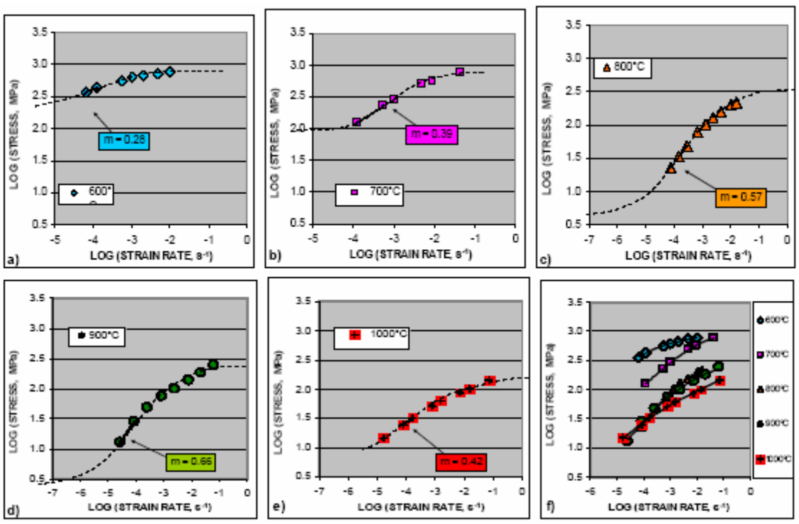

Figure 9. Sensitivity of true stress with true strain rate at the nominal stress peaks, using a single specimen subjected to changes in crosshead speed at: **a)**600°C; **b)** 700°C; **c)**800°C; **d)**900°C; **e)**1000°C; **f)**comparison between the sensitivity of stress with strain rate using same specimens at different temperatures and strain rates.

by Toscano,[2] as illustrated in Figure 2b. According to this author, however, the increase in hardness is noticed only from 500°C and a much slower decrease in hardness observed from 900°C. The present results were obtained using the Vickers Hardness method with a load of 50 kgf which produced reliable hardness values considering the minimum thickness required by the sample. The results by Toscano,[2] however, were obtained using the Rockwell C method which does not seem appropriate for the thickness of the samples used in his research (0.6 mm).

Figure 12 also illustrates the micro-structural changes noticed on the material from its original condition after cold rolling and after subsequent hot tensile tests with V_c=0.5mm/min, using optical microscopy on the grip regions of each specimen. Figure 12a represents the original condition of the material after the last cold rolling pass of 75% reduction in thickness. The sample shows regions with many deformation bands typical of a highly strain hardened austenite structure. The samples tested at 220°C and 420°C also showed basically the same characteristics of cold worked structure in agreement with the hardness results. Figure 12b (600°C) and 12c (700°C) shows significant micro-structural changes in the material with the presence of austenite, and probably finally dispersed ferrite with type (Fe,Mn)AlC_x carbide (Kayak,[8] Krivonogov et al.,[9] Hale[10]). During its first stage of precipitation this carbide is identified as the k-phase (having an FCC ordered structure). In Figure 12c (700°C) the highly deformed austenite structure seem to have been partially re-crystallized. Figure 12d (800°C) reveals the complete recrysta-
llization of the material, which changed to a mixed fine-grained austenite ferrite / micro-structure.

Figures 12e and 12f show progressive austenite grain growth, with the ferrite phase remaining along the grain boundaries. Grain size estimation of the austenite structure in these specimens were as follows: 2 - 3μm at 800°C, 10 -15μm at 900°C, 100 -150μm at 1000°C. Nassour[11] investigated the aging behaviour of a Fe-32Mn-8Al-1.5Si-1C alloy at 500°C, 600°C, 700°C and 800°C, for times varying from 0.25 h to 1000h. The alloy was subjected to a final cold rolling pass of about 45% reduction in thickness, but before the aging experiments it was treated at 925°C for 1 hour to exhibit a single phase austenitic structure with average grain size of 47μm and 303 HV_5 hardness. At 500°C, 600°C and 700°C Nassour[11] noticed the presence of Fe_3AlC and ferrite during the process of hardening of the material. The presence of the $FeMn_4$ compound was also verified during first stages of hardening at 500°C and 600°C, but for longer times during overaging, this phase was absent. At 800°C only the presence of austenite and ferrite was detected. No evidence for the β-Mn phase was verified in his work. The sequence of microstructures observed by Nassour[11] were very similar to those shown in Figure 14. Toscano[2] attributed the appearance of superplastic behaviour in Fe-Mn-Al steel to the movement of the rigid particles of β-phase in the "non-Newtonian fluid" of the γ matrix. In the present research the best superplastic condition happened between 800°C and 900°C, where **m** varies from 0.57 and 0.66 respectively. According to Figures 12d and 12e only austenite and ferrite seem to be present in the microstructures at these temperatures.

Therefore, superplastic behaviour noticed in the present work seems to be related to the fined grained mixed austenite/ferrite structure. As substantial grain growth occurs with increase in temperature, the material exhibits lower superplastic performance, as noticed at 1000°C, where the strain rate sensitivity exponent decreased to **m**=0.42 according to Figure 10f. The trend of the lines fitted through the data at the lower strain rate levels in Figure 6a, revealing presence of minimum UTS values around 850°C, seems to be quite reasonable. The pattern of distribution

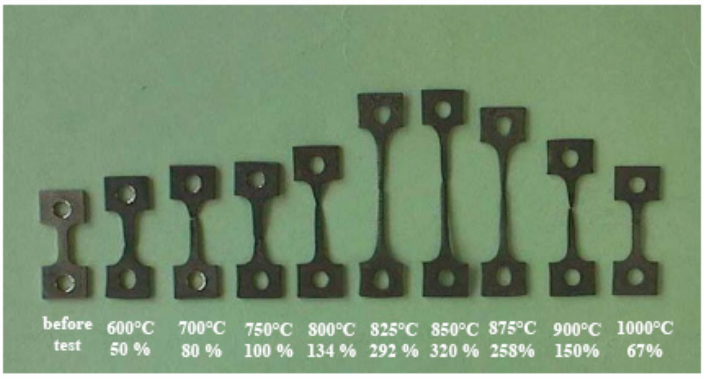

Figure 11. Tensile specimens having different rupture elongation at different temperatures and same crosshead speed V_c= = 0.05 mm/min.

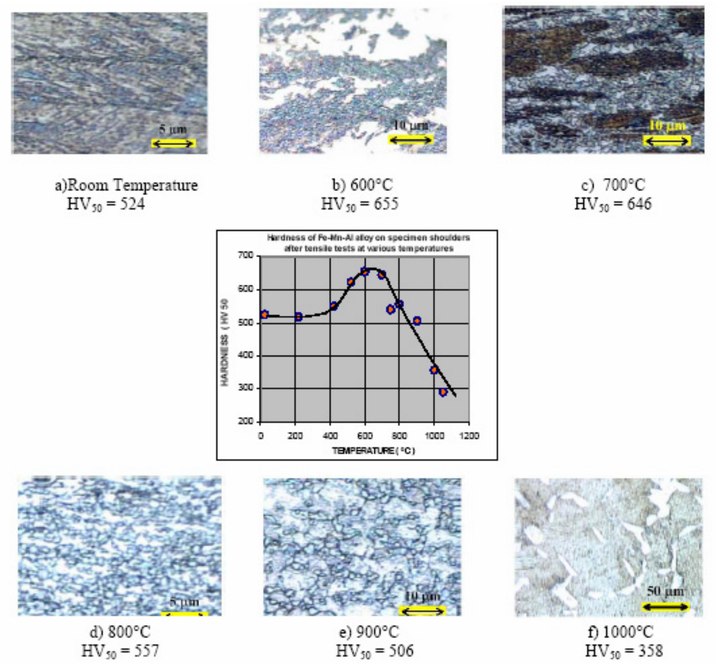

Figure 12. Variation of hardness and microstructure of the Fe-Mn-Al alloy observed on specimen shoulders after tensile testing at various temperatures, with V_c= 0.5mm/min, i.e. about 25 min under test at each temperature.

of the points at each temperature level in this Figure is remarkably well connected with the shape of the S curves shown in Figures 7a,7b,7c,7d and 7e and also in Figures 9a,9b,9c,9d and 9e, which were used to determine the maximum **m** values of the material. The S curves in Figures 7 and 9, however, are defined by the true stress values at the UTS point in each test.

4 CONCLUDING REMARKS

This work represents a first assessment of the superplastic properties in this material and is still in progress. It will be complemented by a new series of tests to define the better combination of temperature and strain rate conditions where the material could show better superplastic behaviour. More studies involving microstructural observation on the deformed regions of the specimens would be necessary to confirm and understand the superplastic effects in this material. Instead of taking the material for the tensile tests directly in the cold worked condition, other annealing treatments could be explored to produce a stable fine grained structure before the tests, as done by Cintho et al.,[3] or possibily the use of adequate thermomechanical treatments for grain refinement, similar to those employed in the case of the Fe-Ni-Cr duplex stainless steels.

The set of results of the present report indicates only a modest superplastic performance for the Fe-Mn-Al steel selected for this study. The maximum elongation of about 320% at 850°C/$8.3x10^{-5}s^{-1}$ is rather below the result reported by Toscano(2) of about 500% at 800°C/$8.3x10^{-4}s^{-1}$. Compared to the superplastic performance of Fe-Ni-Cr duplex steels the results are still more disappointing. However, more investigation is needed to confirm the present results and certainly other alloy versions of the Fe-Mn-Al system need to be considered to assess the superplastic potential of these kind of material.

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