MATERIAL PARAMETERS AND MICROSTRUCTURE ON SUPERPLASTIC BEHAVIOR OF AN AUSTENITIC Fe-Mn-Al STEEL

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Abstract. This work aims is a discussion of relationship between the characteristics and microstructure of an austenitic Fe-Mn-Al steel, presenting two methods of approach the characterization of superplastic behavior. The first method using a a solubilized and cold rolled material, without annealing, obtaining around 1 mm thickness sheets with equiaxial fine grain, dual phase austenitic/ferritic structure, prior to prepare samples for mechanical tests. The second approach added annealing treatment to thermo mechanical processing route. Since the strain rate sensitivity of flow stress (m), which is well known and recognized as an important factor in determining superplastic ductility, with the use of tensile and creep tests procedure. The first approach determined m and the second one the stress exponent (n) related to m. This second method of approach made rationally some improvement on characterization of superplastic behavior, comparatively with results obtained from performed tensile tested samples at temperatures higher than 700°C (of previous work results using the first method). It was possible getting appreciable improvements using results obtained from samples prepared through this second thermo mechanical processing route showing varied results observed in characterization of superplastic behavior from different test procedures used.
Keywords: Fe-Mn-Al steel, microstructure, strain-rate sensitivity, superplasticity

1.INTRODUCTION

The most important characteristics associated to high elongations occur in region II, with progressive characteristics drop in both I and III regions of a usually three-stage relationship in the steady-state strain rate ($\dot{\varepsilon}$) dependence of the applied stress (σ), according to Langdon [(1)],where region II is the superplastic region.

The austenitic Fe-Mn-Al steel may exhibit good combination of mechanical properties as strength, ductility, lower density and corrosion/oxidation resistance.This material has being considered an alternative to materials as Fe-Ni- Cr stainless steels in some applications. At high temperatures however, this mechanical behaviour still remains few explored in creep and superplastic areas, for instance. The preliminary study of superplasticity in those steels, as presented by Toscano [(2)], shows some possibility of exploring its potential use for temperatures higher than 700°C. The studied steel with a chemical composition (wt %) Fe-32Mn-11Al-1.5Si-1.0C, a fully austenitic structure submitted to high reductions using hot-rolling was finished by 40% cold-rolling to obtain 1mm thickness sheets. Tensile isothermal tests were carried out at a fixed crosshead speed of Vc = 0.5 mm/min, showing a drop in yield and tensile strength, with increase in hardness in temperature range from 800 to 1000K. This author [(2)] suggested to occur austenite in grain boundaries, due to the β-manganese phase precipitation, which promotes a very a fine grain size structure from 913K.

This present work aims is discussion of relationship between microstructure and characteristics of superplasticity behavior in austenitic Fe-Mn-Al steel . So two test methods were used to characterize such behavior.

The first method using an approach comprising of solubilization and cold rolling, without annealing processing route, obtaining around 1 mm thickness sheets. This process let an equiaxial fine grain structure with austenitic/ferritic dual phase. Thus the prepared samples were used for a set of mechanical hot tensile tests. Followed by the second set of tests using another thermo-mechanical processing routes, a combination of tensile and creep tests.

This second combined test method rationally made some improvement on characterization of austenitic Fe-Mn-Al steel superplastic behavior. It was possible to obtain appreciable improvements in such characterization, comparatively the previous work results (first method). The performed samples, in this case, were prepared from solutionized, cold rolled and annealed material, hot tensile and creep tested at experimental temperatures higher than 700°C showing varied results observed from different test procedures used.

2. EXPERIMENTAL PROCEDURES

The first method used to characterization, the material, with chemical composition (wt %) determined as: Fe– 24.5Mn– 6.5Al– 1.5Si– 1.1C– 0.009P– 0.016S was prepared in the form of ingots weighing about 3.5 kg with approximately 50 x 50 x 220 mm each. Such ingots were first submitted to solution heat-treatment at 1050^{o}C for 24 hours, followed by quenching in oil. These ingots were grinded to square all the faces before sectioning in two slabs sample each with about 25 x 50 x 200 mm, with hardness of 286 HV_{30}. The slabs were subjected to three series of cold rolling steps 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 a stripe shape with 1mm of final thickness. Samples were machined in the rolling direction from these stripes, with width w = 3.0 mm and nominal gauge length Lo = 10 mm, dimensions similar that samples used by Toscano[(2)]. Tensile tests were carried out mainly at 600^{o}C, 700^{o}C, 800^{o}C, 900^{o}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 experiments were performed in a universal Instron machine model 5500R with tubular electric resistance furnace, with temperature stability maintained in $\pm$ 1°C by P.I.D controllers during all tests. The variation of sensitivity stress with strain rate for each combination of crosshead speed with temperature was observed using *distinct specimens*, and those variation of stress x strain rate subjected to several crosshead speed changes during a certain temperature level were observed using *single specimens*. A previous work, with similar steel rolled by the same procedure, depending on the time and temperature level, could be used to estimate the grain size of the annealed material, as reported by Cintho et al.[3].

The second method used the same procedure plus annealing (850°C/1 hour) chosen of previous annealing and grain growth treatments performed at 800°C, 850°C and 900°C for 1 hour each, to prepare the material. Then the ingots were grinded to square all the faces before sectioning the sample in two slabs, with hardness 286 HV_{30} , and 25 x 50 x 200 mm each. The slabs were subjected to three series of cold rolling deformation steps followed by heat treatments of 1050°C/1 hour. The accumulated deformation levels after each step corresponded to 25, 50 and 75% reduction in thickness, followed by a continuous cold rolling, after the last solution treatment, until a stripe with final shape of 1mm thickness. The samples were machined from these stripes in the rolling direction, with nominal gauge length Lo = 10 mm and width w = 3.0 mm. Tensile tests were carried out at temperature range from 600°C to 1000°C with crosshead speed levels in a range from 0.05 to 50 mm/min, corresponding to initial strain rates range from $8.3x10^{-5}$ to $8.3x10^{-2}$ s^{-1} respectively. These tests were carried out in a universal Instron machine also with same conditions of the first method. The same procedure was used for variation in sensitivity of stress with strain rate. The sample for Creep tests were machined in rolling direction with rectangular shape and fixing role at edge, similar those such used in tensile tests. 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 $\dot{\varepsilon}$ x σ (strain rate versus stress). Tests 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 corresponding weight were verified with a VEB dynamometer of 400kgf using norm ASTM E-139 (1990). The applied stress used in these creep tests were chosen of adopted criterion to associate the experimental tensile peak stresses with respective crosshead speed (V_C) obtained at the same temperature of the tensile tests. These stresses were performed in a wide range of rupture time as load test increase [4].

3. RESULTS AND DISCUSSION

3.1 The first method

Figure 1 shows experimental study results of an austenitic Fe-Mn-Al steel using the first method. This figure shows the hardness variation on specimen shoulders, after tensile tests using V_c=0.5mm/min at various temperatures, which correspond to 25 min under test at each temperature. From 400°C is noticed a remarkable increase in hardness, which is reaching a maximum between 600 and 700°C, with a continuous decrease in hardness from 700°C up to 1000°C (the maximum tensile test temperature for V_c=0.5mm/min).

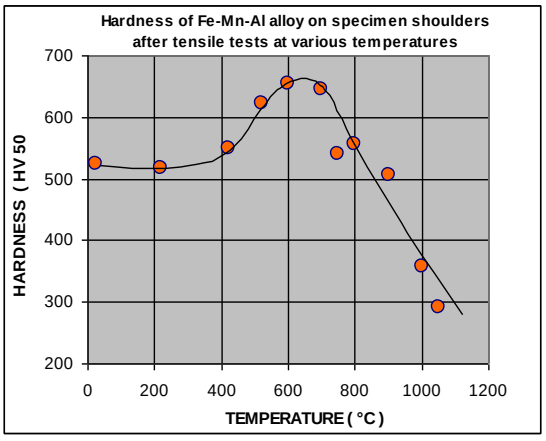
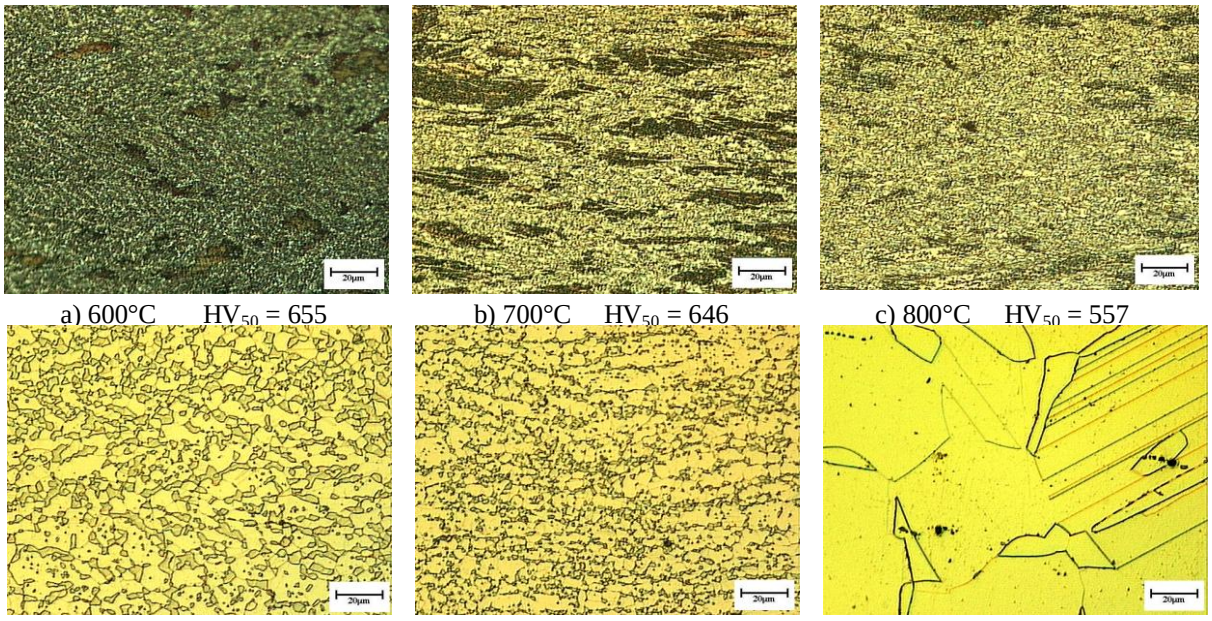


Figure 1. Hardness variation of a Fe-Mn-Al alloy observed on specimen shoulders, after tensile testing at various temperatures, i.e. under test at each temperature.

The data of Figure 1 represents the material condition after last cold rolling pass with 75% of thickness reduction, showing reasonable agreement with those similar ones obtained by Toscano[2]. According this author however, only from 500°C the increase in hardness is noticed and observed from 900°C a much slower decrease in hardness. The results of present work were obtained using a 50 kgf load and Vickers Hardness method, considering the minimum thickness required to produce reliable hardness values by the sample. Those Toscano´s work [2] results, however were

obtained with Rockwell C method, which does not seem appropriate for the sample thickness used in his research (0.6 mm). 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.

Figure 2 illustrates the micro-structural material changes from original condition, on each grip region specimen, after cold rolling and subsequent hot tensile with V_c=0.5mm/min tests using optical microscopy. Figure 2(a) (600°C) and 2(b) (700°C) shows significant micro-structural changes due to the presence of austenite, and probably finelly dispersed ferrite with carbide (Fe,Mn) AlC_x type, according to Kayak [5], Krivonogov et al [6], Hale [7]. During the first stage of precipitation this carbide is identified as k-phase, with an ordered FCC structure. The highly deformed austenite structure seems to have been partially recrystallized, Figure 2(b) (700°C). The change to a mixed fine-grained austenite ferrite / micro-structure with complete recrystallization of material is revealed in Figure 2(c) (800°C). The progressive austenite grain growth, with the ferrite phase remaining along the grain boundaries can be seen in Figures 2(d) and 2(e). The austenite grain size estimation of structure in these specimens as shown at 800°C of 2 - 3μm in Figure 2(d); micrograph at temperature 900°C of 10 -15μm in Figure 2(e) and at 1000°C the average grain size range of 100 - 150μm, this latter illustrated in Figure 2(f).



a) 600°C HV_{50} = 655 b) 700°C HV_{50} = 646 c) 800°C HV_{50} = 557

d) 850°C e) 900°C HV_{50} = 506 f) 1000°C HV_{50} = 358

Figure 2**.** microstructure variation of a Fe-Mn-Al alloy observed of shoulders region under tensile with Vc = 0.5mm/min tested specimen at each temperature.

The sequence of microstructures shown in Figure 2 is very similar to those observed by Nassour[8] who investigated a Fe-32Mn-8Al-1.5Si-1C alloy treated at 925°C for 1 hour subjected to a final cold rolling pass of 45% reduction in thickness. Such material exhibited a single phase austenitic structure with average grain size of 47μm and 303 HV_5 hardness, after ageing treatments at temperature range from 500°C to 800°C, for times varying from 0.25 h to 1000 h. This author [8] noticed at temperatures 500°C, 600°C and 700°C the presence of Fe_3AlC and ferrite during hardening process. It was verified at temperatures 500°C and 600°C during first hardening stages, the presence of the $FeMn_4$ compound, but this phase was absent for longer times during over aging. The presence of austenite and ferrite was detected only at 800°C. No evidence of β-Mn phase was verified in his work.

The present research shows the best superplastic condition found between 800°C and 900°C, with m values varying from 0.57 to 0.66 respectively. At these temperatures only austenite and ferrite seem to be present in the microstructures, according to Figures 2(c) and 2(d).

Therefore, the superplastic behaviour noticed in these first method results seems to be related to the fine grain mixed austenite/ferrite structure. Figure 2(f) exhibits lower superplastic performance with occurence of substantial grain growth with increasing temperature as noticed at 1000°C, with decreased in strain rate sensitivity exponent to m = 0.42. The trend of fitted lines at the lower strain rate levels revealing the presence of minimum UTS values around 850°C, which seems to be quite reasonable. The pattern of distribution points at each temperature level, characteristic of such behaviour is remarkably well connected with the usually three-stage S shape curves of steady-state strain rate (έ) x applied stress (σ) relationship. These curves were used to determine the maximum m values defined in each test by true stress values, at UTS point. These results showing evidence of greater strain rate sensitivity values between 800°C and 900°C also agree to each other notably well.

3.2 The second method

3.2.1 Study of recrystallization and grain growth of the material

The desired parameter of characterization superplastic behavior (ε_r), obtained using the first method results were not the wished target. Thus, they were followed by a preliminary study of recrystallization and grain growth treatment performed at several temperatures and time, prior to continue the work using the second method. The recrystallization model letting to refine processing route with increase of strain rate ($\dot{\varepsilon}$), and explains the formation of a more refined equiaxial structure. Thus improving the material characterization as a desired aid to understand such condition, but even so not completely explaining superplastic behavior.

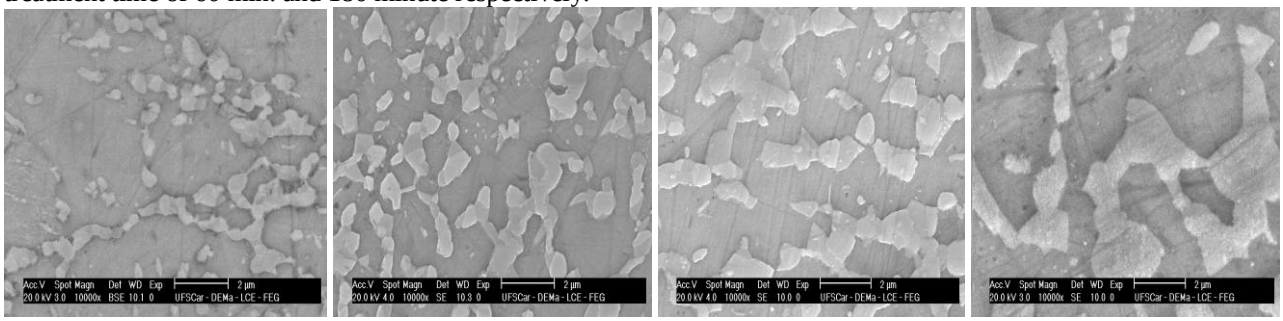
Table 1 illustrates this recrystallization and grain growth heat treatment study showing the used temperature and time with 7x10x1mm samples, where chose 5 sample measured / hardness at face with 7 x 10 mm. Hence, after data analysis better treatment conditions of lower hardness value were chosen to subsequent series of thermo-mechanical treatment.

Table 1. Temperature and time used in study of recrystallization and grain growth of 7x10 mm dimension sample with average hardness HV10 take at room temperature, source: [4] adapted.

T (°C)	AGEING TIME (min)			
	5	20	60	180
25	514	514	514	514
600	576	588	689	798
700	598	624	653	694
800	667	486	459	399
900	336	365	325	333
1000	346	319	320	370

The grain boundary sliding (GBS), which occurs initially with an equiaxial grain structure formation in boundary is considered one of the main superplastic deformation mechanisms, Maehara [9]. So the initial deformation form of recrystallized equiaxial grain structure is not a dynamic recrystallization [10,11]. This condition kept through grain sliding mechanism, without the occurrence of strain hardening structure during whole deformation is sufficient to a new recrystallization. Tthe probable existence of ferritic phase at grain boundary however, was verified during studies using SEM. The phase transformation, during cooling process, have been studied quite a lot recently with attention to austenitic phase formation process, well known for Fe-Mn-Al alloy [12]. Here Mn and Al act as formation elements of austenite and ferrite respectively. Higher proportion of Mn causes appearance of higher austenitic phase at low temperature. Such steel at temperature 900°C, the alloy has proportional component range of 20 to 30% Mn and 5 to 20% Al (% weigth), the same steel frame consisting of ferrite and austenite phases of Fe-Mn-Al alloy studied in this work. Some are of unique ferrite or austenite phase and another are of two phases. It could be observed in Fe-Mn-Al alloy containing low concentration of Al (6,5%) and high concentration of Mn (24,5%) a completely austenitic phase, even at room temperature [12].

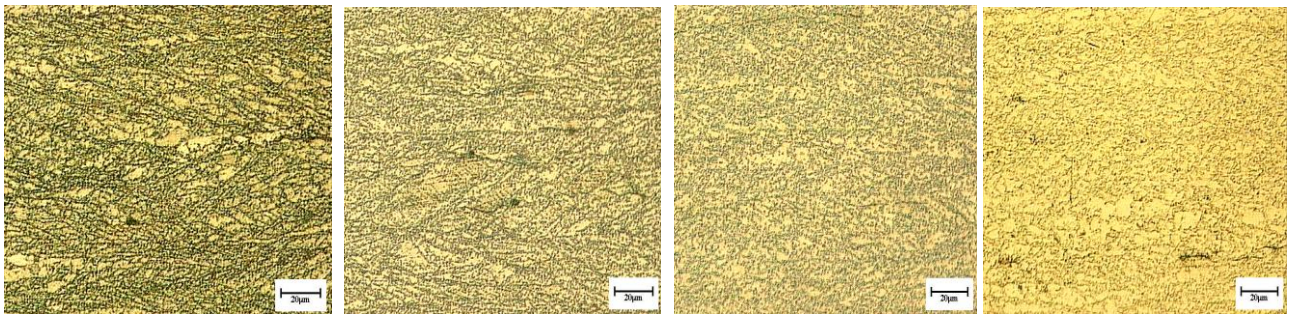
Figure 3 shows FEG/EDS of the same Figure 2 micrograph of austenitic Fe-Mn-Al SRA1, SRA2 and SRA3 steel, observed the precipitation phase among microstructure of samples tested at several temperatures and time through FEG/SE micrography. Figures 3 shows SRA1 materials treated at temperature 900°C in: 3(a) for 5 min. and 3(b) for 20 min. The probable austenite phase precipitation of FCC structure, visible in lighter color is distributed in discontinuous form alongside grain size of ferritic phase, this last visible in dark color in micrograph, which is in accordance with Cheng e Lin [13]. Figures 3 (c) and 3 (d) shows tested samples micrograph at same condition, but with treatment time of 60 min. and 180 minute respectively.



a) 5 min b) 20 min c) 60 min d) 180 min.

Figure 3 treated sample microstruture of FEG/BSE micrograph at temperature 900°C during: a) 5 min.; and FEG/SE micrograph in: b) 20 min., c) 60 min. and d) 180 min

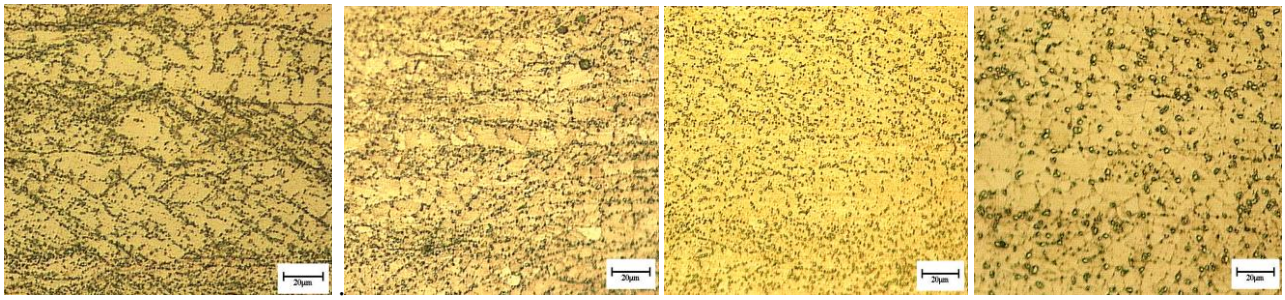
Figure 4 shows austenitic Fe-Mn-Al (SR) steel, after annealing microstructure of sample optical micrograph at 800°C with respective treatment time: a) 5min., b) 20 min., c) 60 min. and d) 180 min. Figure 4(a) shows microstructure after 5 minute tested sample at 800°C where observes average grain size of d ≈ 7, 94 ± 0.3 µm (G11), which structure stands out.The recrystallization process started in some step of the treatment of grain boundary, with new phase precipitation of a fine structure. As the treatment time increases observe a stable structure with not much pronounced grain growth from G11 to G9, seen in Figures from 4(b) to 4(d).



a) 5 min. G = 11 b) 20 min. G = 10 c) 60 min. G = 10 d) 180 min. G = 9

Figure 4. Sample micrograph of austenitic Fe-Mn-Al (SR) steel after heat treatment at temperature 800°C, with time: a) 5 min.; b) 20 min.; c) 60 min. e d) 180 minute, source: [4] adapted.

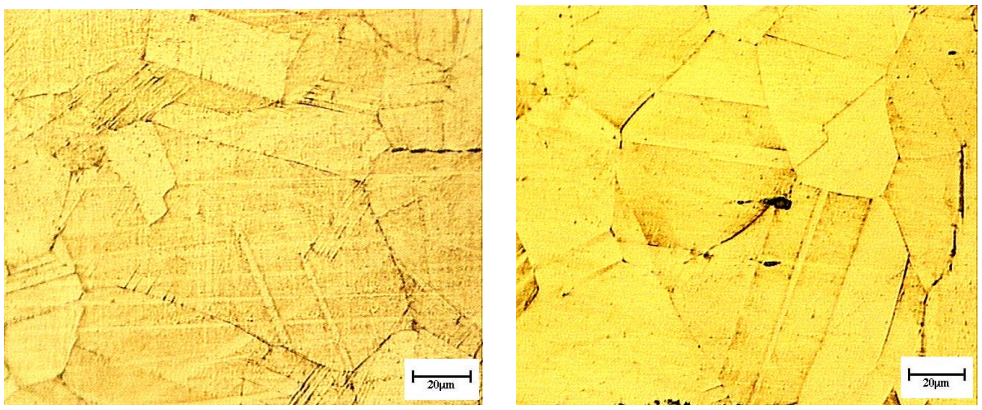
Figure 5 shows an austenitic Fe-Mn-Al (SR) after annealing at 900°C microstructure sample example, of studied recrystallization time and grain growth treatment (Table 1) with time range from 5 min. to 180 min. Figure 3(c) showing in a fine structure with coarse grain stands out grain growth of an annealing results, and also the start of a new phase precipitation in grain boundary during treatment [14].



a) b) c). d).

Figure 5. austenitic Fe-Mn-Al (SR) steel heat treated sample at 900°C, with time: a) 5 min.; b) 20 min; c) 60 min. and d) 180 minute, source: [4] adapted.

Figure 6 shows an treated sample at temperature 1000°C, after annealing treatment optical micrograph (500X) performed at time: a) 60 min. and b) 180 min..The material microstructure, in such temperature and time tests condition, shows a standed out grain growth, indicating a beliavable corresponding region out of superplastic behavior condition. At this same micrograph a probable annealing twin is visible at microstructure.



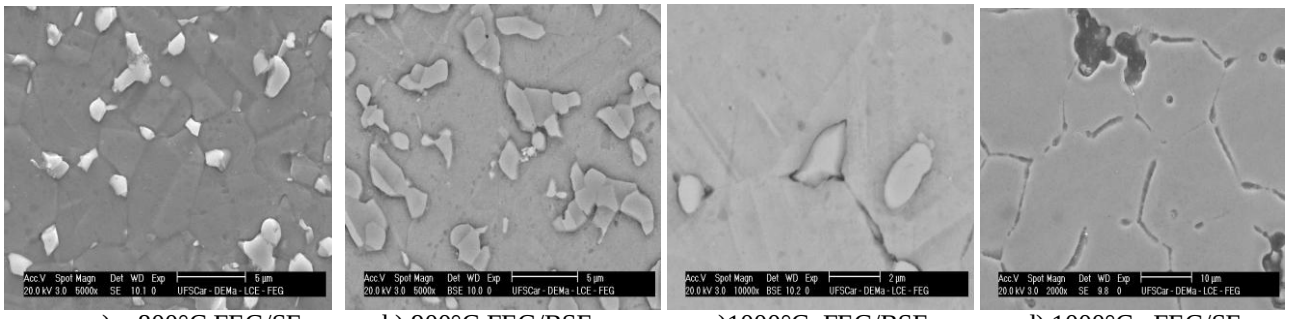
a) 60 min. b) 180 min

Figure 6. Optical micrograph (500X) of treated sample at temperature1000°C with treatment time: a) 60 min. and b) 180 min. source: [4] adapted.

3.2.2 Microstruture of SRA2 material after experimental test

These named SRA materials were performed at 3 annealing treatment used in the next set of experimental tests of SRA1(800°C), SRA2(850°C) and SRA3(900°C) for 60 minute each. The SRA2 hot tensile and creep tested samples, showing up an homogeneous fine grain structure in temperature range from 800°C to 900°C confirming the superplastic characteristic correspondence, and observing dynamic grain growth with coarsening grain structure.

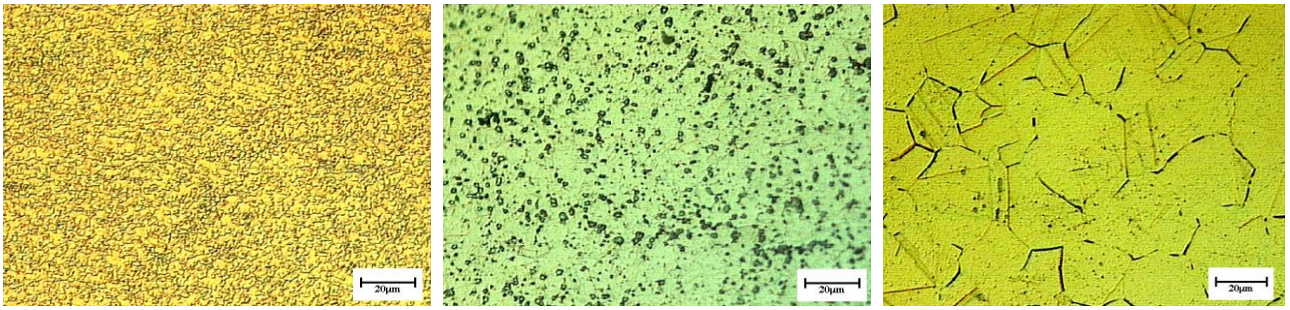
Figure 5 shows micrography of hot tensile tested samples with: Figure 5(a) shows (FEG/SE micrograph) with enhance phase precipitation at temperature 800°C on microstructure, the same as in Figure 3, which observe besides dynamic grain growth of ferritic phase (in dark color), an FCC phase structure probable austenite (visible in lighter color) distributed in discontinuous form alongside grain boundary; Figure 5(b) shows a FEG/BSE micrograph of hot tensile with change V_C tested sample at 900°C; Figure 5(c) shows (FEG/BSE) of hot tensile with change V_C sample tested at 1000°C observing precipitation with angle of grain boundary at triple junction and annealing twin and finally in Figure 5(d) shows a FEG/SE of hot tensile with change V_C tested sample micrograph at same 1000°C, where is noticeable cavitations at grain boundary region.



a) 800°C FEG/SE b) 900°C FEG/BSE c)1000°C FEG/BSE d) 1000°C FEG/SE

Figure 5. SEM micrography of tensile with change V_C sample tested SRA2 material microstructure, at temperatures: a) FEG/SE at 800°C; b) FEG/BSE at 900°C; c) FEG/BSE at 1000°C and d) FEG/SE at 1000°C.

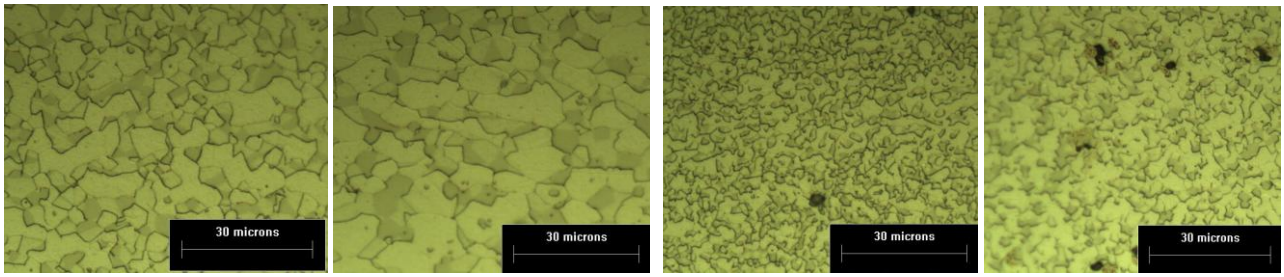
Figure 6 shows micrography of tensile with change V_C tested samples SRA2 material in temperature range from 800°C to 1000°C. Figure 6 (a) shows a fine grain microstructure at temperature 800°C. Figure 6 (b) an enhance dynamic grain growth structure at 900°C and in Figure 6 (c) a pronounced homogeneous coarsening grain microstructure at temperature 1000°C with clear sign of probable annealing twin.



a) 800°C G = 12 b) 900°C G = 10 c) 1000°C G = 6

Figure 6. tensile with change V_C test microstructure of SRA2 material at temperature and grain size (ASTM) of respectively: a) 800°C, G = 12; b) 900°C, G = 10; c) 1000°C, G = 6.

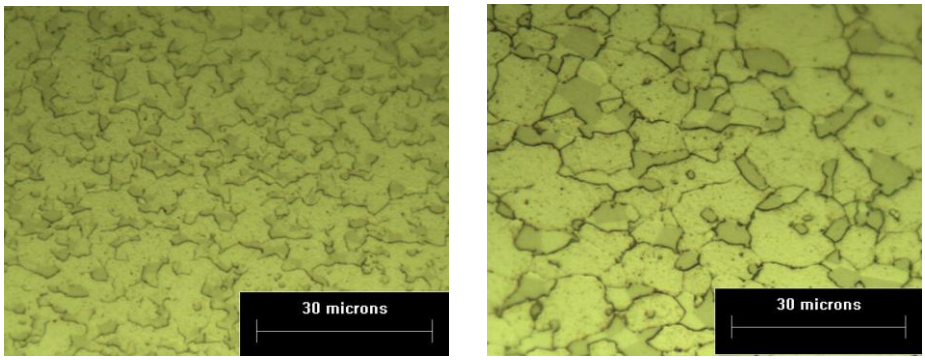
Figure 7 show head grip and gage length (strained) microstructure comparison of tensile tested samples in such condition as: with V_C=0,01mm/min 7(a) head grip and 7(b) gage length (strained) at temperature 800ºC. After t_r = 99,7 hour and ε_r = 598% after rupture, which observed some grain growth after 100 hour test. The tensile with V_C = 0,5 mm/min test results after t_r =2,2 h and ε_r = 660% on Figures 7(c) head grip and 7(d) gage length (strained) . However, there are not any noticeable difference in structure on both grip head and gage length regions.



(a) Head grip (b) gage length (c) Head grip (d) gage length

Figure 7 tensile with constant V_C tested samples microstructure of SRA2 material, V_C = 0,01mm/min, at 800ºC, t_r = 99,7 h,ε_r=598%. a) not strained region G = 10; b) strained region G = 9, and tensile with constant V_C test at 800ºC, at V_C = 0.5 mm/min, t_r =2.2 h, ε_r = 660%; c) G =12 not strained region; d) G =11 strained region, and V_C = 200 mm/min

The SRA2 material after creep with constant load tested carried out with σ = 20 MPa at temperature 800°C showing ε_r = 433% until rupture after t_r = 2,75h. The SRA2 material creep with constant stress tested sample at same temperature carried out with σ = 30MPa, showing ε_r = 737% (without rupture) after t_r = 19,9h. The microstructure of both results are shown in Figures 8 (a) and 8 (b) respectively.



(a) (b)

Figure 8. SRA2 material after creep with constant load tested sample microstructure of: a) (gage length) carried out with σ = 20 MPa at 800°C,with ε_r = 433% until rupture after t_r = 2,75h,; b) same material creep tested with constant σ = 30MPa at 800ºC, with ε_r = 737% (without rupture) after t_r = 19,9h and G = 8.

4. CONCLUDING REMARKS

This work represents the second assessment of characterization of superplastic properties in a Fe-Mn-Al material, which was complemented by a new set of tensile and creep tests, with correction of values during experiments due to define and keep a material with better conditions of superplastic behaviour. The present set of results was based upon different thermo-mechanical processing route, achieving the main objective of improve a thin sheet austenitic steel followed by annealing treatment at 850ºC, generating an equiaxial fine grain structure, with austenite / ferrite phases, and average grain size around 3 µm to characterize a material with superplastic behavior.

The maximum elongation results were obtained on both methods at 800ºC, the first a set of tensile with constant V_C= 0.5 mm/min tests, reached ε_r = 660% after t_r =2.2 h, and the second set of creep with constant σ = 30MPa tested with ε_r = 737%, after t_r = 19,9h (without rupture). Such second method results if compared with those superplastic results of the first work[4] around 320% performance at 850°C/8.3x10^{-5}s^{-1} shows increasing improvement, even better than Toscano[2] results [1] were about 500% at 800°C/8.3x10^{-4}s^{-}. The related microstructure also confirm superplastic potential of this austenitic steel Fe-Mn-Al system with precipitation of new phase from grain boundary due to annealing (recrystallization), steady state fine grain (< 10 µm) structure. Both method results observed with optical and SEM microscopy indicating a practically equiaxial with stable grain structure. These results were obtained even after higher elongation, reached through tensile and creep tested samples SRA2 material in each experimental temperature. The cavitation phenomenon was present in both tensile and creep tested samples, which is another characteristic indication of superplastic behavior. So this desired superplastic condition was reached in the studied austenitic Fe-Mn-Al steel.

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