

PROCESSING OF AN ULTRAFINE GRAINED C-Mn STEEL AND ITS CORRELATION WITH MECHANICAL BEHAVIOR

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Abstract. *In recent years, several studies concerning ultra refinement of ferrite grains have been conducted by different methods (ECAP, ARB, HPT) in order to provide an optimized relationship between mechanical properties and microstructure of the steels. The present work deals with strain induced dynamic formation of ferrite. Samples of low C-Mn steel were deformed in hot torsion aiming at the production of ultrafine ferrite grains. After soaking during 300sec at 900 and 1200°C, the samples were quenched and then reheated and submitted to hot torsion deformation at temperatures of 700 and 740°C. The torsion schedule consisted of 7 isothermal passes leading to a total strain of ≈ 1 , generating an ultrafine grain sizes of the order of 1 μ m after intercritical annealing at 800°C. The shape of stress-strain curves so obtained suggested that ferrite refinement occurred by dynamic recrystallization. The various constituents present in the microstructure, as well as ferrite grain size and morphology, were examined by optical, scanning and transmission electron microscopy. The mechanical properties were evaluated by microhardness tests. The use of multiple regression analysis allowed the establishment of quantitative relationships between the microstructural parameters and mechanical properties of the steel.*

Keywords: *ultrafine grains, ferrite, mechanical properties, multiple regression*

1. Introduction

Ferrite grain refinement by various processing techniques has attracted a significant attention in the last decade. Intense plastic deformation, involving processes such as ECAP (equal channel angular pressure (Shin *et al.* 2001)), HPT (high pressure torsion) and ARB (accumulative roll bonding) are now currently in use in experimental setups throughout the world (Choo, 2001, Niihara *et al.* 2001). ECAP was developed about 10 years ago, as presented in the studies of Valiev *et al.* (1993), demonstrating to be a method capable to promote a great refinement in the steel microstructure. Alternative methods such as ARB and martensitic steel rolling were presented later by Hayashi *et al.* (1999), Saito *et al.* (1999) and Ueki *et al.* (2001, 2002), respectively. Some of these processes, however, are disadvantageous either due to their complexity or to their inapplicability to large scale/volume production of materials. On the other hand, a controlled thermomechanical processing that involved intense deformation, phase transformation and deformation induced ferrite formation, if feasible, could be a suitable way to obtain a more refined microstructure at plant level, producing the superior results compared to the controlled rolling products (Hawkins and Shuttleworth, 1979) (Santos and Barbosa, 1987, 2003), (Hayashi *et al.*, 1999), (Hurley *et al.*, 1999), (Hickson, *et al.*, 1999), (Mabusushi, *et al.* 1999), (Rodrigues *et al.*, 2001), (Kaibyshev, 2001), (Beladi *et al.*, 2004).

The industrial standard processing of these steels is done by controlled rolling with accelerated cooling in order to reach austenite microstructure refinement. This class of HSLA steels can reach yield strength from 350 up to 500 MPa, because grain refinement has a limit around 15 μ m. These steels are employed at automotive industry, to fabricate high diameter gas and oil transportation pipe lines for low temperatures works and plates to naval industry (Tanaka, 1984; Tamura, 1988).

On the other hand, ultrafine grained ferrite microstructure with grains from 1 up to 5 μ m in low carbon-manganese steels has been object of many works (Hayashi *et al.*, 1999), (Mabusushi *et al.*, 1999), (Kelly and Hodgson, 2000), (Kaibyshev, 2001), (Ueki *et al.* 2002). In these steels with ferrite structure grains of 1 μ m, it was observed yield and tensile strength around 400MPa and 600MPa, respectively. The ductility has showed very good results, with data for total elongation around 27% (Santos *et al.*, 2003).

The objective of the present work was to obtain an ultrafine grained ferrite matrix with 1 μ m grain size in the low carbon manganese steel; as long as evaluating its mechanical behavior. The cold rolling of martensite and annealing in

plain carbon steel has been carried out in the work of Ueji *et al.* (2002) or Morito *et al.* (2001), but not the warm work. The processing by warm rolling and intercritical annealing has the advantage of industrial process application and the material processed is enough to machine specimens for mechanical tests. Otherwise this route of warm rolling has been only applied to C-Mn steel (Santos, 2003). The objective of using warm torsion here is to closely simulate the industrial rolling process in the laboratory conditions. The deformation was performed at temperatures where the material is composed of a mixture of ferrite and austenite. The subsequent intercritical annealing was applied to modify the substructure remaining after deformation in the two-phase region and to improve ductility. It was also the objective here to evaluate the mechanical behavior of the material using empirical equations, obtained by multiple regression analysis stepwise method, to correlate the microstructural parameters with the strength of the steel.

2. Experimental Procedure

Tubular specimens (16mm length, 6.35mm outer diameter and 2mm inner diameter) were machined parallel to the rolling direction of the industrial plates. The chemical composition of steels used in this investigation is given in Table 1. Figure 1 describes a sequential scheme of the experimental procedure.

Table 1 - Chemical composition of as received material (% weight)

Elements	C	Mn	Si	Al	P	S	N2
(%Wt)	0.15	1.39	0.39	0.039	0.016	0.009	0.0042

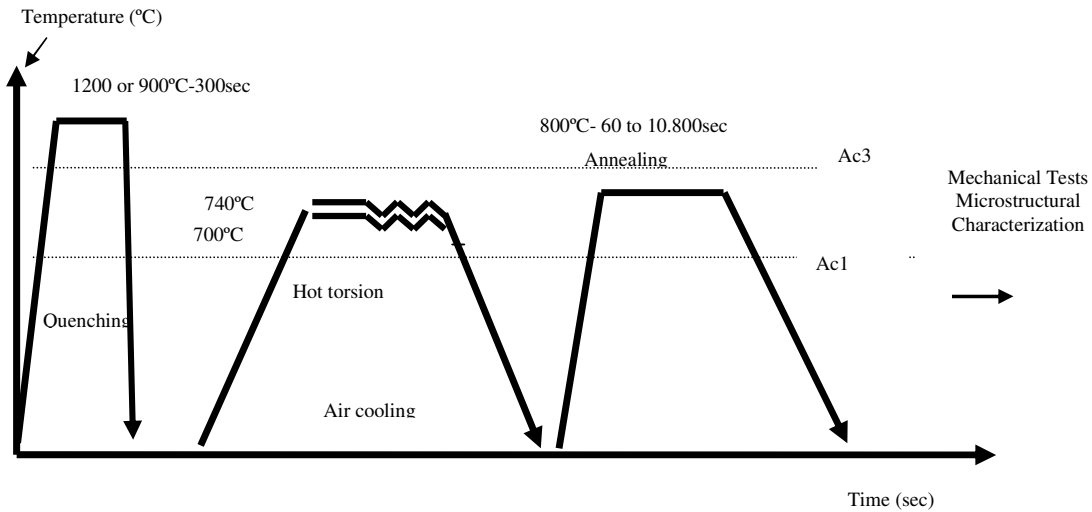


Figure 1 - Sequential paths of the experimental procedure.

The thermomechanical treatment employed in this work (Fig. 1) consisted of three stages: a) austenitization followed by ice brine quenching; b) intercritical warm torsion deformation, and c) an intercritical annealing. Samples were first austenitized at 1220 or 900°C for 300sec and then ice brine quenched. The specimens were then subjected to an intercritical deformation by heating them up either to 700 or 740°C. Ac1 and Ac3 temperatures being for this alloy calculated according to Honeycombe (1980) as 719 and 849°C. Samples were let to homogenize at these temperatures for 300sec prior to straining. This occurred by applying 7 passes of approximately equal deformation per pass leading to a final equivalent true strain at least about 1 at a strain rate of 2sec^{-1} . During warm torsion the specimens were protected by an argon atmosphere into quartz tubes. Details of the torsion equipment employed in the experiments are given elsewhere (Azevedo *et al.*, 2003). All samples were then air cooled to room temperature. After that, the samples were heated to 800°C and soaked for 60 up to 10800sec, then cooled in air.

Nital 2% and LePera etchant (Santos *et al.*, 2003) was used to reveal microstructure, which was analyzed under optical and scanning electron microscopes. Ferrite grain size was measured according to ASTM E562-83 (ASTM, 1983) using image analyzer for grain area determination. Measurements of Vickers microhardness were carried out with a load of 2.94 N (300gf) at a specimen surface parallel to the sample axis.

Selected samples were studied using a Philips CM20 transmission electron microscope (TEM) equipped with an Oxford Link Model MK 6 ultra-thin window Energy Dispersive X-ray spectroscopy detector. Thin foils for TEM were prepared from mechanically ground to 0.25-0.3 mm slices. The 3mm discs were first punched out and then were electropolished in 5wt% perchloric acid in methanol using a twin-jet Tenupol unit, operating at 30V and 0.2A. The

polishing solution was cooled to -20 - 30°C using liquid nitrogen. TEM examination was carried out at 200kV using bright field, dark field and selected aperture electron diffraction modes.

3. Results

3.1. Flow curves

The work hardening and the dynamic softening are two competitive process during torsion, where the balance can be established between them. The generation of dislocations and its intersections during the plastic deformation lead to a hardening, while the recovery and the recrystallization attenuate these effect, for the coalescence, destruction and reconstruction by cross slipping and climbing with aid of the applied tension and the thermal activation, as affirmed by Semiatin *et al.* (1985). Fig. 3 shows examples of stress-strain curves for samples deformed to a total strain of 1.0 and 1.15 at temperatures of 740 and 700°C , respectively. Interpass softening was small in both experiments since the time lag between deformations was set as 1 s, not allowing recrystallization to take place between straining intervals. Analysis of the envelope curves embracing the multi-pass stress-strain curves shows a peak strain of about 0.1 at 700 and of 0.3 to 0.4 at 740°C . The level of stresses at these strains is around 225 MPa. However, as deformations proceeds, the stress level decreases more rapidly for the 700°C samples than for the 740°C one. The stress at the last pass of the sample deformed at 700°C is 200 MPa, about 10% smaller than the 220 MPa measured for the sample deformed at 740°C . This occurs as function of the strain hardening excess that in turn also provokes the dynamic recrystallization of the ferrite present in samples with the remaining austenitic grains. Differences in the shape and level of the envelope stress-strain curves may have resulted from differences in the starting microstructure since the fraction of austenite prior to deformation varied from samples deformed at two different temperatures.

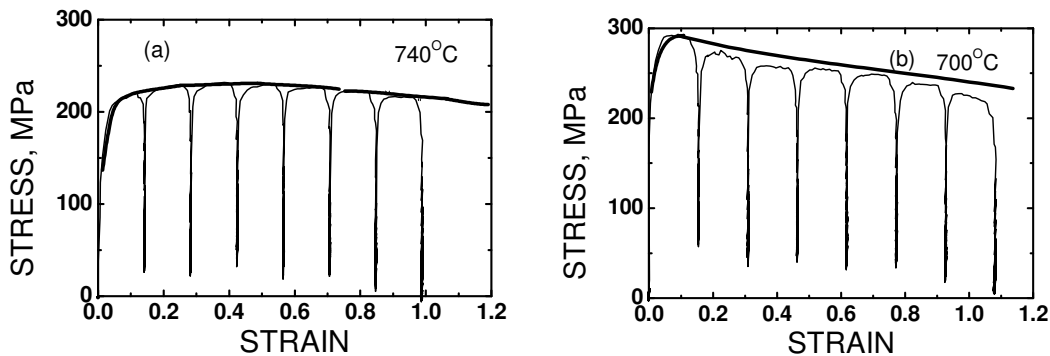


Figure 2. Flow curves for intercritical C-Mn steel deformed samples, (a) 740°C and (b) 700°C .

3.2. Microstructural evolution

The microstructure of the as-received hot rolling steel can be seen in the Fig. 3a. It is a usual mixture of pearlite and ferrite, with grain size around $15\mu\text{m}$. After austenitization at 900 or 1200°C and quenching, the microstructure consists mainly of martensite. Figure 3b shows the microstructure resulting after soaking for 300sec at 900°C and quenching. The prior austenite grains are fairly equiaxed with an average size of $15\mu\text{m}$. This size is similar to that obtained at the end of an industrial hot rolling of C-Mn steel. Fig. 3b shows the characteristic lath martensite of low carbon steel. The objective of starting the intercritical deformation with this microstructure was to obtain a homogeneous and metastable condition, in order to increase ferrite nucleation rate during the deformation. On heating to 700 and 740°C the lath martensite is tempered and becomes mixed with austenite, ferrite and cementite particles. After torsion, the microstructure comprises a highly misoriented ultrafine ferrite grains (Fig. 3c). Figure 3d-e shows the microstructure evolution with annealing time for samples subjected to intercritical annealing at 800°C . It is also evident a slight increase in the ferrite grain size with annealing time. It is possible to identify some heterogeneous distribution of ferrite grain sizes varying from less than 1 to almost 4 to $5\mu\text{m}$. As the annealing time increases, the ferrite grain sizes became more evenly distributed and some carbide dissolution also occurred. During the prior warm working microbands are formed. These microbands coalesce, resulting in sub-grain formation and finally the recrystallized nucleus. Whereas their observation by optical and SEM not grains revealed their presence in some grains, Fig. 3d, f. Otherwise the observation by TEM confirm their distribution in a uniform manner (Beladi,2004). On the other hand, MA starts to appear in the background of the microstructure(Fig. 3d, e).

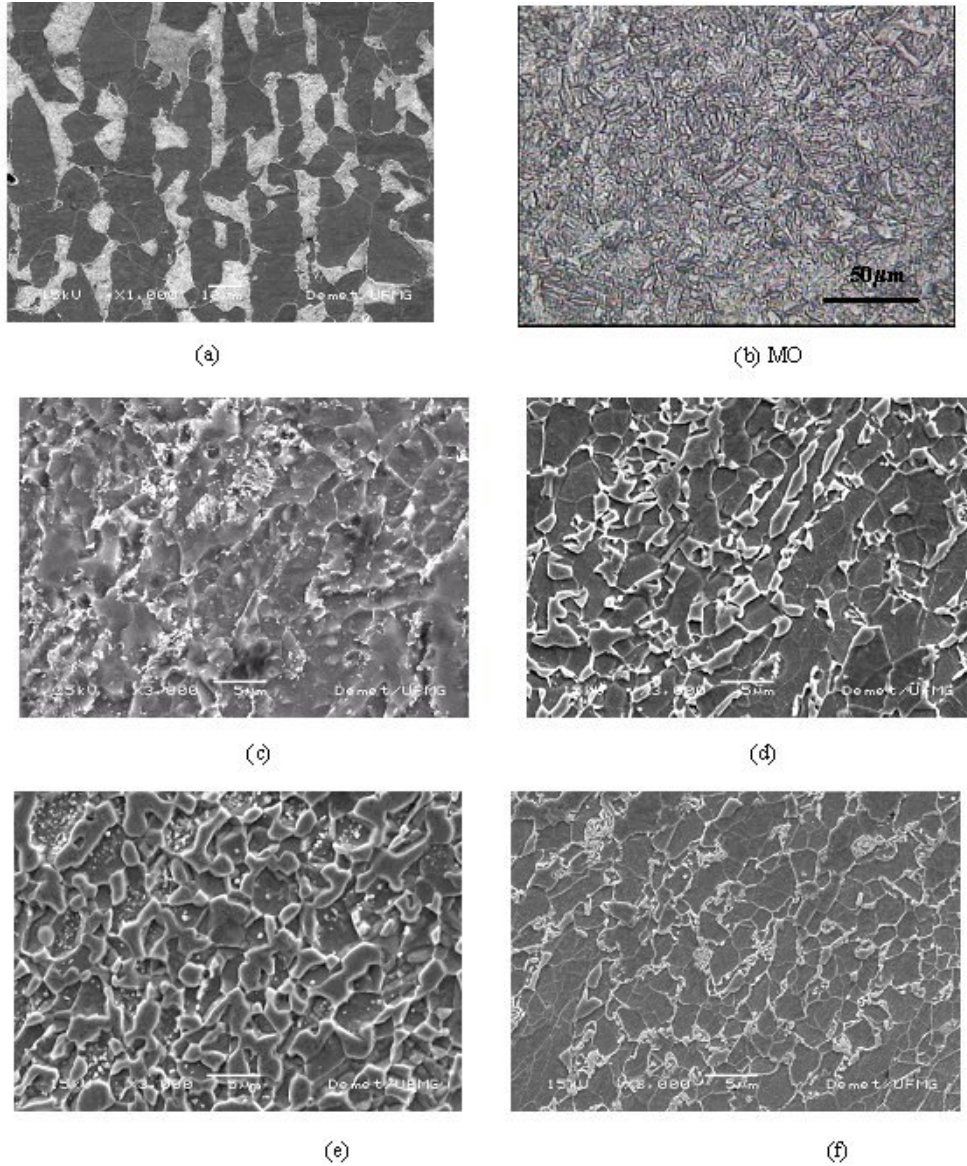


Figure 3. Microstructure evolution with annealing time for samples: as-received (a), quenching from 900°C (b), subjected to hot torsion at 740°C (c) intercritical annealing at 800°C for 60sec (d) for 1800sec (e) for 3600sec (f). (a,c-f) SEM; (b) Optical Microscope.

3.2.1. Transmission electron microscopy

TEM study has shown that the microstructure of C-Mn steel after warm torsion consists of a mixture of elongated polygonal ferrite grains with dislocations within grains interior and nearly equiaxed polygonised grains containing a very low number of dislocations. In some grains, the dislocations formed the cell structure by rearrangement into the dislocation tangles in cell walls and nearly completely free of dislocation cells. This is a result of deformation of ferrite grains during torsion. The latter group of ferrite grains is a product of strain-induced transformation and dynamic recrystallization. In addition to the ferrite, a significant amount of Fe_3C carbides located predominantly at the grain boundaries and triple junctions was observed. Some of the carbides were also found within the ferrite grains (Fig. 4). Located at grain boundaries carbides might pinned them, slowing the movement of grain boundary and leading to the finer ferrite grain size. The shape of carbides varied from the globular to elongated to cuboidal. Energy dispersive X-ray spectroscopy of carbides has shown the substitution of Fe by Mn. Islands of retained austenite were also present in the microstructure (Fig. 4a) and granular bainite (Fig. 4b).

Fig. 5 shows the samples etched with LePera etchant in which the clear constituents appears as almost well delineated islands. It can be seen that MA, distinguished by the bluish colour in the photomicrograph, is widely

present in the sample along with pearlite and isolated carbides. The remainder of the sample, an area of deep gray, represents the ferrite grains. The processed samples after warm working condition show higher microhardness values resulted from the residual strain hardening. This situation in turn makes possible the occurrence of ferrite dynamic recrystallization, which in turn led to a refinement of grain size. Some variations in microhardness also occurred due to the influence of the constituents, as MA and carbides. The volume fraction of MA increases for both steels with annealing time, as it can be seen in the Fig. 6, samples quenching from 900°C. This is a result of austenitization during intercritical annealing period.

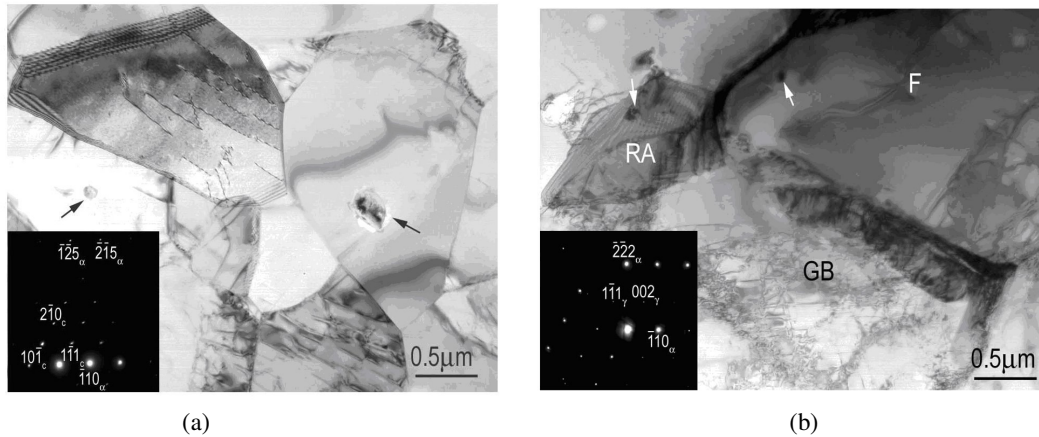


Figure 4. TEM micrographs of C-Mn steel after annealing at 800°C for 600sec (a), 10800sec (b). Zone axis of SAED inset in b is $[553]_{\alpha} // [121]_{\text{C}}$; zone axis of martensite is $[113]_{\text{M}}$ in c and zone axis is $[112]_{\alpha} // [110]_{\gamma}$ in e, where γ is retained austenite, M is martensite, C is carbide and α is ferrite.

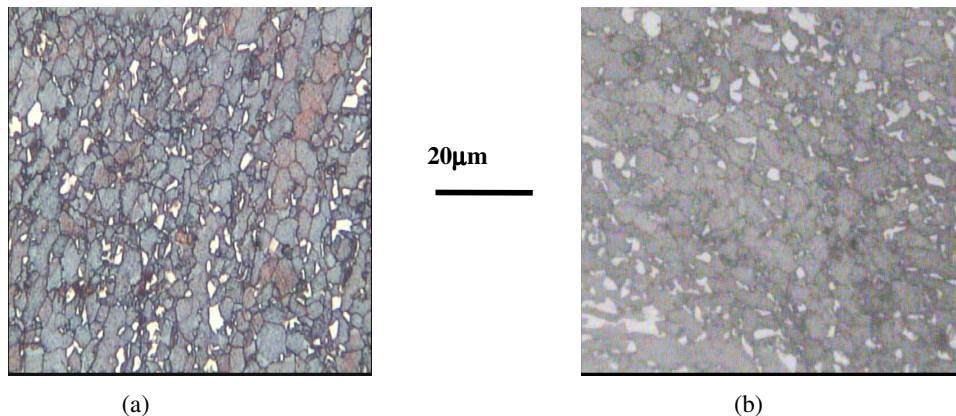


Figure 5. C-Mn steel samples deformed at 740°C (a) and 700°C (b) and annealed during 7200sec. LePera etching, light photomicrographs.

3.3. Microhardness and grain sizes

Fig. 7 shows the dependence of the microhardness on ferrite grain size. Clearly, the hardness drops from approximately 220 to about 185 HV as the ferrite grains coarsen from 0.8 to around 1 μm for both deformation temperatures. As expected, the initial drop in hardness was larger for the sample treated at 740 than for 700°C, that is, 7200sec of annealing led to a drop of less than 10 HV for the sample deformed at lower temperature. The same annealing time led, however, to a drop of almost 20 HV for the sample deformed at 740°C. In fact, the microstructure at 740°C consisted of an aggregate of ferrite grains and martensite in a manner similar to that found at 700°C. Higher temperatures, however, presented, additionally, a fraction of MA and carbides which, together with the original martensite, may have restricted further the growth of ferrite grains as will be discussed at TEM topic.

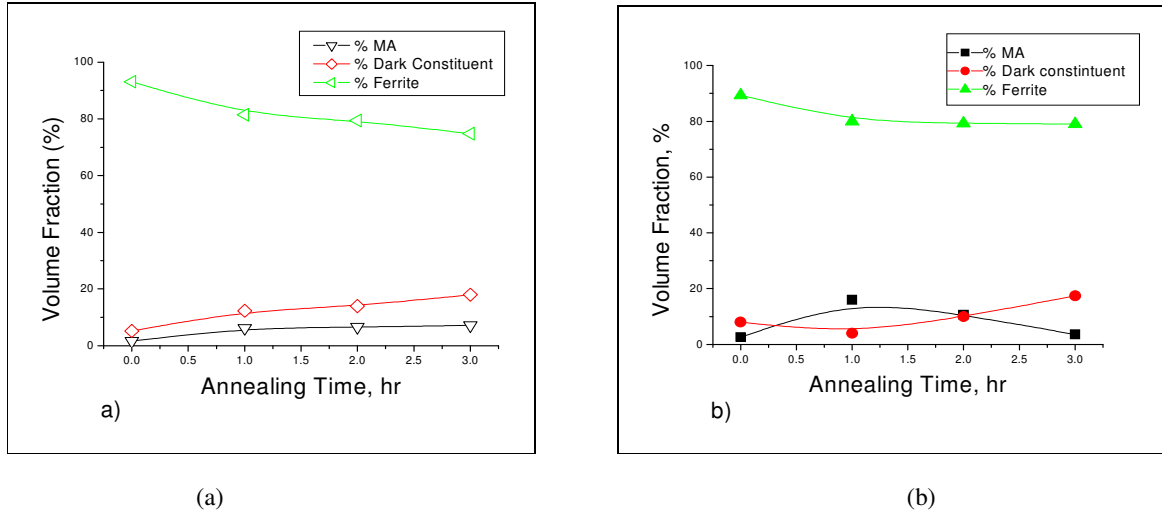


Figure 6. Microstructural evolution as a function of annealing time.
(a) Deformed at 700°C and (b) Deformed at 740°C.

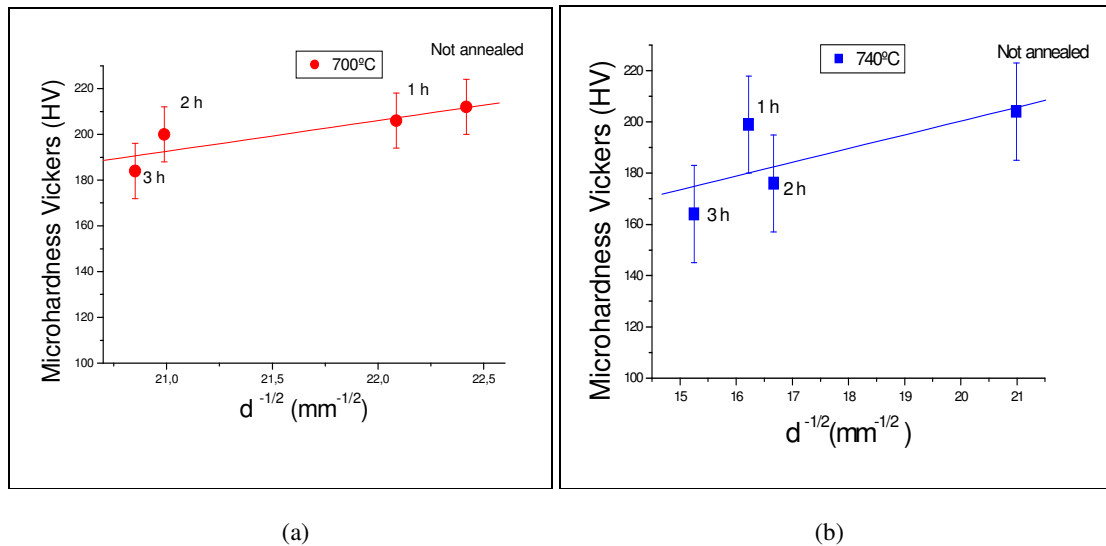


Figure 7. Microhardness as a function of grain size and annealing time for samples deformed at 700 (a) and 740°C (b).

4. Discussion

4.1. The austenite-ferrite transformation and the ferrite recrystallization

The volume fraction of ferrite and how it recrystallizes depend on the reduction given per a pass Hurley *et al.* (1999). The strain rate influences the critical amount of deformation to promote dynamic induced ferrite formation according to Yang, (2001). So does the accumulation of deformation in the unrecrystallized austenite by the increase of possible sites for nucleation of ferrite grains. Intercritical deformation therefore enhances the effects of straining on transformation, thereby refining considerably the ferrite grains originated from the deformed austenite. This is in agreement with results in the literature from torsion experiments (Beladi *et al.*, 2004) where the similar effect was observed, as well as a saturation of austenite grain boundaries prior to the initiation of transformation to ferrite.

The final ferrite grain size does not only depend on nucleation and growth rates, but also it is affected by the holding time at the test temperature, as a consequence, the efficiency of grain refinement that can be reached by thermomechanical processing can be limited. A way to reduce this growth rate during the transformation is to induce the transformation to occur in a short period of time and to quickly reduce the temperature during the transformation, as suggested by Hurley (1999) and Kelly (2000).

The influence of annealing time, ferrite grain size and volume fraction of constituents after the transformation can be accounted for the variation in the mechanical properties, as shown by equations in table2; giving fitting indicator

R2 of 89-97% in these cases. Details of the coefficients of these multiple non-linear fitting are also shown in table 2, only the best values found are presented. The coefficients of HV and d have the expected behavior and sign in agreement with experimental results, as they very well documented in the literature, i.e., increasing the annealing the ferrite grain size increase and the hardness decrease (Morrison, 1966).

Table 2. Microhardness as a function of the annealing time, ferrite grain size and volume fraction of constituents

900°C deformed at 740°C	$HV = 205 - 13.6 \cdot T \text{ (hr)}$	R2= 93.5%
1200°C deformed at 740°C	$HV = 52.9 + 6.63 \cdot d^{-1/2}$	R2= 96.5%
1200°C deformed at 740°C	$HV = 72 + 4.73 \cdot d^{-1/2} + 2.81 \cdot (\%MA) + 0.39 \cdot (\%DC)$	R2= 89.8%
1200°C deformed at 740°C	$HV = 47.3 + 7.09 \cdot (d^{-1/2}) - 0.80 \cdot (\%MA) + 0.217 \cdot (\%DC)$	R2= 97.4%

where 900 and 1200°C correspond to the austenitization temperatures, and 700 and 740°C are the torsion deformation temperatures. Where HV = microhardness, $d^{-1/2}$ = ferrite grain size (mm)^{-1/2}, %MA= volume fraction of MA and %DC= volume fraction of dark constituent.

The work of Priestner *et al.* (2000) report an equation relating microhardness as function of ferrite grain size in ultrafine grained low carbon steel: $HV = 130 + 3.25 d^{-1/2}$. The comparison of the equations presented here reveal the same behavior. but with different values for coefficients. The expression for 900°C and deformation at 740°C is the closest to Priestner equation.

These equations show that the volume fraction of MA constituent has a dubious effect on the mechanical behavior. One reason is their small amount. but the grain size of ferrite and dark constituent. cementite particles, volume fraction have a marked influence on this mechanical properties. The value of R², multiple correlation coefficients, has reached a high value for some specified conditions.

5. Conclusions

Hot torsion testing with multipass deformation was used to study the effects of thermomechanical parameters on ultrafine grain ferrite formation by dynamic recrystallization and intercritical annealing of the C-Mn steel.

The final grain size reached dimensions around 0.8 - 4µm. There seems to be a limit of saturation to the nucleation of new ferrite grains. indicating that the processing is the preponderant factor in the attainment of these results. Ferrite grain size can be considerably refined by the thermomechanical treatment presented here. However not severe accumulation of strain was applied of around 1. Ferrite grain growth during intercritical annealing is not excessively, for the conditions of temperature and deformation employed here. Ferrite grains varied from about 0.8 to 1.2µm and just above 4µm for prolonged annealing periods of 3600sec and higher quenching temperature.1200°C; leading to microhardness values between 220 and 185 HV, or expected UTS values of 660-555 MPa.

Using a regression analysis it was possible the establishment of quantitative relationships between the microstructural parameters and mechanical behavior of steel that to permit foresee the mechanical properties as a function of thermomechanical processing applied to the steel. This approach avoids the complex simulation experiments and spending of time.

6. Acknowledgments

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