JP&M Engineering

Journal of Petroleum and Mining Engineering

Industrial Production of Fine Grained Ferrite in Low-Carbon Steel

Ahmed El-Kawas[a]*, Sabbah Ataia[b,c] and Samir Ibrahim[b]

[a] Ezz Aldekhela (EZDK) Steel Company, Alexandria, Egypt

[b] Department of Metallurgy and Materials Engineering, Faculty of Petroleum and Mining Engineering, Suez University, 43721, Suez, Egypt

[c]Department of Mechanical Engineering, College of Engineering, Al Imam Mohammad Ibn Saud Islamic University, Riyadh 11432, Saudi Arabia

*Corresponding author: e-mail: aelkawas@ezzsteel.com.eg

Telfax:(002) 033082667

Mob.: (002) 01223449755

Abstract

Refinement of the ferrite grains provides a promising approach to simultaneously improving both the strength and the toughness of steels. Among recent techniques to obtain ultrafine ferrite, dynamic strain induced transformation (DSIT) is used. This type of treatment is sensitive to steel composition, deformation temperature, the prior austenite grain size, strain and cooling rate.This work is aiming to improve the mechanical properties of steels using ferrite grain refining through trials on industrial scale using a compact strip production plant (CSP) with hot strip mill (HSM) containing six stands F1 – F6 in EZDK Steel Company. The chemical composition of the trials was ~0.05% C, 0.05% Si and 0.5% Mn. The effect of the finishing deformation temperature at the last rolling stand (F6) on the microstructure and mechanical properties was studied. In addition the effect of changing strain at the final two rolling stands (F5 and F6) was carried out during trials. Comparing the traditionally processed steel which resulted in a ferritic grain size of 11µm with the processed fine grained (~5µm) steel, the yield stress has increased by 23% and the tensile strength increased by 12% on the expense of only 5% decrease in the ductility. However, the impact energy has improved by 7.5 %.

Keywords

Ferritic rolling; Grain refining; Dynamic strain induced transformation.

Introduction

Grain refinement is considered as one of the most effective strengthening methods, since it improves both the strength and the fracture resistance. Moreover, it hardly has a deteriorating effect on ductility and weldability, despite the achievement of considerable strengthening [1]. Ultrafine grained structure in plain carbon steels is gaining high research interest and considered as means of lowering the cost of steel production and opening up the window of high band mechanical properties of steels [2]. There are several methods to obtain fine grains such as severe plastic deformation, dynamic recrystallization, dynamic strain induced transformation and intercritical rolling [3]. Ferrite grain size refinement has been achieved commercially through dynamic strain-induced transformation (DSIT), in which the austenite-to-ferrite transformation is enhanced by the dynamic deformation within an appropriate temperature range. However, DSIT is a complex procedure involving interactions of diffusion of solute atoms, evolution of dislocation, propagation of grain boundaries and phase transformation. Furthermore, it is important to relate this critical condition to the thermo-mechanical parameters that can be controlled during a process [4].

The aim of the present work is to increase the strength and toughness of the rolled commercial steels by grain refining of the ferritic steel structure by dynamic strain induced transformation through on-line industrial production trials. Mechanical properties are planned to be improved by changing the thermo-mechanical processing parameters such as rolling temperature, strain rate, and strain especially at the two last rolling stands.

Experimental work

Material and production route

Trials were carried out using aluminum killed low carbon steel. The steel chemical composition of the conducted trials is listed in **Error! Reference source not found.**. The rolling process was performed using a compact strip production plant (CSP) with hot strip mill (HSM) containing six mill stands F1 to F6 followed by early laminar cooling. The produced strip thickness and width were 3 mm and 1220 mm, respectively. The temperatures after each of the six rolling stands were measured by pyrometer. The temperature at the last mill stand (finishing temperature) was varied between

890°C and 750°C. While coiling temperature through trials was kept around 400°C except the first trial which carried out at a coiling temperature of 580°C representing the processing condition of the commercially produced steel. Strain and strain rate at the final two stands were also changed, as will be indicated later from the measured process parameters.

Table 1 Trials chemical composition (in wt %)

C	Si	Mn	P	S	Ti	Nb
0.05	0.05	0.5	0.007	0.002	0.01	0.001
Al	Ca	Cr	Cu	Ni	N	V
0.03	0.002	0.01	0.02	0.01	0.004	0.001

Microstructure and Mechanical tests

Quasi-static tensile test was carried out according to the standard EN 10002-1 using the tensile testing machine Zwick/Roll (250 KN). Test samples with a width of 20 mm and a length of 43.4 mm was applied. Impact test was conducted on small size impact samples (10 mm height, 2.5 mm thick, 55 mm length and 2mm V-notch depth) according to the standard EN 100045-1. Charpy impact testing machine Type RKP 300/450 was used. Tensile and impact specimens were cut in the rolling direction. Samples for microstructure and grain size measurement were taken from sheets cross section perpendicular to rolling direction. Grain size was measured according to ASTM E112. Scanning electron microscopy SEM (Type JEOL model JSM 5410) and field emission SEM quanta SEG 250 were used to investigate deeply the microstructures.

Results and discussion

Processing parameters

The processing parameters for production of fine grained ferritic steel were varied and observed. Sheet height at each stand, temperature at the final rolling stand (finishing temperature) and coiling temperature were given to the system. Consequently, the strain and strain rate were calculated and the temperature at each stand was measured. **Error! Reference source not found.** includes the various processing parameters. The different processing parameters (the temperature, strain, and strain rate) at the six rolling stands F1-F6 during deformation are presented for the second trials group as shown in **Error! Reference source not found.**.

Table 2 Main process parameters in the industrial scale production trials.

Trial group	1	2				3
Trial number	1	2	3	4	5	6
Nominal finishing temp., °C	890	750	775	800	815	800
Temp. at F6, °C	886	761	780	805	817	807
Coiling temp.,°C	580	400	400	400	400	400
Strain at F5	0.18	0.21	0.21	0.22	0.22	0.27
Stain at F6	0.14	0.15	0.15	0.16	0.16	0.22
F5 Strain rate, s^{-1}	38	34	35	43	43	49
F6 Stain rate, s^{-1}	52	43	43	54	54	54

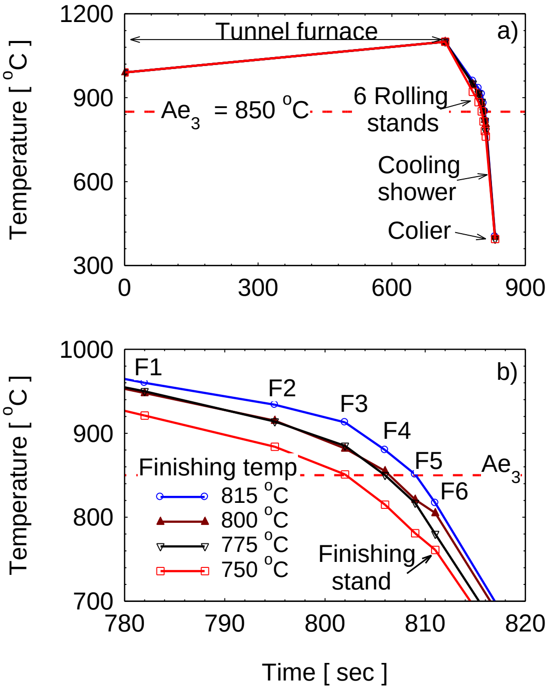


Figure 1 (a) Temperature profile against rolling line and (b) closer description of the 6 rolling stands F1-F6 shown in (a).

The equilibrium transformation temperature of austenite to ferrite $A_{e3}$was estimated from the chemical composition [5] and found to vary between 844 to 850°C according to the slight changes in the chemical compositions. However, this is not the exact temperature of transformation during deformation; it could give only a rough estimation of the amount of deformation at the austenite zone before the start of transformation, as well as the possible deformation in the two-phase zone.

Microstructure evaluation of first trial

In this trial, hot deformation is finished at 890°C high above A_{r3} and coiling temperature is kept at 580°C and the produced microstructure which indicates the presence of polygonal ferrite structure. The average grain size was measured to be ~ 11µm.It is equally important to indicate that only 30% of grains have actually this size of 11µm. Moreover, the grain size above 20µm represent about 10% of total grain size and grain size which is less than 5µm is about 13%, this indicates that a high degree of heterogeneity of the size distribution exist in the microstructure. Relatively fine second phase precipitates are observed both inside and at grain boundaries.

The plastic energy stored in the deformed microstructure is expected to increases with decreasing the deformation temperature and increasing the strain rate. Therefore, a smaller stored energy is expected after a higher temperature of 890°C compared with lower deformation temperature used in other trials, e.g. the second trial. The nucleation and growth kinetics of the ferrite formed from the deformed austenite are expected to be enhanced by the plastic energy stored in austenite, and the final grain size depend on nucleation rate rather than the growth velocity due to the limited time for the process. The total driving force for the deformed austenite decomposition is directly related to interface mobility. The carbon equilibrium concentration in austenite is likely to be increased with the stored deformation energy given rise to austenite free energy to be above this of ferrite phase [6, 7]. Therefore, an early ferrite nucleation could be expected enhanced by the accumulated strain. This type of reaction is called “dynamic induced ferrite transformation” and which occurs above A_{e3}. The applied high strain rates of ~52 s^{-1}will reduce the time needed for defects recoveries., increase the accumulated strain. On the other hand, a longer process time will be necessary for carbon diffusion to complete the austenite transformation.

The second trials group

Processing parameters

The current investigated thermo-mechanical process which is consisting of multi-deformation schedules can be separated into mainly four different stages as the deformation temperature decreases through the various rolling stands.

In this trials group, four different finishing hot deformation temperatures are examined. The low finishing temperatures are carried at 750 and 775°C and high temperatures up to 800 and 815°C and coiling temperature is kept at 400°C. However, the total strain is almost constant for each of them, the amount of reduction increases gradually and reaches maximum at last stand and the same for strain rate

At the start of the deformation at high temperature, coarse austenite grains are expected to be refined by the repetition of deformations. At this stage, the initial austenite grains under the effect of accumulated strain, the process of dynamic recrystallization is quite possible and will take place and this has been confirmed by simulation studies for industrial processing to produce a thin plate by rolling [8]. This resulted in an almost fully dynamic recrystallized austenite microstructure, however, due to the relatively higher interpasses times up to the third stand which vary between 10~8 seconds in the processing line, static recrystallization could also take place in the interpass times. This possibility which could not be rolled–out and expected to contribute to the further drop in processing temperature leading to more refinement in austenite.

The second stage is considered when the processing goes further down in temperature and austenite will not be able to be fully recrystallize due to accelerated strain rate and a smaller interpass time; 3~5 seconds. The accumulated strain will then lead to addition changes in austenite grain shape and also to the formation of dislocation substructure as well as dislocations band structures. The strain accumulation from pass to the next will then take place. Therefore, the deformed Austenite may only undergo a partial recrystallization with inhomogeneous prior austenite grain size, which would be the outcome of this stage of deformation. Intergranular defects introduced by the continuous hot deformation increases by increasing austenite grain size and the strain will be very effective on the DIF kinetics. It has been indicated that the number of intergranular nucleation in steel having larger prior austenite grain size will be more and finally accelerate the ferrite refinement process [9]. This is against the accepted view that fine austenite grain size will lead to fine ferrite grains. Further down in temperature scale, the third stage could be at temperature range between the start of ferrite formation A_{r3} and the end of ferrite transformation A_{r1}. Depending on the inter-critical deformation temperature, ferrite continues to nucleate within the un-transformed austenite grains and at grain boundaries. Simultaneously, austenite continues to deform, and the transformed ferrite grains begins to be strained.

The fourth stage where a variety of microstructures can be obtained during cooling depends on the cooling rate and the stage in which the hot deformation was interrupted. There is a delay between final deformation and subsequently cooling of about 2~5 seconds which could allow for a part of ferrite fraction in the final microstructure to be formed during the laminar cooling.

In the next section, the different microstructure obtained after those various stages of deformation will be examined and correlated with final hot deformation temperature.

Microstructure at 800°C

Changing the final hot deformation temperature during the sheet processing is very effective in varying the microstructure for the same steel composition. The microstructures in **Error! Reference source not found.**, **Error! Reference source not found.** and **Error! Reference source not found.**are a typical representation of such hot deformation temperatures effects mainly on grain size distribution.

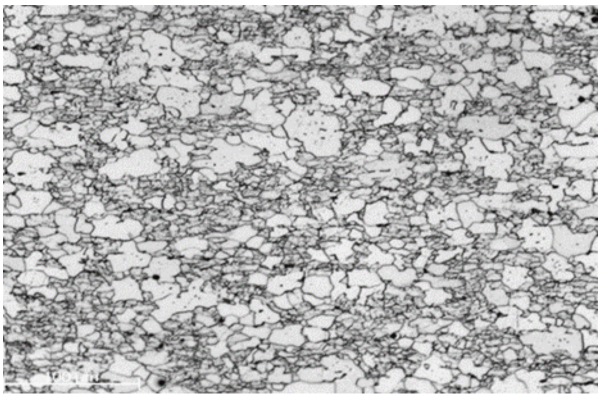


Figure 2 Optical microstructure of second trial, 750°C

A higher finishing deformation temperature 800 and 815°C produced a relative homogeneous ferrite microstructure for grain distribution , as shown in **Error! Reference source not found.** and a fuzzy substructure appears inside some of the ferrite grains. It is difficult using optical microscopy observation to distinguish between the recovery and incomplete recrystallization to be as source for those substructure formations.

Choi et al [10] indicated that for the austenite transformation to ferrite to take place during hot deformation, the strain should exceed a critical level. However, in this temperature range dynamic

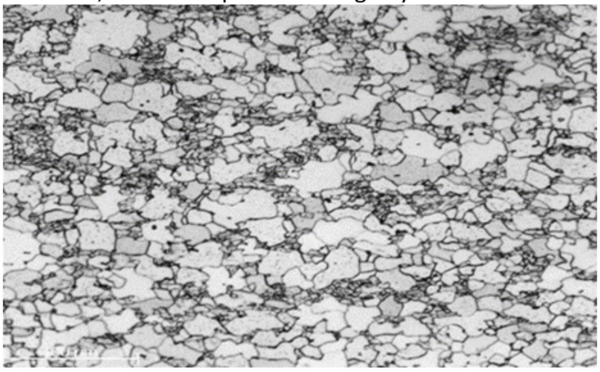


Figure 3 Optical microstructure of the third trial, 775°C

recrystallization can be competing with DIF process and one of them, which has the smaller critical strain, will take place. Similar to trial one the austenite grains in the strip after DRX are expected to undergo heavy deformation with process continuing. The accumulated strain will develop dislocation substructures in austenite and intergranular deformation features become active sites for ferrite nucleation. The heterogeneous distribution of dislocation produced by hot-rolling deformation had been reported to permit the untransformed austenite to continuous work hardening, allowing transformation to be possible up to 100°C above A_{e3}[11]. On the other hand, it has been argued that estimated stored energies of 21.6 to 63.4 J.mol-1 assuming 10 % of work done has been stored in a form of dislocation cannot account for the observed DIF transformation at high temperature above A_{e3} [12].

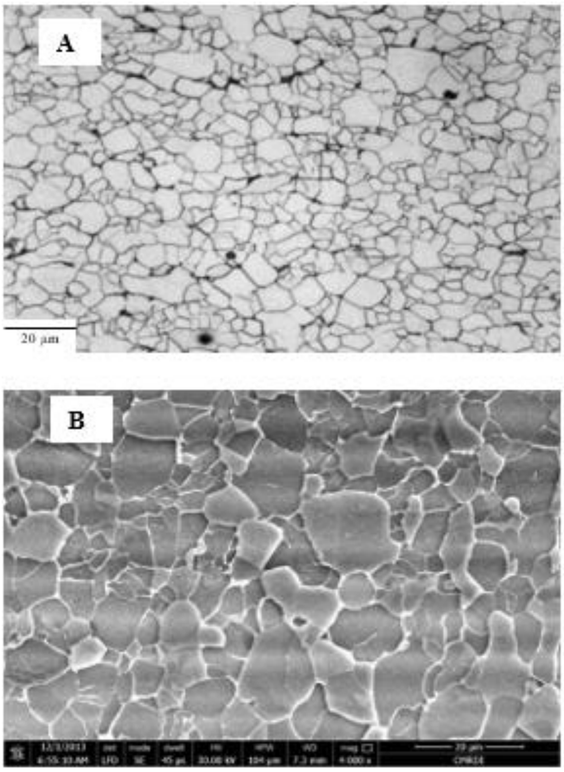


Figure 4 Optical microstructure and SEM of the fourth trial, 800°C.

The SEM observation confirmed the polygonal ferrite morphology and more precisely shows that some ferrite grains contain sub-boundaries structure, as shown in **Error! Reference source not found.**. This observation suggested the possibility that two mechanisms for ferrite formation are involved during the processing. The first is presumably acting through the effect of strain inducing ferrite transformation as indicated above. However, the very short time available for transformation due to high strain rate used during processing, makes the complete of DIF is rather difficult. The amount of the transformation fraction will be then strain, strain rate and temperature dependent [13]. For further deformation up to final temperature 800˚C, it is possible that a second mechanism can be acting. The untransformed austenite will start decomposition with a higher degree of super- cooling compared to the one of DIF transformation.

The resulted ferrite due to this second type of transformation is expected to have a relative higher grain size. On the other hand, the initially deformed DIF grains will continue to be deformed up to the interruption of deformation and could be responsible of the substructure formation in the ferrite grains. It should be noted that the last stage of hot deformation occurs in the inter-critical zone (α + γ). Further coarsening is quite possible during cooling to coiling temperature however, the presence of second phase precipitates can limit such process.

Microstructure at 750°C

The microstructures obtained after a lower interrupted deformation temperature of 750 and 775˚C are shown in **Error! Reference source not found.**and **Error! Reference source not found.**, a high degree of similarity is observed for both microstructures. The two microstructures show a bimodal grain size distribution where small grains are located next to larger ferrite grain. **Error! Reference source not found.**shows the grain size frequency distribution after final deformation temperature of 775˚C, with the average grain size estimated to be~10µm compared to 5.9µm at higher temperature of 800˚C. In this low-temperature finished microstructure the grain distribution shows that only ~9% of grains are above 20µm, 40% for grain sizes between 8~4µm and about 20% of grain with less than 5µm. For these small grains of less than 5µm surrounding the large ferrite is difficult to distinguish whether they are grains or they rather sub-grains. It has been recently indicated that those very small grains could be rather a sub-grains than individual grains, this has been confirmed using back scattering diffraction technique [14].

The effect of prior austenite grain size on the transformation mechanism cannot rule out also, the accelerated cooling following the hot deformation is likely to contribute to the final obtained microstructure. It has been argued [15] that for low carbon steel dynamic recrystallization of prior austenite grain size could have a small retarding effect on the strain induced ferrite transformation in the zone above A_{r3}. As indicated before, during second stage of austenite deformation, partial recrystallization will produce a rather high degree of structural heterogeneity due to different strain distribution.

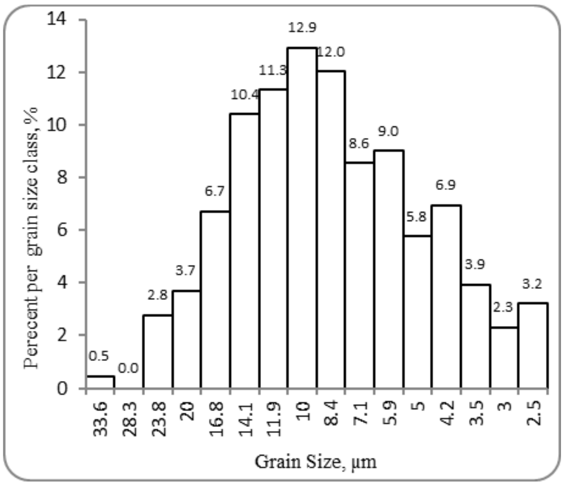


Figure 5 The grain size distribution after a final deformation temperature of 775˚C.

Error! Reference source not found.shows a detailed SEM morphology after deformation up to 750˚C. It is possible to observe that the previous small grains surrounding large ferrite grain appears as been just sub-grains and are limited to large ferrite grains zones. Moreover, the precipitates which confined to grain boundaries has a pearlite morphology as shown in **Error! Reference source not found.**. The pearlite formation could be enhanced by hot deformation through accelerating carbon diffusion in the late stage of process.

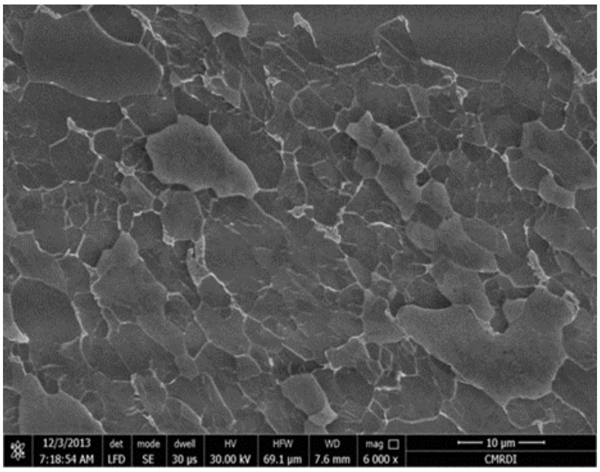


Figure 6 SEM micrograph for steel after finishing temperature of 750°C.

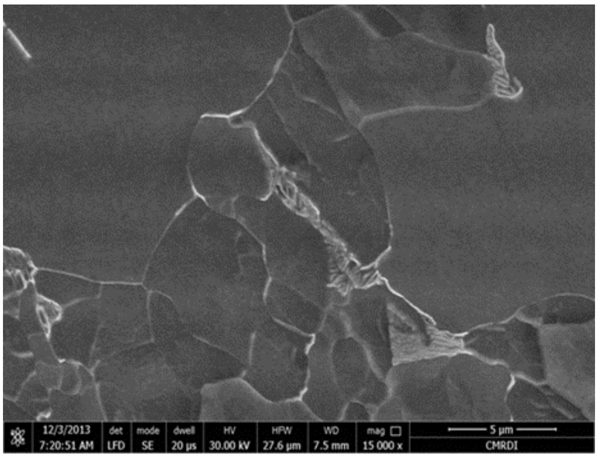


Figure 7 SEM of lamellar pearlite morphology in after low finishing temperature (750°C); higher magnification from Figure 6.

Growth of Ferrite grains

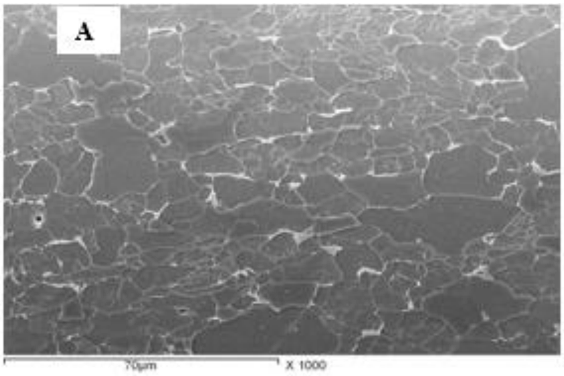
The induced ferrite grains by DIF process can continuously grow after the deformation is interrupted during the subsequent cooling. The limited growth during DIF is mainly attributed to carbon diffusion, which couldn't proceed sufficiently due to very short time available for austenite transformation and hence, the number of diffusion channels formed by dislocation bands is decreased during the formation of DIF [16]. Consequently, only short-range carbon diffusion can be expected to take place during DIF owing to time constrains. This been confirmed using simulation process which shows that high carbon atoms concentration will reach the interface (γ/α) and will have a drag effects on interface migration [17]. This mechanism can lead to the formation of islands of austenite surrounded by fine ferrite grains. During the next cooling stage or even during deformation in the inter-critical zone, below A_{r3}, carbon atoms at the interface can then diffuse by long range diffusion. Another possible mechanism which could contribute to the final microstructure mainly in the last stage of deformation during processing is dynamic recrystallization, DRX. However, it has been argued that DRX is not possible to take place in ferrite, since it has high stacking fault energy. More recently [8], it was suggested that another mechanism compatible with dynamic recrystallization and called “continuous dynamic recrystallization” CDRX can take place. This phenomenon is possible with increasing strain until the sub-structure formation occurs and subsequently changes it to a high angle boundary and finally becomes complete grains, without passing by classical nucleation and growth of DRX process. Many researchers have reported the occurrence of CDRX following the high strain and high temperature deformation [16, 17].

Trial at higher strain and strain rate

This sixth trial was carried out with the aim to achieve a further grain refinement of the ferrite microstructure. This trial was conducted at only one final hot deformation temperature of 800˚C and strain rate in each of the six stands were similar to the fourth trial for the same deformation temperature. However, it was thought that by increasing the amount of strain in the last stage of deformation further grain size reduction would be possible. The amount of strain was increased from 0.16 in the second trials group to be 0.22 in the sixth trial and the coiling temperature is kept constant at 400˚C.

The resulted microstructures are shown in **Error! Reference source not found.**, and consisted of fine equiaxed ferrite grain with the mean ferrite grain size being less than 5µm and fine pearlite colonies and/or carbide precipitates is observed to be confined at grain boundaries. Moreover, the density and the number of grains having such sub-boundaries inside the ferrite grains are higher compared with the fourth trial. Therefore, it is possible the sub-boundaries formation is strain dependent. Cementite and/or pearlite were be formed mainly at grain boundaries during a subsequence cooling after the interruption of hot deformation, as shown in **Error! Reference source not found.**.The critical strain above Ae_3 increases with decrease deformation temperature however, below Ae_3, ε_c decreases with decreasing deformation temperature [18]. Since, both strain rates and austenization pre-deformation at the rolling stands F1-F4 are almost the same in both fourth and sixth trials, it is then possible to suggest that different in strain in sixth trial is responsible for the slight improvement of grain refinement.

At the last stand, temperature which is expected to be below Ae_3, the DIF process will be inhibited to some extent due to preferential formation of pre-eutectoid ferrite. Moreover, the formed DIF grains would undergo further deformation and once the strain accumulated to some extent, (CDRX) continuous dynamic recrystallization would occur in DIF ferrite grains. This mechanism can explain the reason behind the presence of sub-boundaries in certain ferrite grains observed in the microstructure and a similar observation had been reported by Beladi et al [19].



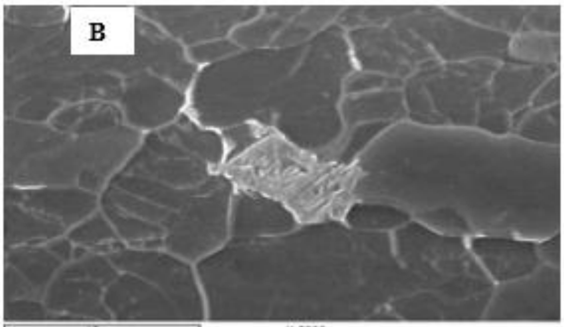


Figure 8 SEM of the sixth trial conducted at highest strain and strain rate at the last two stands.

Microstructure indicates the presence of another phase which occupied the grain boundaries, appears white with an ellipsoid shape. This phase could be the result of cooling of the residual austenite left between high rates formed ferrite grains. L.H. Hao et al [20] confirmed using TEM that a similar shape of formed precipitates is cementite [20].This phase formation is due to high carbon concentration in the remaining austenite with accelerating cooling following the end of deformation. This cementite phase can be generated a long ferrite grain boundaries.

Mechanical properties

Tensile stress- strain curves of all the tested specimen of the second trial for different finishing deformation temperatures steel are shown in **Error! Reference source not found.**. A better tensile ductility is observed with increasing final deformation temperature which could be related to an improvement of the work hardening rate. Yield stress values have been calculated from the lower yield point and each point is the average of three measurements **Error! Reference source not found.**.

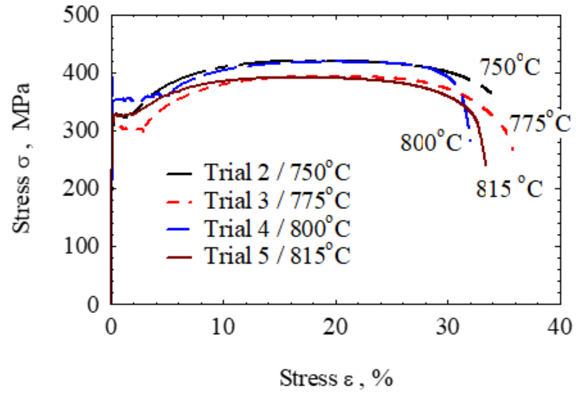


Figure 9 Stress-strain curves for the trials group conducted at same strain and strain rate and at different finishing deformation temperatures.

Table 3 Yield stress values.

Group	1	2				3
Trial # Temp., °C	1 890°C	2, 750°C	3, 775°C	4, 800°C	5, 815°C	6 800°C
Yield stress, MPa	288	329	307	335	329	354
Tensile stress, MPa	377	419	390	401	391	421
Elongation, %	34	32	35	33	37	32

The results showed that the flow stress increases from 288 to 354 MPa with grain size reduction corresponding to 18.6% increase in the yield stress. However, present experimental results showed that only 2% yield stress increased when grain size produced in the second trial group reduced from 10µm to 5.9µm. In that respect, it is clear that grain size reduction by the present refinement process do not fit with Hall Petch correlation. The discrepancy between calculated and measured flow stress can be attributed to the presence of a bimodal grain size distribution which have been confirmed by metallographic observation mainly at lower finishing deformation temperature. The bimodal grains, mixed coarse and fine grains, have been found to provide a useful concept in order to explain the improvement in tensile ductility [21].In this case it would be appropriate to use different average for ferrite grain size in Hall Petch relation. This average can be based on the area fraction for each average ferrite grain size large and small size.

The strain hardening ability has been usually related to grain size. With the high fraction of coarse grains regions in the structure, ductility improvement could be expected and is related to a possible higher mobile dislocations density inside the grains. Therefore, the bimodal grain distribution with high fraction of coarse ferrite grains will be expected to possess a relatively high ductility. **Error! Reference source not found.**shows the average grain sizes distribution at 800˚C finishing temperature.

From the tensile tests results; it is possible to point that there are increase in Lüder's strain with decreasing the grain size. This phenomenon can be related to lower density of mobile dislocation due to locking of dislocation source within the grains.

The results of the impact test conducted at room temperature indicate that higher impact energy is observed with decreasing the grain size. The impact energy increased from 211 Joule for the steel with grain size of 11µm to be 227 Joule for the steel with grain size of 5 µm., i.e., smaller grain size microstructures exhibit a higher resistance to fracture.

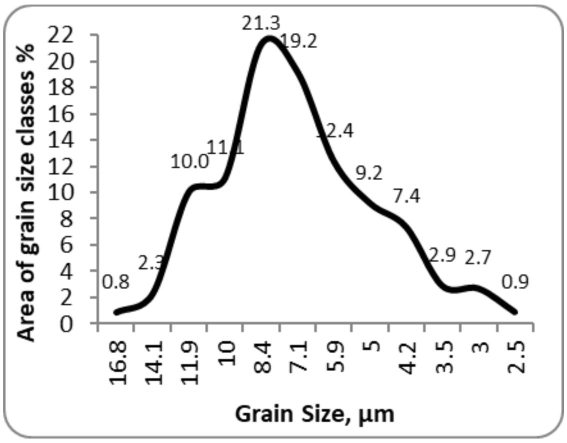


Figure 10 Area percent of grain sizes against grain size classes for the fourth trial (at finishing temperature of 800°C).

Conclusions

The current work focused on the metallurgical aspects related to the production of refined ferrite low-carbon steel. Three thermo-mechanical treatment trials were carried out on a hot deformation processing line to obtain the best rolling conditions for grain refinement of low carbon steel and the following conclusions can be made:

- The produced microstructure after a high finishing deformation temperature of 890°C has a polygonal ferrite microstructure with average grain size ~ 11µm.
- At low finishing deformation temperatures of 750 to 775°C microstructures show a bimodal grain size distribution with an average grain size of 10µm, while, at higher finishing temperatures of 800 to 815°C a relative homogeneous ferrite microstructure for grain distribution and a fuzzy substructure appeared inside some of the ferrite grains with average grain size of ~5.9µm.
- The resulted microstructures at high strain (0.27 and 0.22) and strain rates (49 and 54 s^{-1}) at the last two stands and at finishing rolling temperature of 800°C, consisted of fine equiaxed ferrite grain of ~5µm. Fine pearlite colonies and/or carbide precipitates are observed at grain boundaries.
- Ferrite grain refining to ~5µmhas improved the tensile properties; the yield stress has increased by 23% and the tensile strength increased by 12% on the expense of only 5% decrease in ductility. This improvement can be attributed to the presence of a bimodal grain size distribution mainly for low-temperatures finishing conditions.

Acknowledgment

The authors express their great thanks and appreciation for EZDK Company for their cooperation and help in executing the steel production trials.

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