Effect of Process Parameters on Microstructures and Mechanical Properties of a Nano/ultrafine Grained Low Carbon Steel Produced by Martensite Treatment Using Plane Strain Compression

Seyed Mehdi HOSSEINI,* Ahmad KERMANPUR, Abbas NAJAFIZADEH and Mostafa ALISHAHI

Department of Materials Engineering, Isfahan University of Technology, Isfahan, 84156-83111 Iran. E-mail: sm.hosseini@ma.iut.ac.ir, Ahmad_k@cc.iut.ac.ir, a-najafi@cc.iut.ac.ir, alishahi@ma.iut.ac.ir

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In this work, the martensite treatment consisting of cold deformation by plane strain compression and subsequent annealing was used for producing the nano/ultrafine grained structure in a low carbon steel. The equivalent strain was varied from 0.1 to 2, while the annealing process was carried out in the temperature range of 400–600°C for 0–180 min. The microstructural evolution and mechanical properties of the as-deformed and annealed specimens were investigated. The results showed that in the as-deformed specimens, increase in strain intensified the volume fraction of the martensite cell blocks and consequently the strength. Fully equiaxed nano/ultrafine grained ferrite was developed from the martensite cell blocks during the annealing at warm temperature around 500°C for sufficient time lengths. It was concluded that the final multi-phased microstructure composed of ultrafine ferrite grains, block-tempered martensite, and fine cementite precipitates was responsible for the obtained superior mechanical properties.

KEY WORDS: nano/ultrafine grained structure; mechanical properties; microstructural evolution; martensite cell blocks.

1. Introduction

The advent of the excellent mechanical properties in ultrafine grained (UFG) materials with submicron order size has been a prominent reason for extensive researches in the last two decades. Such an interest has been enhanced by developing new methods for production of UFG materials.1) Generally, there are two main approaches to producing UFG materials including “bottom-up” and “top-down”.2) It should be noted that a similar classification was performed by Valiev et al.3) In the “bottom-up” approach, UFG materials are developed by assembling or consolidating nano-structured components. Inert gas condensation, electro-deposition and ball milling with subsequent consolidation are the well-known examples of this technique. By contrast, “top-down” approach follows a different procedure intended to establish an ultrafine structure in a bulk material with macroscopic dimensions through severe plastic deformation (SPD). In a review paper conducted by Song et al.,4) the SPD processes such as accumulative roll-bonding (ARB), equal channel angular pressing (ECAP), and high-pressure torsion (HPT) were considered as methods which usually apply intense strain ($\varepsilon = 4$). It is worth pointing out that these methods often demand large amount of plastic working energy and special pieces of equipment.5) Another method related to the “top-down” approach is the advanced thermomechanical processes, in particular the martensite treatment which includes three steps: (1) quenching, (2) cold/warm rolling of martensite, and (3) final annealing.6) This process has considerable benefits, because according to the Kurdjumov-Sachs orientation relationship, there are four habit planes with six variants between austenite and martensite, giving a maximum 24 subdivisions in a given austenite grain.7) According to the investigations conducted by Ueji et al.,8) the martensite that includes a multilevel subdivision structure acts as an intermediate structure and reduces large required strain for production of the UFG low carbon steels. Consequently, increase in nucleation sites through quenching and fragmentation of the obtained martensite laths in plastic deformation stage is the basis of grain refinement in this method.

In the martensite treatment, the conventional multi-pass rolling is usually used for fragmentation of martensite laths.5,9) The present authors have recently introduced an experimental method of deformation, plane strain compression (PSC), using a single pass compression. The microstructural evolution and mechanical properties of a low carbon steel subjected to strain of about 1.5 by the PSC were investigated.10) In the present work, the effects of the PSC strains and annealing parameters on the microstructures and mechanical properties were studied to investigate the relationship between mechanical properties and the ultrafine-grained microstructures.

2. Experimental Procedures

2.1. The Martensite Treatment

In this study, a hot rolled low carbon steel sheet with
chemical composition of Fe–0.13C–0.13Si–0.49Mn–0.013P–0.015S (wt. %) was used. Details of the martensite treatment are presented in Fig. 1. According to this figure, specimens were heated up to 950°C for 35 min, quenched in methanol alcohol ice brine solution, and then deformed at room temperature up to the thickness reductions of 65, 75, and 85% ($\varepsilon_{\text{VM}}$ of 1, 1.5, and 2, respectively) by the PSC process. The deformed specimens were then annealed in the temperature range of 400–600°C for 0–180 min.

Figures 2(a) and 2(b) illustrate schematic of the PSC assembly and the PSC tested specimen along with the axis of deformation (dash lines indicate the cross section corresponding to the microscopic observations). In the PSC test, plastic deformation occurs in the direction of platen motion (ND), and normal to the length of the platen (LD). Based on the extensive investigations of Friedman et al., to make sure that plane strain condition has been supplied, the value of $w$ should be 6–10 times more than $b$, and $b/t$ should be between 2–4 times. Hence, after precise design of the platens with respect to the existing dimensional ratio, PSC process was carried out. An industrial grease lubricant was applied to the top and bottom of the specimen where subjected to compression by platens. Afterward, the specimen was clamped between the platens. The rate of the platens motion was considered to be 0.5 millimeter per minute. After imposing compression upon the specimen, the deformed pieces were cut off and collected. They were then prepared for subsequent annealing treatment. Although the elastic constraints of the undeformed shoulders of the sheet on each side of the platens prevent extension of the strip in the width dimension, a negligible increase in that dimension is unavoidable (extension parallel to TD shown in Fig. 2(b)).

2.2. Characterizations

To evaluate martensite phase percentage in as-quenched specimens, a color etchant was used. The etching solution contained 3 g potassium metabisulfite ($K_2S_2O_5$), 10 g sodium thiosulfate anhydrous ($Na_2S_2O_3$), and 100 ml distilled water. The microstructure of specimens was revealed by optical and scanning electron microscopes (TESCAN VEGA2 and SEM Philips XL30). The mean grain size was calculated by Clemex Image Analysis software.

In order to evaluate the mechanical properties, the shear punch test (SPT) was used. The SPT is essentially a blanking operation in which a punch (3 mm in diameter) is driven at a constant rate (0.2 mm/min) through a thin-sized disk (0.3–0.8 mm). The load on the punch is measured as a function of the specimen displacement. The curve obtained from SPT data is similar to that in a tensile test. Consequently, effective yield shear stress ($\tau_{\text{YS}}$), and ultimate shear strength ($\tau_{\text{US}}$) may be calculated from the yield and maximum loads, using the following empirical equation:

$$\tau = \frac{p}{2\pi r l} = C \sigma \quad \text{.................................. (1)}$$

where $p$ is the applied load, $r = (r_{\text{punch}} + r_{\text{die}})/2$, $t$ is specimen thickness, $C$ is a correlation coefficient, $\tau$ and $\sigma$ are the corresponding shear and tensile yield or maximum stress, respectively. The correlation of the form $\sigma = \alpha \tau$, ($\alpha = \frac{1}{C}$) between tensile stress $\alpha$ and shear stress $\tau$ has been evaluated for shear punch testing by several authors. Briefly put, they are:

$$\sigma_{0.02} = \alpha \tau_{1.00}, \quad \text{where} \quad \alpha = 1.77 \quad \text{..............(2a)}$$

$$\sigma_{\text{USS}} = \beta \tau_{\text{USS}}, \quad \text{where} \quad \beta = 1.8 \quad \text{.......... (2b)}$$

$$\left(\frac{n_{\text{e}}}{0.002}\right)^{0.22} = \frac{\tau_{\text{US}}}{\tau_{\text{YS}}}, \quad \text{.................................. (2c)}$$

$$e = 2.26n_{\text{e}} - 0.15 \quad \text{.......................... (2d)}$$

where $e$ is the uniform elongation, $n_{\text{e}}$ is the strain hardening exponent in SPT test, $\sigma_{0.02}$ and $\tau_{1.00}$ represent the 0.02% tensile and the 1.00% SPT normalized displacement offset yield point, respectively.

3. Results and Discussion

3.1. Microstructures before Plastic Deformation

Figure 3(a) shows the optical micrograph of the as-received specimen composed of the pearlite and ferrite grains with an average grain size of about 25 μm.

In the martensite treatment, it is important to select the appropriate austenitizing temperature in order to get maximum amount of martensite. Based on the Fe–C phase diagram and chemical composition of the steel used in this study, the thermal regime of 950°C for 35 min was selected as a suitable condition for austenitizing. To insure that the martensitic structure has completely been formed through quenching, the color metallography was used. In this method, each phase reveals a different color under the polarized light based on its chemical composition. In Fig. 3(b), the
martensite phase was revealed as blue and bluish brown color in the optical micrograph. Orange and yellow colors show pearlite and ferrite phases.

3.2. Characterization of the PSC Tested Specimens

3.2.1. Microstructural Observation

Figure 4 shows morphological features of the PSC tested martensitic specimen after 50% reduction ($\varepsilon_{vM} = 0.7$). The LD and ND are parallel to the horizontal and vertical directions of the micrograph, respectively. The location of the provided micrograph has also been shown by dash lines in Fig. 2(b). Three kinds of morphologies can be generally detected in the PSC tested martensitic specimens. The martensite cell block or lamellar dislocation cell (LDC) structure consists of the lamellar boundaries mostly elongated to a direction with 20° deviation from LD. This kind of structure has previously been observed in severely deformed metals. The irregularly bent lamellar (IBL) structure is a group of laths, tending to orient in different directions. The kinked lath (KL) structures where laths of martensite are kinked by shear bands are mainly parallel to ND. Ueji et al. reported that these morphologies can be also observed in cold-rolled martensitic specimens.

To investigate the microstructural evolution during the PSC test for the reduction amounts ranging from 10 to 85% ($\varepsilon_{vM} = 0.1–2$), SEM micrographs with a smaller scale were applied (Fig. 5). It is seen that lath martensite (LM) is a dominant structure in the micrograph corresponding to 10% reduction ($\varepsilon_{vM} = 0.1$), as shown in Fig. 5(a). With an increase in the reduction up to 35% ($\varepsilon_{vM} = 0.35$), the LDC structure became partially apparent within the large amount of remaining LM structure (Fig. 5(b)). In Fig. 5(c), up to 50% reduction ($\varepsilon_{vM} = 0.7$), three aforesaid morphologies including LDC, IBL, and KL became detectable. Increase in reduction up to 65% ($\varepsilon_{vM} = 1$) led exclusively to domination of the LDC structure. This tendency was also expected for the specimens with reduction amounts more than 65% (Figs. 5(e) and 5(f), corresponding to 75 and 85% reductions, respectively). It is noteworthy to mention that these micrographs were provided from the central areas of the PSC tested specimens. These areas were considered to be suitable for taking images, because it was proven that there was a decreasing slope of strain concentration from the center to the surface of the PSC tested specimens, causing complexity to investigate the subsequent microstructural evolution. The details of this scientific matter were discussed elsewhere. To characterize the above microstructural evolution, some points should be noted. First of all, for this experimental method of deformation even 35% reduction ($\varepsilon_{vM} = 0.35$) is unable to supply the required strain for particular changes in microstructure. Secondly, with an increase in the reduction up to 50%, each block containing laths tends to get a preferred orientation. Most of them show this tendency by rearranging parallel to LD (the direction parallel to the length of specimens and normal to the loading direction). It seems true that some blocks whose orientations are parallel to ND (the direction parallel to loading direction) resist deforming during plastic deformation. These blocks which form the areas corresponding to KL structure require more strain to fragment and to become parallel to LD. Finally, 65% reduction ($\varepsilon_{vM} = 1$) seems to provide adequate strain to dominate the LDC structure which is responsible for the formation of the UFG during recrystallization.

3.2.2. Mechanical Properties

The plastic deformation was carried out on the martensitic specimens by the PSC test. Figure 6 illustrates the engineering stress-strain curve of the martensitic specimens during the PSC test. This figure shows three distinctive regions. The first region is characterized by an initial linear increase in stress. In the second zone, strain increases while stress remains constant. This region is chiefly located between 10–35% reduction ($\varepsilon_{vM} = 0.1–0.35$). This occurrence can be mostly related to the initial deformation of the soft pearlitic and ferritic structures remaining between martensite laths (as shown in Fig. 3(a)), though formation of a partial amount of the LDC was observed in Fig. 5(b) corresponding to 35% reduction ($\varepsilon_{vM} = 0.35$). The third region (after 35% reduction) is identified by a considerable increase in both stress and strain. It seems true that after complete formation of the LDC structure (after $\varepsilon_{vM} = 1$), these lamellar dislocation cells start to compress and fragment more effectively.

Figure 7 reveals the shear stress-normalized displace-
ment curves of the as-received, as-quenched, and PSC tested martensitic specimens for different reduction values. The shape of the curves, especially after 35% reduction ($\varepsilon_{VM} = 0.35$), is similar for all specimens as fracture in the specimens was suddenly followed by the maximum strength. It is important to note that the uniform elongation of these specimens is very small due to their little work-hardening. According to this figure and relevant correlations,^{10} as-received steel represents a suitable strain-hardening associated with 27% uniform elongation. After quenching, the yield and ultimate strength rose up to 940 and 1 274 MPa, respectively. Therefore, the yield strength was approximately 4 times higher than that of the initial steel, while it was 2.2 times higher for the ultimate strength. In addition, the uniform elongation decreased to about 2% for the martensitic specimens. An increase in reduction led to an increase in both yield and ultimate strengths and a decrease in uniform elongation. Furthermore, maximum strength was observed in 85% deformed specimen (1 730 MPa), which was 3.2 higher than that of initial steel.

3.3. Characterization of the PSC Tested Specimens after Annealing

Based on the above results, adequate fragmentation of lath martensite for production of lamellar dislocation cell structure was provided after 50% reduction in thickness ($\varepsilon_{VM} = 0.7$). Hence, the annealing treatment was conducted on the 65–85% PSC tested specimens ($\varepsilon_{VM} = 1–2$) and their microstructures and mechanical properties were evaluated after annealing.

3.3.1. Microstructural Evolution

Figure 8 shows SEM micrographs of the PSC tested...
specimens annealed at various times and temperatures. A combination of remaining lamellar dislocation cell structure, partially recrystallized ferrite grains (shown with arrows), and precipitates of cementite (bright spots) can be observed in the 85% deformed specimens ($\epsilon_{\text{AM}} = 2$), annealed at 450°C for 180 min (Fig. 8(a)). This annealing condition was unable to provide sufficient driving force for the occurrence of complete recrystallization. The lamellar dislocation cell structure and cementite precipitates can mostly be observed in the 85% PSC tested specimens ($\epsilon_{\text{AM}} = 2$), annealed at 500°C for 5 min. Formation of these uniformly dispersed precipitates before the development of UFG structure provides a useful pinning effect which prevents the migration of grain boundaries, and thereby helps to achieve a fine microstructure after adequate annealing time. By increasing the annealing time up to 25 min at 500°C, the recrystallized ferrite grains became apparent in the microstructure (Fig. 8(c)). This microstructure is composed of the nearly equiaxed nano-ferrite grains with mean diameter of 125 nm. It is noteworthy to say that in some areas elongated grains are also visible. This feature is considered to be one of the main microstructural properties of the UFG steels achieved by martensite treatment. Furthermore, precipitation of uniform cementite in the obtained microstructure is predictable as martensite is a supersaturated solid solution of carbon. Abnormal grain growth of ferrite was unavoidable for the specimen annealed at temperature of 600°C for 2 min. It should be emphasized that by annealing at higher temperatures the role of cementite pinning in prevention of grain boundaries migration decreases. Actually, due to annealing at high temperature for short time, recrystallization of ferrite grains in some areas may be simultaneous with precipitation of cementite particles. Existence of fine cementite dispersed within the coarse grains can confirm this reality. Figures 8(e) shows a fully recrystallized structure associated with precipitates of cementite for the 75% deformed specimen ($\epsilon_{\text{AM}} = 1.5$), annealed at 500°C for 65 min. It is expected that a decrease in cold deformation leads not only to the decrease in driving force for recrystallization, and accordingly to an increase in annealing time, but also to the increase in mean ferrite grain size ($\approx 140$ nm). This trend is in excellent agreement with the measured mean grain size of 65% deformed specimen ($\epsilon_{\text{AM}} = 1$), annealed at 500°C for 105 min (Fig. 8(f)). The mean grain size of ferrite was evaluated to be almost 275 nm. To characterize the above microstructural evolution, some cases should be pointed out. Firstly, it should be noted that increasing the applied strain leads to increasing the driving force for the recrystallization. Therefore, the time length during which completion of recrystallization occurs becomes lower with enhancing the strain (stored energy). Secondly, intensive difference between the obtained mean grain sizes of the annealed specimens with strains of 1 and 1.5 indicates that after strain of 1, fragmentation of lath martensite continues and therefore latter examined specimen with strain of 1.5 contains much more nucleation sites. Meanwhile, after strain of 1.5 up to 2 fragmentation of lath martensite is not considerable. This result can be confirmed by comparison of the reported mean grain sizes for each specimen. Finally, the present authors have already studied morphological changes through thickness of the PSC tested specimens after annealing of the 75% compressed specimens ($\epsilon_{\text{AM}} = 1.5$). It was concluded that the existence of block-tempered martensite caused by KL structure in the areas near the surface of fully recrystallized specimens is an inevitable morphology though it disappears in the central regions. This finding was attributed to the nature of the PSC test with inhomogeneous distribution of strain through the thickness of compressed specimens, and thereby the absence of the kinked lath structure at the center of deformed specimens with the strain higher than 1. It should be noted that all micrographs shown in Fig. 8 are provided from the regions near the center of specimens. Consequently, the absence of block-tempered martensite is reasonable in these photographs, but it can be surely found in the areas near the surface of the examined specimens.

3.3.2. Mechanical Properties

The shear stress-normalized displacement curves of the PSC tested specimens corresponding to the above mentioned UFG microstructures are illustrated in Fig. 9. For the martensitic specimens plane strained to 85% reduction ($\epsilon_{\text{AM}} = 2$), annealing at 500°C for 25 min led to a considerable combination of excellent strength (1180 MPa) and good uniform elongation (9.8%). For the 75% PSC tested specimen ($\epsilon_{\text{AM}} = 1.5$), the ultimate strength and uniform elongation of the specimen were estimated about 1135 MPa.
The Hall-Petch (H-P) relationship, which connects the mechanical properties (yield strength) to the microstructural features (grain size), has frequently been used to examine the contribution of grain boundary strengthening. The typical form of the H-P relation for yield strength is given by the following equation:

\[
\sigma_y = \sigma_0 + kD^{-1/2}
\]

Where \(\sigma_y\) is the yield strength, \(\sigma_0\) is the friction stress, \(k\) is the grain boundary resistance, and \(D\) is the grain size in \(\mu m\). In Fig. 10, a linear relationship with a good correlation coefficient \(R^2 = 0.995\) can be seen for yield strength as a function of square root of the inverse grain size ranging from 0.125 to 0.65 \(\mu m\), where \(\sigma_0 = 211.91\) MPa and \(k = 186.75\) MPa. It is observed that the slope of H-P line is much smaller than that of the previous investigations. As an instance, Morrison\(^{17}\) illustrated that for the steel sheets (Fe–0.13 wt.% C–0.14 wt.% Si–0.67 wt.% Mn) produced by cold rolling and annealing, the friction stress and the grain boundary resistance were calculated to be 100 MPa and 551 MPa, respectively. However, the grain size ranged from 30 to 1.6 \(\mu m\). A similar trend has been reported by other researchers as well. According to the results reported by Han and Yue,\(^{17}\) it can be suggested that if H-P relationship in steels could be subjected to the submicron range, \(k\) may take a smaller value. Meyers \(^{2,18}\) have also expressed that the Hall-Petch relationship recorded at large grain sizes cannot be extrapolated to grain sizes of less than 1 \(\mu m\). There is a significant decrease in the slope for small grain sizes. In a more comprehensive investigation, Song \(^{18}\) analyzed the effect of a broad range of grain size of bcc steels developed by diverse methods of thermomechanical processes on the Hall-Petch \(k\) value. They concluded that decimation of \(k\) in submicron steels compared to that of coarse microstructure can be explained by three major theories for the Hall-Petch equation including pile-up models, hypotheses based on work hardening, and grain boundary source theories. However, it seems true that as the grain size is decreased, an increasing fraction of atoms can be found in grain boundaries. On the other hand, a decrease in grain size results in increasing of the atoms located in grain boundaries compared to those within the grains, which influence the properties of UFG materials.\(^{2,18}\)

![Fig. 9. Shear stress-normalized displacement curves of the PSC tested martensitic specimens for different annealing conditions.](image1)

![Fig. 10. Dependence of yield strength on the grain size.](image2)

and 11.6%, respectively. In third specimen with 65% reduction annealed at 500°C for 105 min, a significant feature was attained. According to the results, over 50% of the uniform elongation (13.2%) compared to that of the as-received steel was recovered, while the estimated value of the ultimate strength was about 1000 MPa. The obtained superior mechanical properties can be ascribed to the presence of the final multi-phased structure caused by appropriate annealing conditions. This multi-phased nano-structure consists of ultrafine ferrite grains, nano-sized cementite participates, and block-tempered martensite. Lots of researches have recently been conducted on the role of precipitates in enhancing of work hardening in UFG low carbon steels. As an example, Ueji \(^{18}\) have found that the cementite precipitates increase the work hardening of the UFG low carbon steels. Ashby\(^{15}\) have suggested a theory about this phenomenon. He stated that the activation of secondary dislocation slips around the undeformed particles results in increasing the work hardening.

The PSC tested specimens with 85% reduction (\(\varepsilon_{FM} = 2\)), annealed at 600°C for 2 min possess the ultimate strength of 810 MPa and uniform elongation of 16.8%, which can be attributed to the simultaneous existence of both large and ultrafine grained ferrites. The mechanical properties obtained by short annealing time at high temperature are comparable to the results reported by Azizi-Alizamini \(^{2,18}\) et al. They used a novel process for fabrication of the UFG low carbon steel containing 0.17% C with bimodal grain size distribution. In their proposed process, a ferritic microstructure with bimodal ferrite grain size distribution was fabricated by 50% cold rolling and subsequent annealing of a ferrite-martensite dual-phase microstructure. They reported the ultimate tensile strength of 550 MPa and uniform elongation of 14% for the annealed specimen. However, in the present study a similar microstructure was obtained with better mechanical properties by short annealing time at higher temperature.

3.3.3. Dependence of Strength on Grain Size

The Hall-Petch (H-P) relationship, which connects the mechanical properties (yield strength) to the microstructural features (grain size), has frequently been used to examine the contribution of grain boundary strengthening. The typical form of the H-P relation for yield strength is given by the following equation:

\[
\sigma_y = \sigma_0 + kD^{-1/2}
\]

4. Conclusions

(1) Increasing the cold deformation intensified both the strength and the fraction of the regions with lamellar dislocation cell structure in such a manner that almost fully lamellar dislocation cell structure was obtained after 65%
reduction ($\varepsilon_{VM} = 1$).

(2) Fully recrystallized structure associated with cementite precipitates was achieved for the 65, 75 and 85% PSC tested martensitic specimens ($\varepsilon_{VM} = 1$–2) after annealing at a temperature around 500°C for 105, 65 and 25 min, respectively. The smallest mean grain size of 125 nm was obtained in the 85% deformed specimens ($\varepsilon_{VM} = 2$).

(3) A good combination of strength (1180 MPa) and uniform elongation (9.8%) was achieved in the 85% deformed specimens ($\varepsilon_{VM} = 2$) after annealing in appropriate conditions.

(4) Short time annealing at high temperature led to simultaneous existence of both large and ultrafine grained ferrites in the 85% PSC tested specimen ($\varepsilon_{VM} = 2$), followed by annealing at 600°C for 2 min, which exhibited the ultimate strength of 810 MPa with uniform elongation of 16.8%.

(5) The resulting data achieved by the investigation of mechanical properties (yield strength) and microstructural features (mean grain size) showed a good consistency with Hall-petch relationship.

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