MICROSTRUCTURE AND MICROHARDNESS OF NANO/ULTRAFINE (n/UFG) GRAINED COLD-ROLLED 0.06C STEEL

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Abstract

This present study is aimed to examine the microstructure and microhardness of nano/ultrafine grained (n/UFG) of 0.06C steel which had been cold-rolled, quenched and annealed. The process started with three different initial microstructures with the condition of 1) austenization at 1000°C for 30 min soaking and air cool (AC), 2) austenization at 1000°C for 30 min soaking and ice-water quench (IQ) and, 3) same treated as IQ but the quench specimen was tempered at 750°C for 30 min soaking and ice-water quench once again (IQT). All quenched specimen, then cold-rolled by 75% and annealed at different temperatures ranging from 500°C to 600°C for 30 min soaking. From the analysis, it was found that the specimens annealed at 550ºC exhibit nano/ultrafine ferrite grained size with similar microhardness value for AC, IQ, and IQT. It has also found that acceptable UFG microstructure formed with of IQT specimen performs faster that IQ and AC specimens in terms of microstructure recovery, re-crystallization and grain growth during annealing after cold rolling.

Keywords: Annealing, Cold Rolling, Microhardness, Nano/Ultrafine Grained, Plain Low Carbon Steel

Introduction

The most effective technique to enhance advanced structural steel that incorporates superior mechanical properties with non-complex chemical content such as grain refinement. In order to establish nano/ultrafine grains with mean grain size of less than 1 µm [1], severe plastic deformation (SPD) methods; for example 1) equal channeling angular press (ECAP) 2) accumulated roll-bond (ARB) and 4) high pressure torsional (HPT) have been identified as prospect processes [2-7]. However, SPD processes for mass production and huge dimension samples, seem do not suit for such viable sampling/component process. A very large amount of strain (above 4 strain value) is required to be applied to the materials in order to obtain nano/ultrafine grain structures [8], however, even if the strength is very high, their tensile elongation is found to be limited [9]. Consequently, Tsuji and co-workers [1] have shown another alternative strategy to produce ultrafine ferrite grained size of 180 nm in low carbon steel (0.13 %C) is by applying only 50% of cold-rolling (0.8 strain value) of martensite starting microstructure and then annealing at warm temperature. Tianfu et al [10] also established an alike method and produced grain size around 20 nm to 300 nm. Although the specimen that contained grain size of 20 nm performed excellent strengths, the ductility at ambient temperature was very poor or almost non-existent. In order to improve the ductility, Azizi-Alizamin et al [11] showed another method to fabricate ultrafine grained (UFG) with bimodal grain size dissemination in low carbon steel (0.17 %C). However, those researchers as mentioned above focused only on tensile properties with changing in grain size structures of the materials, but in the reality steels usage, hardness is one of the most important factors that has to be considered in order to choose the right steel for suitable application.

Hence, the present study is aimed to investigate the evolution of microstructure and corresponding change in microhardness of 0.06C steel processed by traditional cold rolling and annealing.

Material and Experimental Procedures

Table 1 shows the chemical composition of the material used in the present study, which was a commercial plain low-carbon steel sheet. As-received sample was a hot-rolled plate with thickness of 5 mm. First of all, specimen of 5 mm thickness, 25 mm width and 100 mm length in size were machined out from hot-rolled plate and subsequently treated with three dissimilar of treatments. The quenched specimens being heat treated with the condition of 1) austenization at 1000°C for 30 min soaking and air cool (AC), 2) austenization at 1000°C for 30 min soaking and ice-water quenched (IQ) and 3) same treated as IQ, but the quenched specimen was tempered at 750°C for 30 min soaking and ice-water quenched once again (IQT) as shown in Table 2. Theses specimens were then cold-rolled to a reduction of 75% thickness in multi-passes at ambient temperature via a laboratory rolling mill (roll diameter: 80 mm, speed: 10 rpm). The cold-rolled specimens were annealed at 500°C - 600°C for 30 min soaking, follow by air-cooling.

Microstructural observations of specimens at each point of the method were investigated by optical microscopy attached with Image Analyzer (OM-IA) and field emission scanning electron microscopy (FESEM). The transverse direction (TD) of all sheets of the specimen was observed the microstructure. The OM-IA was examined and characterized under MT Meiji Techno optical microscopy and the FESEM observations were conducted in ZEISS SUPRA 35PV equipment. The microstructures observation by OM-IA and FESEM were etched with 2% Nital reagent. The Magnisci Software was used for calculating the percentage of ferrite, martensite and pearlite volume fraction and grain size. The microhardness was evaluated by Vickers Microhardness Tester (Model: LM 2448 AT) performed with a load of 100gf for 10 seconds.

Table 1. Chemical Compositional Analysis of Plain Low Carbon Steel (wt%)

C	Mn	P	S	Fe
0.06	0.14	0.01	0.01	Bal.

Table 2. Sample Codes and Heat Treatment

Sample code	Heat treatment
AC	Austenization at 1000°C for 30 min soaking and air cool.
IQ	Austenization at 1000°C for 30 min soaking and ice-water quench.
IQT	Austenization at 1000°C for 30 min soaking and being ice-water quench. The quenched sample was then tempered at 750°C for 30 min soaking and ice-water quench for the second time.

Results and Discussion

The microhardness values of specimens in the various stage processes are shown in Figure 1. It can be observed that as quenched specimens, the microhardness of the AC, IQ and IQT are increased from 94.1 HV (as-received) to 103.7 HV, 152.3 HV and 211.6 HV, respectively. The microhardness of AC is lower than IQ and IQT because of the AC is comprised of largely ferrite phases with minor amount of pearlite phases as indicated in Table 3. The grain size of ferrite phases is approximately 18.6 µm as revealed in Figure 2a, but IQ illustrates ferrite-martensite phases volume fraction (Table 3) with the grain size of ferrite phase is about 4.8 µm (Figure 2b). However, IQT shows the highest microhardness value of the as-quenched specimen (Figure 1) which is 125% higher than as-received condition. The reason could be due to tempered dual phase ferrite-martensite with fine martensite as second ice-water quenching. The grain size of the ferrite is about 11.2 µm (Figure 2c). According to Figure 1, the microhardness of the AC, IQ and IQT then increased to 193.7 HV, 210.0 HV and 286.2 HV, respectively after 75% cold-rolled.

It is well known that microstructure wills greatly affecting the material properties such as microhardness. In current finding it is found that the treated AC specimen (Figure 3a) displays mostly the grains of ferrite and pearlite colonies which are elongated along the rolling direction of the sheet. Similar microstructures finding also observed by previous works by Li et al, 2013 [12] and Yang et al, 1985[13]. Figure 3b shows the ferrite and martensite structures that are totally smashed after cold-rolled. Figure 3c indicates the microstructure of a wavy the ferrite matrix (light gray region) elongated in the direction of rolling and bent intra-grain of the martensite islands (dark region). Zakerinia et al. [14] stated that during cold working, it creates areas with high dislocation density due to the deformation of martensite phase. In the annealing process, these areas initially start to form the formation of recrystallized nuclei, which were then reacts as the source of steel grain refinement. On the other hand, the enhancement of microhardness after cold deformation is due to the effect of strain hardening, achieving by the movement of one dislocation to other adjacent dislocation interaction together with the interaction of dislocation within twin boundary [15].

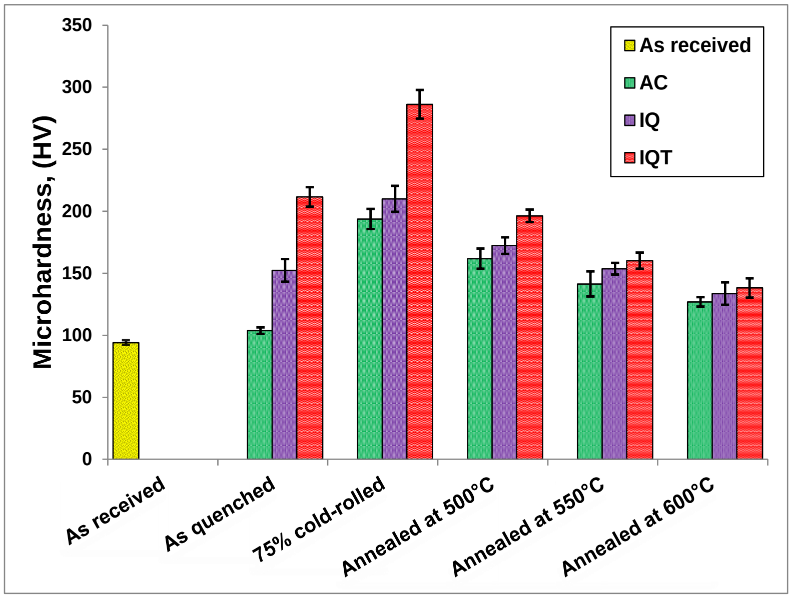

Figure 1. The microhardness values of specimen in various stage processes.

Table 3. Fraction Volume (vol.) of Detected Phases in the Quenched Samples

Samples	Ferrite vol. fraction(%)	Martensite vol. fraction (%)	Pearlite vol. fraction(%)
AC	91.1	-	0.8
IQ	58.2	41.7	-
IQT	92.9	7.1	-

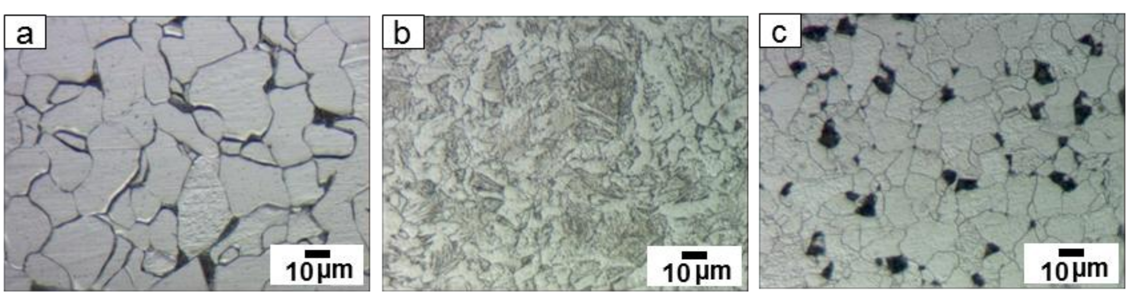

Figure 2. Optical microstructure of specimens before cold rolling, (a) AC, (b) IQ, (c) IQT

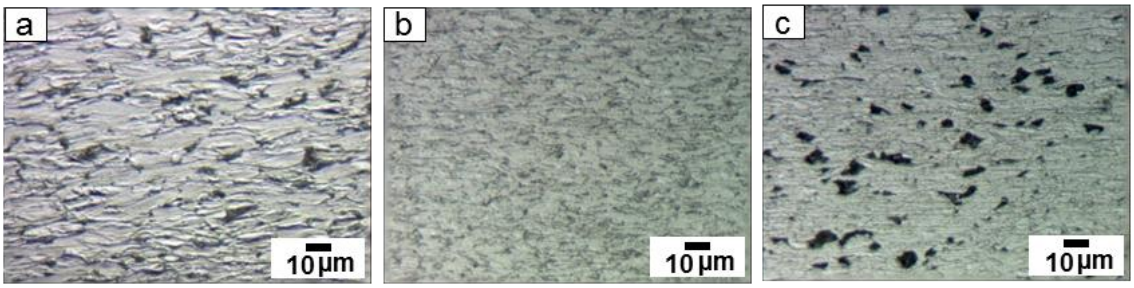

Figure 3. Optical microstructure of specimens after 75% cold rolling of (a) AC, (b) IQ, (c) IQT

Figure 4(a-i) illustrates the microstructural evolutions of specimens subjected to 75% cold-rolled and annealed at different temperatures of 500ºC, 550ºC and 600ºC for 30 min. Theoretically, cold-worked with destruct the existence of microstructures and forming dislocation and as a surplus, the formation of dislocation will increase the strength of the steel. High strength is favorable but the lost of ductility is not favorable for steel application. In order to compromise with this, microstructure needs to be growing again and sequence of recovery, recrystallization and grain growth need to be performed. Recovery microstructure looked like the nuclei flaky type [16]. On the other hand, recrystallization structure looked like the initiation of nuclei flaky to grain boundary formation. Microstructures recovery started to form and can be clearly observed in Figure 4a for specimens annealed at 500°C which performing AC. But IQ (Figure 4b) and IQT (Figure 4c) exhibit recrystallization with some evidence of grain boundary formation as indicated with arrow signs. However, both of IQ and IQT recrystallization for 500°C has not been completed. On the other hand, it can be observed in the Figure 4c that IQT starts to recover and continue recrystallize faster than IQ and AC. In Figure 4d-f, ultrafine ferrite grains size are obtained in IQ (Figure 4e) and IQT (Figure 4f) which are about 300 nm and 500 nm respectively, after annealed at 550°C. But microstructure of AC (Figure 4d) still has a flaky-like shape with some formations of ferrite grain (200 nm). As annealing temperature is increased to 600°C (Figure 4g-i), the microstructures are mostly consisted of the ferrite grains size have been growing, with mean grain size of about 1.1 µm, 1.8 µm and 2.5 µm are formed in AC (Figure 4g), IQ (Figure 4h) and IQT (Figure 4i), respectively. Further observation in Figure 4, it is noteworthy that IQT microstructure performs faster than IQ and AC in term of recovery, re-crystallization and grain growth during annealing after cold rolling due to IQT creates regions with high dislocation density and absorb more energy during cold rolling compared with IQ and AC. These high dislocation regions with more energy absorbing will propagate and enhance in recrystallization of nuclei to subsequently form grains during annealing process.

The decreases in microhardness of the annealed specimens with increasing temperature are shown in Figure 1. The decline in microhardness with increasing annealing temperature is because of the reduction of dislocation density [1]. As can be seen that similar microhardness are obtained for AC, IQ and IQT specimens after annealing at and above 550°C, but microhardness of the IQT is higher than IQ and AC lower than 550°C. This is due to the recrystallization completed at 550°C. On the other hand, IQT specimen consists of tempered ferrite-martensite and martensite volume fraction phase after ice-water quenched as the second time. Therefore, the microhardness of IQT is higher than IQ and AC specimens.

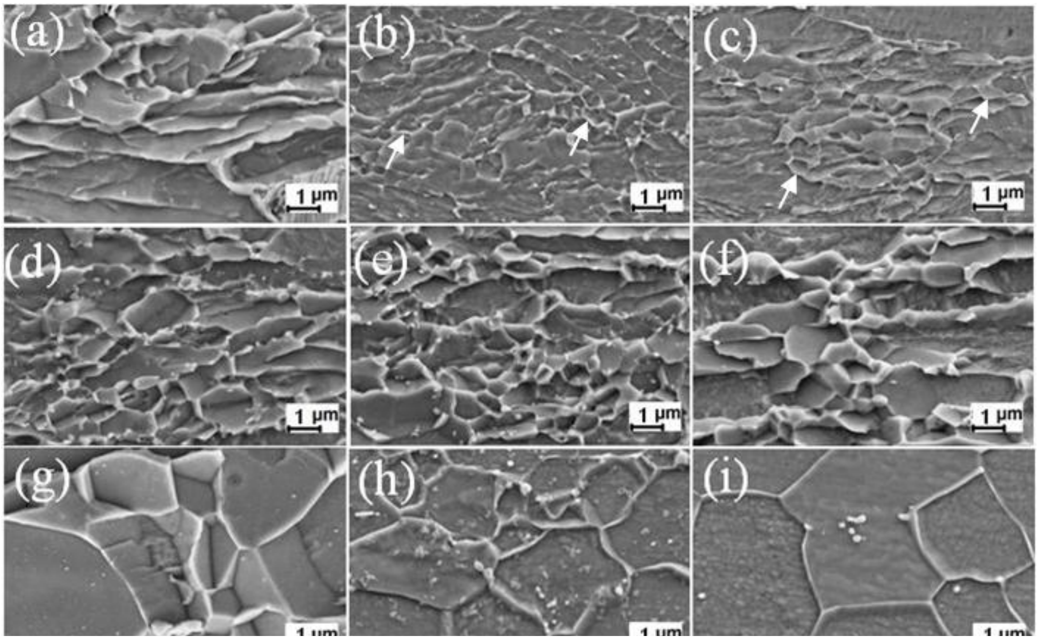

Figure 4. FESEM microstructure of annealing specimens after 75% cold rolling for 30 min, (a) AC annealed at 500°C, (b) IQ annealed at 500°C, (c) IQT annealed at 500°C, (d) AC annealed at 550°C, (e) IQ annealed at 550°C, (f) IQT annealed at 550°C, (g) AC annealed at 600°C, (h) IQ annealed at 600°C, (i) IQT annealed at 600°C

Conclusion

Microstructure and microhardness properties on the formation of nano/ultrafine grained (n/UFG) of 0.06C steel were investigated. It was found that AC, IQ and IQT had achieved nano/ultrafine grained structure which are 200 nm, 300 nm and 500 nm, respectively, through 75% conventional cold-rolled and subsequently annealed at 550°C, without SPD. Microhardness value increased significantly by 75% cold-rolled. In contrary, as annealing temperature increases resulting in the decreasing of microhardness value. The formation of martensite and tempered ferrite-martensite are related to the increase and decrease of microhardness. The similar microhardness are obtained for AC, IQ and IQT specimens after annealing at and above 550°C due to the completion of recrystallization. IQT specimen performs faster than IQ and AC specimens in terms of recovery, re-crystallization and grain growth during annealing after cold rolling.

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