The Effect of Niobium on the Formation of Nanostructured Low Carbon Steel Using Martensite Treatment

M. Abbasi[1]*, A. Kermanpur[2], A. Najafizadeh[3], S. Saeedipour[4] and Y. Mazaheri[5]
Department of Materials Engineering, Isfahan University of Technology, Isfahan 84156-83111, Iran

Abstract

The formation of nano/ultrafine grained ferrite in low carbon steels containing different amounts of niobium was investigated using thermomechanical treatment which consisted of annealing of 85% cold rolled martensite with different parameters. The specimens were characterized by optical and scanning electron microscopy and Vickers hardness test. A lamellar dislocation cell structure was formed during cold rolling. An increase in hardness was found during annealing with the addition of the Nb element. The mean grain size of the specimens annealed at 550 ºC for 300 s was approximately similar (ranging from 79 to 87 nm) for both chemical compositions. Increasing annealing temperature to 600 ºC or annealing time to 2.7 ks led to a severe grain growth in the steel without Nb, but no considerable changes in Nb containing steel were observed.

Keywords: Ultrafine/Nano grained structure, Niobium, Cold rolled martensite, Grain growth.

1. Introduction

It is well known that grain refinement is a desirable approach in metallic materials production due to simultaneous increase in strength and toughness. In recent years, researchers have paid considerable attention to the formation of ultrafine structure materials because of their excellent properties such as good fracture toughness at low temperatures, high strength, adequate elongation and high dynamic strength [1)]. Two main approaches have been developed to decrease the grain size to a degree smaller than a few micrometers. The first is severe plastic deformation (SPD) such as accumulative roll-bonding (ARB) [2)], high-pressure torsion (HPT) [3)], equal channel angular pressing (ECAP) [4)] and bi-directional large strain deformation (BLSD) [5)]. The second is thermomechanical processing such as recrystallization of austenite during hot deformation [5)], strain induced ferrite transformation [5)] and plastic deformation and recrystallization (PDR) [6)]. Between these two methods, thermomechanical processing is more applicable in the mass production of ultrafine grain materials due to its lower plastic deformation energy and equipment [7)]. As revealed by Tsuji et al. [8)], little amount of strain (ε=0.8) is enough to produce ultrafine ferrite grains in low carbon steels using martensite treatment. In this process, the as-quenched martensite can be cold rolled followed by warm temperature annealing. The as-quenched martensite, which is subdivided by up to 83% high angle boundaries, is an ultrafine grain structure which plays an important role in the formation of deformation induced boundaries (DIBs), which subdivide the original grains during cold rolling. The DIBs are low angle boundaries that are composed of or decorated by dislocations. Subsequent annealing turns such unclear boundaries to clear boundaries and causes the formation of ultrafine ferrite grains. Researchers have carefully investigated some important parameters on martensite processing such as rolling reduction [9)], annealing condition (time and temperature) [10)] and the type of cold deformation in the last decade [11)].

In the present paper, the effect of niobium addition as a microalloying element and annealing condition after 85% rolling at room temperature on ferrite grain refinement has been investigated using martensite treatment.

* Corresponding author:
Tel: +98 936 7726475 Fax: +98 311 3912752
Email: mec_abbasi85@yahoo.com
Address: Department of Materials Engineering, Isfahan University of Technology, Isfahan 84156-83111, Iran
1. M.Sc.
2. Associate Professor
3. Professor
4. M.Sc.
5. Ph.D. Student

2. Materials and experimental procedures

Two steels with chemical compositions shown in Table 1 were used in the present investigation. The steels were in the form of hot forged plate with 6 mm thickness. Hot forged sheets were cut to several specimens with 50×30×6 mm dimensions. The applied heat treatment and plastic deformation on samples are shown in Figure 1. Austenitization treatment was carried out at 1000 and 1225 ºC for plain carbon steel

Table 1. Chemical composition of steels used in the present work (wt. %)

Steel No	C	Mn	S	Co	Cr	Ti	Nb	Fe
Nb .00	0.14	0.56	0.004	0.006	0.02	0.002	0.00	Balance
Nb .04	0.14	0.63	0.004	0.006	0.2	0.002	0.045	Balance

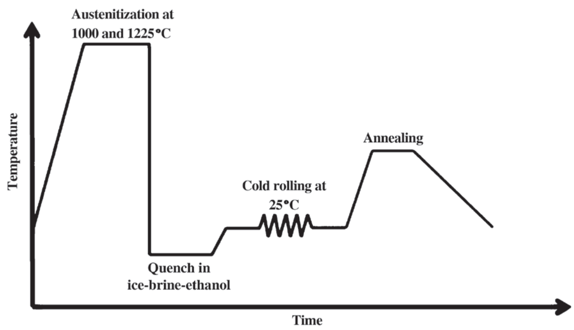

Fig. 1. Schematic diagram of applied heat treatment and plastic deformation.

and microalloyed steel, respectively, in an electrical furnace at the heating rate of 10 ºC/s for 50 minutes. In order to prevent severe decarburization, cast iron swarfs were used to protect samples during heating. Rapid quenching was done in ice-brine-ethanol at -15 ºC to obtain the maximum amount of martensitic structure. The quenched samples were cold rolled to 85% reduction in thickness at room temperature. Rolling operation was performed without any cracking using machine oil as the lubricant and rolling speed of 350 rpm with the reduction of about 0.1 mm at each pass. In order to perform warm annealing treatment, microscopic characterization and hardness measurement, 20×10 mm test pieces were separated from cold rolled sheets.

The hardness measurement was done on a Vickers hardness tester using load of 10 N. Optical microscopy (OM) and Philips XL30 scanning electron microscopy (SEM) were used for microscopic characterization. Samples were etched with 3% nital to reveal the microstructure. The amount of martensite was determined using a solution composed of 3 g potassium metabisulfite ($K_2S_2O_5$), 10 g sodium thiosulfate ($Na_2S_2O_3$) and 100 ml distilled water.

3. Results and discussion

The high austenitization temperature of 1225 ºC was employed in order to dissolute Nb during heating. The solubility of Nb can be calculated as follows (T in K) [12]:

$$\mathrm{Log}_{10}[\mathrm{Nb}][\mathrm{C}]=-6770/T+2.26 \quad (1)$$

By using above equation, solubility of Nb in the presence of 0.14 wt% C at 1225 ºC was ~.04 which was close to Nb content of microalloyed steel in the present investigation. Thus, almost the complete dissolution of Nb in austenitization treatment was expected.

Figure 2(a) represents the as quenched microstructure of Nb .04 steel. This martensite structure was lath type because of its carbon content

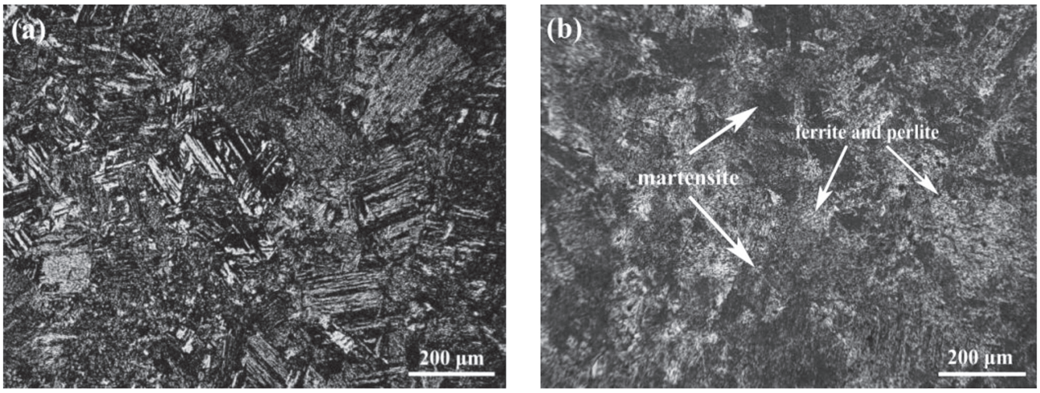

Fig. 2. (a) Monochrome and (b) color OM micrograph of as quenched microstructure of Nb .04 specimen. Up to 85% of microstructure is mirtensite.

which was lower than 0.6 wt% [13]. Achieving the high amount of martensite in low carbon steels was difficult due to their low hardenability. So, -15ºC quench solution was employed to enhance the cooling rate and subsequently access the higher amount of martensite phase. The tetragonality of martensite unit cell of steels with the carbon content of lower than 0.2 wt% was very low, close to cubic unit cells. Thus, it is impossible to determine the volume fraction of martensite using XRD patterns. Color metallography, which reveals each phase with specific color under polarized light based on its chemical composition, is a useful method in such circumstances. Figure 2(b) shows the color metallography image of Nb .04 as quenched microstructure. Martensite appeared as blue and bluish brown using the mentioned etching solution agent, while pearlite and ferrite seemed orange and yellow [14]. Hence, there was up to 85% martensite phase in as quenched steels.

The lath martensite in low carbon steels contained three-level hierarchy microstructure, i.e., martensite laths, which were single crystals of martensite, martensite block, which was the aggregation of laths, and martensite packet, which was the aggregation of blocks. The laths in a block had the same crystallographic orientation and the blocks in a packet had the same habit plane [15]. Up to 83% of block boundaries and packet boundaries were composed of high angle boundaries [16]. Cold rolling of such ultrafine microstructure enhanced the dislocation density and the beginning martensite fragmentation. As reported by Ghasemali et al [10], more than 65% reduction in thickness was required for martensite fragmentation and Hosseini et al [14] proved that increasing cold deformation to 85% reduction in thickness led to achieving finer ferrite grains. Figure 3 reveals SEM image of 85% cold rolled martensite in Nb .04 sample, which was composed of fully lamellar dislocation cells (LDCs).

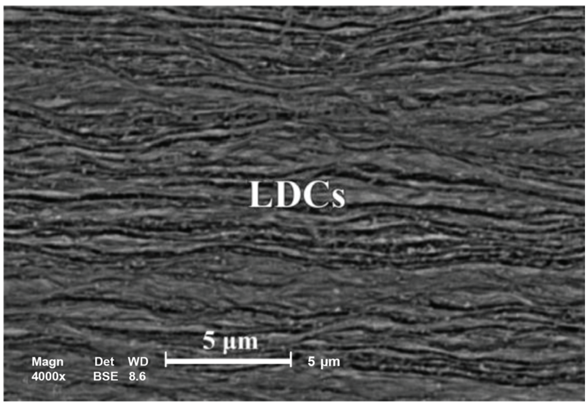

Fig. 3. SEM image of 85% cold rolled martensite.

Ferrous martensite is a supersaturated solid solution of carbon in iron obtained by quenching from temperature at which there is austenite single phase. The result of tempering of such microstructures is the removal of carbon from solid solution and precipitation of Fe_3C carbides. If carbide-forming elements are present at chemical composition, alloy carbides can replace Fe_3C carbides during annealing almost above 500 ºC [17].

The hardness evaluation during warm temperature annealing of samples used in the present investigation has been shown in Figure 4. The cold rolled martensite had the maximum hardness due to the fragmentation of structure including the high density of defects such as dislocations, vacant lattice sites, prior austenite grain boundaries, twins, etc. Hardness drop at the initial times of annealing is related to the recovery of high-density dislocations. As it is obvious, the recovery rate was accelerated with increasing the temperature. After the intense drop, the hardness was reduced with a light gradient. At 450 and 500 ºC, there was no different behavior in hardness decrease between the simple carbon steel and Nb microalloyed steel (Figures 4(a) and 4(b). However, increasing temperature to 550 ºC caused the variation in hardness procedure (Figure 4(c)). During annealing time between 300 s to 500 s, hardness of Nb .04 sample was increased with increasing the heating time. This can be attributed to the precipitation of niobium carbides because of negligible Nb solubility at such a temperature. Such increases in hardness had been observed in high-speed tool steels, which were tempered after rapid cooling to precipitate fine carbides [17]. At 600 ºC annealing temperature (Figure 4(d)), in spite of the increase in hardness distinction of the two chemical compositions, the mentioned stages were enhance and the second drop in hardness was shifted to lower times. This is related to approaching the temperature to the precipitation-time-temperature nose of steels with almost the same chemical composition [18].

It is not logical to consider increasing and decreasing hardness as precipitation start and final, because of the unsteady grain size during heating. Nevertheless, such curves can show precipitation when considering the different behavior of plain carbon and microalloyed steels and secondary hardening effect, which occurred in Nb .04 samples.

As discussed earlier, grain subdivision during cold rolling and the formation of DIBs, followed by the recovery of dislocation during warm temperature annealing were the main reasons for the formation of nano ferrite grains. It is worth noting that martensite fine microstructure is the main factor in achieving DIBs under the lower amount of cold deformation in comparison with SPD methods [16].

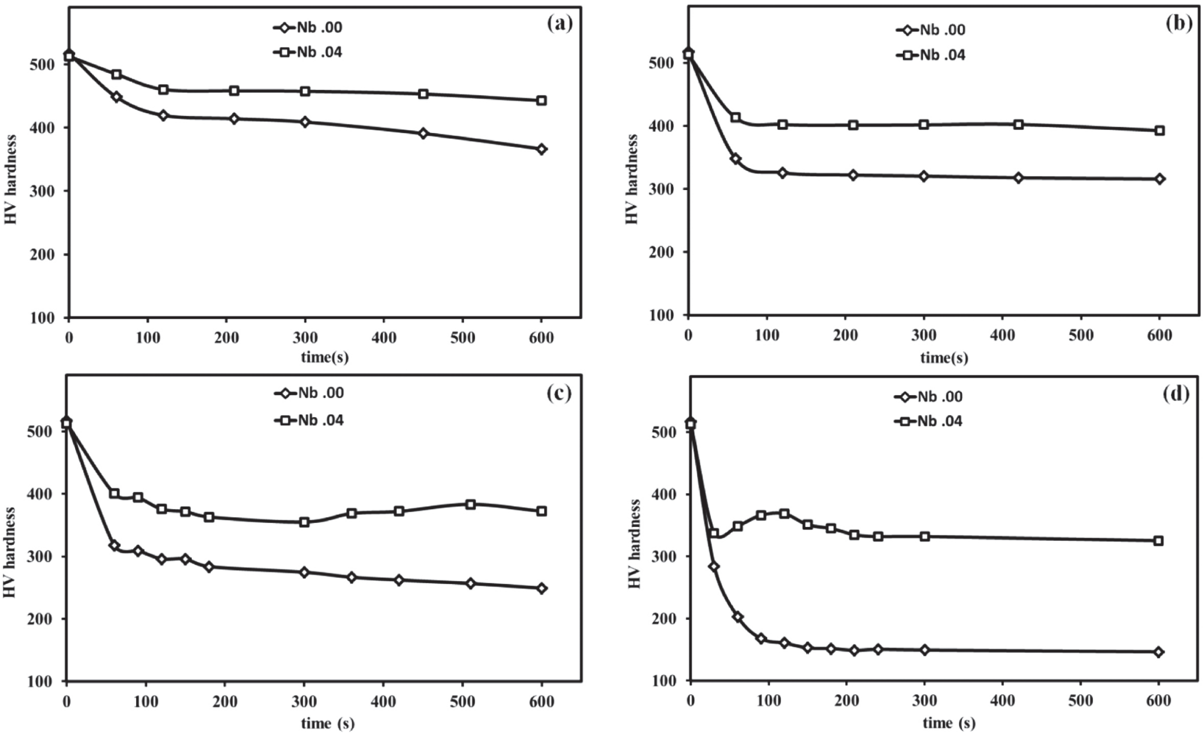

Fig. 4. Hardness variation as a function of annealing time at (a) 450 ºC, (b) 500 ºC, (c) 550 ºC and (d) 600ºC temperatures.

Figure 5 reveals SEM microstructure of 85% cold rolled samples after warm temperature annealing at 550 ºC for 300 s. Annealing of deformed Nb .00 sample in such circumstances led to the complete formation of ferrite with mean grain size of 87 nm, but there were some grains bigger than others (Figure 5(a)). This phenomenon had been reported by Hoseini et al [14] and it is called "abnormal grain growth". The reason for this occurrence is minimizing the role of cementite carbide in the prevention of grain boundaries migration at higher temperatures. The presence of niobium in chemical composition, in spite of lowering the mean grain size to 79 nm, prevented abnormal grain growth (Figure 5(b)) and resulted in the formation of nano ferrite grains with uniform distribution. This difference in microstructure evolution illustrates the unequal role of Fe_3C and NbC carbides in the prevention of grain boundaries migration.

Microstructural images of samples annealed at 550 ºC for 2.7 Ks and 600 ºC for 300 s have been shown in Figure 6. As can beseen, mean grain size of plain carbon steel after being 2.7 Ks at 550 ºC (Figure 6(a)) or 300 s at 600 ºC (Figure 6(b)) reached to a degree bigger than a few micrometers. On the other hand, according to Figures 6(c) and 6(d), there was no considerable change in ferrite grain size in microalloyed steel (mean grain size of 104 nm and 167 nm, respectively). Thus, the existence of niobium carbides strongly affects the grain growth and grain boundaries migration at higher temperatures or longer annealing times.

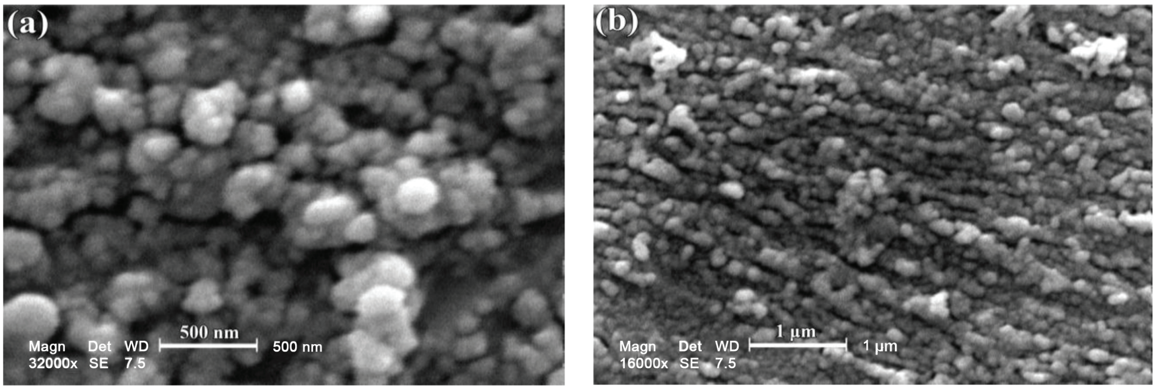

Fig. 5. SEM images of (a) Nb 0.00 and (b) Nb 0.04 after 85% cold rolling followed by annealing at 550ºC for 300 s.

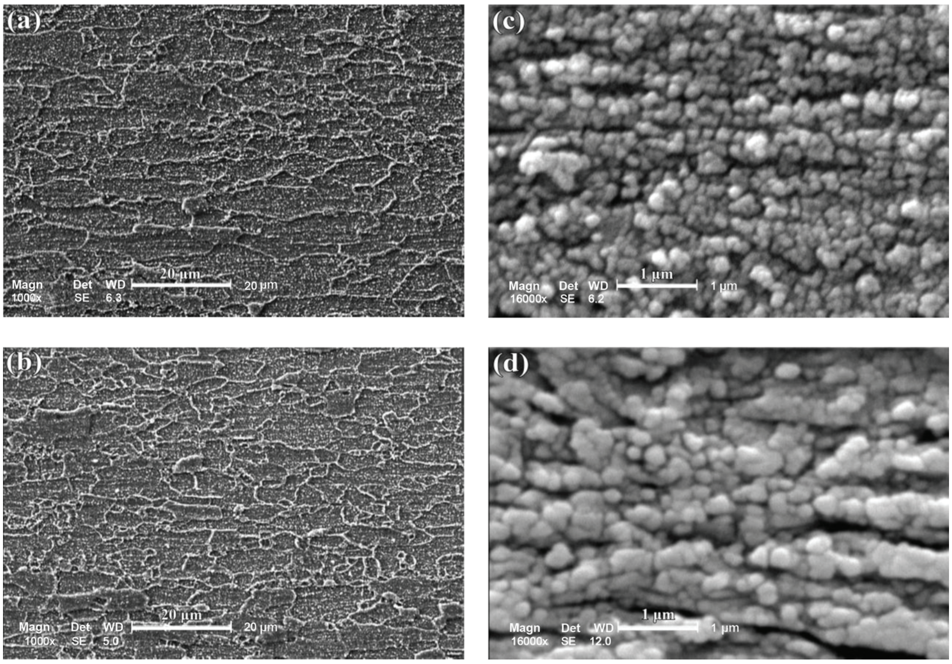

Fig. 6. SEM images of Nb 0.00 cold rolled samples annealed at (a) 550 ºC for 2.7 Ks and (b) 600 ºC for 300 s, and Nb 0.04 cold rolled samples annealed at (c) 550 ºC for 2.7 Ks and (d) 600 ºC for 300 s.

4. Conclusion

In the present work, the effect of Nb on the formation of nanostructured steel was investigated using martensite treatment . It was shown that the presence of 0.045 %wt Nb in chemical composition led to uniform ferrite nano grains with mean grain size of about 79 and 167 nm after annealing of 85% cold rolled martensite at 550 and 600 ºC for 300 s, respectively. In addition, it could prevent abnormal grain growth after annealing at 550 ºC for 300 s. The hardness measurements proved precipitation of NbC carbides during tempering and SEM images of annealed samples showed that these carbides were relatively effective in thermal stability of ultrafine steel and the prevention of severe grain growth.

References

[1] Y. Okitsu, N. Takata and N. Tsuji: Scripta Mater., 60(2009), 76.

[2] Y. Saito, H. Utsunomiya, N. Tsuji and T. Sakai: Acta Mater., 47(1999), 579.

[3] E. Schafler and M.B. Kerber: Mater. Sci. Eng. A, 462(2007), 139.

[4] A.P. Zhilyaev, K. Oh-ishi, G.I. Raab and T.R. McNelley: Mater. Sci. Eng. A, 441(2006), 245.

[5] R. Song, D. Ponge, D. Raabe, J.D. Speer and D.K. Matlock: Mater. Sci. Eng. A, 441(2006), 1.

[6] T. Jing, Y. Gao, G. Qiao, Q. Li, T. Wang, W. Wang, F. Xiao, D. Caiiao, X. Song and X. Zhao: Mater. Sci. Eng. A, 432(2006), 216.

[7] H.F. Lan, W.J. Liu and X.H. Liu: ISIJ Int., 47(2007), 1652.

[8] N. Tsuji, R. Ueji, Y. Minamino and Y. Saito: Scripta Mater., 46(2002), 305.

[9] R. Ueji, N. Tsuji, Y. Minamino and Y. Koizumi: Sci. Tech. Adv. Mater., 5(2004), 153.

[10] E. Ghassemali, A. Kermanpur and A. Najafizadeh: J. Nanosci. Nanotechnol., 10(2010), 6177.

[11] S.M. Hosseini, A. Najafizadeh and A. Kermanpur: J. Mater. Process. Technol., 211(2011), 230.

[12] A. Ghosh, S. Das, S. Chatterjee, B. Mishra and P. Ramachandra: Mater. Sci. Eng. A, 348(2003), 299.

[13] H. Kitahara, R. Ueji, N. Tsuji and Y. Minamino: Acta Mater., 54(2006), 1279.

[14] S.M. Hosseini, A. Kermanpur, A. Najafizadeh and M. Alishahi: ISIJ Int., 52(2012), 464.

[15] F. Foroozmehr, A. Najafizadeh and A. Shafyei: Mater. Sci. Eng. A, 528(2011), 5754.

[16] R. Ueji, N. Tsuji, Y. Minamino and Y. Koizumi: Acta Mater., 50(2002), 4177.

[17] W.C. Leslie and E. Hornbogen: Physical Metallurgy of Steels, Physical Metallurgy, ed. by R.W. Cahn and P. Hassen, fourth reviesed, Elsevier science B.V., Amsterdam, (1996), 1555.

[18] Q. Yong, X. Song, G. Yung and Z. Zhung: Solution and Precipitation of Secondary Phase in Steels: Phenomenon, Theory and Practice, in advanced steels, ed. by Y. Weng, H. Dong and Y. Gan, Springer, (2011), 109.