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TEMPERING OF DEFORMED AND AS-QUENCHED MARTENSITE IN STRUCTURAL STEEL

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Abstract

Annealing of deformed martensite (high-temperature tempering) in ST37 steel was studied. Different reductions in thickness were considered and compared with the behavior of as-quenched martensite during tempering. Tempering of the as-quenched martensite was accompanied by the formation of carbide particles, incomplete disappearance of the lath martensite morphology, and continuous decrease in hardness until reaching low values. However, during tempering of the cold rolled martensite, the precipitation of carbides in the lamellar structure, development of distinct equiaxed ultrafine grains through a continuous recrystallization mechanism, and a sudden hardness drop were characterized. The importance of cold rolling reduction and its amount were also discussed.

Keywords: *Low carbon steels; Quench and tempering; Cold rolling; Continuous recrystallization; Hardness.*

1. Introduction

As overviewed by Zhao and Jiang [1] and Song et al. [2], the thermomechanical processing routes are viable approaches on the industrial scale for grain refinement of steels, where successful implementation of these methods has been reported by Goryany et al. [3, 4], Cabrera and coworkers [5, 6], and Naghizadeh and Mirzadeh [7, 8]. Other techniques such as plasma nitriding [9] and integration of the deep rolling process into the hardening treatment [10] have been recently used for enhancement of properties of steels. On the other hand, the severe plastic deformation (SPD) techniques have been developed for more intense grain refinement of steels as summarized by Umemoto [11]. While the focus of the SPD techniques is on the applied high strain, there is an increasing interest to use advanced thermomechanical processing routes for grain refinement with less required strains, where these techniques have been discussed by Tsuji and coworkers [12, 13]. In this respect, lath martensite characterized by pockets, blocks, and laths with its high dislocation density has been reported to be quite advantageous by Najafi et al. [14], Wang et al. [15], Azizi-Alizamini et al. [16], and Morito et al. [17].

The martensite phase is known as one of the main constituent phases in the advanced high-strength steels, which has been extensively studied by Bleck and coworkers [18-20] and Mirzadeh and coworkers [21-23]. However, owing to their high strength and brittleness, the martensitic steels are normally used in the quenched and tempered condition. Interestingly, the martensitic low-carbon steels show high work-hardening rate and can be directly used without tempering heat treatment. The low-carbon martensitic steels might be also subjected to plastic deformation as shown by Morito et al. [17], Nouroozi et al. [24], and Alibeyki et al. [25]. Based on this fact, an advanced thermomechanical processing has been developed by Tsuji et al. [26], which is based on the rolling of a martensite starting microstructure followed by short tempering to produce an ultrafine ferrite/carbide aggregate. Several excellent works have been reported on this subject: the effects of rolling reduction and tempering temperature have been studied by Ueji et al. [27, 28]; the effect of microalloying elements has been reported by Lan et al. [29]; and the effect of tempering time has been revealed by Malekjani et al. [30]. However, the formation mechanism of ultrafine microstructure has received less attention and it is reported to be still under debate by Lan et al. [29] and Najafi et al. [14]. Therefore, this subject, especially the importance of the cold rolling reduction, deserves much more experimental works, which is the subject of the present paper.

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2. Experimental details

A 0.12 wt%C - 0.16 wt%Si - 1.11 wt%Mn steel was austenitized and quenched to obtain the as-quenched structure. Afterward, rolling reductions of 30%, 50%, and 70% were applied on the as-quenched sheet. The sheets were then tempered at 550 °C for various holding times. The tempering temperature of 600 °C was also considered for comparison purposes. Finally, SEM images and hardness test (load of 0.5 kg) were used for characterization.

3. Results and discussion

Figure 1 shows the evolution of hardness as a function of tempering time for the martensitic microstructure at 550 °C and 600 °C. It can be seen that there is a continuous decrease in hardness and this decrease is faster at 600 °C when compared to 550 °C.

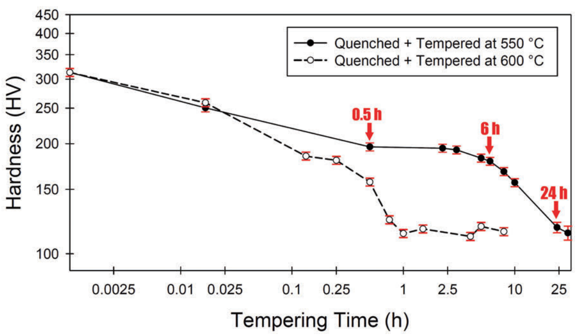

Figure 1. *Hardness evolutions during tempering of the as-quenched martensite (double-logarithmic plot)*

Figure 2a shows SEM images of the starting martensitic microstructure at two different magnifications, which represents typical lath morphology as shown by Morito et al. [31], Maleki et al. [32] and Mirzadeh et al. [33]. As can be seen in Figures 2b and 2c, tempering is accompanied by the formation of carbide particles (white particles) and incomplete disappearance of the lath martensite morphology. In fact, as shown by Krauss [34], the decrease in tetragonality by loss of carbon and the recovery of martensitic microstructure during tempering are the main reasons for the decrease in hardness. At longer tempering durations, these effects become more pronounced as shown in Figure 2d. Accordingly, tempering at a higher temperature (600 °C) is expected to be more rapid, which results in the rapid decrease of hardness (Figure 1).

Figure 3 shows the microstructures of the 70% rolled martensite and those of as-quenched martensite for comparison. It can be seen that the microstructure consisting of prior austenite grains with the lath-type martensitic structure is replaced by a lamellar structure along rolling direction (RD) after cold rolling. The evolution of the lath martensite morphology can be better realized based on the higher magnification images shown in Figure 3, where the deformation of laths is evident. Similar observations have been reported by Ueji et al. [27]. These

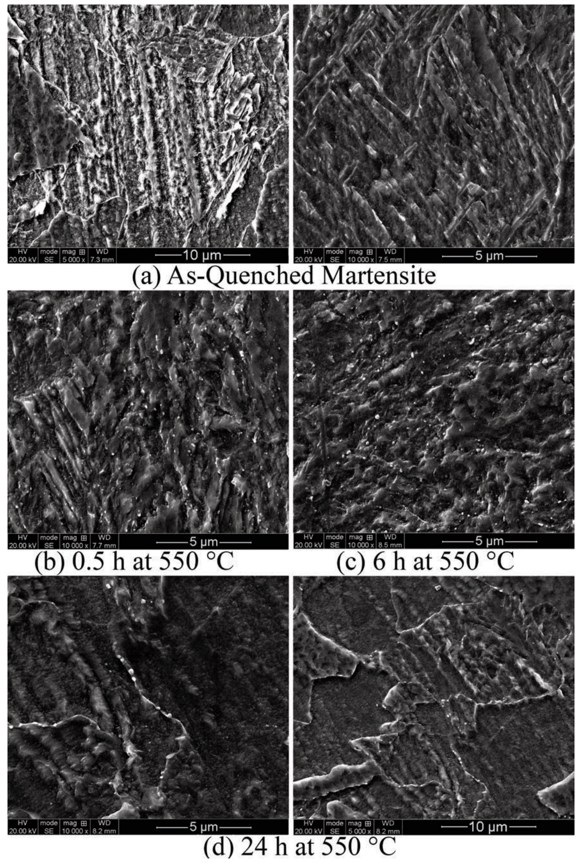

Figure 2. *Microstructural evolutions during tempering of the as-quenched martensite*

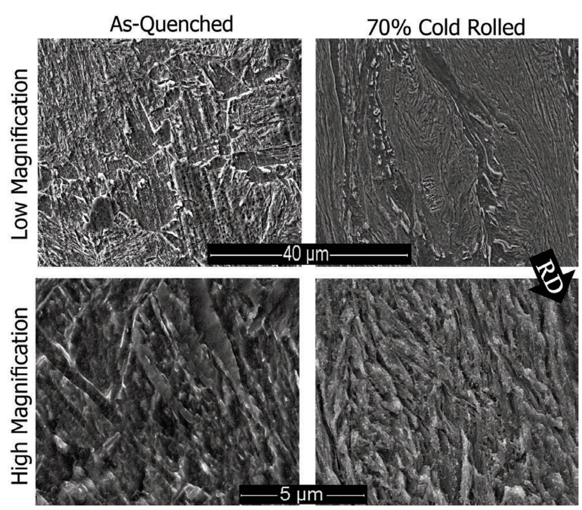

Figure 3. *SEM images of the as-quenched and 70% cold rolled sheets at low and high magnification*

microstructural changes might have a significant effect on the microstructure and hardness during subsequent tempering treatments as will be discussed below.

Figure 4 shows the microstructures of the rolled and annealed samples (550 °C). After 0.5 h at 550 °C, many carbide particles precipitated (bright particles) in the cold-rolled lamellar structure. By continued tempering up to 1.5 h, fading of this structure and formation of equiaxed ultrafine grains is obvious. After 2.25 h, larger grains (~ 3.5 μm) can be seen. These microstructural evolutions can be adequately described by the model proposed for the continuous recrystallization as shown in Figure 4. In this mechanism, as summarized by Humphreys and Hatherly [35] and Najafi et al. [14], the collapse of the lamellar structure followed by tendency toward equiaxed morphology can be identified.

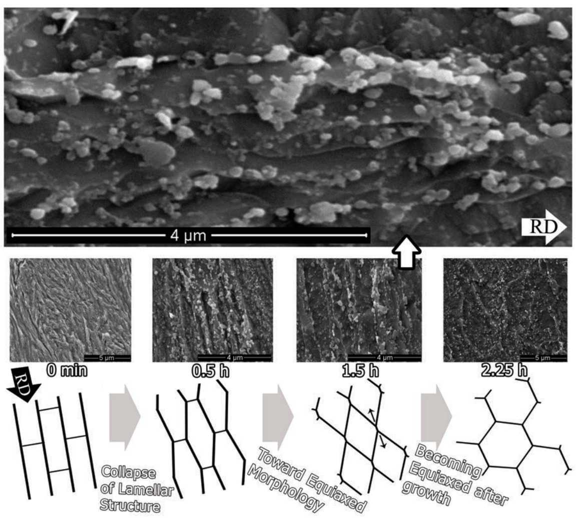

Figure 4. *Microstructures of the rolled and annealed samples at 550 °C along with the corresponding schematic drawings representing the continuous recrystallization mechanism (adapted from [35])*

Figure 5 shows the evolution of hardness as a function of tempering time at 550° C for the 70% cold rolled sheet and as-quenched sheet as well. For the as-quenched sheet, there is a continuous decrease in hardness until reaching low values (note that the time axis is logarithmic). However, for the cold rolled sheet, there is a sudden hardness change from 1.5 h to 2.25 h. This sudden change is characteristic and relates to the formation of the fine-grained microstructure from the cold rolled martensitic one (Figure 4). Therefore, this difference is related to the occurrence of continuous recrystallization mechanism, where it can be deduced that its occurrence is related to the cold deformation before the tempering treatment.

Moreover, it can be seen that the decrease in hardness until reaching low values takes place in a much shorter time. Another interesting observation is related to the values of hardness, showing that the hardness of the cold rolled sample is higher, which is an expected result. During tempering, the hardness decreases, and then, after the sudden hardness change from 1.5 h to 2.25 h, the hardness of the cold rolled sheet falls below that of the as-quenched one. This is another evidence for the occurrence of an important softening mechanism like recrystallization.

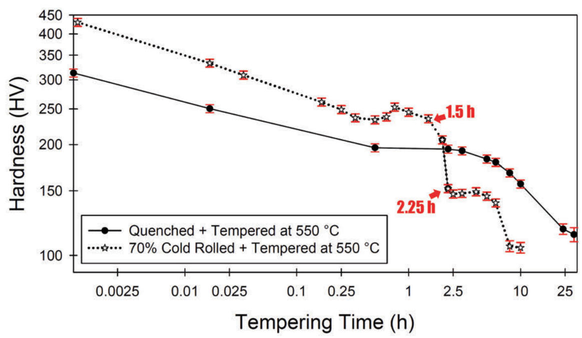

Figure 5. *Hardness evolutions during tempering of the cold rolled and as-quenched martensite (double-logarithmic plot)*

Based on Figures 4 and 5, it was concluded that the sudden decrease in hardness of the 70% cold rolled sheet during tempering is related to the continuous recrystallization mechanism. However, for the as-quenched sheet (0% cold rolled), a monotonic decrease due to the conventional tempering in hardness can be seen. Therefore, it is interesting to investigate the effect of reduction in thickness.

Figure 6a shows the evolution of hardness as a function of tempering time at 550° C for the 0%, 30%,

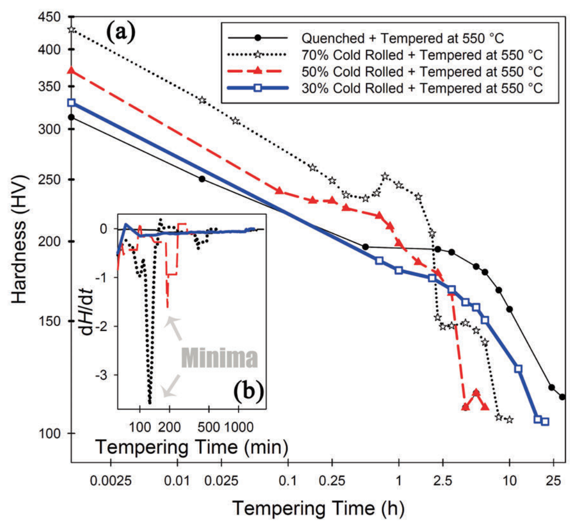

Figure 6. *(a) Hardness evolutions during tempering of the cold rolled martensite (double-logarithmic plot) and (b) the hardening rate versus tempering time*

50%, and 70% cold rolled sheets. It can be seen that the initial hardness increases by increasing the reduction in thickness. During tempering, while the decrease in hardness until reaching low values takes place in a shorter time, the 30% cold rolled sheet exhibits a behavior similar to that of the as-quenched sheet. The 50% cold rolled sheet, however, shows the sudden drop in hardness like the 70% cold rolled sheet. To better evaluate these changes, the hardening rate (dH/dt) versus tempering time is shown in Figure 6b. The sudden hardness drops in Figure 6a are identical to temporary low hardening rates that form minimum points. It can be seen that the 50% and 70% cold rolled sheets clearly show these minimum points.

Therefore, the continuous recrystallization mechanism happens in the 50% and 70% cold rolled sheets, and hence, 30% cold reduction is not adequate. As discussed for Figure 3, cold rolling resulted in the deformation of laths in the 70% cold rolled sheet. Based on Figures 4 and 5, it can be realized that the adequate deformation of the laths is a prerequisite for the continuous recrystallization mechanism to happen. It is well-known in plastic deformation studies that if the mean low angle boundary misorientation is increased (via increasing strain), the boundary properties become more uniform, and the structure cannot recrystallize discontinuously. In this way, it can recrystallize continuously, which happens at large plastic strains as shown by Humphreys and Hatherly [35] and Najafi et al. [14]. However, in the case of martensite starting microstructure, the high dislocation density of the martensite is advantageous to decrease the amount of required strain as shown by Ueji et al. [27]. Anyway, some amount of strain is required to replace martensite laths with dislocation cells as shown by Morito et al. [17]. The present work provided another evidence for this fact.

4. Conclusions

Tempering of deformed martensite at high-temperature in st37 steel was studied. Different reductions in thickness were considered and compared with the behavior of as-quenched martensite during tempering. The following conclusions can be drawn from this work:

(1) Tempering of the as-quenched martensite was found to be accompanied by the formation of carbide particles, incomplete disappearance of the lath martensite morphology, and continuous decrease in hardness until reaching low values.

(2) During tempering of the cold rolled martensite, the precipitation of carbides in the lamellar structure, development of distinct equiaxed ultrafine grains through a continuous recrystallization mechanism, and a sudden hardness drop were characterized.

(3) Before tempering, the hardness of the cold rolled samples was higher. During tempering, the hardness decreases, and then, after the sudden hardness change, the hardness of the cold rolled sheet fell below that of the as-quenched one due to the softening effect of recrystallization.

(4) The importance of cold rolling reduction and its amount on the occurrence of continuous recrystallization mechanism were discussed. It was revealed that the adequate deformation of the laths is a pre-requisite, and hence, a minimum amount of cold deformation is necessary.

References

[1] J. Zhao, Z. Jiang, Prog. Mater. Sci., 94 (2018) 174-242.
[2] R. Song, D. Ponge, D. Raabe, J.G. Speer, D.K. Matlock, Mater. Sci. Eng. A, 441 (2006) 1-17.
[3] V. Goryany, T. Khlyntseva, I. Mamuzić, V. Radsinsky, J. Min. Metall. Sect. B-Metall., 40 (2004) 75-88.
[4] V. Goryany, V. Radsinsky, J. Min. Metall. Sect. B-Metall., 38 (2002) 171-177.
[5] J.M. Cabrera, J. Ponce, J.M. Prado, J. Mater. Process. Technol., 143 (2003) 403-409.
[6] M.B. dos Reis Silva, J.M. Cabrera, O. Balancin, A.M. Jorge Jr, Mater. Charact., 127 (2017) 153-160.
[7] M. Naghizadeh, H. Mirzadeh, Metall. Mater. Trans. A, 49 (2018) 2248-2256.
[8] M. Naghizadeh, H. Mirzadeh, Vacuum, 157 (2018) 243-248.
[9] M. Keddam, T. Thiriet, G. Marcos, T. Czerwiec, J. Min. Metall. Sect. B-Metall., 53 (2017) 47-52.
[10] P. Donhongprai, P Juijerm, J. Min. Metall. Sect. B-Metall., 54 (2018) 67-71.
[11] M Umemoto, Mater. Trans., 10 (2003) 1900-1911.
[12] N. Tsuji, Adv. Eng. Mater., 12 (2010) 701-707.
[13] L. Zhao, N. Park, Y. Tian, S. Chen, A.Shibata, N. Tsuji, Mater. Res. Lett., 5 (2017) 61-68.
[14] M. Najafi, H. Mirzadeh, M. Alibeyki, Mater. Sci. Eng. A, 670 (2016) 252-255.
[15] X. Wang, R. Ding, J. He, A.Zhao, R. Liu, Metall. Mater. Trans. A, 49 (2018) 1439-1443.
[16] H. Azizi-Alizamini, M. Militzer , W.J. Poole, ISIJ Int., 51 (2011) 958-964.
[17] S. Morito, T. Ohba, A.K. Das, T. Hayashi, M. Yoshida, ISIJ Int., 53 (2013) 2226-2232.
[18] W. Bleck, S. Papaefthymiou, A. Frehn, Steel Res. Int., 75 (2004) 704-710.
[19] U. Prahl, S. Papaefthymiou, V. Uthaisangsuk, W. Bleck, J. Sietsma, S. van der Zwaag, Comput. Materi. Sci., 39 (2007) 17-22.
[20] C. Thomser, V. Uthaisangsuk, W. Bleck, Steel Res. Int., 80 (2009) 582- 587.
[21] Z. Nasiri, H. Mirzadeh, Materwiss. Werksttech., 49 (2018) 1081-1086.
[22] M. Zamani, H. Mirzadeh, M. Maleki, Mater. Sci. Eng. A, 734 (2018) 178-183.
[23] S. Ghaemifar, H. Mirzadeh, Steel Res. Int., 89 (2018) 1700531.
[24] M. Nouroozi, H. Mirzadeh, M. Zamani, Mater. Sci.

Eng. A, 736 (2018) 22-26.
[25] M. Alibeyki, H. Mirzadeh, M. Najafi, Vacuum, 155 (2018) 147-152.
[26] N. Tsuji, R. Ueji, Y. Minamino, Y. Saito, Scr. Mater. 46 (2002) 305-310.
[27] R. Ueji, N. Tsuji, Y. Minamino, Y. Koizumi, Acta Mater., 50 (2002) 4177-4189.
[28] R. Ueji, N. Tsuji, Y. Minamino, Y. Koizumi, Sci. Tech. Adv. Mater., 5 (2004) 153-162.
[29] H. F. Lan, W. J. Liu, X. H. Liu, ISIJ Int., 47 (2007) 1652-1657.
[30] S. Malekjani, I.B. Timokhina, I. Sabirov, P.D. Hodgson, Can. Metall. Q., 48 (2009) 229-236.
[31] S. Morito, H. Tanaka, R. Konishi, T. Furuhara, T. Maki, Acta Mater., 51 (2003) 1789-1799.
[32] M. Maleki, H. Mirzadeh, M. Zamani, Steel Res. Int. 89 (2018) 1700412.
[33] H. Mirzadeh, M. Alibeyki, M. Najafi, Metall. Mater. Trans. A 48 (2017) 4565-4573
[34] G. Krauss, Steel Res. Int., 88 (2017) 1700038.
[35] F.J. Humphreys, M. Hatherly, Recrystallization and Related Annealing Phenomena, second ed., Elsevier, Amsterdam, The Netherlands 2004.

OTPUŠTANJE DEFORMISANOG MARTENZITA U KALJENOM KONSTRUKCIONOM ČELIKU

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Apstrakt

Žarenje deformisanog ST37 čelika (otpuštanje pri visokim temperaturama) sa martenzitnom strukturom je ispitivano. Razmatran je uticaj različitih stepena redukcije debljine čelika i upoređen sa ponašanjem martenzita u kaljenom stanju čelika tokom postupka otpuštanja. Postupak otpuštanja martenzita dobijenog kaljenjem čelika je praćen formiranjem čestica karbida, nepotpunim nestankom strukture martenzitnih pločica, kao i kontinuiranim smanjenjem tvrdoće do dostizanja niskih vrednosti. Međutim, tokom postupka otpuštanja hladnovaljanog martenzita primećeno je taloženje karbida lamelarne strukture, nastanak posebnih ultra-finih ravnoosnih zrna putem mehanizma kontinuirane rekristalizacije, kao i iznenadni pad tvrdoće. Ispitivana je i važnost redukcije kod hladnog valjanja, kao i u kojoj se meri ona dešava.

Ključne reči: *Niskougljenični čelici; Kaljenje i otpuštanje; Hladno valjanje; Kontinuirana rekristalizacija; Tvrdoća.*