IJMF, Iranian Journal of Materials Forming, Vol.5, No. 1, pp 19-25
Printed in The Islamic Republic of Iran, 2018

Grain Refinement of Dual Phase Steel via Tempering of Cold-Rolled Martensite

M. Najafi, H. Mirzadeh* and M. Alibeyki

School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, P.O. Box 11155-4563, Tehran, Iran.

Abstract: A microstructure consisting of ultrafine grained (UFG) ferrite with average grain size of ~ 0.7 µm and dispersed nano-sized carbides was produced by cold-rolling and tempering of the martensite starting microstructure in a low carbon steel. Subsequently, fine grained dual phase (DP) steel consisting of equiaxed ferrite grains with average size of ~ 5 µm and martensite islands with average size of ~ 3 µm was produced by intercritical annealing of this microstructure. Coarse grained DP steel with average ferrite grain size of ~ 20 µm and average martensite island size of ~ 5 µm was also produced by intercritical annealing of the as-received ferritic-pearlitic microstructure. The UFG microstructure showed high strength, low ductility, and poor work hardening response due to intense grain refinement. The fine grained DP steel had higher tensile strength and total elongation compared with the coarse grained one, which was related to the improved work-hardening behavior by microstructural refinement.

Keywords: DP steels, Microstructure, Mechanical properties, Thermomechanical processing.

1. Introduction

Dual phase (DP) steels, as a class of advanced high strength steels, are low carbon steels having a microstructure consisting of hard martensite phase in a matrix of soft ferrite. They possess high strength-ductility balance, which make them attractive for automotive and other industries [1-5].

At a given martensite fraction, the mechanical properties of the DP steels are largely determined by the morphology, size, and distribution of the martensite and also ferrite grain size [6, 7]. In this regard, fine grained DP steels have received a considerable attention [1, 8-11]. The majority of processing routes to obtain fine grained DP steels are based on the (1) intercritical annealing preceded by cold rolling of DP microstructure (in carbon steel with 0.17 wt.% C) [9], (2) intercritical annealing preceded by cold rolling of martensite (in carbon steel with 0.18 wt.% C) [10], or cold rolling of tempered martensite (in carbon steel with 0.17 wt.% C) [1].

It has been recently shown that by the tempering of cold rolled martensite in a low-carbon steel with ~ 0.12 wt.% C, it is possible to obtain a wide range of ferrite grain sizes and dispersed carbides [12-15]. Moreover, the responsible mechanisms for the evolution of ultrafine grained (UFG) structure during the tempering step have been identified [13]. Therefore, it seems that no tempering stage before cold rolling is required for low-carbon steel with ~ 0.12 wt.% C and it is possible to directly produce fine-grained structures by intercritical annealing cold rolled martensite based on the optimum tempering time [13], which has not been considered so far. The present work aims to deal with this subject using a low-carbon steel with 0.121 wt.% C.

2. Experimental Materials and Procedure

A 0.121C-0.16Si-1.11Mn (wt.%) steel was used in this work. The A_1 and A_3 temperatures were respectively estimated as ~ 730 °C and ~ 897 °C based on Trzaska and Park equations (1) [16]. Two

Received by the editors August 9, 2017; Revised September 12, 2017 and Accepted September 25, 2017.
*Corresponding author (hmirzadeh@ut.ac.ir)

different processing routes were followed to obtain different DP microstructures, as schematically represented in Fig. 1. The as-received sheets (Fig. 1a) were austenitized at 1050 °C followed by water quenching to develop a martensitic microstructure (Fig. 1b) which was subsequently cold rolled by reduction in thickness of 70% followed by tempering for 30 min at 550 °C [13] to develop an UFG ferrite-carbide aggregate (Fig. 1c). As-received ferritic-pearlitic and UFG microstructures were soaked at 770 °C for 1 min followed by water quenching to obtain DP microstructures with martensite volume fraction (V_M) of ~ 0.2 (Fig. 1d and 1e). Etching in the LePera's reagent (1g $Na_2S_2O_5$ in 100 ml H_2O + 4 g picric acid in 100 ml ethanol) followed by 2% Nital solution was used to reveal microstructural features for optical microscopy and scanning electron microscopy (CamScan MV 2300 SEM). The tensile specimens were prepared according to the JIS Z 2201 standard with a gage length of 6.2 mm for the 70 pct cold-rolled sheet and 11.2 mm for other samples to conform to the requirements of Barba's law [17].

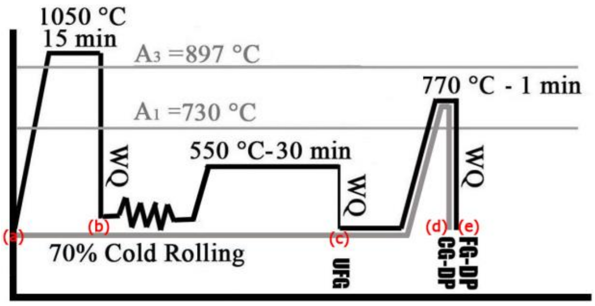

Fig. 1. Schematic representation of the processing routes used in this work: (a) as-received, (b) as-quenched, (c) ultrafine grained, (d) coarse-grained DP, and (e) fine-grained DP states.

3. Results and Discussion

3.1. Microstructures

The microstructures before intercritical annealing as obtained based on Fig. 1 are shown in Fig. 2. The as-received microstructure consists of coarse ferrite grains with average grain size of ~ 15 µm and pearlite islands. The as-quenched sample has a typical martensitic lath morphology and the so-called UFG microstructure shows average ferrite grain size of ~ 0.7 µm alongside with nano-sized carbides due to the occurrence of continuous recrystallization as described elsewhere [13,14].

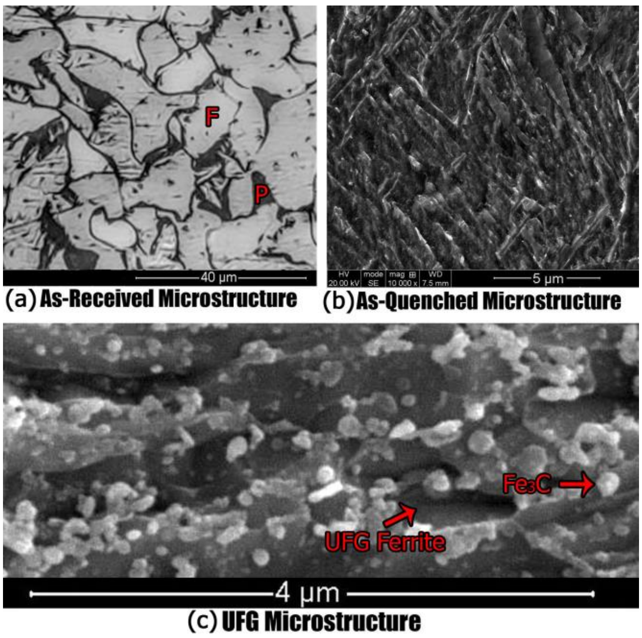

Fig. 2. Optical micrograph of the as-received specimen (a) as well as SEM images of the as-quenched (b) and UFG (c) microstructures. F and P denote the ferrite and pearlite respectively.

After intercritical annealing, in the as-received microstructure, the pearlitic areas and part of the neighboring ferrite transform to austenite during intercritical annealing and then to martensite after quenching (shown in Fig. 3 as CG-DP). In this microstructure, the average size of ferrite grains and martensite islands are ~ 20 µm and 5 µm respectively. Therefore, it can be deduced that the ferrite grain size in this DP steel is somewhat large, which was considered as a coarse-grained microstructure in the present work. Conversely, by intercritical annealing of the UFG microstructure, a DP microstructure consists of equiaxed ferrite grains with average size of ~ 5 µm and martensite islands with average size of ~ 3 µm was obtained (Fig. 3). During intercritical annealing, austenite normally forms in place of fine carbide particles and grows outward in the adjacent ferrite. It can be seen that during intercritical annealing, the ultrafine ferrite grains grows even in 1 min. Evidently, this DP microstructure is much finer, and was therefore named fine-grained DP microstructure (FG-DP).

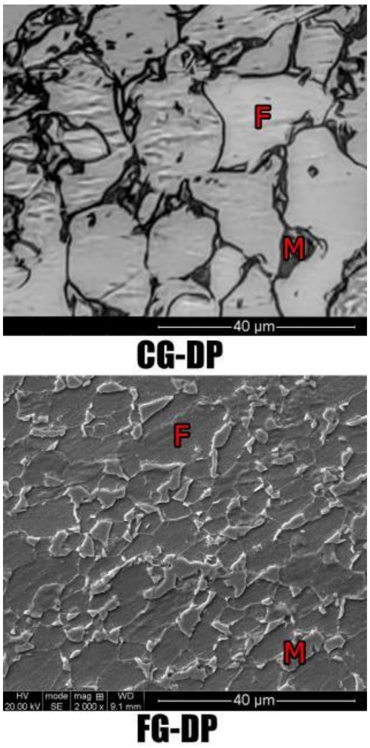

Fig. 3. Microstructures of the DP steels processed in the present work. F and M denote the ferrite and martensite phases respectively.

3.2. Mechanical properties

Figure 4 shows the corresponding tensile test results. The as-received specimen exhibits low strength, high ductility, and the yield point phenomenon, where the latter is believed to be related to the Cottrell atmospheres produced by interstitial atoms around the dislocations [17]. However, the UFG microstructure (produced by cold rolling of martensite followed by tempering) exhibits very high strength and low ductility due to intense grain refinement and also shows the yield point phenomenon as it was expected. The stress-strain behavior of this specimen does not show appreciable work-hardening regime that is typical to ultrafine grained materials [18].

After intercritical annealing the yield point phenomenon disappeared for both samples (Fig. 4), which can be related to the volume change during quenching after intercritical annealing that induces plastic deformation of adjacent ferrite grains, and therefore, creates a high density of unpinned dislocations in the vicinity of martensite. These martensite-induced dislocations move at low stresses creating low yield strengths. They interact to produce high rates of strain hardening and prevent the occurrence of yield-point phenomenon [19].

By comparing the two DP steels in Fig. 4, it can be surmised that the FG-DP has higher tensile strength and total elongation compared to CG-DP steel. This reveals that this sample with finer microstructure has much better strength-ductility balance, which overcomes the conventional trend of data shown in Fig. 4 as a dotted line. This enhancement is mainly related to the improved work-hardening behavior as demonstrated in the following.

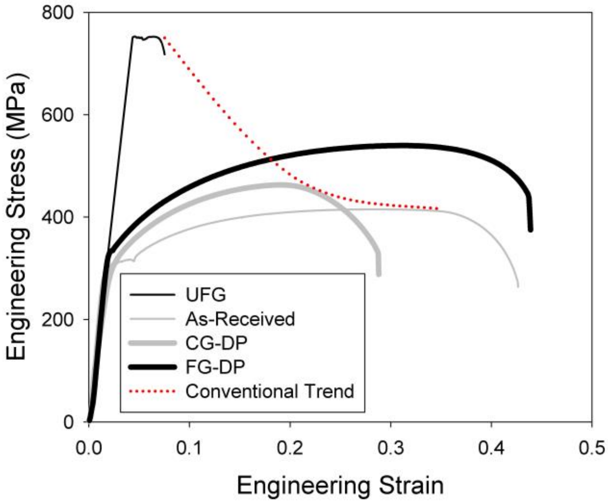

Fig. 4. Tensile stress-strain curves.

3.3. Work-hardening rate analysis

To calculate the instantaneous work-hardening exponents (n), the Hollomon equation was expressed as $\ln\sigma = \ln k + n\ln\varepsilon$, and at a given strain, the slope of the plot of $\ln\sigma$ versus $\ln\varepsilon$ was determined. So, $n = d\ln\sigma / d\ln\varepsilon$. To obtain the slope, the central difference approach based on $n_i = \{\ln\sigma_{i+1} - \ln\sigma_{i-1}\}/\{\ln\varepsilon_{i+1} - \ln\varepsilon_{i-1}\}$ was utilized. The results are shown in Fig. 5a, where it can be seen that the FG-DP steel has maintained its high n-values up to higher strains. Based on the Considére criterion, plastic instability of strain-rate-insensitive materials occurs when the strain-hardening rate coincides with the flow stress, i.e. $\sigma \geq d\sigma/d\varepsilon$ [17]. Therefore, maintaining high work-hardening exponents up to high strains is in favor of uniform elongation (and probably total elongation). The variation of work hardening rate ($d\sigma/d\varepsilon$) versus true strain is shown in Fig. 5b and reveals that the work hardening rates of FG-DP steel are generally higher than those of CG-DP steel.

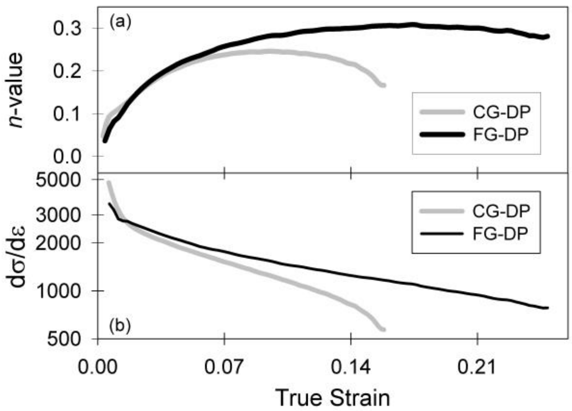

Fig. 5. Work-hardening rate analysis: (a) incremental *n*-values and (b) work-hardening rate.

Briefly, these results unveil the opportunities that fine-grained DP steels for structural applications can offer without changing the initial microstructure. Moreover, it can be surmised that cold working step is required before intercritical annealing to obtain DP steel sheets with required mechanical properties.

4. Conclusion

Grain refinement of DP steel via tempering of cold-rolled martensite was dealt with and the following conclusions can be drawn from this work:

(1) A microstructure consisting of ultrafine grained (UFG) ferrite with average grain size of ~ 0.7 µm and dispersed nano-sized carbides was produced by cold-rolling and tempering of the martensite starting microstructure. Subsequently, a fine grained dual phase (DP) steel consisting of equiaxed ferrite grains with average size of ~ 5 µm and martensite islands with average size of ~ 3 µm was produced by intercritical annealing of this microstructure.

(2) The fine grained DP steel had higher tensile strength and total elongation compared with the coarse grained one. These enhancements were related to the improved work-hardening behavior by microstructural refinement in ferritic-martensitic DP steel. The incremental work-hardening exponents (*n*-values) were used for these analyses, where the fine grained DP steel maintained its high *n*-values up to higher true strains.

5. References

[1] H. Azizi-Alizamini, M. Militzer, W.J. Poole, Formation of ultrafine grained dual phase steels through rapid heating, *ISIJ International* 51 (2011) 958-964.

[2] C.C. Tasan, M. Diehl, D. Yan, M. Bechtold, F. Roters, L. Schemmann, C. Zheng, N. Peranio, D. Ponge, M. Koyama, K. Tsuzaki, D. Raabe, An overview of dual-phase steels: Advances in microstructure-oriented processing and micromechanically guided design, *Annual Review of Materials Research* 45 (2015) 19.1-19.41.

[3] H. Mirzadeh, M. Alibeyki, M. Najafi, Unraveling the initial microstructure effects on mechanical properties and work-hardening capacity of dual phase steel, *Metallurgical and Materials Transactions A*, in press.

[4] N. Saeidi, M. Karimi, M.R. Toroghinejad, Development of a new dual phase steel with laminated microstructural morphology, *Materials Chemistry and Physics* 192 (2017) 1-7.

[5] Y. Okitsu, N. Takata, N. Tsuji, Ultrafine ferrite formation through cold-rolling and annealing of low-carbon dual-phase steel, *Materials Science and Technology* 31 (2015) 745-754.

[6] M. Mazinani, W.J. Poole, Effect of martensite plasticity on the deformation behavior of a low-carbon dual-phase steel, *Metallurgical and materials transactions A* 38 (2007) 328-339.

[7] R. Kuziak, R. Kawalla, S. Waengler, Advanced high strength steels for automotive industry, *Archives of Civil and Mechanical Engineering* 8 (2008) 103-117.

[8] M. Calcagnotto, D. Ponge and D. Raabe, Effect of grain refinement to 1 µm on strength and toughness of dual-phase steels, *Materials Science and Engineering A* 527 (2010) 7832-7840.

[9] Y. Mazaheri, A. Kermanpur, A. Najafizadeh, Strengthening Mechanisms of Ultrafine Grained Dual Phase Steels Developed by New Thermomechanical Processing, *ISIJ International* 55 (2015) 218-226.

[10] H. Ashrafi, M. Shamanian, R. Emadi, N. Saeidi, A novel and simple technique for development of dual phase steels with excellent ductility, *Materials Science and Engineering A* 680 (2017) 197-202.

[11] S.A. Etesami, M.H. Enayati, Microstructural Evolution and Recrystallization Kinetics of a Cold-Rolled, Ferrite-Martensite Structure During Intercritical Annealing, *Metallurgical and Materials Transactions A* 47 (2016) 3271-3276.

[12] R. Ueji, N. Tsuji, Y. Minamino, Y. Koizumi, Ultragrain refinement of plain low carbon steel by coldrolling and annealing of martensite, *Acta Materialia* 50 (2002) 4177-4189.

[13] M. Najafi, H. Mirzadeh, M. Alibeyki, Toward unraveling the mechanisms responsible for the formation of ultrafine grained microstructure during tempering of cold rolled martensite, *Materials Science and Engineering A* 670 (2016) 252-255.

[14] H. F. Lan, W. J. Liu, X. H. Liu, Ultrafine Ferrite Grains Produced by Tempering Cold-rolled Martensite in Low Carbon and Microalloyed Steels, *ISIJ International* 47 (2007) 1652-1657.

[15] S. Malekjani, I.B. Timokhina, I. Sabirov, P.D. Hodgson, Deformation Behaviour of Ultrafine Grained Steel Produced by Cold Rolling of Martensite, *Canadian Metallurgical Quarterly* 48 (2009) 229-236.

[16] B. Pawłowski, Critical points of hypoeutectoid steel - prediction of the pearlite dissolution finish temperature Ac1f, *Journal of Achievements in Materials and Manufacturing Engineering* 49 (2011) 331-337.

[17] G.E. Dieter: Mechanical Metallurgy, 3rd ed., McGraw-Hill, New York, 1988.

[18] R. Song, D. Ponge, D. Raabe, Improvement of the work hardening rate of ultrafine grained steels through second phase particles, *Scripta Materialia* 52 (2005) 1075-1080.

[19] G. Krauss, Steels Processing, Structure, and Performance, 2nd edition, ASM International, 2015.

ریزدانه سازی فولاد دوفازی توسط تمپر شدن مارتنزیت نورد سرد شده

مصطفی نجفی، حامد میرزاده*، محمد علی بیکی

دانشکده مهندسی متالورژی و مواد، دانشکده فنی، دانشگاه تهران، تهران، ایران

چکیده: ریزساختاری شامل فریت فوق ریزدانه با متوسط اندازه دانه 0/7 میکرومتر و ذرات نانومتری و پراکنده کاربید توسط نورد سرد و تمپر ساختار اولیه مارتنزیتی در یک فولاد کم کربن به دست آمد. در ادامه، فولاد دوفازی ریزدانه شامل دانه های هم محور فریت با متوسط اندازه دانه 5 میکرومتر و جزیره های مارتنزیتی با متوسط اندازه 3 میکرومتر توسط آنیل درون بحرانی این ریزساختار تولید شد. فولاد دوفازی درشت دانه با متوسط اندازه دانه فریت 20 میکرومتر و جزیره های مارتنزیتی با متوسط اندازه 5 میکرومتر نیز توسط آنیل درون بحرانی ریزساختار فریتی-پرلیتی به دست آمد. ساختار فوق ریزدانه، استحکام بالا، انعطاف پذیری پایین و قابلیت کارسخت شدن ضعیفی از خود نشان داد که به ریزدانه سازی شدید به دست آمده مرتبط شد. فولاد دوفازی ریزدانه استحکام و درصد ازدیاد طول بالاتری نسبت به فولاد دوفازی درشت دانه داشت که به بهبود رفتار کارسختی در اثر بهبود ریزساختار نسبت داده شد.

واژه های کلیدی: فولادهای دوفازی، ریزساختار، خواص مکانیکی، عملیات ترمومکانیکی.