Materials Science & Engineering A 575 (2013) 30–34

Contents lists available at SciVerse ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Achieving ultrafine dual-phase structure with superior mechanical property in friction stir processed plain low carbon steel

P. Xue, B.L. Xiao, W.G. Wang, Q. Zhang, D. Wang, Q.Z. Wang, Z.Y. Ma*

Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, 72 Wenhua Road, Shenyang 110016, China

ARTICLE INFO

Article history:
Received 23 January 2013
Received in revised form
13 March 2013
Accepted 16 March 2013
Available online 25 March 2013

Keywords:
Friction stir processing
Steel
Dual phases
Ultrafine grained microstructure

ABSTRACT

Ultrafine dual-phase structure was successfully prepared in a plain low carbon steel via friction stir processing (FSP) at a tool rotation rate of 400 rpm and a travel speed of 50 mm/min under rapid water cooling. Ultrafine polygonal ferrite grains (~1 μm) were distributed around the martensite phase consisting of nanostructured laths with a width of ~200 nm. FSP steel exhibited a good combination of ultrahigh yield strength of ~1100 MPa and large uniform elongation of ~7%. This work provides an effective strategy to prepare high strength low carbon steels.

1. Introduction

Grain refinement is an essential strengthening mechanism that enhances both strength and toughness of metal materials simultaneously. Therefore, ultrafine grained (UFG) steels with relatively simple chemical compositions have attracted much interest in replacing some conventional high strength alloyed steels [1]. In recent years, a variety of methods have been developed to produce UFG steels, and these methods can be divided into two categories: (i) advanced thermomechanical processing (ATMP) routes, which aim at improving conventional processing routes in commercial large-scale rolling mills and (ii) severe plastic deformation (SPD) techniques, which are essentially confined to laboratory-scale sample dimensions. Compared to the SPD techniques, the ATMP methods can be operated in various temperature ranges conveniently; thereby they are beneficial to exploiting phase transformation and controlled cooling. However, tender plastic deformation is usually achieved in the ATMP methods compared to the SPD techniques [1,2].

As is expected, the hardness and tensile strength of the UFG steels are drastically enhanced due to the structure refinement. However, the tensile ductility decreases dramatically, especially for the UFG steels prepared by the SPD techniques [1]. Furthermore, the UFG steels exhibit a very low strain hardening rate, which marks the main limitation with respect to commercial applications. Therefore, it is of ongoing interest to overcome the restricted ductility of the UFG steels without sacrificing the strength. One possible way to restore the strain hardenability of the UFG steels is the introduction of layered structure, such as twin boundaries, martensite laths, in the ultrafine structure [1–3]. Based on this principle, UFG ferrite/martensite dual-phase (DP) steels with higher mechanical properties than conventional coarse grained (CG) DP steels have been designed [4,5]. However, the strength, especially the yield strength (YS), of these UFG DP steels prepared by the ATMP methods is much lower than that of the UFG steels prepared by the SPD techniques [1].

Recently, a new processing technique–friction stir processing (FSP) – was developed for microstructral modification, based on the basic principles of friction stir welding (FSW) [6]. During this process, the material in the processed zone (PZ) undergoes intense plastic deformation at elevated temperatures, resulting in significant grain refinement [6,7]. Recently, FSP has been successfully used to prepare bulk UFG materials, such as aluminum, copper and magnesium alloys [8–11]. After FSP, the microstructure of the PZ is characterized by equiaxed dynamically recrystallized grains with a large fraction of high angle grain boundaries (HAGBs, misorientation angle $>15^{\circ}$) [6–11]. FSP caused more intense plastic deformation than the ATMP process, and the processing temperature and the cooling rate can be controlled during the FSP process. In principle, FSP is able to prepare UFG steels, and the grain size can be further refined compared to that obtained by the ATMP process. Unfortunately, there are still no studies on preparing the UFG steels via FSP, though steels are major structural materials nowadays.

A thorny problem that restricted the wide application in FSW/P of steels is the lack of suitable tool materials [6,12–14]. Usually, the

* Corresponding author. Tel./fax: +86 24 83978908.
E-mail address: zyma@imr.ac.cn (Z.Y. Ma).

0921-5093/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.msea.2013.03.033

stirring tool materials for steels are expensive polycrystalline cubic boron nitride (PCBN) and tungsten-based alloys, and this is obviously not beneficial to the industrial application. However, the chosen scope of the tool materials can be enlarged if the processing temperature during FSW/P is reduced to a value which can adequately ensure the plastic flow of steels. Previous studies have proved that besides changing the rotation rate and the traverse speed, employing additional rapid cooling is an effective method of reducing the processing temperature [8–11,15]. More importantly, UFG microstructure can be obtained in FSP steels in this case if suitable parameters are chosen.

In this study, plain low carbon steel plate was subjected to FSP using the low-cost stirring tool under the additional rapid cooling. The aim is to investigate if UFG steel with a good combination of strength and ductility can be achieved by an FSP route.

2. Experimental procedures

A tool with a shoulder 14 mm in diameter without stirring pin was machined in order to avoid the pollution in the PZ. The whole tool was made of common tool steel except the end of shoulder part which was made of TiC-based cermet, so the cost of the tool is very low. Hot rolled plain low carbon steel plate 5 mm in thickness was used, and the chemical composition is shown in Table 1. FSP was conducted at a low heat input parameter with a tool rotation rate of 400 rpm and a traverse speed of 50 mm/min. In order to obtain a very low heat input and a rapid cooling rate, the steel plates were first fixed in water and additional rapid cooling with flowing water was used during the FSP process, and the detailed parameters about water cooling have been described in our previous study [15]. For comparison, quenched martensite steel was also prepared in this study. The same plain low carbon steel plates were first held at 950 °C for 1200 s, and then quenched quickly in the water.

Microstructural characterization and analysis were carried out by optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The specimens for OM and SEM were cross-sectioned perpendicular to the processing direction, polished and then etched in 5% nital for 5 s. SEM observation was carried out on a FEI Quanta 600 SEM and a LEO Supra 35 FEG SEM operating at 20 kV. TEM specimens were prepared by means of double-jet electrolytic polishing in an electrolyte consisting of 100 ml perchloric acid and 900 ml alcohol. TEM observation was carried out on a FEI Tecnai G^2 20 microscope operating at 200 kV.

The Vickers microhardness test was performed using a 200 g load for 10 s. Tensile specimens of 5 mm gauge length, 1 mm gauge width and 0.6 mm gauge thickness were machined from the PZ perpendicular to the FSP direction, and the gauge section is shown in Fig. 1 by the rectangle. Meanwhile, tensile specimens with the same scales were machined from the parent material (PM) and as-quenched samples. The tensile tests were carried out at an initial strain rate of 1×10^{-3} s^{-1}.

Table 1
Chemical composition of the plain low carbon steel (wt%).

C	Mn	Si	P	S	Fe
0.17	1.30	0.35	0.017	0.018	Bal.

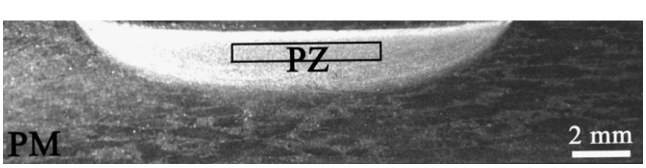

Fig. 1. Cross-sectional macrograph of the friction stir processed sample. The rectangular region shows the gauge area of the tensile specimen (PM: parent material, PZ: processed zone).

3. Results and discussion

The plain low carbon steel was successfully friction stir processed in this study, and no obvious tool abrasion was observed. Though no stirring pin was used, an obvious PZ was achieved in the FSP plain low carbon steel, as shown in Fig. 1. Under the effect of the shoulder, the width of the PZ was comparable to the shoulder diameter. More importantly, the depth of the PZ was as large as 2 mm, which reached the similar effect obtained using the tool with pin [16,17]. Except for a very thin layer (~100 μm) with ultrafine equiaxed grains at the surface, relatively uniform microstructure was achieved in the upper part of the PZ with a thickness of ~1.5 mm. Microstructure and mechanical properties of the material in this area were investigated in this study, and “the FSP steel” indicates this area hereafter. The initial PM exhibited a typical hot-rolled microstructure, which consisted of coarse equiaxed ferrite grains and pearlite grains, as shown in Fig. 2a. However, the grains were greatly refined in the FSP steel (Fig. 2b), and it was difficult to discern the exact microstructure under OM.

Fig. 3 shows the detailed microstructure of the FSP steel under SEM and TEM. It is clear that the FSP steel exhibited ultrafine DP structure, which was composed of very fine polygonal ferrite (~1 μm) and relatively coarse martensite phases (Fig. 3a). From the TEM bright field (BF) image shown in Fig. 3b, the martensite phase was identified as lath martensite with a lath width of ~200 nm. Low dislocation density was achieved in the ferrite grains; however, high density of dislocations existed in the nanostructured martensite laths.

Clearly, solid phase transformation of austenite to martensite occurred in the FSP steel during the FSP process, indicating that the processing temperature was above Ae_1. Moreover, the obvious DP structure in the FSP steel indicated that the processing temperature should be lower than Ae_3, i.e., within a (γ+α) 2-phase field [18]. Previous studies have proved that the microstructure could be refined effectively by combining phase transformation and plastic deformation in the 2-phase field [2,18,19]. It is suggested that during the FSP process, part of the original ferrite phase transformed to austenite phase, and then transformed into martensite phase during subsequent rapid water cooling. Furthermore, dynamic recrystallization of austenite phase occurred during the FSP process due to the intense plastic deformation, resulting in the refined martensite phase.

The formation mechanism of the ultrafine ferrite phase, however, should be very complex during the FSP process. It is proposed that the original ferrite phase (untransformed part) was refined by dynamic recrystallization during FSP. Simultaneously, ultrafine ferrite grains could be also obtained from the transformation of the austenite phase in the 2-phase field during deformation, and one possible mechanism is strain-induced dynamic transformation (SIDT) [18,20]. Ok and Park [19] indicated that continuous dynamic recrystallization of transformed massive ferrite phase also occurred in the ferrite grain refinement process. Anyhow, the intense plastic deformation within the 2-phase field during FSP obviously enhanced the refinement of the ferrite phase by SIDT and/or dynamic recrystallization.

Table 2 summarizes the mechanical properties of the PM, as-quenched and the FSP steels, and the typical tensile curves are shown in Fig. 4. The hardness value of the PM was about 150 Hv;

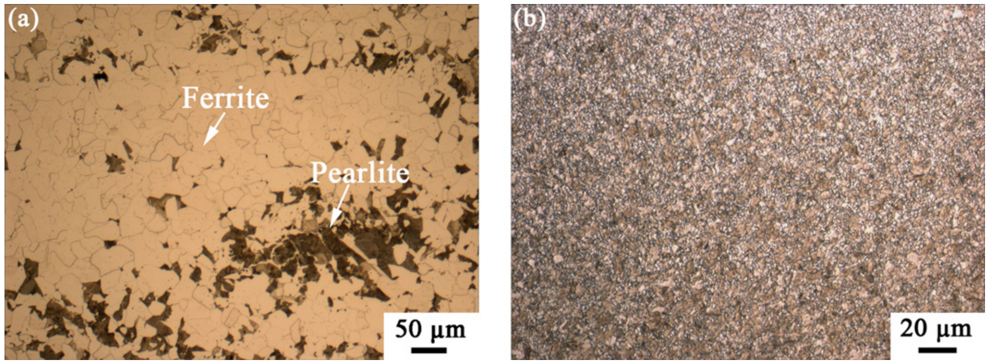

Fig. 2. Optical microstructure of (a) parent material and (b) friction stir processed steel.

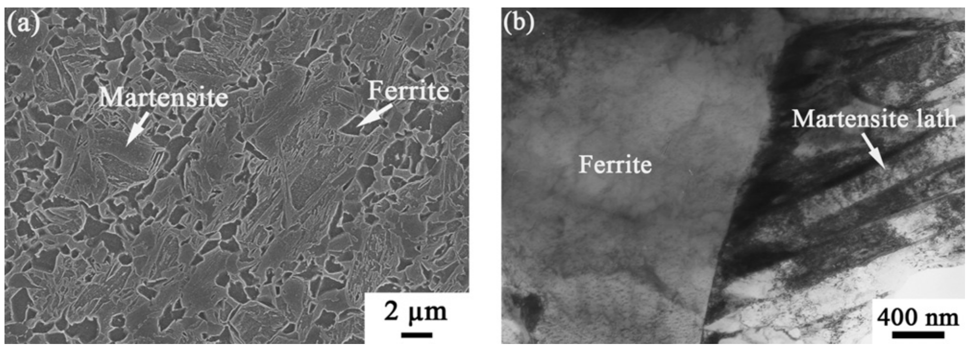

Fig. 3. Microstructure of friction stir processed steel: (a) SEM and (b) TEM bright field images.

Table 2
Mechanical properties of the PM, as-quenched and FSP steel samples.

Sample	Hardness (Hv)	YS (MPa)	UTS (MPa)	Elongation (%)
PM	150 ± 5	350 ± 10	520 ± 5	24 ± 2
As-quenched	425 ± 10	1100 ± 100	1150 ± 150	1.0 ± 0.2
FSP	410 ± 10	1100 ± 50	1350 ± 50	11 ± 1

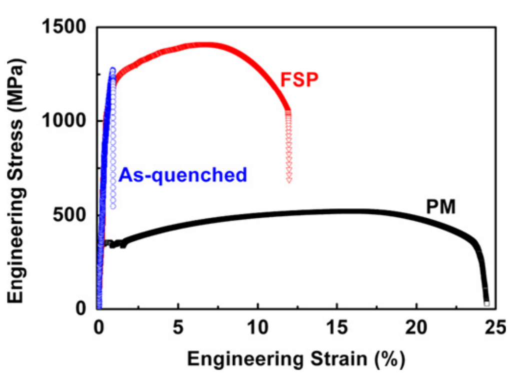

Fig. 4. Typical tensile curves of the parent material (PM), as-quenched and friction stir processed (FSP) steels.

however, very high hardness values of about 425 and 410 Hv were achieved in the as-quenched and FSP steels, respectively. From the tensile curves in Fig. 4, the PM exhibited an obvious yield point elongation with a YS of ~350 MPa and an ultimate tensile strength (UTS) of ~525 MPa. For the as-quenched steel, very high YS of ~1200 MPa was achieved, but, the sample tended to fail very quickly once the yield stress was reached and even before yielding in other tests, resulting in extremely low ductility. Compared to the PM and as-quenched steel, obvious continuous yielding was observed from the tensile curve of the FSP steel. Further, very high

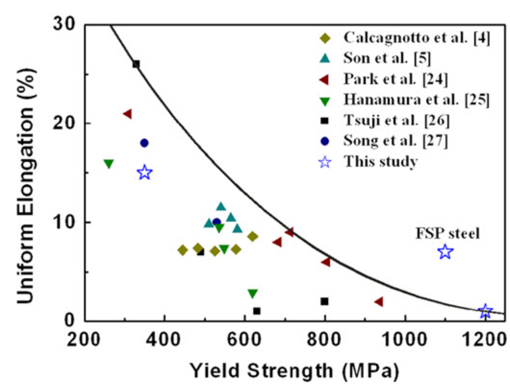

Fig. 5. Uniform elongation and yield strength of the low carbon steels prepared by severe plastic deformation and advanced thermomechanical processing methods [4,5,24–27], and by friction stir processing (FSP) in this study.

YS of ~1100 MPa and UTS of ~1400 MPa were obtained in the FSP steel. More importantly, the FSP steel exhibited obvious strain hardening during the tensile test with a uniform elongation (UE) of ~7%, which was higher than that of the utilizable UE in industrial application (5%).

Strength and ductility are the key mechanical properties of steels, but they are typically opposing characteristics. In recent years, various advanced steels, such as twining-induced plasticity (TWIP) steels, transformation induced plasticity (TRIP) steels, and also DP steels have received high interest due to their abilities in enhancing strain hardening [4,5,21–23]. UFG steels prepared by the SPD and ATMP techniques can reach a very high UTS of ~1000 MPa. However, these UFG steels usually exhibit very weak or no strain hardening during tensile tests, resulting in very low UE which was usually <2% [1,24–26]. On the other hand, conventional UFG DP steels exhibit a low YS of ~600 MPa, though a relatively high UE of ~10% can be achieved [1,4,5].

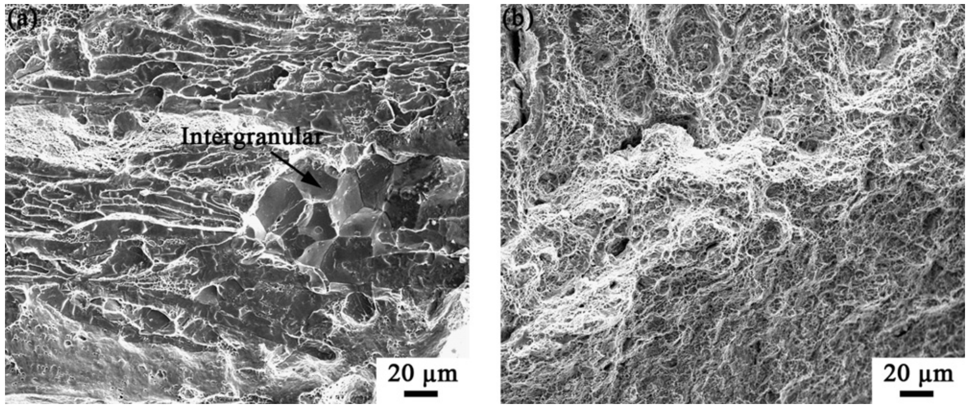

Fig. 6. SEM microstructure of fracture surfaces of (a) as-quenched and (b) friction stir processed steels.

Fig. 5 summarizes the experimental data on UE versus YS for various low carbon steels prepared by the SPD and ATMP methods [4,5,24–27]. It is clear that all the results obey the high strength–low ductility rule, but the FSP steel in this study exhibited a sound combination of strength and ductility. Compared to the UFG DP steels with similar composition prepared by Calcagnotto et al. [4], similar UE can be obtained in the FSP steel. However, the ultrahigh YS of ~1100 MPa was achieved, which was larger than that obtained in most UFG low carbon steels [1,24–26].

The sound mechanical properties of the FSP steel should be related to the special ultrafine DP structure, and can be attributed to the following factors. First, ultrafine ferrite and martensite phases were obtained in the FSP steel, and the martensite phase was composed of abundant laths with a width of ~200 nm. It is well accepted that the ultrafine ferrite and martensite phases together with the nanostructured martensite laths, and the solute C atoms in the martensite phase caused the ultrahigh hardness and strength [1,2].

Second, very low dislocation density was obtained in the ferrite phase, and sufficient room for dislocations also existed in the martensite laths though relatively higher dislocation density was achieved. Therefore, dislocations can be accumulated in the ferrite phase and the martensite laths during the tensile test, which resulted in the enhanced strain hardening.

Third, compared to the as-quenched martensite steel, the martensite phase was greatly refined in the FSP steel. Moreover, the martensite grains were surrounded by the ductile ferrite phase with a grain size of ~1 μm. This special structure inhibited the propagation of the large scale cracks along the grain boundaries, martensite laths or blocks, and it can be observed from the SEM fractographs.

For the as-quenched sample, brittle intergranular fracture easily to occur during tension (Fig. 6a). However, obvious dimples were observed on the fracture surface of the FSP steel (Fig. 6b), indicating good ductile fracture. Wang et al. [28] have also proved that the cleavage crack could be arrested in the martensite phase with refined grains and then the propagation direction was changed. Therefore, brittle fracture observed in as-quenched martensite sample was eliminated in the FSP steel, so good ductility with UE of ~7% could be achieved.

Clearly, FSP with additional rapid water cooling is an effective method of producing ultrafine DP steels with superior mechanical properties. In this study, combined intense plastic deformation and phase transformation were achieved during the FSP process, resulting in the ultrafine DP structure in the FSP steel. Considering that FSP can prepare large area fine-grained materials via multi-pass overlapping [6,29,30], and low-cost tool material can act well in FSP of steels if appropriate temperature is chosen, this study undoubtedly provides an effective processing method for preparing large area UFG steel plates, especially for the thin plates. Meanwhile, this method should be more effective in the surface treatment of the thick steel plates. Moreover, based on the same principle of FSW and FSP, this study also provides a feasible method of joining advanced high strength UFG low carbon steels.

4. Conclusions

In summary, the following conclusions are reached:

1. Plain low carbon steel was successfully friction stir processed at a tool rotation rate of 400 rpm and a travel speed of 50 mm/min with additional rapid water cooling using a low-cost cermet tool without pin, and an obvious PZ with a depth of about 2 mm was obtained.
2. The FSP steel was characterized by ultrafine ferrite/martensite DP structure. The ultrafine polygonal ferrite grains (~1 μm) were distributed around the martensite phase which consisted of nanostructured laths with a width of ~200 nm.
3. The FSP steel exhibited a good combination of strength and ductility, and an ultrahigh YS of ~1100 MPa and a UE of ~7% was achieved.

Acknowledgments

This work was supported by the National Natural Science Foundation of China under Grant nos. 50890171 and 51071150, and the National Basic Research Program of China under Grant no. 2011CB606301.

References

[1] R. Song, D. Ponge, D. Raabe, J.G. Speer, D.K. Matlock, Mater. Sci. Eng. A 441 (2006) 1–17.
[2] N. Tsuji, T. Maki, Scr. Mater. 60 (2009) 1044–1049.
[3] G. Frommeyer, U. Brüx, P. Neumann, ISIJ Int. 43 (2003) 438–446.
[4] M. Calcagnotto, Y. Adachi, D. Ponge, D. Raabe, Acta Mater. 59 (2011) 658–670.
[5] Y.I. Son, Y.K. Lee, K.T. Park, C.S. Lee, D.H. Shin, Acta Mater. 53 (2005) 3125–3134.
[6] R.S. Mishra, Z.Y. Ma, Mater. Sci. Eng. R 50 (2005) 1–78.
[7] R.S. Mishra, M.W. Mahoney, S.X. McFadden, N.A. Mara, A.K. Mukherjee, Scr. Mater. 42 (1999) 163–168.
[8] J.Q. Su, T.W. Nelson, C.J. Sterling, Scr. Mater. 52 (2005) 135–140.
[9] C.I. Chang, X.H. Du, J.C. Huang, Scr. Mater. 57 (2007) 209–212.
[10] P. Xue, B.L. Xiao, Z.Y. Ma, Mater. Sci. Eng. A 532 (2012) 106–110.
[11] P. Xue, B.L. Xiao, Z.Y. Ma, Scr. Mater. 68 (2013) 751–754.
[12] H.K.D.H. Bhadeshia, T. DebRoy, Sci. Technol. Weld. Joining 14 (2009) 193–196.
[13] R. Rai, A. De, H.K.D.H. Bhadeshia, T. DebRoy, Sci. Technol. Weld. Joining 16 (2011) 325–342.
[14] R. Nandan, T. DebRoy, H.K.D.H. Bhadeshia, Prog. Mater. Sci. 53 (2008) 980–1023.

[15] P. Xue, B.L. Xiao, Q. Zhang, Z.Y. Ma, Scr. Mater. 64 (2011) 1051–1054.
[16] Y.S. Sato, H. Yamanoi, H. Kokawa, T. Furuhara, Scr. Mater. 57 (2007) 557–560.
[17] H. Fujii, L. Cui, N. Tsuji, M. Maeda, K. Nakata, K. Nogi, Mater. Sci. Eng. A 429 (2006) 50–57.
[18] J.K. Choi, D.H. Seo, J.S. Lee, K.K. Um, W.Y. Choo, ISIJ Int. 43 (2003) 746–754.
[19] S.Y. Ok, J.K. Park, Scr. Mater. 52 (2005) 1111–1116.
[20] P.D. Hodgson, M.R. Hickson, R.K. Gibbs, Scr. Mater. 40 (1999) 1179–1184.
[21] I. Gutierrez-Urrutia, S. Zaefferer, D. Raabe, Mater. Sci. Eng. A 527 (2010) 3552–3560.
[22] I. Gutierrez-Urrutia, D. Raabe, Acta Mater. 60 (2012) 5791–5802.
[23] H. Ding, H Ding, D. Song, Z.Y. Tang, P. Yang, Mater. Sci. Eng. A 528 (2011) 868–873.
[24] K.T. Park, Y.S. Kim, J.G. Lee, D.H. Shin, Mater. Sci. Eng. A 293 (2000) 165–172.
[25] T. Hanamura, F. Yin, K. Nagai, ISIJ Int. 44 (2004) 610–617.
[26] N. Tsuji, Y. Ito, Y. Satio, Y. Minarmino, Scr. Mater. 47 (2002) 893–899.
[27] R. Song, D. Ponge, D. Raabe, Acta Mater. 53 (2005) 4881–4892.
[28] C.F. Wang, M.Q. Wang, J. Shi, W.J. Hui, H. Dong, Scr. Mater. 58 (2008) 492–495.
[29] J.Q. Su, T.W. Nelson, C.J. Sterling, Scr. Mater. 52 (2005) 135–140.
[30] Z.Y. Ma, S.R. Sharma, R.S. Mishra, Scr. Mater. 54 (2006) 1623–1626.