

## Fabrication of Ultrafine Grained Steels without Severe or Heavy Plastic Deformation

Nobuhiro Tsuji, Rintaro Ueji and Yoritoshi Minamino

Department of Adaptive Machine Systems, Graduate School of Engineering, Osaka University.

2-1 Yamadaoka, Suita, 565-0871, Japan.

Fax: 81-6-6879-7434, e-mail: tsuji@ams.eng.osaka-u.ac.jp

Ultrafine grained low-carbon steel was fabricated by a new and simple process named the martensite process. The key of the martensite process is to start from martensite structure in the thermomechanical processing. The 0.13C steel specimen cold-rolled only by 50% from the martensite starting structure showed the lamellar boundary structure which have been typically observed in heavily deformed materials. The interval of the lamellar boundaries was much smaller than that formed by severe plastic deformation up to 4.0 of strain. Warm temperature annealing turned the deformation structure into nearly equiaxed ultrafine grains with mean diameter of 180nm. Because martensite is a supersaturated solid solution of carbon, a number of fine carbides precipitated uniformly within the matrix during annealing. The fine and uniformly distributed carbides played an important role to realize the good mechanical properties of the UFG material having both high strength and adequate ductility.

Key words: low carbon steel, martensite, thermomechanical process, accumulative roll bonding, mechanical properties

### 1. INTRODUCTION

Ultrafine grained (UFG) steels whose mean grain sizes are smaller than  $1\mu\text{m}$  have been energetically studied in recent years, because they are expected to perform superior mechanical properties [1,2]. The UFG bulk steels can be fabricated by either severe plastic deformation (SPD) [1,3] or thermomechanically controlled processing (TMCP) under extreme conditions [1,2]. However, the SPD processes need large plastic strain and the ultimate TMCP also requires one-pass heavy rolling at relatively low temperature, for which huge amount of plastic working energy and special equipment with large capacity are necessary. On the other hand, the present authors recently developed a new and simple way to obtain UFG low-carbon steels without severe or heavy plastic deformation, as is illustrated in Fig.1 [4,5]. The key of the new process, named *the martensite process*, is to use martensite as a starting structure of TMCP. The purpose of the present study is to compare the microstructures and the mechanical properties of the UFG steels fabricated by

two different methods, i.e., the SPD and the martensite process.

### 2. EXPERIMENTAL

A plain low-carbon steel (JIS-SS400: 0.13C-0.0043N-0.01Si-0.37Mn-0.020P, wt%) was used in this study. Two different starting microstructures were prepared for two different processes. The hot-rolled sheets 2mm in thickness having ferrite-pearlite structure were provided for the SPD process. As SPD, the accumulative roll-bonding (ARB) [3,6,7] was conducted. For the ferrite-pearlite starting structure, the ARB at room temperature without lubrication was carried out up to 4.0 of total equivalent strain ( $\epsilon$ ) by the use of the same facilities reported previously [3,8]. The ARB processed sheets were annealed at various temperatures ranging from  $300^\circ\text{C}$  to  $700^\circ\text{C}$  for 1.8ks. The other starting microstructure was martensite. The hot-rolled sheets of SS400 were austenitized at  $1000^\circ\text{C}$  for 1.8 ks in Argon + 10vol% Hydrogen atmosphere, and then water-quenched immediately. The quenched sheets with lath-martensite structure were cold-rolled by 50% reduction in thickness ( $\epsilon=0.8$ ) at ambient temperature. The rolling was carried out in three passes with machine-oil as lubricant, using a two-high mill having a roll-diameter of 310 mm. The cold-rolled sheets were annealed at various temperatures from  $300^\circ\text{C}$  to  $700^\circ\text{C}$  for 1.8ks. Whole these procedures are named the martensite process. Microstructures of the specimens obtained by the ARB and the martensite process were characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Mechanical properties of the specimens were measured by tensile test at ambient temperature.

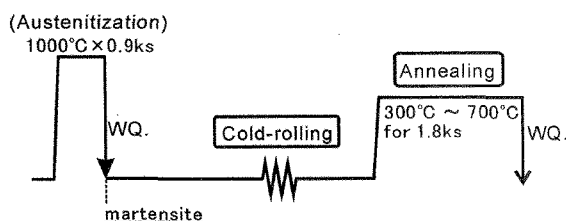


Fig.1 Thermomechanical history in the martensite process.

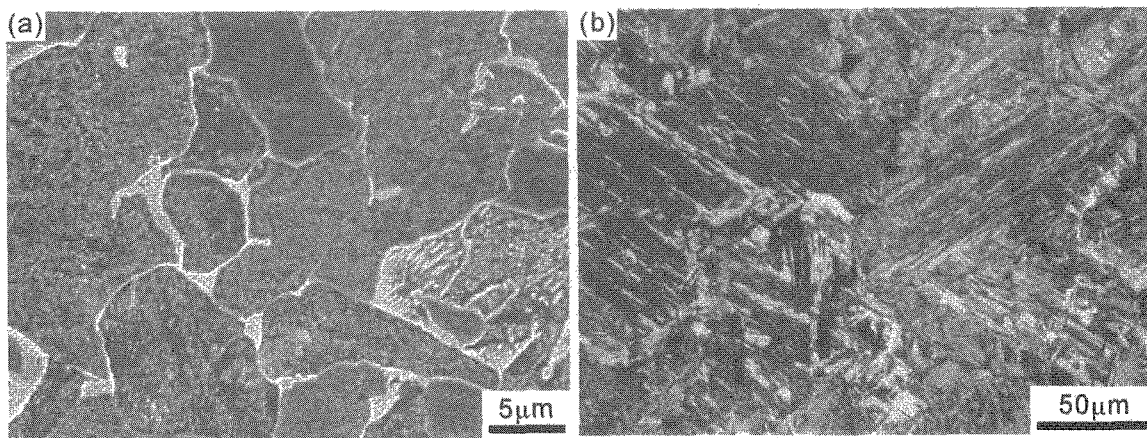


Fig.2 Micrographs of two different starting microstructures of the SS400 steel. (a) SEM microstructure of ferrite-pearlite for the ARB. (b) Optical microstructure of Lath martensite for the martensite process.

### 3. RESULTS AND DISCUSSION

Figure 2 shows the SEM micrographs of the two different starting microstructures of the SS400 steel. Ferrite-pearlite structure in Fig.2 (a) was provided for the SPD by the ARB process. The microstructure is mostly composed of equiaxed ferrite grains whose mean diameter is 5.4 $\mu$ m. The fraction of pearlite is not so much in this low-carbon steel. The quenched sheet for

the martensite process was filled with typical lath martensite structure shown in Fig.2 (b). The martensite of the low-carbon steel was quite ductile and there was no problem in subsequent cold-rolling.

TEM microstructures of the SS400 specimens after rolling deformation are shown in Fig.3. The microstructures were observed from the transverse direction (TD) of the sheets. The ferrite-pearlite specimen severely deformed up to 4.0 of strain by the ARB showed the lamellar boundary structure elongated along the rolling direction (TD) as shown in Fig.3 (a). This is the typical deformation microstructure which has been observed in heavily rolled metallic materials [9] and in the ARB processed materials [1,3]. The average interval of the lamellar boundaries was 110nm. The selected area electron diffraction pattern was not spotty but ring-like, indicating that large misorientations exist in the lamellar boundary structure. The 50% cold-rolled specimen which had martensite starting structure also showed a lamellar boundary structure having large misorientation in most parts, though there were another kinds of deformation structures in part of the specimen [5]. The mean interval of the lamellar boundary was 60nm, which is much smaller than that of the ARB processed specimen (Fig.3 (a)). It should be noted that in the martensite process the finer structure was formed by much smaller strain ( $\epsilon=0.8$ ) than in the ARB ( $\epsilon=4.0$ ).

Annealing the severely deformed specimen turns the lamellar boundary structure into the UFG structure by the aid of recovery [1,3,10,11]. The TEM microstructure of the ARB processed specimen after annealing at 540°C for 1.8ks is shown in Fig.4. Most of the lamellar boundary structure turned into the pancake-shaped UFGs whose diameters are smaller than 1 $\mu$ m. At the same time, however, several coarse grains having grain size over a few microns are observed in Fig.4. Such a heterogeneous grain structure has not been observed in the ARB processed and annealed interstitial free (IF) steel having ferrite single phase. It is noteworthy that there are a number of fine spheroidized particles of cementite derived from the initial pearlite structure in the UFG regions while

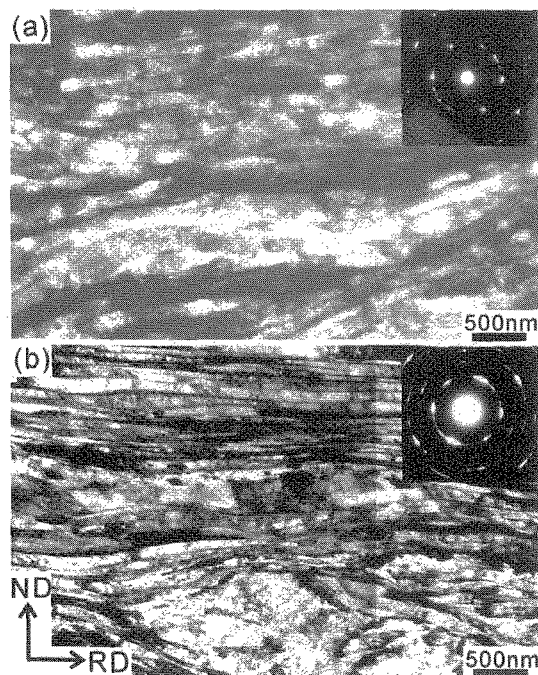


Fig.3 TEM microstructures and corresponding electron diffraction patterns of (a) the ferrite pearlite specimen ARB processed up to 4.0 of strain at room temperature and (b) the martensite specimen cold-rolled up to 0.8 of strain (50% reduction). Observed from TD.

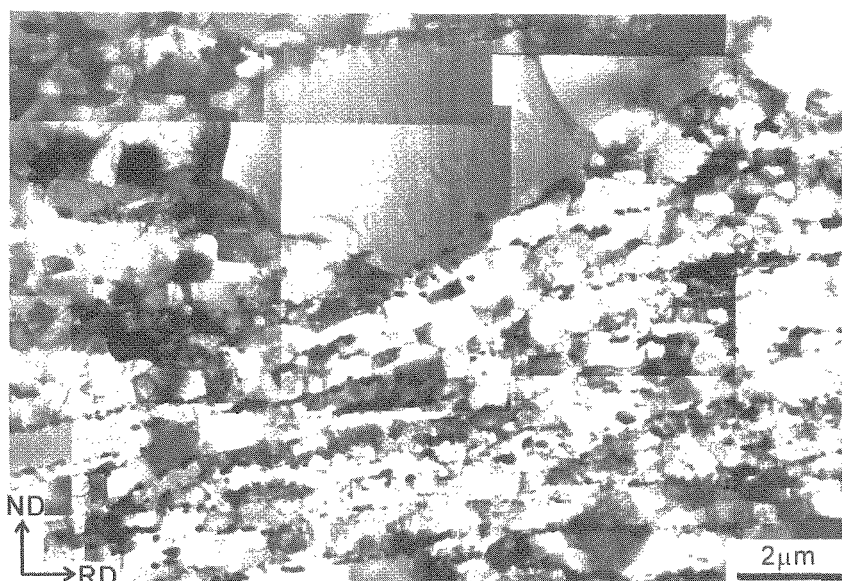


Fig.4 TEM microstructure of the ferrite-pearlite specimen ARB processed up to 4.0 of strain and then annealed at 540°C for 1.8 ks. Observed from TD.

cementite is not seen around the coarse grains. It can be concluded, therefore, that the inhomogeneous distribution of cementite, which can act as pinning particles to inhibit grain growth during annealing, in the ferrite-pearlite starting structure resulted in heterogeneous grain structure in the ARB and annealed specimens.

Figure 5 is a TEM micrograph of the martensite-processed SS400 specimen after annealing at 500°C for 1.8 ks. Most of the regions showed nearly equiaxed UFGs whose mean diameter is 180nm, though tempered martensite having block-like morphology was partly observed in the specimen. Because martensite is a supersaturated solid solution of carbon, fine cementite precipitated uniformly within the UFG structure during the annealing. The fine and uniformly distributed precipitates are effective to stabilize the UFG structure by inhibiting grain growth [12].

Nominal stress-strain curves of the SS400 specimens processed in two different ways are shown in Fig.6. The ARB processed specimen severely deformed up to 4.0 of strain showed 1027MPa of tensile strength, which is 2.5 times higher than that of the starting material having ferrite-pearlite structure (Fig.6 (a)). However, the flow stress reached its maximum at very early stage of the tensile test followed by macroscopic necking, so that the uniform elongation was limited within a few percents. The strength decreased with increasing the annealing temperature, but the uniform elongation recovered only after the strength significantly dropped below 500MPa. It has been clarified by Tsuji et al. [10] that the limited uniform elongation in the UFG IF steel having ferrite single phase is explained by early plastic instability caused by the lack of work-hardening. It has been also expected that to enhance work-hardening by any means, such as uniform distribution of fine second phase particles, would improve the ductility even in the UFG structures.

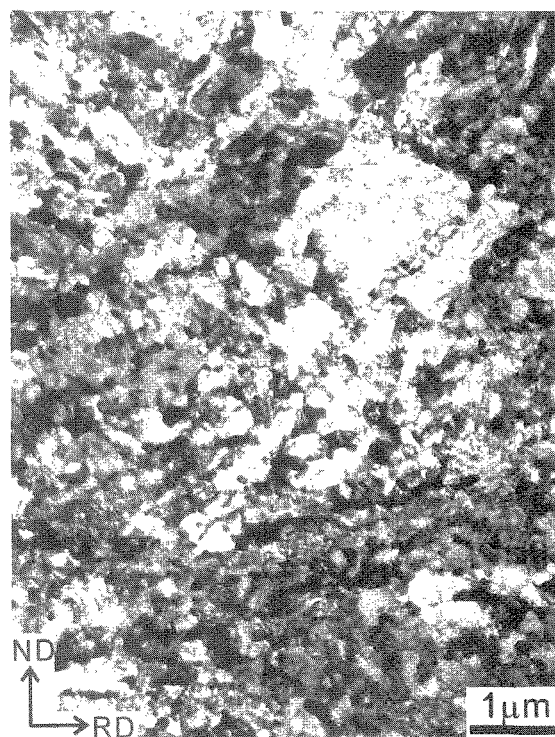


Fig.5 TEM microstructure of the SS400 steel cold-rolled by 50% reduction ( $\epsilon=0.8$ ) and annealed at 500°C for 1.8 ks. Starting microstructure was martensite. Observed from TD.

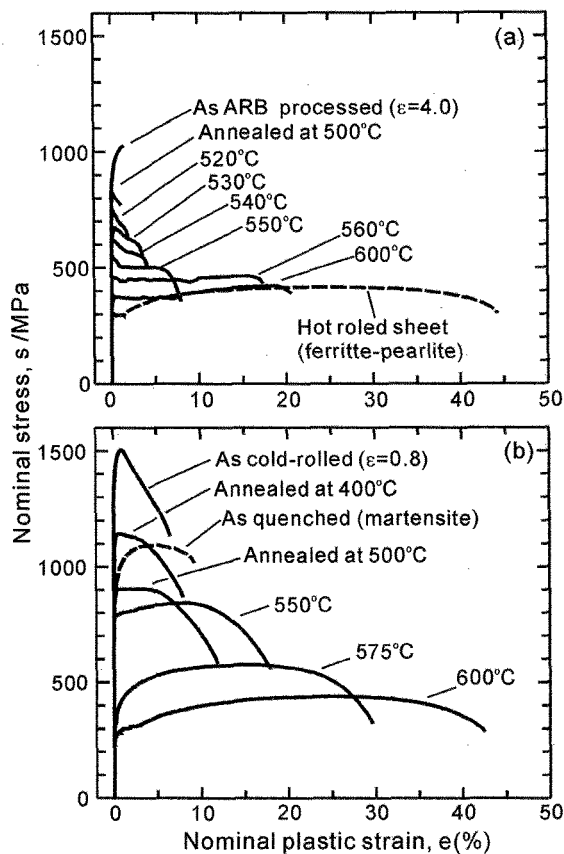


Fig.6 Nominal stress-strain curves of the SS400 specimens. (a) The ferrite-pearlite specimens ARB processed up to 4.0 of strain at room temperature and annealed at various temperatures for 1.8ks. (b) The martensite specimen cold-rolled up to 0.8 of strain (50% reduction) and annealed at various temperatures.

Though the present low-carbon steel includes a certain portion of carbide, the carbide concentrated within the pearlite region in the initial specimens for the ARB, as was shown in Fig.2 (a) and Fig.4. Consequently, the ARB and annealed specimens showed similar tensile behavior to that of the IF steel having ferrite single phase [10]. The 50% cold-rolled martensite showed very high strength above 1.5GPa (Fig.6 (b)). However, it showed limited uniform elongation same as the ARB processed specimens. However, the martensite processed specimens showed good strength-ductility balance after warm temperature annealing at around 500°C, where the relatively uniform UFG structure formed (Fig.5). For example, the 550°C annealed specimen showed 870MPa of tensile strength together with 9% of uniform elongation and 20% of total elongation. This is completely different from the mechanical property of the ARB processed specimen started from ferrite-pearlite structure. The adequate ductility in the martensite-processed specimens is probably attributed to the enough work-hardening enhanced by fine carbides uniformly precipitated during annealing (Fig.6) [12]. That is, the martensite process is a simple and promising method to fabricate the multiphased UFG steels having

excellent mechanical properties.

#### 4. CONCLUSION

The microstructure and mechanical properties of the ultrafine grained 0.13C steel fabricated by the martensite process were compared with those of the ARB processed specimens. The martensite starting structure could easily fabricate the ultrafine deformation structure usually obtained only after severe deformation. Because martensite is a supersaturated solid solution of carbon, fine carbide precipitated uniformly within the matrix during annealing. The fine and uniformly distributed carbide played important roles not only to stabilize the ultrafine grained structure but also to enhance work-hardening to realize superior strength-ductility balance. It could be concluded that the martensite process is a promising method to fabricate advanced low-carbon steels with the multiphased nano-structure performing excellent mechanical properties.

#### ACKNOWLEDGMENT

This study was financially supported by the Grant-in-Aid for Young Scientists (A) under contract No.14702052 and the Center of Excellence for Advanced Structural and Functional Materials Design in Osaka University as the 21st Century COE program, both through the Ministry of Education, Sports, Culture, Science & Technology of Japan, which are gratefully appreciated by the authors.

#### REFERENCES

- [1] N.Tsuji, *Tetsu-to-Hagane* (J. of Iron and Steel Inst. Jpn.), 88, 359-369 (2002).
- [2] M.Niikura, *Bull. of Iron and Steel Inst. Jpn.*, 8, 718-724 (2003).
- [3] N.Tsuji, Y.Saito, S.H.Lee and Y.Minamino, *Advanced Eng. Mater.*, 5, 338-344 (2003).
- [4] N.Tsuji, R.Ueji, Y.Minamino and Y.Saito, *Scripta Mater.*, 46, 305-310 (2002).
- [5] R.Ueji, N.Tsuji, Y.Minamino and Y.Koizumi, *Acta Mater.*, 50, 4177-4189 (2002).
- [6] Y.Saito, N.Tsuji, H.Utsunomiya, T.Sakai and R.G.Hong, *Scripta Mater.*, 39, 1221-1227 (1998).
- [7] Y.Saito, H.Utsunomiya, N.Tsuji and T.Sakai, *Acta Mater.*, 47, 579-583 (1999).
- [8] N.Tsuji, Y.Saito, H.Utsunomiya and S.Tanigawa, *Scripta Mater.*, 40, 795-800 (1999).
- [9] N.Hansen and D.Juul Jensen, *Phil. Trans. R. Soc. Lond. A*, 357, 1447-1469 (1999).
- [10] N.Tsuji, Y.Ito, Y.Saito and Y.Minamino, *Scripta Mater.*, 47, 893-899 (2002).
- [11] N.Tsuji, R.Ueji, Y.Ito and Y.Saito, *Proc. of the 21st RISØ Int. Symp. on Mater. Sci.*, RISØ National Laboratory, Roskilde, Denmark, 607-616 (2000).
- [12] R.Ueji, N.Tsuji, Y.Minamino and Y.Koizumi, *Sci. Tech. Adv. Mater.*, 4 (2003), in press.

(Received December 1, 2003; Accepted June 23, 2004)