Effect of rolling reduction on ultrafine grained structure and mechanical properties of low-carbon steel thermomechanically processed from martensite starting structure

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Abstract

The present authors have invented a novel and simple thermomechanical processing to realize the ultrafine grained microstructure in carbon steels. The key of the process is to start from martensite structure. In the previous study, it has been clarified that conventional cold-rolling to a reduction in thickness of only 50% (equivalent strain of 0.8) and subsequent annealing at warm temperature around 500 °C fabricates the multi-phased ultrafine grained structure composed of the ultrafine ferrite grains with mean grain size of 180 nm, uniformly precipitated nano cementite and tempered martensite. In this study, the effect of the rolling reduction ranging from 25 to 70% (equivalent strains of 0.3–1.5) on the ultrafine grained structure and the mechanical properties of the plain low-carbon steel (Fe–0.13 wt% C) processed from martensite starting structure was studied. In the as-deformed specimen, the area fraction of the region showing the lamellar structure, which is typical for severely rolled metals, increased with increasing the rolling reduction and the strength also increased. After annealing at warm temperature around 500 °C, the multi-phased ultrafine grained microstructures were obtained in all the examined rolling reductions. The area fraction of the region showing the ultrafine ferrite grains increased with increasing the rolling reduction. At higher temperature, conventional recrystallization took place, and the recrystallization temperature became lower with increasing the reduction. Tensile test exhibited that the specimen rolled to the intermediate reduction (50%) performed the best strength-ductility balance (870 MPa of tensile strength and 9% of uniform elongation). The reason for the good strength-ductility balance of the specimen rolled to the intermediate reduction was discussed on the basis of the observed microstructures.

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1. Introduction

Nowadays, ultrafine grained (UFG) materials with mean grain size smaller than 1 μm are studied actively, because the ultrafine grained materials are expected to perform superior mechanical properties such as high strength [1], enhanced superplasticity [2], low ductile–brittle transition temperature [3], and so on. In order to obtain the UFG structures, the severe plastic deformation (SPD) process [4,5], for example accumulative roll-bonding (ARB) [6–10] equal channel angular pressing (ECAP) [11–13] high pressure torsion (HPT) [14,15] and mechanical milling [16] is occasionally used. These processes have successfully achieved ultra grain refinement [6–16]. However, the SPD processes also have the disadvantages that they need large amount of plastic working energy and the special equipments.

On the other hand, the present authors have invented a new route to fabricate the UFG steels, named the martensite process, which does not need SPD [17,18]. The key of this process is to use martensite structure as a starting microstructure. Cold-rolling of the martensite starting structure to a conventional reduction and annealing of it at warm temperatures fabricate the multi-phased ultrafine grained microstructure, which is mainly composed of ultrafine ferrite grains, uniformly precipitated carbides and tempered martensite blocks existing partially. The multi-phased ultrafine microstructure performs superior mechanical properties. For example, the plain low carbon steel with
the multi-phased ultrafine grained structure fabricated by this process performs high strength with adequate elongation: 710 MPa of 0.2% proof stress, 870 MPa of tensile strength, 9% of uniform elongation and 20% of total elongation [17].

The present authors [18] have been studied that the microstructural change during the martensite process in case of the 50% cold rolling (equivalent von Mises strain: \( \varepsilon_{vM} = 0.8 \)) in 0.13% C plain carbon steel. The 50% cold-rolling of martensite mainly developed the ultrafine lamellar structure, which is typical for the severely deformed materials, and the subsequent annealing at warm temperature around 500 °C brought the change from the lamellar structure to the equiaxed ultrafine grains with mean grain size of 180 nm [18]. The previous study [19] also indicated that the formation mechanism of the lamellar structure is a kind of grain subdivision: the deformation induced grain boundaries subdivide the initial grains to ultrafine regions. It had been believed that SPD is necessary for ultrafine grain boundaries, but the present study [18] suggested that one of the main reasons for the quick grain subdivision is that the martensite starting structure has a kind of fine grained structure in the as-transformed state, although the detailed mechanism of the quick grain subdivision has not been clarified yet. Furthermore, only 50% cold-rolling was used in the previous studies [17,18], so that the effect of rolling reduction (strains) on the deformed and final microstructure in the martensite process has not yet been studied.

The aim of this study is to clarify the effect of rolling reduction (strain) on the mechanical properties and the microstructures of the martensite processed low-carbon steel. Especially, the strength-ductility balance of the obtained material will be noticed to determine the preferable reduction for good mechanical properties and discussed on the basis of the microstructural observations.

2. Experimental

A plain low carbon steel (JIS-SS400; Fe–0.13 wt% C–0.004Ni–0.37Mn–0.020P–0.004S–0.043sol.Al) was studied in the present investigation. As-received hot rolled sheet had ferrite–pearlite structure. The size of the sheet was 2 mm in thickness, 25 mm in width and 250 mm in length. First of all, the sheet was austenitized at 1000 °C for 1.8 ks in Ar + 10 vol% H_2 atmosphere, followed by water quenching to get martensite structure. The quenched sheet showed typical lath martensite structure. The prior austenite grain size estimated from the microstructure after quenching was 270 μm [18]. The quenched sheets were cold-rolled to a reduction of 25, 50 or 70% (\( \varepsilon_{vM} = 0.3, 0.8 \) or 1.5) by 1 pass, 3 passes and 25 passes, respectively. The cold-rolling was conducted by a two-high mill with the roll diameter of 310 mm at a roll peripheral speed of 17.5 m/min. Machine oil was used as lubricant. The cold-rolled sheet was annealed at various temperatures from 300 to 700 °C for 1.8 ks in Ar atmosphere.

The mechanical properties of the cold-rolled sheets and the cold-rolled and annealed sheets were examined by room temperature tensile test. The size of the tensile test piece was 10 mm in gage length and 5 mm in gage width, which is 1/5 size of JIS-5 specimen. The initial strain rate in the tensile test was 0.8 × 10^{-4} s^{-1}. The tensile direction was parallel to the rolling direction (RD) of the sheets. The microstructure was observed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). All the microstructures were observed from the transverse direction (TD) of the sheets. The SEM observation was conducted in a Philips XL30-PEG microscope operated at 20 kV, and the TEM observation was carried out in a Hitachi H-800 microscope operated at 200 kV. The specimen surfaces for the SEM observation were electro-polished and then etched by 3% nital, whereas the specimens to identify carbides were prepared only by electro-polishing. Thin foils for TEM were prepared by twin-jet type electro polishing in a 90 vol% CH_3COOH + 10 vol% HClO_4 solution.

3. Results

3.1. Cold-rolled specimen

Fig. 1 shows the nominal stress–nominal strain curves of the as-received specimen with ferrite + pearlite structure, the as-quenched specimen with lath–martensite structure and the specimens cold-rolled to various reductions. The 0.2% offset stress, tensile strength, uniform elongation, and total elongation (elongation to fracture) analyzed from
the stress–strain curves were summarized as a function of rolling strain in Fig. 2. The as-received specimen shows a typical stress-strain curve of low-carbon steels with ferrite–pearlite microstructure; yield drop and Lüders elongation followed by work hardening were recognized. After quenching, the strength rises to 870 MPa in 0.2% offset stress and 1100 MPa in tensile strength, both of which are approximately two times higher than those of the as-received specimen. The as-quenched specimen exhibits no yield-drop. Both the uniform elongation and the total elongation decreases from the as-received state. The curve of the as-quenched sheet indicates nearly the same as that previously reported [21]. The curves of all the cold-rolled specimens show their maximum strength at small strain followed by necking and fracture. As a result, the uniform elongation is limited to a few percent and the total elongation is within 10%. Such stress–strain curves are similar to those of the as-SPD processed materials having single phase [9]. The strength increases but the elongation decreases with increasing the rolling reduction, and the tensile strength of the 70% cold-rolled specimen reaches to 1640 MPa, which is four times higher than that of the as-received specimen.

Fig. 3 shows the typical TEM microstructures of the 50% cold-rolled specimen ($\epsilon_{0.2} = 0.8$). The RD and the normal direction (ND) of the rolled sheet are parallel to the horizontal and the vertical directions in the micrographs, respectively. As the present authors [18] have pointed out in the previous paper, there are three kinds of typical microstructures observed in the 50% cold-rolled martensite as shown in Fig. 3(a) the lamellar dislocation cell (LDC) structure mainly composed of the lamellar boundaries elongated to RD, (b) the irregularly bent lamella (IBL) structure, and (c) the kinked lath (KL) structure where martensite lath is kinked by shear bands. The previous investigation [18] has shown that the LDC structure and the IBL structure have large local misorientation. Especially, it is noteworthy that the LDC structure is similar to the microstructure in the materials severely deformed [19, 20, 22]. In case of the severely deformed materials, the most lamellar boundaries are high angle grain boundaries [19,20, 22]. It should be emphasized that the structure typical for
the SPD processed materials is obtained by the conventional cold-rolling when the starting microstructure is martensite.

The three types of morphology, i.e. LDC, IBL, and KL, were clearly recognized in the SEM observations for the present specimens as well [18]. Fig. 4 shows the SEM microstructures of the as-quenched specimen [lath–martensite; (a)] and the 25% ($\varepsilon_{M} = 0.3$); (b), 50% ($\varepsilon_{M} = 0.8$) (c) or 70% ($\varepsilon_{M} = 1.5$) (d) cold-rolled specimens. The symbols of M, LDC, IBL, and KL indicate martensite, LDC, IBL, and KL structures, respectively. The IBL and the LDC structures were easily categorized by their characteristic morphology. On the other hand, the distinction between the LDC structure and the martensite structure was sometimes difficult, because both show similar lamellar structures. They were distinguished by the direction of the lamellar structure. When the lamellar structure is elongated to a direction within 20$^\circ$ from RD, it is regarded as the LDC structure, while in the other case, the structure was categorized as the martensite structure. The area fraction of each structure was measured from the SEM microstructures by point counting method, and the results are shown in Fig. 5. All the three kinds of deformed structures (LDC, IBL, and KL) exist in the 25% ($\varepsilon_{M} = 0.3$) cold-rolled specimen and fairly large amount of the martensite structure still remains. The LDC structure shows the largest area fraction of the three. The martensite structure was no longer observed in the 50% cold-rolled specimen. The area fractions of the LDC, IBL and KL structures, in the 50% ($\varepsilon_{M} = 0.8$) cold-rolled specimen were 50, 43 and 7%, respectively. The area fraction of the LDC increases with increasing the reduction. After 70% ($\varepsilon_{M} = 1.5$) cold-rolling, almost all the area show the LDC structure.

3.2. Annealed specimen

Fig. 6 shows the nominal stress–strain curves of the tempered martensite (i.e. the 0% ($\varepsilon_{M} = 0$) cold-rolled and annealed specimens) (a), and the specimens cold-rolled to various reductions and then subsequently annealed at various temperatures from 300 to 700 $^\circ$C for 1.8 ks (b)–(d). As far as the specimens tempered below 400 $^\circ$C are concerned (Fig. 6(a)), the shapes of their curves are similar to each other; yielding takes places without yield-drop, small work-hardening occurs, the maximum strength is achieved at relatively early stage of the tensile test, and then flow stress gradually decreases to fracture. It should be noted that the uniform elongation of these specimens is very small because of their poor work-hardening. The strength decreases and total elongation gradually recovers with increasing temperature. The 700 $^\circ$C tempered specimen performs nearly the same mechanical behavior as that of the ferrite + pearlite specimen. On the other hand, some of the cold-rolled and annealed specimens exhibited desirable shapes of stress–strain curves completely different from those of the quenched and tempered specimens. The curves of the specimens 25 or 50% cold-rolled and annealed at temperatures lower than 400 $^\circ$C show nearly the same tendency as the as-cold-rolled specimens, so that the elongation is still limited (Fig. 6(b) and (c)). The strength decreases but the elongation recovers with increasing the annealing temperature. When the specimen is cold-rolled and subsequently annealed at temperatures higher than 500 $^\circ$C, the shape of the curves significantly changes. The curves show enough work hardening, so that large uniform deformation takes place. In case of the 70% cold-rolled specimen (Fig. 6(d)), the elongation is still limited after warm temperature annealing and uniform deformation along with work-hardening is recognized only after
the strength significantly decreases at temperature above 540 °C.

The strength (0.2% offset stress and tensile strength) and the elongation (uniform elongation and total elongation) of the specimens cold-rolled and annealed at various temperatures are shown as a function of annealing temperature in Figs. 7 and 8, respectively. The data of the tempered specimens are also plotted in the figures. Concerning the strength, both the 0.2% offset stress and the tensile strength increase with increasing annealing temperature. The slopes of these curves are almost the same at temperatures below 500 °C, that is, the specimen cold-rolled to the larger reduction keeps the higher strength in this region. In the case of the 25% ($\epsilon_{vM} = 0.3$) or 50% ($\epsilon_{vM} = 0.8$) cold-rolled specimens, the strength does not decrease significantly at temperatures ranging from 500 to 550 °C, followed by large drop of strength at higher temperature, whereas the 70% ($\epsilon_{vM} = 1.5$) cold-rolled specimen does not show such a plateau but the sudden decrease in strength takes place at about 540 °C. The temperature at which the strength significantly drops decreases with increasing the reduction. On the other hand, both the uniform and total elongation of the cold-rolled specimens does not recover when the specimens are annealed at low temperatures. The elongation recovers when the specimens are annealed at temperatures higher than 400 °C for the 25% ($\epsilon_{vM} = 0.3$) cold-rolled specimen or higher than 500 °C for the 50% ($\epsilon_{vM} = 0.8$) cold-rolled specimen. Concerning the 70% ($\epsilon_{vM} = 1.5$) cold-rolled specimen, the elongation recovers at temperature higher than 550 °C at which the strength already significantly decreases.

Fig. 6. Nominal stress–nominal strain curves of the 0.13 wt% C steel (JIS-SS400) tempered at various temperatures for 1.8 ks. (a) or cold-rolled to a reduction of 25% ($\epsilon_{vM} = 0.3$) (b), 50% ($\epsilon_{vM} = 0.8$) (c) or 70% ($\epsilon_{vM} = 1.5$) (d) and subsequently annealed at various temperatures for 1.8 ks. Starting microstructure was martensite.

Fig. 7. Offset stress (0.2%) (a) and tensile strength (b) of the 0.13 wt% C steel (JIS-SS400) cold-rolled to a reduction of 25% ($\epsilon_{vM} = 0.3$), 50% ($\epsilon_{vM} = 0.8$) or 70% ($\epsilon_{vM} = 1.5$) and subsequently annealed at various temperatures for 1.8 ks. Starting microstructure was martensite.
Fig. 9 shows the TEM microstructures of the specimens cold-rolled to various reductions and annealed at temperatures ranging from 500 to 600 °C where significant changes in mechanical properties are observed. The tempered specimens showed typical tempered martensite structures composed of recovered lath–martensite and carbides precipitated on or within the laths. All the specimens cold-rolled and annealed at 500 °C show the nearly equiaxed ultrafine grains. The carbides precipitated uniformly because martensite is a supersaturated solid solution of carbon. The specimen cold-rolled to a reduction of 25% (a) or 50% (d) and then annealed at 500 °C partially exhibited the tempered martensite structure, whereas the specimen 70% cold-rolled and annealed at 500 °C (g) showed only the ultrafine grained microstructure with carbides. This microstructural difference corresponds well with the fractions of three kinds of deformation microstructures in the cold-rolled specimen. As was shown in Fig. 5, the LDC structure was dominant in the 70% rolled specimen. That is, the ultrafine structure in the specimens annealed at the warm temperature around 500 °C is evolved mainly from the LDC structure in the cold-rolled specimens. When annealing temperature increases, the coarse grains with grain size of a few micrometers were observed in addition to the ultrafine grained microstructure. Concerning the 25% (ε_M = 0.3) cold-rolled specimen, the coarse grains appeared after 600 °C annealing (c). Few dislocations can be found in the coarse grains, which indicates that they are the conventionally recrystallized grains. The temperature at which the recrystallized coarse grains appeared becomes lower as the rolling reduction
The 25% and annealed specimen mainly shows equiaxed carbides. The 50% (\(e_{vM} = 0.8\)) and 70% (\(e_{vM} = 1.5\)) cold-rolled specimens show elongated shapes, while the cold-rolled specimen (a) show elongated shapes, and annealed at 550 °C for 1.8 ks. Starting microstructure was martensite. Observed from TD.

As was mentioned above, a number of fine carbides precipitate in the matrix during annealing, because martensite is a supersaturated solid solution of carbon. It is expected that the carbides play an important role for the microstructural change in annealing and in turn they greatly affect the mechanical properties. Fig. 10 shows the SEM microstructures of the 550 °C tempered specimen and the specimens cold-rolled to various reductions and annealed at 550 °C for 1.8 ks. Concerning the 25% (\(e_{vM} = 0.3\)) or 50% (\(e_{vM} = 0.8\)) cold-rolled specimens, these are the conditions under which the specimens performed both high strength and adequate ductility.

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4. Discussion

The effect of reduction on the microstructure and the tensile property of the martensite-processed low-carbon steel was clarified in this work. When the starting microstructure was martensite, the cold-rolled specimen performed higher strength with increasing the rolling reduction. Cold-rolling of the low-carbon steel with martensite starting microstructure showed mainly the LDC structure, typical for the severely deformed materials, even after 25% reduction. The area fraction of the LDC structure increased with increasing the reduction. After warm temperature annealing around 500 °C, the deformed microstructure changed to the ultrafine multi-phased structure which consists of the ultrafine grains with grain size about 100 to 200 nm, uniformly precipitated carbides and tempered martensite. The high temperature annealing brought about conventional recrystallization to result in coarse grained microstructure. The recrystallization temperature becomes lower with increasing the rolling reduction. The strength of the material increased with increasing the rolling reduction, and the 70% cold-rolled specimen exhibited very high strength. However, the elongation of the as-rolled specimens was limited. Both the 0.2% offset stress and the tensile strength decreased with increasing annealing temperature. After warm temperature annealing, the elongation recovered significantly keeping high strength in the specimen cold-rolled up to 50% (\(e_{vM} = 0.8\)), whereas, in the 70% (\(e_{vM} = 1.5\)) cold-rolled specimen, it was difficult to find the coexistence of high strength and adequate elongation.

First of all, the effect of the reduction on the strength-ductility balance is discussed because it is one of the most important features of the UFG materials for structural use. Though there are some benchmarks of ductility, the uniform elongation is used as the indication of ductility in this discussion, because the uniform elongation is important for many situations, such as the products design and the secondary metal-working.

Fig. 11 shows the relationship between the uniform elongation and the tensile strength of the present steel.
The recent study of the ultrafine grained microstructure with single phase [9] clarifies that the ultra grain refinement provides high strengthening but the work hardening rate does not increase, so that early necking takes place and uniform elongation is limited. However, it should be noted that these results have been confirmed in the single phase materials which have originally small work-hardening ability. That is, it would be possible to realize adequate ductility by enhancing work-hardening even in the UFG materials having very high strength. It can be expected, for example, that the undeformable precipitates in the matrix enhance the work hardening. Ashby et al. [24] have suggested that the enhanced work hardening is caused by the activation of secondary dislocation slips around the undeformable particles. The present multi-phased ultrafine structure includes fine carbides as shown in Figs. 9 and 10. It should be reasonable to consider that the fine carbides enhance the work hardening of the matrix. Actually, the stress–strain curves of the specimen with high strength and adequate ductility shows large work hardening (Fig. 6) compared with the UFG materials having single phase [9], which is thought to be the reason for the good strength-ductility balance of the present material.

As shown in Fig. 9, conventional recrystallization (discontinuous recrystallization) takes place at higher temperatures to result in the coarse grained microstructure. The microstructural change during annealing is schematically summarized in Fig. 12. The temperature at which conventional recrystallization occurs became lower with increasing the rolling reduction. As is well-known, the recrystallization temperature becomes lower with increasing the reduction since the stored energy (the driving force for recrystallization) becomes larger [25]. On the other hand, precipitation inhibits growth of recrystallized nuclei. Such a pinning effect should be considered in the present materials, because a number of fine carbides precipitate from a supersaturated solid solution of carbon (martensite) during annealing, as was described above. As is well-known, the Zener pinning force becomes larger when the size of the precipitates becomes smaller and the volume fraction larger [25]. As shown in Fig. 10, the size of the precipitates in the present specimens became slightly larger...
with increasing strain, whereas the volume fraction of the precipitates seems to be constant. Therefore, the pinning force would slightly decrease with increasing the rolling reduction. Combining the driving force for recrystallization and the pinning force for grain boundary migration, it can be expected that the recrystallization in the present cold-rolled specimen would be inhibited compared with the single phase material but an abrupt recrystallization (grain growth) would occur under a certain condition (reduction and temperature). It is noteworthy that the large drop of the strength, as shown in Fig. 7, corresponds well with the occurrence of the conventional recrystallization. In case of the 70% rolled specimens, the recrystallization took place at considerably low temperature (500 °C) due to the large driving force (stored energy) which overcomes the pinning force. Because of this enhanced recrystallization, the specimen cold-rolled to higher reduction (70%, \( \varepsilon_{\text{M}} = 1.5 \)) cannot keep high strength until enough ductility recovers. On the other hand, the 25% (\( \varepsilon_{\text{M}} = 0.3 \)) cold-rolling was not enough to realize high strength. In case of the intermediately (50%) rolled specimens, recrystallization is adequately retarded due to the balance between the driving force and the pinning force, so that it could perform superior strength-ductility balance.

Fig. 13 shows the relationship between the total elongation and the tensile strength. Roughly speaking, there is no significant difference between the quenched and tempered specimens and the cold-rolled and annealed specimens, except for the 70% (\( \varepsilon_{\text{M}} = 1.5 \)) cold-rolled and annealed specimens which perform inferior strength-ductility balance. The reason why the 70% rolled specimens showed worse strength—total elongation balance has not been clarified yet. Originally there is no theoretical explanation for total elongation. It should be, further, emphasized that, in structure materials, uniform elongation is usually considered much more important than total elongation. Consequently, it can be concluded that the martensite process is a good process for realizing desirable strength-ductility balance (high strength and adequate uniform elongation (Fig. 11)).

5. Conclusion

The effect of the rolling reduction ranging from 25 to 70% (equivalent strains, \( \varepsilon_{\text{M}} \), of 0.3–1.5) on the ultrafine grained structure and the mechanical properties of the plain low-carbon steel (Fe–0.13 wt% C) processed from martensite starting structure (martensite process) was studied. The major results are summarized as follows:

1. In the as-deformed state, three kinds of the deformation microstructures were observed. The LDC structure, which is the typical microstructure of the severely deformed materials, mainly developed together with other two types of deformation microstructures. The area fraction of the LDC structure increased with increasing the rolling reduction. Almost fully LDC structure was evolved in the 70% (\( \varepsilon_{\text{M}} = 1.5 \)) cold-rolled specimen. The strength increased but the elongation decreased with increasing the reduction.

2. After annealing at warm temperature around 500 °C, the multi-phased ultrafine grained structure which mainly consists of the ultrafine ferrite grains were obtained. A number of fine carbides uniformly precipitated in the matrix, because martensite is a supersaturated solid solution of carbon. The area fraction of the region showing the ultrafine ferrite grains increased with increasing the rolling reduction. At higher temperature, conventional recrystallization took place, and the recrystallization temperature became lower with increasing the reduction. The microstructural change was interpreted in terms of the balance between the driving force (stored energy) for recrystallization and the pinning force for grain growth owing to the precipitates.

3. Tensile test exhibited that the specimen rolled to the intermediate reduction (50%, \( \varepsilon_{\text{M}} = 0.8 \)) performs the best strength-ductility balance (Fig. 11). The 50% rolled specimen showed 870 MPa of tensile strength and 9% of uniform elongation after 550 °C annealing. Because of the enhanced recrystallization, the specimen cold-rolled to higher reduction (70%, \( \varepsilon_{\text{M}} = 1.5 \)) cannot keep high strength until adequate ductility is realized. On the other hand, the 25% (\( \varepsilon_{\text{M}} = 0.3 \)) cold-rolling was not enough to realize significant high strength. The strength-ductility balance of the materials was explained by the plastic instability in consideration of the multi-phased ultrafine grained structure.
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