Effect of Heat Treatment on Microstructures and Tensile Properties of Ultrafine Grained C–Mn Steel Containing 0.34 mass% V

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(Received on October 27, 2003; accepted in final form on February 20, 2004)

An ultrafine grained (UFG) C–Mn steel containing a relatively large amount of vanadium was fabricated by equal channel angular pressing (ECAP) and its microstructures and tensile properties were examined. This investigation was aimed at demonstrating the effect of precipitation stage of vanadium precipitates in the course of the material processing on the tensile properties of the ultrafine grained C–Mn steel, especially strength and strain hardening capability. For this purpose, the two different heat treatments were carried out on the present steel: (a) conventional normalization for vanadium precipitation before ECAP, and (b) isothermal transformation for vanadium precipitation during ECAP and subsequent annealing. The results showed that the second heat treatment was more effective on improving the thermal stability and the overall tensile properties of the steel by better uniform distribution of nano-sized vanadium precipitates which yielded an extensive interaction with lattice dislocations inside ultrafine ferrite grains. In addition, in this report, the feasibility enhancing the strain hardening capability of the UFG C–Mn steel was explored by comparing the microstructure and stress–strain curve of the steel prepared by the second heat treatment with those of the UFG C–Mn steel without vanadium.

KEY WORDS: C–Mn steel; ultrafine grain; equal channel angular pressing; heat treatment; microstructure; tensile properties; vanadium.

1. Introduction

The ultrafine grained (UFG) carbon–manganese (C–Mn) steels have a great potential as advanced structural material by their ultrahigh strength and enhanced toughness.1,2 However, as proven by recent extensive experimental studies,3–7 strain hardening capability and ductility of the UFG C–Mn steels, especially when they are fabricated by severe plastic deformation, are much inferior to those of coarse grained ones. These deficiencies are the primary factors limiting the practical application of the UFG C–Mn steels. In general, the poor strain hardening capability of UFG materials is often explained by relatively rapid dynamic recovery associated with the ultrafine grain size during deformation.5,6 As well, the value of the strain hardening exponent affects uniform elongation. One of the effective methods improving the strain hardening capability is an employment of effective obstacles for dislocation motion at the ultrafine grain interior.

Recently, the present authors9,10 examined the tensile properties of UFG C–Mn steel containing a small amount of V (0.06 mass%) for improving its thermal stability and strain hardening capability by embedding fine V precipitates into UFG ferrite matrix: in that study, the steel was normalized before equal channel angular pressing (ECAP), a severe plastic deformation method introducing an UFG structure. The results showed that the thermal stability of the 0.06 mass% V added UFG C–Mn steel was dramatically improved but its strain hardening capability was hardly improved, compared to the one without V. The lack of the strain hardening capability in the 0.06 mass% V added UFG C–Mn steel may be attributed to the fact either that the V content was too small or that V precipitates formed during normalization before ECAP could not pin the lattice dislocations effectively. In order to address this issue more comprehensively, the two different approaches were attempted in this study: (a) the addition of relatively large amount of V (0.34 mass%) and (b) heat treatment designed to form V precipitates during ECAP and subsequent annealing, rather than during normalization before ECAP. In this article, the microstructures and tensile properties of the UFG C–Mn steel with 0.34 mass% V prepared by the present approaches are discussed and compared to those of the steel processed by the previous route.

2. Experimental

2.1. Material

A steel used in the present investigation was prepared as a 50 kg ingot using a vacuum induction furnace. The
The chemical composition of the steel was Fe–0.15C–0.25Si–
1.12Mn–0.34V–0.012N (in mass%). No deliberate alu-
minum addition was made in order to avoid the undesired
effect from aluminum nitride precipitation. For the present
chemical composition, the Ae₃ temperature and the dissolu-
tion temperature of V precipitates were predicted as 1122
and 1306 K respectively from the Thermo-Calc program.

2.2. Heat Treatment before ECAP
The ingot was homogenized at 1523 K for 1 h and size-
rolled to the plate of 50 mm thickness and 150 mm width.
After machining the rods of 10 mm diameter and 130 mm
length from the plate, the two different heat treatments were
conducted on the rods before ECAP. In the first heat treat-
ment, the rods were austenitized at 1473 K for 1 h, oil-
quenched and then normalized. Normalization consisted of
a soaking at 1173 K for 1 h and the subsequent air cooling
to ambient temperature. This route is anticipated to yield
the interphase precipitation of V precipitates in ferrite be-
fore ECAP. (Hereafter, the sample subjected to the first
route is denoted as the CSV1.) The second heat treatment
was designed for V precipitates to form during ECAP
and/or subsequent annealing treatment. The rods were
austenitized at 1473 K for 1 h, direct-quenched at 873 K,
maintained for 4 h and air-cooled. (Hereafter, the sample
subjected to the second route is denoted as the CSV2.)
Compared to the first one, the second heat treatment was
expected to result in more uniform distribution of finer pre-
cipitates after ECAP due to accelerated precipitation kinet-
ics and a larger number of precipitate nucleation sites asso-
ciated with very high internal energy and dislocation densi-
ty induced by severe plastic deformation.

2.3. ECAP and Subsequent Annealing Treatment
ECAP was carried out on the preheat-treated samples at
623 K up to 4 passes. As described elsewhere, the pre-
sent ECAP die was designed to yield an effective strain of
−1 per pass: the inner contact angle and the arc of curva-
ture at the outer point of contact between channels of the
die were 90° and 20°, respectively. During ECAP, the sam-
ple was rotated 180° around its longitudinal axis between
the passes, i.e. route C. The samples for the subsequent
annealing treatment were encapsulated in a glass tube with
Ar atmosphere in order to minimize the possible decarbur-
zation. Annealing of the ECPed samples was conducted for
1 h in the temperature range of 753–93 K. During an-
nealing, temperature was controlled within ±2 K.

2.4. Microstructural Observation and Mechanical
Testing
The microstructures were examined by optical micro-
scope and transmission electron microscope (TEM). For
TEM observation, thin foils were prepared by a twin-jet
polishing technique using a mixture of 20% perchloric acid
and 80% methanol at an applied potential of 40 V and at
233 K. TEM micrographs were obtained by utilizing a
JEOL 2010 TEM operating at 200 kV. Tensile tests were
carried out at room temperature using an INSTRON model
1125 machine with the initial strain rate of 1.33×10⁻³ s⁻¹
on the full scale tensile samples with the gage length of
25.4 mm. Three or four tensile specimens were tested for
each experimental condition.

3. Results and Discussion

3.1. Microstructure
3.1.1. Before ECAP
The optical microstructures of the CSV1 and CSV2
steels are shown in Fig. 1. The CSV1 steel (Fig. 1(a)) ex-
hibited the typical ferrite–pearlite duplex structure. The
mean linear intercept size of the ferrite grains and pearlite
colonies was about 12 μm. For the CSV2 steel, the ferrite
grains were irregular in shape and their average size was
about 9 μm. Unlike the CSV1 steel, the pearlite colony size
of the CSV2 steel consisted of degenerated cementite lamellae. At a first glance, the pearlite fraction of
the CSV1 steel seemed to be higher than that of the
CSV2 steel. However, the detailed quantitative 2-dimen-
sional measurement using an image analyzer revealed that
the pearlite area fraction of the CSV2 steel (~26%) was
slightly higher than that of the CSV1 steel (~23%); an ex-
ample of the image analysis is shown in Fig. 2. The higher
pearlite volume fraction and degenerated cementite lamellae in the CSV2 steel were mainly attributed to the fact that
higher supercooling results in less carbon content in
pearlite and, resultantly, a larger fraction of pearlite in the
hypoeutectoid composition.

Figure 3 shows TEM micrographs of the CSV1 and
CSV2 steels. The presence of parallel rows of array of the
precipitates with equal spacing was evident in the CSV1
steel (Fig. 3(a)), i.e. interphase precipitation. The size of in-
terphase precipitates was about 10 nm or less. On the
contrary, precipitates were hardly observed in the CSV2 steel (Fig. 3(b)). In addition, no distinct extra spots for precipitates appeared in the selected area diffraction pattern (SADP). Instead, unidirectional streaking was evident in the SADP, indicative of the supersaturated state. From the comparison between Figs. 3(a) and 3(b), it is not erroneous to conclude that the ferrite phase of the CSV2 steel was in the solid solution state supersaturated by excessive carbon and vanadium contents.

3.1.2. After ECAP

The TEM microstructure of the CSV1 (with interphase precipitates) and CSV2 steels (without precipitates) after ECAP (623 K and an effective strain of ~4) are shown in Fig. 4. Both steels exhibited several similar features: (a) the grain size of 0.2–0.3 μm, (b) ill-defined grain boundaries, (c) dense dislocation debris, (d) near-ring type SADP, etc. Very recently, it was addressed that the existence of the relatively coarse second phase particles accelerated the grain refinement process during ECAP. However, in the present case, such a trend was not observed: the grain size of the CSV1 and CSV2 steels was comparable each other at the identical ECAP strain.

It was noticed that, as indicated by the arrows in Fig. 5(a), the extremely fine precipitates of 5–10 nm were observed at the area of high dislocation density in the as-ECAPed CSV2 steel which did not contain the precipitates before ECAP. Accordingly, it is obvious that these precipitates, which were identified as V carbides, mainly V₃C₄, by the energy dispersive spectra analysis (Fig. 5(b)), were formed during ECAP at 623 K. These precipitates are believed to result from the strain induced precipitation of which nucleation sites were the heterogeneous ones such as dislocations of high density formed by ECAP. Figure 6 shows the TEM replica micrographs showing the distribution and size of V carbides in the CSV1 and CSV2 steels annealed after ECAP. In the CSV1 steel annealed at 873 K for 1 h (Fig. 6(a)), the distribution of V carbides became random from the initial distribution of parallel rows and considerable carbide coarsening occurred, from less than
(10 nm (Fig. 3(a)) to mostly larger than ~50 nm. In the CSV2 steels annealed at 933 K for 1 h (Fig. 6(b)), V carbides were uniformly distributed in the ferrite matrix and their size, 10–30 nm, was slightly larger than those in the as-ECAPed sample, but still smaller than those in the CSV1 steel even at higher annealing temperature. Accordingly, it is certain that precipitation during ECAP and subsequent annealing resulted in more uniform distribution of finer precipitates than precipitation before ECAP. This microstructural feature is anticipated to improve thermal stability and strain hardening capability of the C–Mn steels having an UFG structure.

3.2. Tensile Properties

3.2.1. Stress–Strain Curves

The representative nominal stress–strain curves for the CSV1 and CSV2 steels at various experimental conditions are shown in Fig. 7 and the mean values of their major tensile properties averaged from three or four identical tests are listed in Table 1. Before ECAP, the normalized CSV1 steel (curve 1) exhibited a discontinuous yielding with some extent of yield point elongation followed by prolonged strain hardening, as typical in the common low carbon ferrite–pearlite steels. The stress–strain curve of the CSV2 steel before ECAP (curve a) was characterized by continuous yielding and extensive strain hardening. For the substitutional solid solution alloys, the stress field around solute atoms more influences on the friction resistance of dislocation motion than the dislocation pinning.14) Besides, there would be an additional interaction between supersaturated carbon atoms and dislocations. Resultantly, yielding becomes continuous and the whole level of the stress–strain curve increases. Along with Fig. 3(b), this finding strongly lends support to the fact that the CSV2 steel before ECAP was in the supersaturated solid solution state. The smaller ferrite grain size and continuous yielding may be responsible for higher flow stress of the CSV2 steel than that of the CSV1 steel.

For the as-ECAPed samples, both CSV1 (curve 2) and...
CSV2 (curve b) steels exhibited very high YS over 900 MPa, no strain hardening, and very little uniform elongation. Of course, the dramatic increase of YS is the combined effects of grain refinement down to the submicrometer level and very high dislocation density introduced by severe plastic deformation.

There was drastic difference in the stress–strain curves of the annealed samples between the CSV1 and CSV2 steels. For the CSV1 steel annealed at 873 K for 1 h after ECAP (curve 3), the YS, UTS, and flow stress decreased to the level lower than those of the sample before ECAP (curve 1), due to significant V carbide coarsening and grain growth. Compared to the annealed CSV1 steel, the CSV2 steel annealed at 933 K for 1 h after ECAP (curve c) revealed several characteristics. First, the YS, UTS, and flow stress were much superior to those of the sample before ECAP (curve a), even at the annealing temperature 60 K higher than that for the CSV1 steel. Second, the uniform as well as total elongations were similar to those of the sample before ECAP. Finally, moderate strain hardening occurred. The last two characteristics along with such high strength level in UFG materials are uncommon and have been rarely reported. TEM micrographs of the annealed CSV2 steel (curve c) after failure are shown in Fig. 8. After annealing, the ferrite grain size of the CSV2 steel (Fig. 8(a)) was about 0.5 μm, indicating no significant grain growth during annealing and there was extensive interaction between lattice dislocations and nano-sized V precipitates (Fig. 8(b)). The above findings clearly demonstrate that the present heat treatment, i.e. strain induced precipitation by ECAP, is very effective on improving not only thermal stability of the V containing UFG C–Mn steel but also its overall room temperature tensile properties, compared to normalization followed by ECAP.

| condition                  | YS, MPa | UTS, MPa | εu, % | εT, % |
|---------------------------|---------|----------|-------|-------|
| CSV1 before ECAP          | 435     | 568      | 17    | 28    |
| after ECAP                | 920     | 920      | 2     | 9     |
| annealed at 873 K×1 hr    | 441     | 516      | 18    | 31    |
| CSV2 before ECAP          | 465     | 643      | 14    | 18    |
| after ECAP                | 925     | 949      | 2     | 10    |
| annealed at 933 K×1 hr    | 718     | 796      | 9     | 19    |

εu: uniform elongation  εT: elongation to failure
The values are the averaged ones from three or four tests under identical experimental condition.

Fig. 8. (a) Deformed microstructure of the CSV2 steel annealed at 933 K for 1 h after ECAP. (b) TEM micrograph showing the interaction between V carbides and lattice dislocations in the annealed CSV2 steel after tensile test.

Fig. 9. A comparison of the nominal stress–strain curves between the CSV steel annealed at 933 K for 1 h after ECAP and the CS steel annealed at 753 K for 72 h after ECAP.

3.2.2. Strain Hardening in the Present CSV2 Steel

The absence of strain hardening in UFG materials is often explained in terms of (a) dynamic recovery balancing the dislocation generation rate with the spreading rate of trapped lattice dislocations at the grain boundaries, and (b) the mean free dislocation length is comparable to the grain size. Under these conditions, no dislocation tangling associated with strain hardening is expected to take place inside the grains of UFG materials. In this section, the feasibility improving strain hardening capability of UFG steels will be explored by comparing the microstruc-
ture and stress–strain curve of the present CSV2 steel with those of the UFG steel without V (hereafter, CS steel) reported previously.

The nominal stress–strain curves of the CSV2 steel (annealed at 933 K for 1 h after ECAP) and CS steel (annealed 753 K for 72 h after ECAP) are shown in Fig. 9. The ECAP conditions were identical in both steels and the basic chemical composition of the CS steel was also the same except the V and nitrogen contents: the detailed information on the CS steel is in Ref. 5). The CS steel annealed 753 K for 72 h after ECAP was selected for the purpose of comparison by the following reasons: (a) the grain size of both steels annealed after ECAP was comparable, ~0.09 μm, (Fig. 8(a) and Figs. 10(a)) and 10(b) YS of both steels annealed after ECAP increased with almost equal ratio compared to that before ECAP. For the annealed condition, the strain hardening exponent of the CSV2 steel, ~0.09, was 50% higher than that of the CS steel, ~0.06: as a first approximation, the strain hardening exponent (m) was estimated by applying the Hollomon equation of $\sigma = K \varepsilon^m$. Figure 10(b) shows a TEM micrograph of the annealed CS steel after tensile test. Unlike the CSV2 steel (Fig. 8(b)) in which dislocations were distributed uniformly at the grain interior, the localized dislocation distribution at the vicinity of grain boundaries was evident in the CS steel. This feature provides the evidence of trapped lattice dislocation at grain boundaries associated with dynamic recovery. Accordingly, it is conclusive that the homogeneous distribution of nano-sized V precipitates which resulted from the strain induced precipitation through the present heat treatment conceives a strong feasibility to improve the strength and strain hardening capability of the UFG C–Mn steel without loss of ductility.

4. Conclusions

(1) The two different heat treatments with ECAP were carried out in the course of fabrication of ultrafine grained C–Mn steel with 0.34 mass% vanadium: (a) conventional normalization for vanadium precipitation before ECAP, and (b) isothermal transformation for vanadium precipitation during ECAP and subsequent annealing.

(2) Annealing after ECAP resulted in the considerable coarsening of vanadium carbides which were precipitated during normalization before ECAP. By contrast, vanadium carbides precipitated during ECAP and subsequent annealing were relatively stable.

(3) The heat treatment designed for vanadium carbides to precipitate during ECAP and subsequent annealing was effective on improving the thermal stability and overall tensile properties of the steel by better uniform distribution of nano-sized vanadium carbides which yielded an extensive interaction with lattice dislocations inside ultrafine ferrite grains.

(4) A strain induced precipitation associated with severe plastic deformation has a strong feasibility to improve the strength and strain hardening capability of the UFG C–Mn steel without loss of ductility.

Acknowledgment

This work was supported by Ministry of Science and Technology of Korea through ‘2000 National Research Laboratory Program’ and ‘The 21st Century New Frontier Research and Development Program’.

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