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.
chemical composition of the steel was Fe–0.15C–0.25Si–1.12Mn–0.34V–0.012N (in mass%). No deliberate aluminum addition was made in order to avoid the undesired effect from aluminum nitride precipitation. For the present chemical composition, the Ae3 temperature and the dissolution temperature of V precipitates were predicted as 1 122 and 1 306 K respectively from the Thermo-Calc program.

2.2. Heat Treatment before ECAP

The ingot was homogenized at 1 523 K for 1 h and size-rolled to a 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 treatment, the rods were austenitized at 1 473 K for 1 h, oil-quenched and then normalized. Normalization consisted of a soaking at 1 173 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 before 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 1 473 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.)

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 present ECAP die was designed to yield an effective strain of about 8 grains were irregular in shape and their average size was 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)) exhibited the typical ferrite–pearlite duplex structure. The mean linear intercept size of the ferrite grains and pearlite colonies was ~12 μm. For the CSV2 steel, the ferrite grains were irregular in shape and their average size was about 5 μm. Unlike the CSV1 steel, the pearlite colony size of the CSV2 steel was smaller than the ferrite grain size. As shown in the inset TEM micrograph in Fig. 1(b), most pearlite colonies of the CSV2 steel consisted of degenerated cementite lamellae. At a first glance, the pearlite faction of the CSV1 steel seemed to be higher than that of the CSV2 steel. However, the detailed quantitative 2-dimensional 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 example 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.

2.4. Microstructural Observation and Mechanical Testing

The microstructures were examined by optical microscope 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 1020 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.
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.13) 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. 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.\(^{15,16}\) 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.

### 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,\(^{5,8}\) and (b) the mean free dislocation length is comparable to the grain size.\(^{17-19}\) Under these conditions, no dislocation tangleing 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-

### Table 1. Tensile properties of the CSV1 and CSV2 Steels.

<table>
<thead>
<tr>
<th>condition</th>
<th>YS, MPa</th>
<th>UTS, MPa</th>
<th>(\varepsilon_u) %</th>
<th>(\varepsilon_{\ell}) %</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSV1 before ECAP</td>
<td>435</td>
<td>568</td>
<td>17</td>
<td>28</td>
</tr>
<tr>
<td>CSV1 after ECAP</td>
<td>920</td>
<td>920</td>
<td>2</td>
<td>9</td>
</tr>
<tr>
<td>CSV1 annealed at 873 K×1 hr</td>
<td>441</td>
<td>516</td>
<td>18</td>
<td>31</td>
</tr>
<tr>
<td>CSV2 before ECAP</td>
<td>465</td>
<td>643</td>
<td>14</td>
<td>18</td>
</tr>
<tr>
<td>CSV2 after ECAP</td>
<td>925</td>
<td>949</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>CSV2 annealed at 933 K×1 hr</td>
<td>718</td>
<td>796</td>
<td>9</td>
<td>19</td>
</tr>
</tbody>
</table>

\(\varepsilon_u\): uniform elongation  \(\varepsilon_{\ell}\): elongation to failure
The values are the averaged ones from three or four tests under identical experimental condition.
ture and stress–strain curve of the present CSV2 steel with those of the UFG steel without V (hereafter, CS steel) reported previously.\(^5\)

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, ~0.5 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 \(\sigma=K\varepsilon^n\). 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 steels 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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