Reverse Austenite Transformation Behavior of Equal Channel Angular Pressed Low Carbon Ferrite/Pearlite Steel

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Reverse austenite transformation characteristics of ultrafine grained ferrite/pearlite low carbon steel were examined by using a dilatometry with a heating rate of 10°C/s, and compared with those of coarse grained counterpart. Ultrafine grained steel was prepared by equal channel angular pressing at 500°C, resulting in a microstructure consisting of ultrafine ferrite grains and pearlite with partially dissolved pearlitic cementite. Reverse austenite transformation start and finish temperatures of ultrafine grained steel were lower than those of coarse grained steel. Reverse austenite transformation of coarse grained steel occurred with the two serial stages of pearlite→austenite followed by proeutectoid ferrite→austenite. In the case of ultrafine grained steel, reverse austenite transformation was manifested by the three serial stages of carbon-supersaturated ferrite→austenite, pearlite→austenite, and proeutectoid ferrite→austenite in the order. The formation of carbon-supersaturated ferrite in ultrafine grained steel is associated with carbon dissolution from pearlitic cementite during equal channel angular pressing. The effect of equal channel angular pressing on reverse austenite transformation temperatures can be explained in terms of the difference in shear stress driving transformation between two steels under assumption that the nature of carbon-supersaturated ferrite in ultrafine grained steel is similar to that of conventional martensite.

KEY WORDS: reverse transformation; equal channel angular pressing; ultrafine grain; low carbon steel; dilatation.

1. Introduction

One of the distinguishing characteristics of the steel from other metallic materials is that a variety of constituent phases including both stable and metastable phases can be obtained at a fixed chemical composition. Accordingly, a combination of such phases makes it possible that the steel with a fixed chemical composition can cover a wide range of applications demanding different mechanical requirements. In order to obtain the desired combination of phases, it is essential to understand the phase transformation characteristics of the steel at a given composition. In addition to phase transformation during cooling from austenite, reverse transformation during heating of ferrite/pearlite has a particular importance on developing the advanced steels such as ferrite/martensite dual phase steels, transformation induced plasticity aided steels, and other multiphase steels. This is because the microstructural characteristics of these steels (i.e. type, shape, size, volume fraction, and distribution of constituent phases) are primarily determined by reverse transformation conditions between austenite transformation start (\(A_{1\text{f}}\)) and finish (\(A_{3\text{f}}\)) temperatures (i.e. ferrite/austenite two phase region).

Since the first development of ferrite/martensite dual phase steel in the mid 1970’s,1) reverse transformation of coarse grained steels (usually grain or phase size larger than \(\sim 10 \mu m\)) has been studied steadily up to now, and its mechanism is reasonably understood. Meanwhile, with the recent advent of breakthrough technologies refining the grain size of metallic materials down to the submicrometer level, innumerable researches have been carried out to develop ultrafine grained (UFG) steels,2–4) mainly focusing on enhancing their mechanical properties. The reverse transformation characteristics of UFG steels are expected to quite differ from those of coarse grained steels since the former contains a higher density of lattice defects that can affect the transformation kinetics than the latter. However, little systematic information on the reverse transformation of UFG steels is available at present although UFG ferrite/martensite dual phase steel showing an excellent combination of ultrahigh strength and extended ductility was developed by reverse transformation of UFG ferrite/pearlite steel.5,6) Therefore, in the present investigation, the reverse transformation characteristics of UFG low carbon ferrite/pearlite steel fabricated by severe plastic deformation (SPD) were investigated. The firsthand purpose is to better understand the effects of the initial UFG structure and SPD on reverse transformation of low carbon ferrite/pearlite steels. In the course of the present investigation, equal channel angular pressing (ECAP)7) was utilized as the SPD technique since (a) it is the most well-established SPD technique producing bulk, contamination/porosity-free, and equiaxed UFG metallic materials,8) and (b) sufficient information on its application to production of UFG ferrite/
pearlite steel is available in the literature.\textsuperscript{4,9,10)}

2. Experimental Procedures

A plain low carbon steel (Fe–0.15C–0.25Si–1.06Mn (wt%)) was austenitized at 1200°C for 1 h and then air cooled: hereafter ‘unECAPed steel’. The microstructure of unECAPed steel (Fig. 1(a)) consisted of ~23 vol% pearlite (dark patches) and the remainder of ferrite. Both ferrite grains and pearlite were nearly equiaxed and their size was ~30 \( \mu \text{m} \). After austenitization, some of the steel was machined into rods of 10 mm diameter and 130 mm length for ECAP. ECAP was conducted at 500°C with a die yielding an effective strain of ~1 by a single pass and a ram speed of 2 mm/s. ECAP was performed up to 4 passes (an accumulated strain of ~4) with route C (180° sample rotation around the sample axis between the pass): hereafter ‘ECAPed steel’. Macroscopically, both ferrite and pearlite were severely deformed seemingly along the shear direction of the final ECAP pass (Fig. 1(b)). After machining the samples of 3\( \times \)10\( \times \)1 mm\(^3\) from both ECAPed and un-ECAPed steels, dilatometric tests were conducted in vacuum by reheating the samples from room temperature to 1100°C with a heating rate of 10°C/s, followed by cooling to room temperature with a cooling rate of 50°C/s. The dilatational strain–temperature data were collected and processed by a data acquisition system. In order to examine austenite formation during reverse transformation, some samples were quenched before completion of reverse transformation at various temperatures belonging to the ferrite/austenite two-phase region. The microstructures were examined by optical microscope, scanning electron microscopy (SEM, JEOL6330F at 20 kV) and transmission electron microscope (TEM, JEOL2010 at 200 kV). 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.

3. Results

3.1. \( A_{c1} \) and \( A_{c3} \) Temperatures

The representative dilatational strain–temperature curves of unECAPed and ECAPed steels during heating are shown in Fig. 2, and their \( A_{c1} \) and \( A_{c3} \) temperatures inferred from Fig. 2 are listed in Table 1. The temperature range of reverse transformation, ~125°C, was almost the same for un-ECAPed and ECAPed steels. However, \( A_{c1} \) and \( A_{c3} \) temperatures of ECAPed steel were shifted to lower temperature by ~10°C compared to those of unECAPed steel: that is, reverse transformation of ECAPed steel started and finished earlier than that of unECAPed steel.

An empirical formula predicting \( A_{c1} \) and \( A_{c3} \) temperatures of the steel based on the type and amount of alloying elements was suggested by Andrews,\textsuperscript{11)} and has been widely quoted:

\[
A_{c1} (°C) = 723 - 10.7Mn - 16.9Ni + 29.1Si + 16.9Cr + 290As + 6.38W \\
A_{c3} (°C) = 910 - 203C^{0.5} - 15.2Ni + 44.7Si + 104V + 31.5Mo + 13.1W
\]

\( A_{c1} \) and \( A_{c3} \) temperatures of the present steel predicted from Eq. (1) are ~718°C and ~842°C respectively, both lower

![Fig. 1](image1.png)  
Optical microstructure of the present steel: (a) before ECAP and (b) after ECAP.

![Fig. 2](image2.png)  
A representative plot of dilatational strain against temperature of unECAPed and ECAPed steels.

<table>
<thead>
<tr>
<th>steel</th>
<th>( A_{c1} ) (°C)</th>
<th>( A_{c3} ) (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>unECAPed</td>
<td>739</td>
<td>865</td>
</tr>
<tr>
<td>ECAPed</td>
<td>730</td>
<td>854</td>
</tr>
</tbody>
</table>
than those measured. The difference between the predicted and the measured is probably attributed to the two main facts that (a) Eq. (1) was developed only on the compositional basis without considering the initial microstructure and thermal and/or mechanical history, and (b) the present heating rate (10°C/s) was rapid enough to decelerate the reverse transformation kinetics.

3.2. Dilatational Response

3.2.1. unECAPed Steel

As seen in Fig. 2, for unECAPed steel, the transformation contraction gradually took place with increasing temperature at the initial stage of transformation (stage A) and then became rapid (stage B). It is known that pearlite→austenite transformation first occurs ahead of ferrite→austenite transformation during reverse transformation of ferrite/pearlite steels. The first occurrence of pearlite→austenite transformation arises from (a) preferred austenite nucleation at pearlite colony boundaries within a pearlite nodule, and (b) austenite growth towards pearlite interior, after being nucleated at ferrite/pearlite interfaces, rather than towards proeutectoid ferrite in association with shorter carbon diffusion distance from pearlitic cementite to pearlitic ferrite than to proeutectoid ferrite.

In order to examine the first occurrence of pearlite→austenite transformation, unECAPed steel was heated to 780°C (belonging to the stage A), and then immediately austenite transformation, unECAPed steel was heated to a carbon diffusion distance from pearlitic cementite to pearlitic ferrite in association with shorter carbon diffusion distance from pearlitic cementite to pearlitic ferrite than to proeutectoid ferrite.

Fig. 3. SEM micrograph showing the presence of martensite at either pearlite colony boundaries (marked by ‘A’) or pearlite/ferrite interface (marked by ‘B’) of unECAPed steel reheated to 780°C followed by immediate quenching.

3.2.2. ECAPed Steel

Contrary to unECAPed steel showing the two serial stages of reverse transformation, ECAPed steel exhibited a sigmoidal dilatational response with increasing temperature which can be divided into the three distinct stages: initial rapid contraction (stage I), gradual contraction (stage II), and re-rapid contraction (stage III). In order to characterize the transformation stages of ECAPed steel, the average slope \( d\varepsilon/dT \) of the curve segment representing each transformation stage was obtained, and compared with that of unECAPed steel in Table 2. The values of \( d\varepsilon/dT \) of the stages II (\(-0.44 \times 10^{-5} \, ^\circ\text{C}^{-1}\)) and III (\(-2.01 \times 10^{-5} \, ^\circ\text{C}^{-1}\)) of ECAPed steel were found to be close to those of the stages A (\(-0.40 \times 10^{-5} \, ^\circ\text{C}^{-1}\)) and B (\(-1.63 \times 10^{-5} \, ^\circ\text{C}^{-1}\)) of unECAPed steel, respectively. This comparison strongly implies that the stages II and III of ECAPed steel correspond to pearlite→austenite transformation and ferrite→austenite transformation respectively, as similar to unECAPed steel.

It is of interest to note that ECAPed steel exhibited the initial rapid contraction, i.e. stage I, at the onset of reverse transformation, which was absent in unECAPed steel. Its value of \( d\varepsilon/dT \) (\(-1.79 \times 10^{-5} \, ^\circ\text{C}^{-1}\)) was very close to that of the stage B (\(-1.63 \times 10^{-5} \, ^\circ\text{C}^{-1}\)) of unECAPed steel, suggesting that the stage I of ECAPed steel is also related to ferrite→austenite transformation. The appearance of the stage I of ECAPed steel is certainly associated with ECAP since the preparation history and experimental conditions were identical for both steels except ECAP. Therefore, it is speculated that, by ECAP, some of ferrite already became carbon-supersaturated enough for austenite formation so that carbon-supersaturated ferrite was transformed to austenite prior to pearlite→austenite transformation during reverse transformation.

4. Discussion

4.1. Origin of Stage I Transformation of ECAPed Steel

Recently, the dissolution behavior of pearlitic cementite of fully pearlitic steels during SPD (for example, cold drawing or high pressure torsion) has been studied by using the advanced analytical tools such as Mössbauer spectroscopy, 3-dimensional atomic probe field ion microscopy, etc. These investigations commonly revealed (a) the carbon enrichment in pearlitic ferrite well above its equilib-

Table 2. The values of \( d\varepsilon/dT \) \((^\circ\text{C}^{-1})\) at each transformation stage of unECAPed and ECAPed steels.

<table>
<thead>
<tr>
<th></th>
<th>unECAPed steel</th>
<th>ECAPed steel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stage A</td>
<td>(-0.40 \times 10^{-5})</td>
<td>Stage I (-1.79 \times 10^{-5})</td>
</tr>
<tr>
<td>Stage B</td>
<td>(-1.63 \times 10^{-5})</td>
<td>Stage II (-0.44 \times 10^{-5})</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Stage III (-2.01 \times 10^{-5})</td>
</tr>
</tbody>
</table>
rium solubility limit, (b) a preferred attachment of enriched carbon atoms on dislocation arrays residing at ferrite/cementite interfaces, and (c) an increase of vacancy concentration. More specifically, Hono and coworkers\textsuperscript{(16)} reported the 0.2–3 at\% carbon concentration in pearlitic ferrite of the fully pearlitic steel cold-drawn to an effective strain of $\sim$ 4.2. Sauvage et al.\textsuperscript{(15)} found that pearlitic cementite was replaced with nanocrystalline ferrite of 2–10 at\% carbon concentration, and the carbon concentration in pearlitic ferrite was about $\sim$ 0.5 at\%, without altering the lamellar structure, by high pressure torsion ($\sim$ 7 GPa) of a fully pearlitic steel. Considering the equilibrium carbon concentration of $\sim$ 0.01 at\% in ferrite, it is evident that a considerable amount of carbon-supersaturated ferrite is formed inside pearlite nodules by SPD. Ivanisenko et al.\textsuperscript{(19)} interpreted that such formation of carbon-supersaturated ferrite is possible by a mechanically driven carbon atom transport process during SPD. In addition to the formation of carbon-supersaturated ferrite inside pearlite nodules by mechanical diffusion, a recent thermal diffusion analysis\textsuperscript{(6)} revealed that proeutectoid ferrite in the vicinity of pearlite nodules is to be carbon-supersaturated concurrently by carbon diffusion through various defects at sufficiently high temperatures.

In Fig. 4, the pearlite morphology of unECAPed and ECAPed steels was studied. While unECAPed steel exhibited the well-developed lamellar structure, pearlitic cementite of ECAPed steel became thinner and even some degree of spheroidization occurred, implying the carbon dissolution from pearlitic cementite during the present ECAP. Accordingly, under the present ECAP conditions, i.e. temperature of 500°C and effective strain of $\sim$ 4, carbon dissolution from pearlitic cementite and carbon supersaturation in proeutectoid ferrite as well as pearlitic ferrite are likely to occur in the present ECAPed steel. A thermodynamic simulation using the Thermo Calc package informs that the equilibrium carbon concentration of austenite for the present chemical composition is $\sim$ 2.4 at\% at 730°C (measured $A_{1}$ temperature of ECAPed steel). Therefore, it is natural that ferrite carbon-supersaturated beyond 2.4 at\% by ECAP is first transformed to austenite ahead of undissolved pearlite$\rightarrow$martensite transformation in which carbon dissolution from cementite lamellae and its diffusion to ferrite to reach the equilibrium carbon content for austenite transformation are required. It is worth mentioning that the equilibrium carbon concentration in ferrite of ECAPed steel is lower than that predicted by the thermodynamic simulation, i.e. 2.4 at\%, since the equilibrium concentration of interstitial atoms is known to decrease significantly under pressure.\textsuperscript{(20)}

### 4.2. Effect of ECAP on $A_{1}$ Temperature

As discussed above, ECAP not only influences the reverse transformation sequences of ferrite/pearlite steel but also lowers its transformation temperatures. As a first speculation, the decrease of transformation temperatures of ECAPed steel seems to be attributed to the fact that various defects, e.g. dislocations with a high density, additional (sub)grain boundaries, etc., generated by ECAP provide more driving force and higher density of austenite nucleation sites for reverse transformation of ECAPed steel.

At this moment, a different approach is suggested to examine the effect of ECAP on $A_{1}$ temperature of the present steel. From the preceding session, it is evident that a certain amount of carbon-supersaturated ferrite exists in ECAPed steel. The nature of this carbon-supersaturated ferrite is to be very similar to that of conventional $\alpha'$ martensite in the three ways of carbon supersaturation, high dislocation density and a large amount of shear, all associated with ECAP. In addition, although the crystallography of both is different, their lattice structure is anticipated to be similar since the tetragonality of conventional $\alpha'$ martensite is close to unity, i.e. body centered cubic rather than body centered tetragonal, when the carbon content is small, typically less than $\sim$ 0.25 wt\%.\textsuperscript{(21)}

According to the Hornbogen’s analysis,\textsuperscript{(22)} the shear stress inducing transformation affects $\alpha'$ martensite$\rightarrow$austenite transformation temperature by

$$
\frac{\tau_{ab}}{\Delta T} = \frac{C \cdot d\tau_y}{\gamma_{ab}} = \frac{s_{ab}}{\gamma_{ab}} \quad \text{................................(2)}
$$

where $\tau_{ab}$ is the shear stress driving the transformation, $\Delta T$ is the change of transformation temperature, $d\tau_y$ is the yield stress increase, $C$ is the proportional constant assuming that $\tau_{ab}$ is linearly proportional to $d\tau_y$, $s_{ab}$ is the transformation entropy, and $\gamma_{ab}$ is the transformation shear strain. Considering the yield stress difference between unECAPed and ECAPed steels with $C$ = 1, Eq. (2) predicted that $A_{1}$ temperature of ECAPed steel is lowered by $\sim$ 26°C compared to that of unECAPed steel. For prediction, the following values are used as a first approximation:

1. $C$ = 1
2. $d\tau_y = 170$ MPa: $\tau_y = \sigma_y / \sqrt{3}$ (von Mises criterion), $\sigma_y$ of unECAPed steel = 300 MPa,\textsuperscript{(4)} and $\sigma_y$ of ECAPed steel = 600 MPa\textsuperscript{(6)}
3. $s_{ab} = -5.6$ J/mol·K: $s_{ab} = dF_{ab}/dT$, where $F_{ab}$ is the free energy change for ferrite$\rightarrow$martensite transformation in pure iron (the variation of $F_{ab}$ with tempera-
ture was taken from the linear portion of the Kaufman and Cohen data[^25]

(4) $\gamma_{ab} = 0.2$: typical shear strain involved in martensite transformation of low carbon steels[^24, 25]

The difference between the predicted and measured $A_{c1}$ temperature decrease, $\sim 26^\circ C$ and $\sim 10^\circ C$ respectively, mainly results from the use of the proportional constant $C = 1$. The actual $C$ is expected to be smaller than 1 since all external stress is not expected to be consumed for driving transformation. In spite of errors involved in the values of the parameters in Eq. (2), the estimation is in good agreement with the measured. This interpretation supports that carbon-supersaturated ferrite formed by ECAP, of which nature is similar to that of conventional $\alpha'$ martensite, transforms to austenite firstly during reverse transformation of ECAPed steel, originating the appearance of the stage I. By using the similar approach, Ivanisenko et al.[^18] reported the $A_{c1}$ temperature decrease by as much as $\sim 500^\circ C$ in a fully eutectoid steel after SPD: in their case, pearlitic cementite was completely dissolved by high pressure torsion with the shear strain of $\alpha' \sim 300$.

5. Summary

(1) Reverse austenite transformation of ultrafine grained low carbon ferrite/pearlite steel prepared by equal channel angular pressing was examined by using a dilatometry, and its behavior was compared with that of coarse grained counterpart.

(2) For coarse grained steel, reverse austenite transformation occurred with the two serial stages of pearlite $\rightarrow$ austenite followed by proeutectoid ferrite $\rightarrow$ austenite. By contrast, reverse austenite transformation of ultrafine grained steel consisted of the three serial stages of carbon-supersaturated ferrite $\rightarrow$ austenite, pearlite $\rightarrow$ austenite, and proeutectoid ferrite $\rightarrow$ austenite in the order. The formation of carbon-supersaturated ferrite in ultrafine grained steel is associated with carbon dissolution from pearlitic cementite and its concurrent diffusion into either pearlitic ferrite or proeutectoid ferrite during equal channel angular pressing.

(3) Reverse austenite transformation start and finish temperatures of ultrafine grained steel were lower than those of coarse grained steel. This can be explained in terms of the difference in shear stress required to drive transformation, which is directly related to yield stress, between two steels.

Acknowledgements

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REFERENCES