Delamination Toughening of Ultrafine Grain Structure Steels Processed through Tempforming at Elevated Temperatures

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The deformation of tempered martensitic structures, namely tempforming treatments, were applied to a 0.6C–2Si–1Cr steel at 500, 600 and 700°C using multi-pass caliber-rolling with an accumulated area reduction of 80%. The tensile and Charpy impact properties were investigated to make clear the relation between the microstructure and the delamination behavior of the tempformed (TF) samples. The tempforming treatments resulted in the evolution of ultrafine grain structures with strong (110)//rolling direction (RD) fiber deformation textures and fine spheroidized cementite particles distributions. In contrast to the ductile-to-brittle transition of the conventional quenched and tempered (QT) samples, the TF samples exhibited inverse temperature dependences of the impact toughness due to the delaminations, where the cracks branched in the longitudinal direction (//RD) of the impact test bars. As a result, high strength with excellent toughness was achieved in the TF samples. A yield strength of 1364 MPa and a V-notch Charpy absorbed energy of 125 J were obtained at room temperature in the sample that was tempformed at 500°C. The delamination was shown to occur due to the microstructural anisotropy of the TF samples, and the dominating factors controlling the delamination toughening were the transverse grain size, the grain shape and the (110)//RD fiber deformation texture. The discussion also indicated that the ultra refinement of the transverse grain structure was the key to enhancing both the yield strength and the toughness of the TF steel while lowering the ductile-to-brittle transition temperature.

KEY WORDS: thermo-mechanical treatment; ultrahigh strength steel; grain size; texture; ductility; strength; toughness.

1. Introduction

The two most important parameters which control the notch toughness of structural materials have long been recognized to be their intrinsic fracture resistance and the operative stress system under which the fracture occurs.1) The techniques that are generally known to improve the intrinsic fracture resistance of steels are: 1) the reduction of impurity elements such as P and S, and inclusions causing embrittlement, 2) the reduction of carbon, 3) the addition of alloying elements such as Ni, and 4) grain refinement. In Al–Li alloys2) and pipeline steels,3,4) delamination is known to relax the triaxial tension stresses generated by the localized plastic constrain at the notch and/or the crack tip ahead of advancing crack tips, leading to the improvement of notch toughness at low temperatures. Among them, the grain refinement5–10) and the delamination toughening3,4) are attractive techniques for lowering the ductile-to-brittle transition temperature (DBTT) without adding alloying elements. However, these techniques3–8) have tended to sacrifice the upper-shelf energy for both strengthening and lowering the DBTT of steels.

We have recently found that delaminations in ultrafine grain (UFG) structures that consist of elongated grains with strong (110)//rolling direction (RD) fiber deformation textures resulted in the enhancement of impact toughness of ultrahigh strength steels with yield strengths of 1400 MPa or more.11,12)
tured and high strength parts such as bolt. Especially in medium carbon steel, fine carbide particles dispersed intentionally in the tempered martensitic structure are helpful for suppressing grain coarsening during deformation at elevated temperatures, where hard and brittle martensitic structure becomes relatively ductile with a release of internal stresses. As a result, a large scale of parts with UFG structures can be obtained. We would like to call this thermomechanical treatment “tempforming” according to Tamura’s classification. The present study was undertaken to make clear the specific microstructural factors which control the delamination toughening in the UFG structure processed by the tempforming treatment at elevated temperature. A 0.6%C–2%Si–1%Cr steel, which does not contain any strong alloy carbide forming elements, was selected for the study. The tempforming treatments using multi-pass caliber-rolling were applied at 500, 600 and 700°C to obtain the UFG structures with different microstructural factors such as the transverse grain size, the austenitizing temperature for controlling the microstructural factors for the UFG structure steel processed by the deformation of a tempered martensitic structure.

Tokizane et al. developed the thermomechanical treatment involving deformation of lath martensitic structure in low carbon steels using cold plate rolling with an area reduction up to 84% mainly from the point of view of austenite grain refinement. Hayashi et al. successfully obtained an ultrafine equiaxed grain structure with an average grain size of 1 μm through multi-axial forging at 640°C for a Fe-2%Mn-0.05%C steel with a tempered martensitic structure. Nowadays, the deformation of martensitic structure at room temperature or tempered martensitic structures at elevated temperatures is gaining a lot of attention as an effective method for grain refinement, and the formation of ultrafine equiaxed grains through the deformation and subsequent annealing has been reported. Meanwhile, the authors have a strong motivation to use the thermomechanical treatment, which involves the deformation of a tempered martensitic structure, for a simultaneous “formation” of UFG structure and high strength parts such as bolt. Especially in medium carbon steel, fine carbide particles dispersed intentionally in the tempered martensitic structure are helpful for suppressing grain coarsening during deformation at elevated temperatures, where hard and brittle martensitic structure becomes relatively ductile with a release of internal stresses. As a result, a large scale of parts with UFG structures can be obtained. We would like to call this thermomechanical treatment “tempforming” according to Tamura’s classification. The present study was undertaken to make clear the specific microstructural factors which control the delamination toughening in the UFG structure processed by the tempforming treatment at elevated temperature. A 0.6%C–2%Si–1%Cr steel, which does not contain any strong alloy carbide forming elements, was selected for the study. The tempforming treatments using multi-pass caliber-rolling were applied at 500, 600 and 700°C to obtain the UFG structures with different microstructural factors such as the transverse grain size, the grain shape, the texture, and the carbide particle distribution. The tensile and Charpy impact properties of the UFG samples were investigated and compared to those of the conventional quenched and tempered samples.

2. Experimental
2.1. Material and Thermomechanical Treatment
A steel with a chemical composition of 0.57% C, 1.96% Si, 0.16% Mn, <0.001% P, <0.001% S, 0.041% Al, 1.02% Cr, 0.002% Mo, 0.0018% N, <0.0005% O and the balance Fe (all in mass%) was used in this study. A 100 kg ingot was prepared by vacuum melting and casting, homogenized at 1200°C, and then hot-rolled to a plate with a thickness of 4 cm. A 12 by 4 cm block was cut out of the plate, heated to 1200°C, hot-rolled to a square bar with a cross section area of 10 cm², solution-treated at 1200°C for 60 min, and water quenched to obtain a martensitic structure without any coarse undissolved carbides. The average prior-austenite grain size of the quenched bars was 210 μm and the hardness was HV 850. The quenched bars were tempered at 500, 600 and 700°C for 90 min, subjected to multi-pass caliber-rolling at the respective temperatures to square bars with a cross section area of 2 cm², and air cooled (tempformed (TF) samples). The accumulative reduction in area through tempforming was 80% in ten passes, which corresponds to an equivalent strain of 1.8. Note that the samples were held for 5 min in a furnace after every three passes during the rolling and passed through twice for the final groove to control the cross sectional shape of the bars, namely the pass schedule was 3-3-4 at each temperature. To obtain conventional quenched and tempered samples with the same tensile strengths for comparison, normalized bars were austenitized at 880°C for 30 min, followed by oil quenching, tempered at 500, 600 and 700°C for 90 min, and then water cooled (QT samples). The QT samples had an average prior-austenite grain size of 26 μm and showed random textures.

The principal axes of the squared bar in this study are defined as follows. The axis that is coincident with the rolling direction is defined as RD, the one that is coincident with the direction of the main working force at the final pass is defined as ND, and the one that is normal to the RD and the ND is defined as TD.

2.2. Microstructure Characterization and Mechanical Testing
The microstructures of the cross sections of the squared bars were observed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Electron back scattering diffraction pattern (EBSP) analysis was performed using the SEM equipped with a field emission gun. The average transverse linear interception was measured on two kinds of planes normal to the RD and the ND for the grain boundaries with misorientation angles of more than 15°. The average transverse grain size was converted by multiplying the transverse linear interception by the correction factor of 1.128. The grain shape aspect ratio distribution was analyzed on an area of 15×15 μm² for the grain boundaries with misorientation angles of more than 2°. To determine the carbide particle size, the sizes of 90–300 particles were measured in two to four fields of view for each sample. X-ray diffraction (XRD) was performed using a Cu target. The elastic strain (ε = σ/E) was estimated by the Hall–Williamson plots of the XRD physical line broadening for (110) and (220) reflections on the planes normal to the RD, where σ is internal stresses and E is the Young’s modulus.

The tensile tests were conducted at room temperature at a crosshead speed of 0.5 mm/min for round specimens with a gage length of 24.5 mm and a diameter of 3.5 mm that were machined in the RD (JIS14A specimens). A yield strength of 0.2% offset was reported. Charpy impact tests were carried out at temperatures from −196 to 227°C for full-size
2 mm U-notch specimens that were machined in the RD, and supplementally for full-size 2 mm V-notch specimens. The striking direction (SD) of the impact tests had an angle of $\sim 45^\circ$ to the TD and the ND. Small plate specimens with a parallel length of 4 mm, a width of 3 mm, and a thickness of 1 mm were also machined from the squared bars in the RD and in the SD and then were used for tensile testing at temperatures from $60$ to $150^\circ$C at a crosshead speed of 0.11 mm/min to investigate the anisotropy.

3. Experimental Results

3.1. Microstructures

Figure 1 shows the matrix ferrite grain structures on the cross sections of the planes that are normal to the RD and the ND in the TF samples. The sample that was tempformed at 500°C shows the feature of an UFEG structure that is composed of rod and ribbon shaped grains that are aligned to the RD. The inverse pole figure for the RD indicates the evolution of a strong (110)//RD fiber deformation texture. A similar (110)//RD fiber texture is usually observed in heavily cold drawn and/or swaged steel wires. As the tempforming temperature increases, both the area fraction and the size of the equiaxed grains increase. The matrix structure that was tempformed at 700°C is characterized by equiaxed grains, although the sample has a strong (110)//RD fiber deformation texture. The average transverse grain size was measured to be 0.34, 0.50 and 0.97 $\mu$m in tempforming at 500, 600 and 700°C, respectively. The XRD indicated that the elastic strain ($\varepsilon_\text{elastic} = \sigma/E \cdot 100$), which is considered to arise from dislocations and lattice distortions, showed a decrease from 0.32±0.04% to 0.22±0.02% as the tempforming temperature increased from 500 to 700°C. The $\varepsilon_\text{elastic}$ of the TF samples was almost similar to that of the QT samples at the same tempering temperature; the $\varepsilon_\text{elastic}$ of the QT samples was 0.36±0.04% and 0.24±0.01% in tempering at 500 and 700°C, respectively.

Figure 2 are TEM and SEM images showing morphologies and distributions of carbide particles of the TF and QT samples. Spheroidized cementite particles are dispersed in all of the TF samples. Relatively large cementite particles exist on the (sub) boundaries of the matrix ferrite grains, while finer spherical cementite particles are homogeneously dispersed inside the matrix grains. In particular, the cementite particles on the (sub) boundaries may play an important role in retarding the grain migration through their pinning effect during tempering and tempforming. The average aspect ratios of cementite particles were measured to be about 1.3 inside the grains and 1.5 on the (sub) grain boundaries,
respectively. The average lengths of the long axis for the cementite particles inside the grains and on the (sub) boundaries in tempforming at 500°C are 28 and 64 nm, respectively, and increased to 177 and 370 nm in tempforming at 700°C. It appears that the number of cementite particles inside the grains tends to decrease with increasing tempforming temperature. When compared at the same tempering temperature, there is no significant difference in the cementite particle size distribution between the QT and TF samples. This indicates that such bimodal distributions for the cementite particles in the TF samples might be inherited from those of prior tempered martensitic structures.

3.2. Tensile Properties at Room Temperature

Typical flow curves and the tensile properties for the JIS 14A specimens that were machined in the RD are displayed in Fig. 3. The upper yield points are observed in all of the TF samples and for the QT sample that was tempered at 700°C. The yield and tensile strengths in the sample tempformed at 500°C are 1364 and 1456 MPa, respectively, and decrease to 840 and 983 MPa in the sample tempformed at 700°C. While the uniform and total elongations and the reduction in area are 7.9, 14.7 and 41%, respectively, in tempforming at 500°C, they increase to 11.4, 21.8 and 58% in tempforming at 700°C. This trend is summarized as a function of tensile strength. When the tensile strength is the same, the yield strength is higher in the TF samples than in the QT sample, while the tensile elongations and the reduction in area of the TF samples are almost comparable to those of the QT samples.

Figure 4 shows the plots of the yield strength of the TF samples against the inverse square root of the average transverse ferrite grain size. The data for the 0.4C–2Si–1Cr–1Mo steel that was developed through the tempforming treatment12) are also shown in the figure. The data points...
fall roughly on a single line following the Hall–Petch relation for the present TF samples and for the 0.4C–2Si–1Cr–1Mo steel that was annealed at 700°C for 60 min after tempforming at 500°C. The Hall–Petch slope of the TF samples is similar to that of the low- and medium-carbon steels \(^{5,22,27–29}\) with the C content from 0.05 to 0.3 wt% and is independent of the C content (0.4–0.6% C), if the solid solution strengthening by 2% Si (~170 MPa \(^{30}\)) is added to the \(\sigma_0\). In the TF samples with UFEG structures that were processed at 500°C, the yield strength tends to deviate upward from the Hall–Petch relation. The deviation is more significant in the 0.4C–2Si–1Cr–1Mo steel with a distribution of nanometer-size carbide particles of 50 nm or less. Such upward deviation in the yield strength is probably related to the dense distribution of nanometer-size carbide particles and the high internal stresses. \(^{22}\) Here, we would like to emphasize that the yield strength along the RD strongly depends on the transverse ferrite grain size for the present TF samples.

3.3. Impact Properties

Figure 5 shows the results of the U-notch Charpy impact tests in the TF and QT samples. The QT samples typically exhibit the ductile-to-brittle transitions, where the uE decreases with testing temperature. Higher tempering temperatures result in higher upper-shelf energy and lower DBTT. The fractography of the QT samples revealed that quasi-cleavage fractures started to occur from 150 and 60°C in tempering at 500 and 700°C, respectively. The uE of both TF samples shows maximum values at temperatures from 20 to –20°C, where the QT samples undergo the ductile-to-brittle transitions. When compared at the same tempering temperature, the TF sample has a higher uE than that of the QT sample over the entire temperature range tested. Note that the delamination is much more striking in tempforming at 500°C than at 700°C, resulting in the marked increase in the uE. Additionally, the upper-shelf energy at 227°C is reduced from 137 to 90 J as the tempforming temperature decreases from 700 to 500°C.

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but the uE of the samples that were tempformed at 500°C is about two times higher than that of the QT samples with the same tempering temperature (uE=38J). Hence, the influence of tempforming on the uE becomes larger with decreasing tempforming temperature.

U-notch Charpy absorption energies at room temperature, where the delaminations are more pronounced in the present TF samples, are plotted in Fig. 6 as a function of yield strength. Data for commercial spring steels with the C content of 0.5–0.6% are also shown for reference. The uE of the quenched and tempered steels decreases dramatically with increasing tensile strength up to 1 300 MPa, then remains at 30–40 J. This trend has been well accepted in conventional low-alloy tempered martensite steels. By contrast, the uE of the TF samples tends to increase with yield strength, as a consequence of the delamination. Table 1 shows the vE and yield strength of the 0.4C–2Si–1Cr–1Mo and the 0.6C–2Si–1Cr steels with the UFEG structures that evolved through tempforming at 500°C. Compared to the 0.6C–2Si–1Cr steel, the 0.4C–2Si–1Cr–1Mo steel exhibits higher yield strength with higher vE in both ductile and delamination fracture regions. This suggests that the transverse planes normal to the RD are necessary to be more ductile and tougher to cause the delamination toughening.

Table 1. V-notch Charpy absorbed energy (vE) and yield strength of the samples with ultrafine elongated grain structures that were processed by tempforming at 500°C. The samples were subjected to impact and tensile testing at 20 and 150°C.

<table>
<thead>
<tr>
<th>Steels</th>
<th>at 20 °C</th>
<th>at 150 °C</th>
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<tbody>
<tr>
<td>0.6C–2Si–1Cr</td>
<td>vE=112, 127, 135 J</td>
<td>vE=63, 75, 84 J</td>
</tr>
<tr>
<td></td>
<td>YS=1364 MPa</td>
<td>YS=1056 MPa</td>
</tr>
<tr>
<td>0.4C–2Si–1Cr–1Mo</td>
<td>vE=216, 221, 241 J</td>
<td>vE=130, 134, 135 J</td>
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<tr>
<td></td>
<td>YS=1840 MPa</td>
<td>YS=1610 MPa</td>
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Figure 7 shows the representative fracture surfaces of the TF samples with and without the delamination toughening. In the TF samples exhibiting the marked delamination toughening “the terraces” are formed on the fracture surfaces roughly parallel to the RD and “the steps” are formed on the surfaces roughly normal to the RD. Such stepwise crack propagations have been observed in ausformed steels exhibiting similar delamination behavior. The fracture mode of the TF samples occurred primarily by a quasi-cleavage for the terraces, while a ductile fracture mode with fine dimple patterns took place for the steps. The presence of many steps provided the evidence for the extensive local plastic flow. The sample still showed a zigzag fracture feature at −196°C, where the uE decreases to 13 J. However, the quasi-cleavage fracture along the RD was less pronounced and the stepwise crack propagation that is accompanied by the ductile fracture was not observed. Therefore, the primary fracture mode is quasi-cleavage and is different from that of the sample with the delamination toughening. Macroscopically, the zigzag fracture paths appear to have an angle of ~45° to the SD and the RD. Such fracture features were commonly observed in all of the TF samples.

4. Discussion
4.1. Microstructural Factors to Control the Delamination Toughening

Thermomechanically treated steels, laminates and laminated composites that were processed through rolling were reported to exhibit delaminations due to their anisotropic microstructures that were oriented to the RD. Figure 8 presents examples of laminate geometries that show delamination toughening. When weak interfaces are present parallel to the longitudinal direction of the impact test bar, the interaction between the weak interfaces and the stress, σx or σy that is generated by the localized plastic constrain at the notch and/or the crack tip can cause
Delaminations. There are two basic geometries termed “crack divider” and “crack arrester”. The delamination at the weak interfaces divides the crack into a series of cracks in the crack divider geometry. This causes a relaxation of the triaxial tension towards a state of biaxial tension, lowering the DBTT. However, the ductile fracture that is accompanied by the delamination results in the reduction of upper-shelf energy, because the effective width of the specimen across the notch decreases. In the UFG structure steel plates that were produced by the plate rolling process, the notch toughness was often evaluated for the crack divider geometry. The delaminations of the UFG steel plates were observed to occur on the fracture surfaces of the impacted specimens, and the upper-shelf energies decreased despite the lowering of the DBTT with decreasing grain size. Meanwhile, the strengthening through the method of ultra grain refinement to 1 μm or less is often accompanied by a marked loss of tensile elongation, especially uniform elongation. Therefore, the occurrence of delamination along with the reduction in the tensile elongation is thought to correlate with the reduction in the upper-shelf energy in the UFG structure plates. On the other hand, in the crack arrester geometry, the delamination is thought to relax the triaxial stress conditions and to blunt the crack tip. To fracture a material, crack re-initiation is necessary and occurs under conditions of nearly uniaxial tension, which is an unfavorable cleavage. Hence, high absorbed energy is obtainable through the delamination. Inverse temperature dependences of VE due to the delaminations were observed in an austrformed 0.2C–3Ni–3Mo steel and austenitic stainless steel. The delamination behavior in the present TF samples is similar to that of the crack arrester geometry. The stepwise crack propagation in Fig. 7 appears to be the trace of the crack re-initiations.

Delamination has often been observed to occur in association with the texture, between the matrix and inclusions such as the elongated MnS and the carbides, that were aligned to the RD, etc. In an austrformed steel, the elongated prior-austenite grain boundaries were reported to act as the crack arrester. In the present TF steels, the amount of P, S, and inclusions was strictly reduced and the boundaries of the prior-austenite grains became difficult to recognize through the deformation of the tempered martensitic structures. The fracture mode for the crack branching planes was observed to occur primarily by a quasi-cleavage. Therefore, the observed delamination is attributed to the ultrafine grain structures that evolved through caliber-rolling based on a multi-directional deformation mode. [001] pole figures that were obtained from the EBSP analysis for the TF samples are shown in Fig. 9. Bcc iron cleaves on {100} planes. The (110)//RD fiber deformation textures provide lots of cleavage {100} orientations parallel to the RD and on the planes with the angle of ±45° to the RD and the SD of the impact bar, while {110} ductile orientations are present on the planes normal to the RD. As the tempforming temperature increases from 500 to 700°C, the texture intensity tends to decrease while the aspect ratio of the grains becomes smaller. Hence, one of the most dominating microstructural factors for crack branching is the texture. In addition, the effective grain size for the fracture is the coherence length on {100}, which corresponds to the cleavage crack length in bcc steel. Therefore, the critical value of the local stress for fracture, σ, and the effective grain size are roughly comparable on the same scale according to the Hall–Petch relation, i.e., σ=Kd−1/2. In the elongated grain structure, the coherence length on {100} is long along the RD due to the elongated grain shape. This means that the grain shape may also have a strong influence on crack branching.

Figure 10 shows the flow curves and tensile properties for the small plate specimens at room temperature. The longitudinal yield and tensile strengths of the small specimens and the JIS 14A specimens in the RD (Fig. 3) are quite similar. The elongation of the small specimens also decreases by increasing the strength, while they tend to be larger than those of the JIS 14A specimens. This may be caused by the differences in the shape and dimension between the specimens. As expected, the TF samples exhibit an anisotropic tensile behavior due to the microstructural anisotropy, in contrast to the QT sample with an isotropic microstructure. When this trend is summarized as a function of the tensile strength of the JIS 14A specimens, the transverse yield strength in the SD shows a downward deviation from the line for longitudinal yield strength in the RD. The decrement in the elongation with increasing yield strength is also more significant in the SD than in the RD. In particular, the poor transverse tensile ductility in the samples that were
tempformed at 500°C indicates a lower fracture stress on the planes that are parallel to the RD for the UFEG structure.

According to the Yoffee diagram, the ductile-to-brittle transition occurs when the peak tensile stress in the process zone of a crack tip ($\sigma_t$) exceeds the cleavage fracture stress ($\sigma_f$). The $\sigma_t$ scales with $\sigma_y$, and is of the order (3–5) $\sigma_y$. The thermal increment in the $\sigma_y$ with decreasing temperature eventually causes the cleavage fracture of bcc steel. Grain refinement increases both the $\sigma_t$ and the $\sigma_f$, however, the effect on the $\sigma_f$ is normally larger with a decrease in the DBTT. In an isotropic grain structure, the longitudinal $\sigma_f$ and $\sigma_t$ are the same as the transverse $\sigma_f$ and $\sigma_t$, and the fracture always initiates and propagates on the SD of the impact bar. As shown in Fig. 11, the $\sigma_y$ of the TF samples is shown to increase with decreasing testing temperature in both the RD and the SD. However, the increment in the $\sigma_y$ is higher in the RD than in the SD. Figure 12 illustrates the Yoffee diagram for the UFEG structure with a strong {110} fiber deformation texture. The $\sigma_f$ is much higher on the transverse planes (//SD) than on the longitudinal planes (//RD) due to the texture and elongated grain shape. Below $T_\gamma$, cleavage fractures on the longitudinal {100} planes preferentially occur, resulting in delamination, where cracks branch normal to the SD of the impact test bars. A thermal increment in the $\sigma_y$ with decreasing temperature can eventually promote the delamination. As a result, the absorbed energy is enhanced with decreasing testing temperature (line (iii)). However, lots of {100} cleavage orientations are also on the planes with an angle of $\pm 45^\circ$ to the RD and the SD (Fig. 9). Below $T_\gamma$, the $\sigma_y$ is even greater, and the cleavage fracture is also induced on the {100} cleavage planes with the transverse components, leading to a diminishing of delamination toughening (line (ii)). This is supported roughly by the fractography in Fig.
7. If the toughness anisotropy between the SD and the RD is smaller, the delamination toughening becomes less pronounced. The influence of the tempforming temperature on the delamination behavior in Fig. 5 can be satisfactory rationalized by considering the toughness anisotropy.

Therefore, we can conclude that the combination of the microstructural factors, i.e., the transverse grain size, the grain shape and the (110)/RD fiber texture can control the delamination toughening in the bcc steel. The ultra refinement of the transverse grain structure is the key to simultaneously increasing the yield strength and the delamination toughening effect of the steel while lowering the DBTT in the UFEG structure.

4.2. Upper-shelf Energy of the UFG Structure Steels

The increase in yield strength is usually accompanied by a loss of upper-shelf energy as well as ductility. However, the principal advantage in the UFEG structure that is processed by caliber-rolling at elevated temperatures is the improvement of delamination toughening at low temperatures without a significant loss of upper-shelf energy. This trend is also observed in the present TF samples compared to that of the QT samples (Fig. 5). Figure 13 shows the fractographs of the TF and QT samples at the tempering temperature 500°C. At 227°C, the TF sample exhibits a fine and homogeneous dimple pattern, while the fracture surface of the QT sample consists of fine dimples and large, shallow dimples. This indicates the occurrence of more extensive plastic deformation in the TF sample. Inoue et al. have recently confirmed in a 0.15C–0.3Si–1.5Mn steel that the UFG sample with a strong (110)/RD fiber texture showed the same upper-shelf energy (=250J) as that of a coarse grained sample, despite the yield strength of the UFG sample (=857 MPa) being 2.4 times higher than that of the coarse grained sample. Baczynski et al. pointed out that in a low carbon pipe line steel (X80), the upper-shelf energy increases as the volume fraction of the {110} ductile planes. Hence, the high upper-shelf energy in the UFEG structure is probably due to the ultrafine transverse grain structure with lots of ductile {110} orientations. Furthermore, the comparison of the vE between the 0.4C–2Si–1Cr–1Mo and the 0.6C–2Si–1Cr steels in Table 1 indicates that the carbide structure has a strong effect on both the upper-shelf energy and the delamination toughening of the UFEG structure, because the transverse grain size (0.29–0.34 µm) and the texture intensities (l_{max}=7–9) are almost the same. In other words, since ductile fracture occurs by the growth and coalescence of voids typically nucleated at carbide particles, both the volume and the size of the carbide particles should be minimized to toughen the transverse plane. However, there is a lack of data on this subject. Thus, more detailed investigations on the toughening mechanism at the ductile fracture region are necessary in association with the microstructural factors of the UFG structure, i.e., the transverse matrix grain size, the texture and the carbide particle distribution.

5. Conclusions

The microstructural factors for controlling the delamination toughening were investigated in ultrafine grain (UFG) structures that were processed by the tempforming treatments at 500 to 700°C using multi-pass caliber-rolling with an accumulative reduction in area of 80% for a 0.6%C–2%Si–1%Cr steel. The main results are summarized as follows:

(1) An ultrafine elongated grain (UFEG) structure with a strong (110)/RD fiber deformation texture and a dispersion of spheroidized cementite particles evolved from the tempforming treatment at 500°C. As the tempforming treatment increased from 500 to 700°C, the transverse grain size increased from 0.34 to 0.97 µm in response to the growth of the cementite particles, and the matrix grain shape changed from an elongated one to an equiaxed one.

(2) The yield and tensile strengths in the sample tempformed at 500°C are 1364 and 1456 MPa, respectively, and decrease to 840 and 983 MPa in the sample tempformed at 700°C. When the tensile strength is the same, the yield strength is higher in the tempformed (TF) samples than in the quenched and tempered (QT) sample, while the tensile elongations and the reduction in area of the TF samples are almost comparable to those of the QT samples.

(3) The inverse temperature dependences of the absorbed energies that were responsible for the delaminations, where the crack branched parallel to the longitudinal direction of the impact test bar, were demonstrated in the UFG samples. The influence of the tempforming temperature on both the upper-shelf energy and the delamination toughening became larger with decreasing tempforming temperature. As a result, higher strength with better impact toughness was achieved in the TF samples than in the QT samples.

(4) The combination of the microstructural factors, i.e., the transverse grain size, the grain shape and the (110)/RD fiber deformation texture were able to control the delamination toughening in the UFG steel. The ultra refinement of the transverse grain structure was the key to increasing the yield strength and the delamination toughening effect of steel simultaneously while lowering the DBTT in the UFG.
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