Brittle Fracture Stress of Ultrafine-Grained Low-Carbon Steel

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The variation of brittle fracture stress on grain size in a wide range of 138 μm to 1 μm was studied in low-carbon steel. The steel bars with an ultrafine elongated grain structure and equiaxed grain structure were fabricated by caliber rolling. The brittle fracture stress was quantitatively estimated through a three-point bending test at 77 K with a notch sample and finite element analysis. A reduction in grain size leads to higher fracture stress, compared with yield strength, and steel with a grain size of 1 μm was estimated to have a very high fracture stress of 6.8 GPa. It has been shown that significant improvement of brittle fracture stress is one of the advantages of grain refinement.

1. Introduction

It is well known that yield strength in structural metallic materials increases with the refinement of crystal grains.1–4) The improvement of brittle fracture stress as well as the yield strength is one of the advantages of grain refinement. In the Charpy impact tests, a refinement in grain size leads to a lower ductile-to-brittle transition temperature (DBTT).5,6) The ductile-to-brittle transition in body-centered cubic steels is interpreted as a result of competition between tensile stress near the crack/notch tip, \( \sigma_t \), related to the yield strength, \( \sigma_y \), and brittle fracture stress, \( \sigma_F \). The \( \sigma_y \) increases at low temperatures, and the \( \sigma_F \) is independent of temperature. The DBTT occurs when the \( \sigma_y \) exceeds the \( \sigma_F \), the steels normally become brittle, and most of their toughness is lost. Since grain refinement effectively enhances the \( \sigma_F \), the DBTT is lowered due to a more significant increase in the \( \sigma_y \).5–7) However, the variations of brittle fracture stress on grain size in fine-grained steels have not been reported systematically, compared with those of the yield strength. This is because it is difficult to create a bulk sample with a fine-grained structure and because the \( \sigma_F \) must be quantitatively evaluated from the results extracted with numerical simulation including sample size and material data.8)

In the present study, a quasi-static three-point bending test on an ultrafine elongated grain low-carbon steel fabricated by multi-pass warm caliber rolling is conducted at 77 K, and the brittle fracture stress is quantitatively estimated through finite element simulation. The relation between brittle fracture stress and grain size in a wide range of 138 μm to 1 μm was studied including data reported by other researchers, on the basis of the Griffith equation.

2. Experimental Procedure

A low-carbon steel with a chemical composition of 0.15 C, 0.3 Si, 1.5 Mn, 0.001 P, 0.001 S, 0.029 Al, 0.0017 N, 0.002 O, and the balance Fe (all in mass%) was used in this study. A 150 kg ingot was prepared by vacuum melting and casting, homogenized at 1473 K, hot-forged to a 100 mm round bar, hot-rolled to a 40 mm-square bar, and then cut to 110 mm in length. The bar was hot-rolled until a 12.9 mm-square bar soaked at 1173 K for 1 h, followed by air cooling (hereafter designated as the CG sample). The average size of the ferrite grain, \( d_{\text{f}} \), in Fig. 1(a) was approximately 18 μm, and the Vickers hardness was HV(9.8N) = 146 ± 4. To create fine grained structures, after the hot-rolled 40 mm-square bar is soaked at 1173 K for 1 h, it was rolled to form a 14.3 mm-square bar, followed by air cooling (hereafter designated as the FG sample). The \( d_{\text{f}} \) in Fig. 1(b) was approximately 10 μm, and the hardness was HV(9.8N) = 147 ± 9. Next, to obtain an ultrafine-grained structure, the 40 mm-square bar was austenitized at 1173 K for 1 h, followed by air cooling and water quenching. The air-cooled and water-quenched bars, 40 mm square and 100 mm long, showed the microstructures of ferrite-pearlite and martensite (and/or bainite). The two bars were soaked at 773 K for 1 h and then subjected to a caliber-rolling simulator of the square/square type without any lubricant.10) Eventually, the 14.3 × 14.3 × 860 mm³ rolled bars were produced under a total reduction in area of about 87%. The bars passed through the final groove were immediately water-quenched. Hereafter, the bar warm-rolled after air cooling is designated as the ART sample (HV(9.8N) = 255 ± 4), and the bar warm-rolled after water quenching is designated as the WRT sample (HV(9.8N) = 266 ± 4). These bars composed of an elongated grain structure, and the average transverse grain sizes, \( d_{\text{trans}} \), were 1.3 μm and 1.0 μm, respectively, in Fig. 1(c), (d). The sizes were measured from the orientation maps through SEM/EBSP analysis with misorientation angles of more than 15°, though the maps are not shown in this paper.

Specimens used in a three-point bending test were taken from all rolled bars, as shown in Fig. 2(a). In the CG and FG samples with equiaxed grains, rectangular bars of 10(W) × 10(B) × 55(L) mm were machined along the RD (Fig. 2(b)), and then a notch with a depth of \( a_0 = 5 \) mm and two root radii of \( r = 0.13 \) and 0.25 mm was introduced by electro-discharge machining. In the case of the ART and WRT samples with elongated grains, it is impossible to evaluate the brittle fracture stress because the crack propagated vertically to the LD, i.e., crack branching occurs.11,12) Hence, specimens in the ART and WRT samples were taken from the rolled bars with 90° rotation along the RD, both ends of the samples

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were bonded to a low-carbon steel through electronic beam welding (EBW), and then a similar initial notch was introduced, as shown in Fig. 2(b). A three-point bending test with a support distance, \( S \), of 40 mm at a crosshead speed of 0.5 mm min\(^{-1}\) was conducted at 77 K. The maximum tensile stress near the initial notch, which corresponds to the fracture stress, was evaluated using finite element analysis (FEA) including sample size and strain-stress curves at 77 K. Full details of the specimen preparation, bending test, and FEA were given earlier.\(^{13,14}\) All the values presented represent an average of two or three measurements.

3. Results and Discussions

Figure 3(a) shows the relation between bending load \( P \) and displacement \( u \) in the WRt sample. The samples catastrophically fractured with a peak loading, \( P_{\text{max}} \), of 2.06 kN for \( \rho = 0.13 \) mm and 3.18 kN for \( \rho = 0.25 \) mm. The increase in \( P_{\text{max}} \) with \( \rho \) is due to the reduction in stress concentration near the notch. It is found that the \( P - u \) relations obtained from the FEA (solid lines) are in good agreement with the experimental results (dashed lines). Figure 3(b), (c) shows the distributions of stresses in each direction and the equivalent strain, \( \varepsilon_{\text{eq}} \), near the notch tip at \( u \), obtained from FEA. The tensile direction of the maximum stress near the notch corresponds to the \( x \) direction, and this \( \sigma_{xx} \) means the brittle fracture stress vertical to RD, \( \sigma_{F:RD\text{WRt}} \). Its value is 1626 MPa at \( \rho = 0.13 \) mm and 1707 MPa at \( \rho = 0.25 \) mm. Three measurements for each \( \rho \) were conducted, and the average was \( \sigma_{F:RD\text{WRt}} = 1595 \) MPa. Similarly, the all samples other than the WRt sample catastrophically fractured with peak loading, and the crack propagated directly across the center part of the test pieces. Figure 1(e), (f), (g), (h) shows SEM micrographs of the fracture surface in each sample. Here, the fracture surface was observed at the position from the notch tip where \( \sigma_{xx} \) becomes maximum, as shown in Fig. 3(b), (c). For the CG and FG samples, Fig. 1(e), (f) shows a typical brittle fracture surface, it can be seen that the facet size in the cleavage fracture for the FG sample is smaller than that for the CG sample. On the other hand, the fracture surface for the ART and WRt samples shown in Fig. 1(g), (h) is characterized by a cleavage attributed to the elongated grain structure. A brittle fracture occurs when the effective yield stress, \( \sigma_{\text{eff}} \), in the process zone at the notch tip exceeds the cleavage fracture stress. According to the Griffith equation, the \( \sigma_{F} \) is a function of the surface energy of the fracture, \( \sigma_{n} \), the crack size (in this case, it is replaced by the effective grain size, \( d_{\text{eff}} \)), Young’s modulus, \( E \), and Poisson’s ratio, \( \nu \).\(^{15}\) It is represented by
Fig. 3 (a) Comparison between bending load $P$ – displacement $u$ relations obtained by experiments and FE analysis in the WRt sample at 77 K and (b) distributions of stresses in each direction and the equivalent strain near the initial notch tip at $u = 0.108$ and 0.166 mm, respectively, obtained by FE analysis. Here, $r$ denotes the root radius of the initial notch.

\[
\sigma_F = \frac{2E\gamma_s}{\pi(1-\nu^2)} \cdot d_{eff}^{-0.5}.
\]

Actually, a test piece is a finite body. Hence, a shape factor depending on the fracture test method must be taken into account in eq. (1). Under the present three-point bending condition, the $\sigma_F$ is expressed as follows:

\[
\sigma_F = F(\xi) \cdot \frac{2E\gamma_s}{\pi(1-\nu^2)} \cdot d_{eff}^{-0.5}.
\]

Here, $F(\xi)$ denotes a shape factor determined by the initial notch length, $a_0$, and specimen thickness, $W$, and under $S/W = 4$ it is defined by:

\[
F(\xi) = 1.090 - 1.735 \xi + 8.2 \xi^2 - 14.18 \xi^3 + 14.57 \xi^4,
\]

where $\xi = a_0/W$. In the present study, $F(\xi) = 1.41$ at $\xi = 0.5$. Generally, in the case of the CG and FG samples with equiaxed grains, the $d_{eff}$ corresponds to the ferrite grain size. The difference in facet size observed in Fig. 1(e), (f) is attributed to the difference in ferrite grain size in both samples. The $\sigma_{FG(CG)}$ and $\sigma_{FG(FG)}$ in these samples were quantitatively determined from FEA. On the other hand, in the case of the ARt and WRt samples with elongated grains, the $d_{eff}$ (in this case, $d_{effL}$) for the fracture stress, $\sigma_{F,LD}$, vertical to the RD shown in Fig. 3(b), (c) corresponds to the average grain size, $d_{avgL}$, in a longitudinal direction parallel to the RD (see inset in Fig. 4). It is difficult to accurately measure the $d_{avgL}$ size using the EBSP maps and SEM images of the fracture surface, as shown in Fig. 1(g), (h). However, it is possible to calculate the $d_{effL}$ from the results of the equiaxed grain sample and $\sigma_{F,LD}$ under the same chemical composition. Assuming that the $\gamma$, $E$, and $v$ in the CG and WRt samples are the same, the effective grain size, $d_{effL(WRt)}$, of the WRt samples is described as

\[
\sigma_{F(\gamma)} \sqrt{d_{effL(WRt)}} = \sigma_{F,LD(WRt)} \sqrt{d_{effL(WRt)}} \rightarrow d_{effL(WRt)} = d_{effL(WRt)} \left( \frac{\sigma_{F(\gamma)}}{\sigma_{F,LD(WRt)}} \right)^2.
\]

Using $d_{effL(CG)} = 18.0 \mu m$, $\sigma_{F(CG)} = 1612 \, MPa$, and $\sigma_{F,LD(WRt)} = 1595 \, MPa$, the $d_{effL(WRt)}$ is estimated to be 18.4 $\mu m$. Next, the brittle fracture stress, $\sigma_{F,RD(WRt)}$, parallel to the RD is expressed by

\[
\sigma_{F,RD(WRt)} = \frac{\sigma_{F,L(\gamma)} \sqrt{d_{effL(WRt)}}}{\sigma_{F,L(RD(WRt))}} \sqrt{d_{effL(WRt)}} \rightarrow \sigma_{F,RD(WRt)} = \frac{\sigma_{F,L(\gamma)} \sqrt{d_{effL(WRt)}}}{\sigma_{F,L(RD(WRt))}} \sqrt{d_{effL(WRt)}}
\]

Hence, using $d_{effL(WRt)} = 1.0 \mu m$, $\sigma_{F,L(RD(WRt))} = 6842 \, MPa$. In the ARt sample, the brittle fracture stress, $\sigma_{F,L(RD(ARt))}$, and the effective grain size, $d_{effL(ARt)}$, parallel to the RD are estimated through a similar approach. In Fig. 4, these fracture stresses are plotted as a function of the inverse square root of $d_{eff}$, together with data for ferritic steel obtained by a fracture test and FEA in Refs. 8) and 9). The effective surface energy is $158 \, J/m^2$ from this linear relation and eq. (2). Here, $E$ is taken as 219 GPa, and $v$ is taken as 0.286, as for a low-carbon steel at 77 K.\(^{(17)}\) This value is included in a range of 90–190 J m\(^{-2}\) for C-Mn steels with a polygonal ferrite microstructure and for micro-alloyed ferrite-pearlite steels.\(^{(18,19)}\)

Interestingly, ultrafine-grained steel with grain size of 1 $\mu m$ has a very large fracture stress of approximately 6.8 GP. If this linear relation, $\sigma_F = 6.8 \cdot d_{eff}^{-0.5}$, is assumed to be maintained for finer grains, the fracture stress becomes about 15 GPa at $d_a = 0.2 \mu m$ and 22 GPa at $d_a = 0.1 \mu m$. These values correspond to the ideal strength, $E/10 – E/15$, of a steel. Moreover, according to the Hall-Petch relationship, the yield stress increases with $k \cdot d_a^{0.5}$. The coefficients $k$, which corresponds to a slope in this relation, was 0.4 in the present study, and this is consistent with the result found in the literature.\(^{(2,3)}\) Hence, a slope, 6.8, in the $\sigma_F – d_{eff}^{0.5}$ relation is ten times larger than $k$ in the Hall-Petch relationship. This means that the DBTT is significantly improved by grain refinement.

4. Conclusion

The present results demonstrated grain size dependence on brittle fracture stress in a 0.15C-0.3Si-1.5Mn steel through a three-point bending test at 77 K with a notch sample and numerical simulation. The fracture stress, $\sigma_F$, and...
effective grain size, $d_{\text{eff}}$, had a relation of $\sigma_F \approx 6.8 \cdot d_{\text{eff}}^{-0.5}$, and ultrafine-grained steel with a grain size of 1 μm was predicted to have a very large fracture stress of 6.8 GP. Significant improvement of brittle fracture stress is one of the advantages of grain refinement, in addition to strengthening.

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