1. Introduction

Strong and tough steels are always in demand to reduce weight and improve safety in transportation and to improve performance in heavy machinery. Thus, embrittlemet of steels has been intensively studied for many years. There are two types of embrittlement in low alloy structural steels, hardening embrittlement and non-hardening embrittlement. Non-hardening embrittlement is mainly caused by the grain boundary segregation of impurity elements during tempering or in long term application at elevated temperatures. Phosphorus (P) is a major embrittler in steels, which always exists at some level, and its strong segregation to the grain boundaries enables the steels to fracture intergranulary by decreasing the grain boundary cohesion, resulting in a decrease in the upper shelf energy and an increase in the ductile-to-brittle transition temperature (DBTT).

Intergranular cracking along prior austenite grain boundaries is generally observed in conventional quenched and tempered (QT) steels with grain boundary P segregation, even though the tempered martensitic steels contain other kinds of high angle boundaries within a prior austenite grain; the so called packet and block boundaries. This cracking occurs due to the continuity of prior austenite grain boundaries. The austenite grain boundaries having formed at a high temperature are a relatively coarse polyhedral network in which the dihedral angles are about 120°. A crack passing along this network is thus deflected through an average angle of 60° whenever it encounters a three grain junction. Thus, the energy absorbed during intergranular fracture of a polycrystal material increases as the grain size decreases and the angle of deflection increases beyond 60°, as reported by Ohtani and McMahon. Moreover, grain refinement has another merit for grain boundary segregation embrittlement to decrease impurity segregation because of increase of grain boundary area per unit volume.

According to the above suggestions, there have been several studies on the effect of grain refinement on grain boundary segregation embrittlement. Tsuji et al. produced an ultrafine equiaxed grain structure by the accumulative roll bonding (ARB) process in a 0.041% P added IF steel and carried out the miniaturized Charpy impact tests. They found that the DBTT is successively decreased by the grain refinement but the upper shelf energy is reduced at the same. A similar result was reported by Jia et al. The reduced upper shelf energy by grain refinement has been reported in the thermo-mechanically processed steel as well as the electrodeposited cobalt. Thus, we need to find a new microstructure to achieve both the suppression of intergranular fracture and the enhancement of upper shelf energy.

Recently, Kimura et al. reported that ultrafine elon-
gated grain (UFEG) structures with a strong (110)// rolling direction (RD) deformation texture were obtained in high strength steel bars through the calibre-rolling of tempered martensite (tempforming) and the UFEG structures showed an increase in the upper shelf energy in addition to a significant decrease in the DBTT for cleavage fracture. Since elongated grain shape along the longitudinal direction of an impact specimen gives rise to much larger deflection angles than 60° when crack propagation along grain boundary takes place,5) the UFEG structure is expected to suppress intergranular fracture markedly. Therefore, in the present study, we have tried to produce the UFEG structure to a P-doped high strength steel in order to investigate whether it is possible to obtain both the suppression of intergranular fracture and the enhancement of upper shelf energy in a brittle high strength steel at a conventional QT condition. Peculiarities of the microstructure and the fracture behavior and their relation are discussed in some details.

2. Experimental

2.1. Material and Heat Treatment

Two steels with the basic chemical compositions of 0.4% C, 0.25% Si, 0.73% Mn, 0.001% S, 1.0% Cr, 0.2% Mo, and P contents of 0.001 and 0.053% (all in mass %) were selected for the present study. Their chemical compositions are shown in Table 1, which are similar to that of a high strength JIS SCM 440 (AISI 4140) steel. Note that 0.053% P is more than twice as high as the typical P content of approximately 0.02% of a SCM 440 steel. Hence the 0.053% P steel was selected as a brittle steel.

Two 100 kg ingots with different P contents were prepared by vacuum induction melting and casting. The ingots were homogenized at 1473 K, and hot-rolled to plates with a thickness of 4 cm. Blocks of 12×4×4 cm³ were cut out of the plates, heated to 1473 K for 1.8 ks, and hot-rolled to squared bars with a cross-section area of 10 cm². The bars were solution-treated at 1193 K for 3.6 ks, followed by quenching to produce martensitic structures. Then they were tempered at 823 K for 3.6 ks, to obtain the same level of P segregation and strength.

2.2. Microstructure Characterization and Mechanical Testing

The microstructures in the central part of the cross-section in the squared bars were observed using the electron backscattering diffraction (EBSD) method with a scanning electron microscope (SEM) equipped with a field emission gun operated at 20 kV. The EBSD analysis was conducted using a LEO Gemini 1550 equipped with a TSL-OIM analytical system. The fracture surfaces were investigated by SEM and light microscopy.

All the mechanical test specimens were cut from the squared bars along the rolling direction (RD). The tensile tests were conducted at room temperature (RT) in an Instron machine for round specimens with a 30 mm gage length and a 6 mm gage diameter at a crosshead speed of 0.85 mm min⁻¹. Three tensile specimens were tested for each sample with the exception of the 0.05% P-QT sample with two specimens. The Charpy impact tests were carried out with full-size 2 mm V-notch specimens at RT. Three specimens were tested for each sample. The striking direction (SD) in the impact test had an angle of approximately 45° to the transverse direction (TD) and the normal direction (ND) of the squared bars. A figure for the principal axes of the rolled bar appears in 21, 22).

3. Experimental Results

3.1. Microstructure

Figure 1 shows the grain boundary maps obtained from the EBSD analysis on the cross-section of the plane along the longitudinal direction for the QT sample. The typical tempered martensitic structures with a prior austenite grain size of 30 μm are seen and any significant difference is not recognized between the 0.001% P and 0.053% P samples. The average linear intercept of high angle grain boundaries with misorientation angles larger than 10° is 453 and 455 nm for the 0.001% P and 0.053% P steels, respectively. The grain boundary area per unit volume (Sv) measured in Figs. 1(a) and 1(b) was 4.80×10⁶ m⁻² and 4.81×10⁶ m⁻², respectively. Note that the tempered martensitic structures

| Table 1. Chemical compositions of steels (mass%). |
|--------|--------|--------|--------|--------|--------|--------|--------|
| C      | Si     | Mn     | P      | S      | Cr     | Mo     | O      |
|--------|--------|--------|--------|--------|--------|--------|--------|
| 0.001% P steel | 0.40   | 0.24   | 0.73   | 0.001  | <0.001 | 1.04   | 0.22   | 0.0006 |
| 0.053% P steel | 0.42   | 0.25   | 0.72   | 0.053  | 0.001  | 0.98   | 0.20   | 0.001  |

Fig. 1. Grain boundary maps of (a) 0.001% P-QT and (b) 0.053% P-QT samples.
before the calibre-rolling were similar to those in Fig. 1.

**Figure 2** shows the grain boundary maps for the TF samples on the cross-section of the plane along the RD (Figs. 2(a), 2(b)) and the plane normal to the RD (Figs. 2(c), 2(d)). Fine and elongated grain structures are observed along the RD and the prior austenite grain boundaries can no longer be recognized in Figs. 2(a) and 2(b). The average transverse linear intercept of the high angle grain boundaries was 490 nm and 411 nm for the 0.001% P and 0.053% P steels, respectively. When the EBSD analysis was conducted on the cross-section plane normal to the RD (Figs. 2(c), 2(d)), it is seen that the grain are still elongated and curved but the aspect ratio of a grain, length to width, is smaller than that in Figs. 2(a) and 2(b). This indicates that the TF samples consist of ribbon-shaped grains. These deformation microstructures must be associated with the original lath martensite structure before the deformation (Fig. 1). The average linear intercept of the high angle grain boundaries was 253 nm and 212 nm for the 0.001% P and 0.053% P steels, respectively. With a consideration for the anisotropic ribbon-shaped grain structure, the $S_v$ was calculated to be $5.32 \times 10^6 \text{m}^{-1}$ and $6.54 \times 10^6 \text{m}^{-1}$ for the 0.001% P and 0.053% P steels, respectively. In this calculation, a rectangular grain shape was assumed.

The texture orientation analysis revealed the evolution of a strong $(110)//\text{RD}$ fibre deformation texture in the TF samples of both steels which is similar to the results of the rolled bars reported by Kimura et al.\textsuperscript{19–21} The typical inverse pole figures for the RD of the TF samples are shown in Fig. 3.

These results indicate that an evolution of a similar UFEG structure with a strong $(110)//\text{RD}$ fibre deformation texture was succeeded in SCM 440 type steels regardless of the P content.

### 3.2. Mechanical Property

The tensile properties at RT of the QT and TF samples are summarized in **Table 2**. Typical engineering stress–strain curves of the QT and TF samples are shown in **Fig. 4**. In general, all the samples showed a similar tensile strength and a total elongation of approximately 1 200 MPa and close to 15%, respectively. However, it is noted as for difference that the TF samples exhibited slightly higher values in strength and elongation and that the 0.053% P-QT sample showed a smaller elongation of 13%.

**Figure 5** shows the absorbed energy in the V-notch impact tests at RT for the QT and TF samples as a function of P content. The absorbed energy decreased considerably from 93 to 23 J with increasing P content in the QT samples. However, the TF samples with the UFEG structures did not show any degradation with increasing P content. A high absorbed energy of 150 J, 1.6 times the value of the 0.001% P-QT sample, was obtained regardless of the P content.

**Figure 6** shows the SEM fractographs in the area beneath the V-notch for the QT and TF samples. Note that the magnification is different among the photos. For the QT samples, the 0.001% P steel showed a dimple fracture (Fig. a).

*Table 2. Tensile properties of the QT and TF samples. W is the plastic work per unit volume for fracture.*

| P Content | Yield strength $\sigma_y$ (MPa) | Tensile strength $\sigma_u$ (MPa) | Uniform elongation $\varepsilon_u$ (%) | Total elongation $\varepsilon_t$ (%) | Reduction in area $\delta$ (%) | W $10^3$/m$^3$ |
|-----------|-------------------------------|---------------------------------|-----------------------------------|-----------------------------------|-------------------------------|----------------|
| 0.001% P  | 999 ±27                       | 1122 ±16                        | 5.4 ±0.5                          | 15.6 ±1.2                        | 59.5 ±1.3                     | 16.140.1       |
| TF        | 1127 ±22                      | 1168 ±27                        | 6.6 ±0.1                          | 16.1 ±0.6                        | 60.1 ±0.9                     | 17.721.1       |
| 0.053% P  | 1051 ±7                       | 1171 ±19                        | 6.2 ±0.1                          | 13.1 ±0.1                        | 47.6 ±2.8                     | 14.961.6       |
| TF        | 1138 ±246                     | 1201 ±27                        | 6.5 ±0.6                          | 15.7 ±0.3                        | 53.9 ±5.8                     | 17.028.1       |
6(a)), whereas the 0.053% P steel exhibited a combination of dimple and intergranular fractures (Fig. 6(b)). The brittle facet size of the 0.053% P steel corresponds to its prior austenite grain size of 30 μm. This result indicates that the embrittlement of prior austenite grain boundaries by P segregation occurred in the present P-doped SCM 440 type steel with a strength level of 1 200 MPa and caused the absorbed energy to decrease to 23 J as shown in Fig. 5. On the other hand, the fracture surface is characterized by a fine dimple structure in the TF samples for both steels (Figs. 6(c), 6(d)). The intergranular fracture observed in the 0.053% P-QT sample (Fig. 6(b)) is completely suppressed in the 0.053% P-TF sample (Fig. 6(d)).

Figure 7 shows a low magnification fractographs taken with a light microscope. Large shear lips with a width of approximately 2 mm are seen for 0.001% P samples (Figs. 7(a), 7(c)) and 0.053% P-TF samples (Fig. 7(d)), while shear lips are reduced to less than 1 mm in width for 0.053% P-QT sample (Fig. 7(b)). Note that delamination/splitting fracture of a crack arrester type\(^23-25\) is seen in the 0.053% P-TF sample. To show the appearance of delamination more clearly, the optical microscope images on the cross-section along the RD and SD in the central part of the TF samples were taken (Fig. 8). Significant delamination of a crack arrester type indicated by arrows is clearly recognized in the 0.053% P-TF sample (Fig. 8(b)) but not in the 0.001% P-TF sample (Fig. 8(a)).

4. Discussion

4.1. Suppression of Intergranular Fracture

The intergranular fracture caused by P segregation to grain boundaries could be suppressed through the introduction of a UFEG structure with a strong (110)//RD fibre deformation texture in a 1 200 MPa-class medium-carbon low-alloy steel with 0.053% P (Figs. 5 and 6). This suppression of intergranular fracture should be discussed with concentration of P at grain boundaries, effective grain size, as well as grain shape.

Since the P content of 0.053% and the final tempering condition of 823 K for 3.6 ks were constant for the QT and TF samples, the P concentration at grain boundaries may be associated only with grain boundary area per unit volume (Sv) of the samples. Note again that the Sv was somewhat larger in the TF sample (6.54×10⁸ m⁻¹) than in the QT sample (4.81×10⁸ m⁻¹) in the 0.053% P steel. Thus, the P concentration at the high angle grain boundaries would be lower somewhat and hence intergranular fracture tend to be
suppressed in the TF sample, if the grain boundary character dependence of impurity segregation is assumed to be small.

Before discussing influences of effective grain size and grain shape, we have to consider the occurrence of delamination in the 0.053% P-TF sample (Figs. 7 and 8). Kimura et al.\textsuperscript{19–21} reported in high strength steels with very low contents of P and S that the delamination of a crack arrester type similar to the present case was caused by cleavage fracture associated with a strong $\langle 110 \rangle$//RD fibre texture, where a lot of $\{ 100 \}$ cleavage planes are aligned along the longitudinal direction parallel to the RD. They\textsuperscript{19–21} also found that the delamination occurred when the strength level becomes high enough to overcome the critical stress for cleavage fracture. The strength level of 1 200 MPa in the present steels is smaller than that reported by Kimura et al.\textsuperscript{19–21} Moreover, the delamination was not observed in the 0.001% P-TF sample although its texture was similar to that of the 0.053% P-TF sample (Fig. 3). Thus, it is possible that the delamination in the 0.053% P-TF sample is caused as a result of (1) intergranular fracture due to reduction of grain boundary cohesion, as we observed more severely in 0.053% P-QT sample, (2) enhanced effect of arrays of second-phase particles, \textit{i.e.} cementite, formed on grain boundary on void nucleation and maked the grain boundary a favourable path for crack propagation due to the reduction of cementite–ferrite interface cohesion,\textsuperscript{2,26,27} and (3) intensified effect of dispersed cementite particles, having the decreased cohesion with matrix, in the grains leading to easier cleavage cracking on $\{ 100 \}$ planes. It should be noted that the second and third cases also present in the 0.001% P-TF sample, in absence of P.

Keeping in mind the low cohesion of grain boundaries even in the 0.053% P-TF steel as emphasized above, let us discuss the effects of effective grain size and grain shape on suppression of intergranular fracture. Intergranular cracking in a conventional QT structure occurs along prior austenite grain boundaries.\textsuperscript{5} This was also confirmed in the present 0.053% P-QT sample (Fig. 6(b)). Thus effective grain size for the intergranular cracking corresponds to the prior austenite grain size, which is 30\unit{\mu m} in the present sample. On the other hand, the prior austenite grain boundaries were no longer recognized in the TF sample. In this case, the effective grain size for intergranular cracking along the striking direction (SD) should be equal to the transverse grain size. It was 411\unit{nm} for the 0.053% P-TF sample, which is much smaller than that for the 0.053% P-QT sample. Moreover, a crack must be deflected with much more angles than 60° since grains are significantly elongated along the RD in the TF sample (Fig. 2). The much smaller effective grain size as well as the larger deflection angle must be responsible for suppression of intergranular fracture in the present steel.

4.2. Enhancement of Upper Shelf Energy

Let us discuss the reason why the TF samples with the UFEG structures showed a high absorbed energy of 150 J, which is 1.6 times that of the 0.001% P-QT sample, even though all three samples exhibited a ductile dimple fracture mode (Fig. 6). One of the reasons would be the higher tensile ductility in the TF samples as shown in Table 2. However, the difference in ductility is not large enough to explain the increase in the absorbed energy. For example, the work for the tensile fracture was $17.7 \times 10^3 \text{Jm}^{-3}$ for the 0.001% P-TF sample, which is 1.1 times that of $16.1 \times 10^3 \text{Jm}^{-3}$ for the 0.001% P-QT sample (Table 2). Another reason can be found in the dimple shape. In the TF samples, the fracture surface consisted of two types of dimples; the fine dimples and the large and shallow dimples indicated by the arrow in Fig. 6(a). On the other hand, the TF samples exhibited a homogeneous fracture surface with fine and deep dimples regardless of the P content (Figs. 6(c), 6(d)). Since the absorbed energy associated with void growth becomes higher as the ratio of dimple height to the dimple diameter increases,\textsuperscript{28–32} the absence of shallow dimples may be responsible for the higher absorbed energy of the TF samples. The fine and deep dimples of the TF samples are thought to be associated with the unique texture of $\langle 110 \rangle$//RD of the UFEG structures, although further studies are required to confirm the relation between the dimple shape and texture in the high strength steels.

Next, we should consider the effects of P segregation on void nucleation in the ductile dimple fracture mode. P segregates to the ferrite/cementite interfaces as well as the grain boundaries, resulting in a decrease in interface cohesion.\textsuperscript{5} Thus, the void nucleation at the cementite particles would become easier in the presence of P. This P segregation effect must decrease the total absorbed energy when the dimple size and shape are identical. In order to confirm it, we further investigated Charpy impact tests at elevated temperatures for obtaining the absorbed energy for a fully dimple fracture mode in the 0.053% P-QT sample. It was found that the fracture mode becomes fully ductile at 523 K, as shown in Fig. 9. Note that feature of the fracture surface is very similar to that of the 0.001% P-QT sample (Fig. 6(a)). The average absorbed energy at 523 K was 67 J and plotted in Fig. 5. It is found that the absorbed energy for a fully ductile mode or the upper shelf energy is smaller in the 0.053% P-QT sample than in the 0.001% P-QT sample. It is likely that the reduced upper shelf energy is caused by easy void nucleation at the cementite particles due to P segregation to the cementite/ferrite interfaces.

It must be noted that the measured absorbed energies were the same for both TF samples (Fig. 5), even though easy void nucleation must operate in the 0.053% P-TF sample. This result suggests that an additional mechanism to increase the absorbed energy had operated in the 0.053% P-TF sample to compensate the easy void nucleation. This mechanism can be found in the delamination fracture, as was observed in Figs. 7 and 8. Significant delamination of a
“crack arrester type” was seen in the 0.053% P-TF sample (Fig. 8(b)) but not in the 0.001% P-TF sample (Fig. 8(a)). The crack arrester type delamination can relax the triaxial stress condition and blunt the crack tip.23–25) Crack re-initiation is necessary to fracture a material and occurs under conditions of nearly uniaxial tension, resulting in higher absorbed energy. It is reasonable that the enhancement of delamination fracture is affected more by the cracking along the grain boundaries with a low cohesion due to P segregation in the 0.053% P-TF sample compared to the 0.001% P-TF sample, when the grain size, grain shape, and texture are similar in both samples. In other words, P segregation may improve the impact toughness through the enhancement of delamination fracture in the case of the UFEG structure at least in the present 1200 MPa-class high strength steel. The allowance of P content in the UFEG structure with respect to impact toughness should be investigated in the future.

5. Conclusions

Microstructure observations and Charpy impact tests were carried out in 1200 MPa-class medium-carbon low-alloy steels with phosphorous (P) contents of 0.001 and 0.053 mass%, in order to investigate whether it is possible to obtain both the suppression of intergranular fracture and the enhancement of upper shelf energy in a brittle high strength steel with a conventional quenched and tempered (QT) condition. The main results are summarized as follows:

(1) A similar ultrafine elongated grain (UFEG) structure with a strong (110)//RD fibre deformation texture can be developed in SCM 440 type steels regardless of P content by warm calibre-rolling with 80% reduction following tempering at 773 K.

(2) The UFEG and the QT structures show a similar tensile strength and a total elongation of approximately 1200 MPa and close to 15% irrespective of the P contents, although the UFEG structures exhibit slightly higher values in strength and elongation and the 0.053% P-QT sample shows a smaller elongation of 13%.

(3) The UFEG structure exhibits almost the same high absorbed energy of 150 J regardless of P content, although the absorbed energy of the QT structure is significantly decreased from 93 J for the 0.001% P steel to 23 J for the 0.053% P steel due to the occurrence of intergranular fracture.

(4) Crack arrester-type delamination occurs only in the 0.053% P-UFEG structure and does not in the 0.001% P-UFEG structure even though the grain structure and the texture are similar, indicating the low cohesion of grain boundaries in the 0.053% P-UFEG structure.

(5) The suppression of intergranular fracture in the 0.053% P-doped steel by the introduction of the UFEG structure is resulted by a smaller effective grain size as well as larger crack deflection angles for crack propagation in addition to a lower P concentration on grain boundaries.

(6) The high absorbed energy of 150 J for the 0.053% P-doped UFEG structure is associated with the fine and deep dimples as well as the delamination fracture, which compensates easy void nucleation at cementite particles due to P segregation to the cementite/ferrite interfaces. This suggests that P segregation may rather improve the impact toughness through the enhancement of delamination fracture in the case of the UFEG structure.

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