Effect of Carbide Size Distribution on the Impact Toughness of Tempered Martensitic Steels with Two Different Prior Austenite Grain Sizes Evaluated by Instrumented Charpy Test

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The effect of tempering temperature on the impact toughness of 0.3 mass% carbon martensitic steels with prior austenite grain (PAG) size of about 6 and 60 µm was investigated. Instrumented Charpy impact test (ICIT) was used to evaluate the impact toughness. The tempering temperature of 723 K gives the largest difference in the Charpy impact energy at room temperature between the specimens with two different PAG sizes, where the finer PAG specimen shows higher impact energy at room temperature (RT). The other tempering temperatures do not show a significant difference as compared with that shown among the 723 K tempered specimens. Investigation of the test temperature dependence of Charpy impact energy in the 723 K tempered steels shows a steep transition at around 200 K for the 6 µm PAG specimen, while it shows a continuous slow transition in a wide range of temperatures for the 60 µm PAG specimen. ICIT waveform analysis of these steels shows that the fracture propagation energy mainly controls the temperature dependence of the impact energy, while the fracture initiation energy stays nearly constant against the variation of the test temperature. The carbide size distribution in these two specimens was investigated by secondary electron microscope (SEM) and transmission electron microscope (TEM). The 60 µm PAG specimen shows distribution of coarser carbide than does the 6 µm PAG specimen, which seems to give rise to the observed difference between them in the Charpy impact energy and the other properties of impact fracture. [doi:10.2320/matertrans.M2013079]

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1. Introduction

Martensitic steels with high strength are playing an important role in state-of-the-art industrial technology in view of the reduction in the environmental load by weight reduction of vehicles on land or sea.1) However, a trade-off balance between strength and ductility or toughness is a matter of concern to prevent brittle fracture in high strength steels.2) Microstructural elements such as grain size and carbide size are critical in the fracture process.3,4) Grain refining is a useful method for improving the toughness.5) Kimura et al.5,6) have presented ultrafine grained fibrous structured steel with excellent toughness. Hui et al.7) investigated the effect of prior austenite grain (PAG) size on impact toughness, where they reported the enhancement in toughness and ductility of fine-grained 1.08Cr–0.39C martensitic steel tempered at 673 K. Decrease in the Charpy impact energy in accordance with the increase in PAG size was observed in the as-quenched and 473 K tempered martensitic steel.8,9) Kawasaki et al.10) used an induction heating method, which makes use of the rapid heating process in grain growth or carbide forming kinetics,11) in austenitizing and tempering of 0.58%C steel to investigate the mechanical properties in relation to the microstructures, and they reported that the finer microstructure gives rise to enhancement in ductility, impact toughness and so forth. However, in tempered martensitic steels, the grain size effect could be diminished by the carbide precipitation.12)

Therefore, the effect of the carbide should be kept in view as well as the PAG size effect.

In steels, the cracking of brittle particles, carbide in many cases, is believed to give rise to nucleation of the cleavage crack.13,14) Thus, the carbide size plays an important role in the fracture process. Bowen et al.15) reported that the carbide size distribution is critical in the fracture process of tempered bainitic or martensitic steels. Tsuchiyama et al.15) controlled the carbide state in 12%Cr–0.3%C steels by partial solution treatment and investigated the impact toughness and concluded that the carbide dispersion mode is critical in the upper shelf of the impact energy.

Charpy impact test is a powerful tool in studying the fracture process of materials.16) Moreover, the instrumented Charpy impact test (ICIT) method17,18) is useful because it gives information on the fracture initiation and propagation as well as the energy absorbed through the impact process.19–21) Hashemi22) used the ratio of the total absorption energy to the fracture propagation energy to correct the conventional fracture model of the pipeline steel. Sreenivasan et al.23) investigated martensitic stainless steel according to the ICIT method and presented three typical waveforms, Type I brittle, Type II ductile-brittle and Type III fully ductile, detail of which shall be discussed later. However, the ICIT waveform has not been systematically investigated in connection with the fracture process of martensitic steel.

The present study investigated the effect of microstructure factors, carbide size and PAG size, upon the impact fracture properties of plain tempered martensitic carbon steels containing simple additives such as Si, Mn and Al through systematic investigation of the ICIT waveforms.

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Table 1 Chemical compositions of the steel in mass%.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.31</td>
<td>0.50</td>
<td>2.0</td>
<td>0.001</td>
<td>0.001</td>
<td>0.05</td>
<td>0.004</td>
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2. Experiment

The factors affecting the PAG size have been investigated extensively.\(^\text{24}\) A repeated austenitizing and quenching process is often employed.\(^\text{7,25,26}\) A thermomechanical process\(^\text{27}\) or induction heating method\(^\text{10,11}\) is also often employed to control the PAG size or grain size of the steel. Meanwhile, the pinning effect by the fine precipitate is also applied in controlling the PAG size.\(^\text{24,28}\) Ushioda et al.\(^\text{29}\) studied the AlN precipitation behavior in low carbon steel. In the present study, AlN precipitate was expected to impede PAG growth making it possible to control the PAG size in a wide range by varying the austenitizing temperature.

Steel with 0.3 mass% C and 0.05 mass% Al, the chemical composition of which is listed in Table 1, was used. The amount of P was kept as low as possible in order to avert the P-derived embrittlement in the tempered steels.\(^\text{30}\) The ingot was prepared by vacuum induction melting. The ingot was homogenized at 1,473 K for 2 h and forged down to a thickness of 15 mm. Bars of 15 mm in width and 180 mm in length were sampled out from the plate. The bars were austenitized at two different temperatures, 1,153 or 1,373 K for 5 min, and then were water quenched. Each bar was tempered at 453, 623, 723 or 923 K for 90 min. The bars for the as-quenched specimens were stored in liquid nitrogen to minimize the carbon diffusion at room temperature (RT). Then tensile test specimens and standard Charpy impact test specimens with 2 mm V-notch were machined out from the bars. The stress–strain characteristic was measured with the strain curve shows an upper yield point with a high yield strength of about 1.5% for the as-quenched specimens and austenitized specimens.\(^\text{37}\) The difference in the yield strength is small between 523 and 723 K.\(^\text{43}\) The difference in the retained austenite fraction stays almost unchanged at the values of 1.2–1.8% for the as-quenched and the 453 K tempered specimens, which is almost consistent with previous results.\(^\text{41,42}\) However, the yield stress is somewhat lower in the 60 µm PAG specimen than that in 6 µm one at every tempering temperature. A close look at the yielding features in the 723 K tempered specimens shows a clear distinction.

3. Results

3.1 Microstructure and mechanical properties

Figures 1(a) and 1(b) show the prior austenite grains (PAG) of the as-quenched specimens austenitized at 1,153 K, giving the PAG size of about 60 µm, and at 1,373 K, giving the PAG size of about 60 µm, respectively. The PAG boundary is almost consistent with previous results,\(^\text{41,42}\) and decreases by an order of magnitude as the tempering temperature rises, which is consistent with the rapid decomposition of austenite between 523 and 723 K.\(^\text{43}\) The difference in the retained austenite fraction is, if any, quite small between the 6 µm PAG and 60 µm PAG steels. The stress–strain curves are shown in Fig. 3. The ultimate tensile stress of the as-quenched steel is larger in the 6 µm PAG steel, however, the difference becomes smaller or disappears in the tempered specimens as reported by Swarr et al.\(^\text{12}\) However, the yield stress is somewhat lower in the 60 µm PAG specimen than that in 6 µm one at every tempering temperature. A close look at the yielding features in the 723 K tempered specimens shows a clear distinction.
subsequent yield elongation, while in the 60 µm PAG specimen, only a round shoulder is shown of its stress-strain curve. The influence of this difference in their yielding behaviors on the fracture property shall be discussed in the later section. However, in the 923 K tempered specimens, both show upper yield points with subsequent yielding elongations. The reduction in area (RA) in Fig. 4 shows that the 6 µm PAG specimens show rather enhanced ductility as compared with the 60 µm PAG specimens.

The microstructure and the mechanical properties of the present steels show that the specimens are plain tempered martensitic steels. Hence, the major factors to be considered could mainly be cementite and PAG size.

3.2 Impact toughness

Figure 5 shows the Charpy impact energy of the present tempered martensitic steels tested at RT. The absorbed energy is rather low, brittle, in the as-quenched, 453 K tempered and the 623 K tempered specimens and then it increases as the tempering temperature rises, either in the 6 µm or 60 µm PAG specimens. Fractographs of some of these specimens are shown in Fig. 6. Typical cleavage fracture is observed in the as-quenched and 453 K-tempered specimens. A clear intergranular fracture is observed in the 623 K-tempered specimen, which corresponds to the low temperature temper embrittlement.\textsuperscript{1,44} The difference in the absorbed energy between the 6 µm PAG specimens and the 60 µm PAG specimens is largest for the tempering temperature of 723 K. At the other tempering temperatures, though the 6 µm PAG specimens tend to be higher in their Charpy impact energy than the 60 µm PAG specimens, the difference is small, by a few J, falling almost in the range of error of the present ICIT device. It is reported by Ritchie \textit{et al.}\textsuperscript{8,9} that for the as-quenched and 473 K tempered martensitic steels, the impact Charpy energy decreases by about 7–8 J as PAG size increases from about 20 to 200 µm. Therefore, it might be difficult to observe the PAG size effect on the impact toughness in the present plain martensitic steels. In the cleavage fracture process of the steels, the coherence length on \{100\} planes is an important measure because iron cleaves on those planes, which could be a measure of the grain size.\textsuperscript{3} However, the coherence length is difficult to measure in many steels, particularly martensitic steels.\textsuperscript{12,39} The yield stress of the martensitic structure is related to the block size as well as the PAG size in Hall-Petch type of manner.\textsuperscript{12,39} The block size might possibly be related to the coherence length in martensitic steels. The temperature dependence of the impact energy for the 723 K tempered specimens is shown in Fig. 7. The 60 µm PAG steel gives a lower upper shelf and a sluggish change against the test temperature. Meanwhile, the 6 µm PAG steel gives a stable upper shelf of about 120 J and a steep change at about 198 K.

In order to investigate the fracturing behavior, ICIT waveforms were analyzed.
3.3 Instrumented Charpy impact test

ICIT is intensively exploited to investigate the impact toughness of the steels used for pressure vessels,\textsuperscript{45} weldment,\textsuperscript{46} fiber composite materials\textsuperscript{47} and embrittled tempered martensitic steel.\textsuperscript{48}

ICIT gives the load-displacement, or load-time, waveform of the specimen in response to the impact given by the striker. The raw waveform is accompanied by noise, which is filtered out by moving average operation.\textsuperscript{17,47} The impact energy deduced from the impact waveform was compared with that deduced from the energy loss of the striker, which is shown in Fig. 8. They coincide with each other fairly well. Sreenivasan et al.\textsuperscript{23} investigated martensitic stainless steel and categorized the ICIT waveforms into 3 types, Type I, II and III, which correspond to brittle, ductile-brittle and fully ductile fractures, respectively. Figure 9 shows some of the waveforms of the 6 µm PAG steel. Figure 9(a) corresponds to the as-quenched specimen struck at \textit{RT}. Figures 9(b) and 9(c)}
correspond to the 723 K tempered specimen, each of which was tested at 153 K and RT, respectively. These waveforms correspond to Type I (brittle fracture) for Fig. 9(a), Type II (ductile-brittle fracture) for Fig. 9(b) and Type III (fully ductile fracture) for Fig. 9(c). The variations of the waveforms of the 723 K tempered specimens against test temperature are shown in Fig. 10. For the 6 µm PAG specimens, the waveforms struck at 298 and 223 K are Type III, fully ductile waveforms. However, the waveform struck at 198 K shows Type II feature with a small tail. Meanwhile, for the 60 µm PAG specimens, the waveform struck at 298 K shows Type III feature, then it shows Type II feature when struck at 223 K. Comparison of these waveforms with the absorbed energy plots in Fig. 7 shows that for the 6 µm PAG specimens, the waveform stays almost unchanged, which is Type III, until the test temperature is lowered to around 200 K, where, as shown in Fig. 7, the absorbed energy decreases steeply and the long tail of the waveform suddenly disappears leading to the Type II waveforms, whose maximal load slightly increases as the test temperature decreases. On the other hand, for the 60 µm specimens, corresponding to the gradual change of the absorbed energy shown in Fig. 7, the waveform gradually transforms from Type III to Type II. In either steel, the waveform does not show Type I brittle feature even where the impact Charpy energy reaches its lower shelf. However, in either steel, the waveform finally turns to Type I feature at the impact test temperature of 123 K. The temperature dependent variation of the impact waveform was also presented of the 9Cr–0.1C heat-resistant steels using precracked specimens, where the clear transition from Type III waveform to Type II waveform was not reported. Some of the fracture surfaces of these specimens are shown in Fig. 11. Figure 11(a) shows the ductile fracture surface of the 6 µm PAG specimen struck at 298 K, while the 60 µm PAG specimen struck at 298 K shows a partly cleavage fracture surface as shown in Fig. 11(b). In Figs. 11(c) and 11(d), both of the two specimens, struck at 198 K, show cleavage brittle fracture. It should be noted that in the 60 µm PAG specimen struck at 298 K, in spite of its Type III fully ductile waveform shown by a solid line in Fig. 10(b), the fractograph shows a combination of brittle fracture and ductile fracture.

As noted previously, the impact fracture process is roughly divided into two parts, one is fracture initiation, where the crack initiates to spread transversally and the load reaches maximum, and the other is fracture propagation, where the
load decreases. The energies absorbed during fracture initiation and propagation, denoted by $E_i$ and $E_p$, respectively, are plotted against the test temperature for the 723 K tempered specimens in Fig. 12. Attention should be paid to that $E_i$ and $E_p$ are given by integrating the load-displacement curve from the initial point up to the load-maximum and then from the load-maximum thereafter, respectively. As expected from the waveforms in Fig. 10, the fracture propagation process contributes to the major part of the temperature dependence of the absorbed energy. Meanwhile, the fracture initiation energy shows only slight temperature dependence in the present temperature region. In spite of the slight increase of the maximal load with the decrease in the test temperature shown in Fig. 10, the width of the waveform corresponding to the fracture initiation decreases, which leads to the small temperature dependence of $E_i$ shown in Fig. 12(b). From Figs. 10–12, the major difference in the temperature dependence between the two specimens is in the fracture propagation behavior.

3.4 Carbide size distribution

The carbides in the 723 K tempered steels were observed by SEM and also by TEM, photographs of which are shown in Figs. 13 and 14, respectively. In most of the SEM images, the difference in the carbide size is hardly observed, however in some images, as shown in Fig. 13, large carbides of 2–3 µm in length are observed in the 60 µm PAG specimen. Meanwhile, also in the TEM observation, the carbide size tends to be larger in the 60 µm PAG specimen than in the 6 µm PAG specimen as clearly seen in Fig. 14. As a result, the carbide size distributions were obtained for the 723 K tempered steels, which are shown in Fig. 15. In the 60 µm PAG specimen, about 10% of the carbides are larger than 1 µm. Meanwhile, in the 6 µm PAG specimen, almost all carbides are smaller than 1 µm. Hereafter, the term “carbide size” in the present steel is referred to as the length of the anisotropically shaped carbide particle.

As shown in Fig. 5, in the present steels, the PAG size effect on impact toughness is not clearly seen except in the 723 K tempered steels. The carbide size difference observed here can give rise to the difference observed in the impact toughness among the 723 K tempered steels. The diffusion length $l$ of carbon in steel at temperature $T$ during the interval of time $t$ could be estimated as follows;
\[ l = \sqrt{Di} \] (1)

\[ D = D_0 \exp(-Q/RT), \] (2)

where \( D \), \( Q \) and \( R \) are diffusion coefficient, activation energy and gas constant, respectively. Then, with \( D_0 = 0.394 \) (mm\(^2\)/s) and \( Q = 80.22 \) (kJ/mol), \( l \) could be about 55 \( \mu \)m at 723 K for 90 min, which seems to be long enough for the carbon atoms in the 60 \( \mu \)m PAG specimen to stably form carbides at the PAG boundaries. Then it can be considered that the larger is the PAG size, the coarser are formed the carbides along the PAG boundaries in the 723 K tempered specimens. The same could be true with the specimens tempered at higher temperatures. However, in the 923 K tempered steels, the spheroidally shaped carbide is observed in either 6 \( \mu \)m PAG\(^{51}\) or 60 \( \mu \)m PAG specimen, where the difference in carbide size is hardly observed between the two specimens. Speich\(^{52}\) reported that spheroidal cementite formation proceeds from tempering at 623 K up to 823 K, and then that Ostwald-type ripening occurs at higher temperature. On the other hand, Lement et al.\(^{53}\) and Yusa et al.\(^{54}\) reported that the low temperature tempering gives rise to coarse film-like cementite formed along the PAG boundaries. Then the
tempering at 723 K in the present steel might possibly give rise to the onset of the cementite morphological transition from film-like to spheroidal, where the carbide size might be influenced by the PAG size. Therefrom, the difference could be derived in the carbide size distribution of the present 723 K tempered specimens.

4. Discussion

It is important to discuss the relationship between the waveforms and the corresponding fracturing process. In the brittle fracture, the process can be roughly pictured as cleavage crack nucleation\(^{55-57}\) and growth.\(^{58}\) The Type I waveform shown in Fig. 9(a), where as soon as the load reaches maximum it quickly goes down, could correspond to such process. However, when the specimen is ductile, soon after the cleavage crack is nucleated, it is blunted by spontaneous emission of dislocations,\(^{59}\) which delays the crack propagation. The thick waveform of Type II could account for such a blunting process. The blunting occurs also in the fracture initiation process of the Type III waveforms. In the fully ductile fracture, the waveform of which is shown in Fig. 9(c) Type III, the crack nucleation and dislocation emission could lead to the microvoid nucleation around the carbide and then they grow and coalesce quickly and then the plastic limit-load failure of the matrix occurs accompanying softening.\(^{60}\) In the Type III waveform, it is estimated that the slant shearing fracture process leading to the shear lip formation near the edges of the specimen contributes to some of the tail in the waveform.\(^{22}\) However, it is difficult to split the propagation energy into the shearing fracture energy and other fracture energy such as void nucleation and growth or softening. Thus, the initiation process of 723 K tempered steels, whose waveforms are Type II or Type III, could be considered to correspond to the process where the crack initiates and blunts by dislocation emission. However, the propagation process could be different between Type II and Type III. In the Type II brittle-ductile process, the crack propagation is steep, almost as steep as Type I brittle process. On the other hand, in the Type III fully ductile process, the crack propagation corresponds to the process where the microvoids nucleate and grow or coalesce.\(^{60}\) However, it could be misleading to view the impact fracture process as the simple process described above. In the 60 µm PAG specimen, e.g. that was struck at 298 K, whose waveform is shaped as Type III fully ductile waveform, the fracture surface is a mixture of the brittle and the ductile fracture accompanied by the slant shear lip fractures. Thus, it is considered that in the fracture propagation process of this specimen, both unstable, brittle crack and ductile crack are propagating.

Pitch\(^{61}\) compared the transition of the impact absorption energy of two ferritic steels with the same grain size, one with 0.6 µm carbide and the other with 2 µm carbide, the major location of which, whether intergranular or intragranular, is not clear, though. He presented experimental results showing that the steel with 0.6 µm carbide shows a steeper change in absorption energy at lower temperature than does the steel with 2 µm carbide, the shapes of the transition curves are somewhat similar to the present results shown in Fig. 7. Meanwhile, Swarr \textit{et al.}\(^{12}\) presented that the PAG size dependence of the yield stress is diminished in the tempered martensitic steels. It is likely that the PAG size dependence, if any, of the fracture process in the tempered martensitic steels may as well be diminished as that of the yield stress. Therefore, the carbide size, rather than the grain size which may correspond to the block size in the present martensitic morphology,\(^{39,62}\) might influence the impact fracture property of the present specimens.

The transition curve shown in Fig. 7(a), or Fig. 12(a) for the 6 µm PAG steel implies that at test temperatures above 223 K, the fracture mode is fully-ductile with Type III waveform with the dimple fracture surface as shown in Fig. 11(a). Below the transition temperature at about 198 K, the fracture mode becomes brittle-ductile with Type II waveform, showing a sudden decrease in the propagation energy and the cleavage fracture surface as shown in Fig. 11(c). It should be noted that in spite of the brittle cleavage fracture surface, the Type II waveform shows that the dislocation emission process might have occurred before the cleavage crack ran. Therefore, the continuous transition of the 60 µm PAG specimens was observed in the energy transition curve shown in Fig. 7 or Fig. 12(a) could imply that the transition temperature continuously differs site by site in the specimen. This behavior is considered to be closely related to the carbide size, especially that in the range of 2–3 µm in the distribution curves shown in Fig. 15.

In the fracturing process, carbide particles or the cracks formed in them are considered to give rise to the initiation of fracture, whether ductile\(^{63}\) or brittle.\(^{57}\) It is reported that in
the tempered martensitic structure, the mechanism of fracture involves the nucleation and propagation of the microcracks from the coarsest carbides.\textsuperscript{4)} Tsuchiyama \textit{et al.}\textsuperscript{15} showed that the coarse carbide lowers the upper shelf energy of the absorbed energy. Petch\textsuperscript{61} showed by a theoretical investigation that the cleavage strength decreases as the carbide size increases. The relatively large carbide particles, larger than 1 µm as shown in Fig. 13(b), could be nucleation sites of cleavage crack or microvoids. In the as-quenched and the 453 K tempered specimens, massive formation of carbide was not observed. However, TEM observation, if tried, would have captured some transitional carbide, as studied by Nagakura \textit{et al.}\textsuperscript{64-66} In the 623 K tempered steels where the fracture surfaces show intergranular fracture, the morphology of the carbide, such as carbide film formation along the PAG boundaries, might bear an important role in the embrittling behavior.\textsuperscript{53,54} Thus, the clear difference in the carbide size distribution observed in the 723 K tempered steels likely brings about the difference observed in the fracture toughness between the specimens with two different sizes. In discussing the brittle fracture, Ritchie \textit{et al.}\textsuperscript{14} introduced a model that relates the microscopically sharp crack tip to the macroscopic fracture toughness through the brittle particles, carbide in the present case, distributed in the microstructure. Curry \textit{et al.}\textsuperscript{67} emphasized the importance of the carbide size distribution to derive a probabilistic model of fracture that relates the fracture toughness to the chance of finding a carbide of a given size at a given distance from the macroscopic crack. Also in the ductile fracture, the carbide size plays a crucial role. The toughness at high test temperature is controlled by the size of the coarsest particle, on the other hand, the brittle cleavage at low test temperature is controlled by the average size of carbide.\textsuperscript{68} The same mechanism could be working in the present results.

Although, the carbide size difference among the 723 K tempered specimens may lead to the difference in their impact fracturing behaviors, attention should be paid to the difference in their yielding behaviors in their stress–strain curves. As shown in Figs. 3(a) and 3(b), in the 723 K tempered specimens, the round shoulder in the stress–strain curve for the 60 µm PAG specimen implies that the dislocation therein is more mobile than that in the 6 µm PAG specimen which shows a clear upper yield point with subsequent yield elongation. When a crack is nucleated, the more mobile the dislocation is, the more likely emitted is the dislocation from the crack tip leading to crack tip blunting.\textsuperscript{59) Then, the 60 µm PAG specimen could be more ductile than 6 µm PAG one, which is not the case in the present study. Therefore, in these specimens, some particle-size-dependent interaction between carbide particles and dislocation might possibly be operative.

5. Conclusions

In summary, 0.3 mass% carbon tempered martensitic steels with two different PAG sizes, 6 and 60 µm, were prepared and the impact toughness was investigated by ICIT method in connection with the carbide size distribution. The systematic variation of the impact waveform was also investigated. The conclusions are as follows:

(1) The tempering temperature of 723 K gives the largest difference in the impact toughness at room temperature between the 6 µm PAG and 60 µm PAG martensitic steels.

(2) Among the 723 K tempered steels, the 60 µm PAG specimen gives continuous change in the impact absorption energy along the test temperature. Meanwhile, the 6 µm PAG specimen gives steep change at lower test temperature.

(3) According to the waveform analysis, the change in the fracture propagation energy contributes to the change in the impact absorption energy. On the other hand, the fracture initiation energy stays almost constant in the range of the present test temperature.

(4) The waveform of either specimen corresponds to the “brittle-ductile” type even at the impact test temperature of 150 K, which is low enough to give the lower shelf.

(5) The observed difference in the impact toughness at room temperature between the two steels tempered at 723 K could be attributed to the difference in the carbide size distribution, which gives almost no carbide larger than 1 µm for the 6 µm PAG specimen while it gives carbide ranging from 2 to 3 µm for the 60 µm PAG specimen.

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