

Tempered Martensite Embrittlement in a 32NiCrMoV125 Steel

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This study focused on tempered martensite embrittlement in a 32NiCrMoV125 steel through examination of the effects of austenite grain size and tempering temperature on the mechanical properties and fracture morphology of this material. Two different austenite grain sizes were obtained by austenitizing at 870 and 950 °C. After quenching, the specimens were tempered in the temperature range of 200-650 °C. The results obtained in this research indicate that by increasing the tempering temperature, the strength and hardness decrease, but ductility increases. However, impact testing indicated that tempered martensite embrittlement occurred when samples were tempered in the range of 250-400 °C. Fractography revealed intergranular and quasi-cleavage fracture. In summary, increasing the austenite grain size decreased strength, but increased impact toughness, except for samples tempered between 200 and 350 °C.

Keywords 32NiCrMoV125 steel, austenite grain size, tempered martensite embrittlement, tempering

1. Introduction

Ultrahigh-strength, low-alloy steels with medium carbon content and various amounts of chromium, molybdenum, nickel, and vanadium have been used for high-performance pressure vessels, rotors, etc. These steels can be successfully used at yield strengths equal to or greater than 1400 MPa (Ref 1, 2).

Generally, quenching and tempering are well-established means to increase the strength of steel. This can be achieved mainly due to the martensitic structure produced by quenching and the precipitation of a fine dispersion of alloy carbides during tempering (Ref 3, 4). To produce the highest level of strength in steel, the martensite structure is rarely used in the untempered condition because the residual internal stresses associated with the transformation can cause the material to have less ductility than needed (Ref 3, 5, 6).

The mechanical behavior of quenched-and-tempered steel depends strongly on its microstructure (Ref 7-9). It has been shown that when these steels are tempered near 350 °C, a loss in toughness occurs due to tempered martensite embrittlement (TME) (Ref 3, 7, 10, 11). This phenomenon is usually characterized by a plot of the impact energy as a function of tempering temperature and by a ductile-brittle transition temperature that exhibits a maximum at the temperature corresponding to the minimum in the impact toughness (Ref 12, 13).

Several researchers have found that the TME phenomena is related to the combined effect of carbide precipitation and impurity element segregation. While impurity elements, primarily

phosphorus and sulfur, segregate into the grain boundaries during austenitization and tempering (Ref 10, 14, 15), the decomposition of lath-boundary-retained austenite and the subsequent formation of interlath cementite films occur during tempering (Ref 11, 16, 17). These interlath carbides may provide crack nucleation sites and easy crack paths.

The 32NiCrMoV125 steel is in the group of ultrahigh-strength low-alloy martensitic steels that provides an advantageous combination of strength, ductility, and toughness. The steels in this group are, however, susceptible to embrittlement as a result of tempering within a specific temperature range. This study focused on the mechanical properties and fracture morphology of 32NiCrMoV125 steel under different austenitizing and tempering temperatures

2. Experimental Procedure

A 32NiCrMoV125 steel, supplied in the form of an as-forged bar 270 mm in diameter, was used in this study. Its chemical composition is presented in Table 1. To obtain different quenched-and-tempered martensitic structures, samples from the as-received steel were austenitized at either 870 or 950 °C for 1 h, followed by oil quenching to produce a quenched martensite structure. Specimens were then tempered at 200, 250, 300, 350, 400, 500, 600, and 650 °C for 1 h.

After heat treatment, specimens were machined for tension and impact testing. The size and geometry of the specimens, as well as the testing procedure, were in accordance with DIN 50,125 and DIN 50,115 for tension and impact testing, respectively. Three specimens were used for each test condition. The toughness was characterized by the absorbed fracture energy of specimens at room temperature. The fracture surfaces of the Charpy specimens were analyzed by scanning electron microscopy (SEM) to determine the mode of fracture.

3. Results and Discussion

Two austenitizing temperatures (870 and 950 °C) were used to produce different grain sizes: 15 and 39 μm , respectively.

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The variations of strength and hardness of the steel with tempering temperature are shown in Fig. 1 and 2, respectively. The data for the as-quenched condition (shown as tempering temperature 0) are indicated in the figures for comparison. These figures indicate that the strength and hardness decreased as tempering temperature increased. Figure 3 shows that the reduction in area and elongation decreased with increasing tempering temperature. As expected, the mechanical behavior of this steel is quite sensitive to the tempering temperature. Under as-quenched conditions, the steel had the highest level of strength and hardness, but its ductility was also the lowest. This can be explained by the phase transformation that occurs in the

steel during quenching, where the lattice structure of steel changes immediately from a face-centered cubic (γ phase) to a body-centered tetragonal (martensite). At the same time, significant distortion occurred during the formation of the martensite, which leads to the rapid increase in strength and hardness.

Figure 4 shows the room-temperature impact energy as a function of tempering temperature for both austenitizing temperatures. It can be seen in this figure that there is loss of toughness in the tempering temperatures from 250 to 400 °C. The loss of toughness after tempering in this temperature range is typical of steels that are susceptible to TME. This result is

Table 1 Chemical composition (in wt.%) of the steel specimens

C	Si	Mn	Ni	Cr	Mo	V	P	S	N ₂ , ppm	O ₂ , ppm	H ₂ , ppm
0.361	0.264	0.567	3.08	1.34	0.496	0.149	0.006	0.004	60	18	0.54

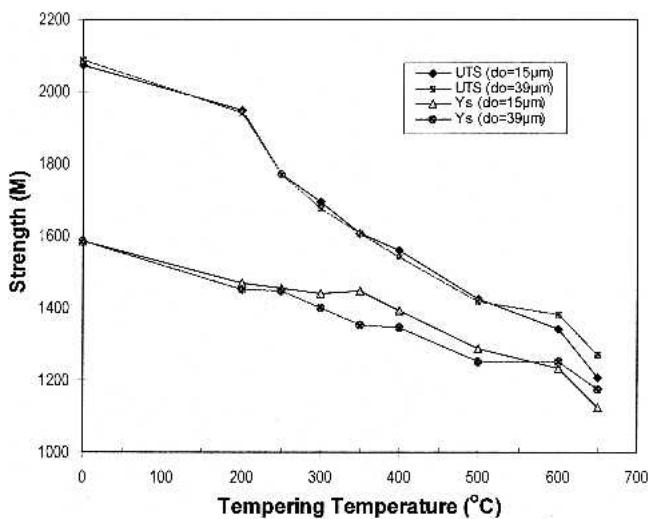


Fig. 1 Variations of yield strength and tensile strength with tempering temperature for two austenite grain sizes

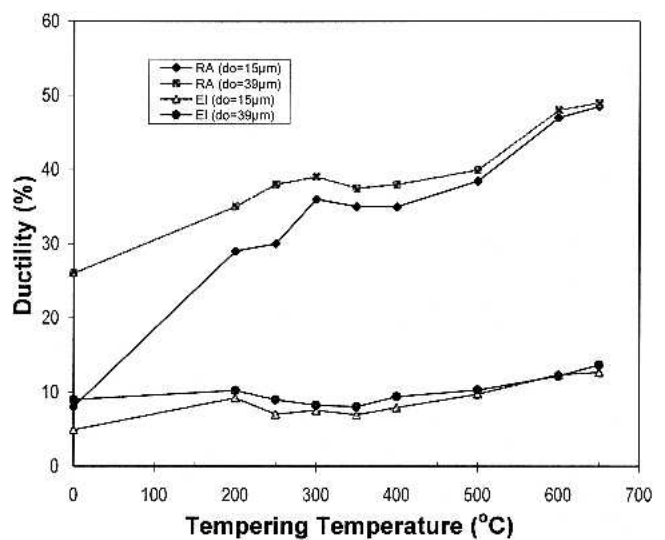


Fig. 3 Variations of reduction in area and elongation with tempering temperature for two austenite grain sizes

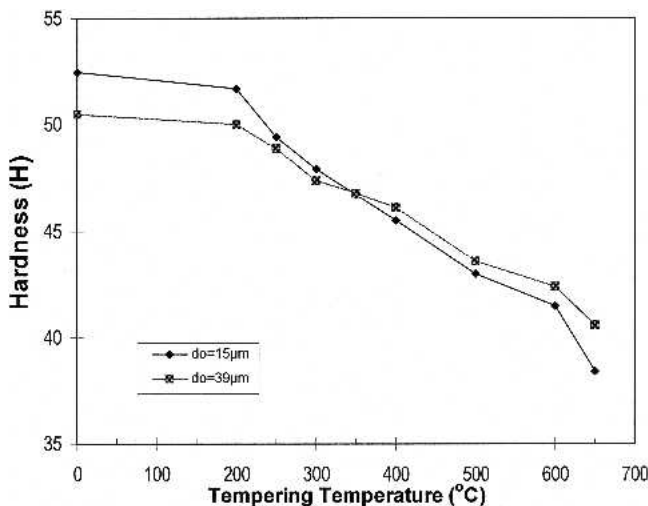


Fig. 2 Variation of hardness with tempering temperature for two austenite grain sizes

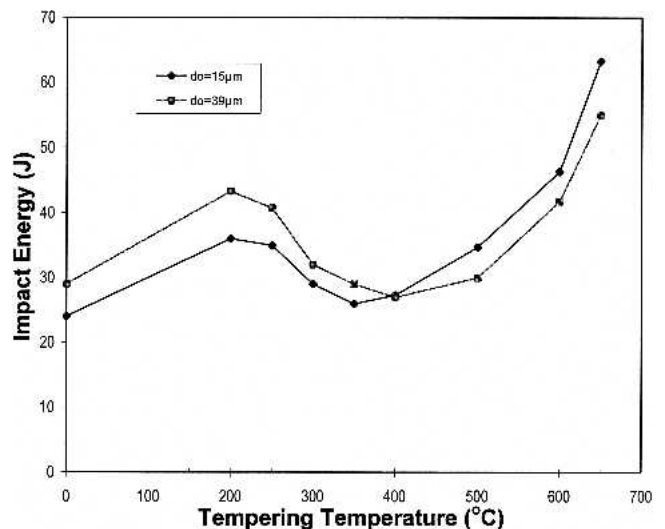


Fig. 4 Variations of impact energy with tempering temperature for two austenite grain sizes

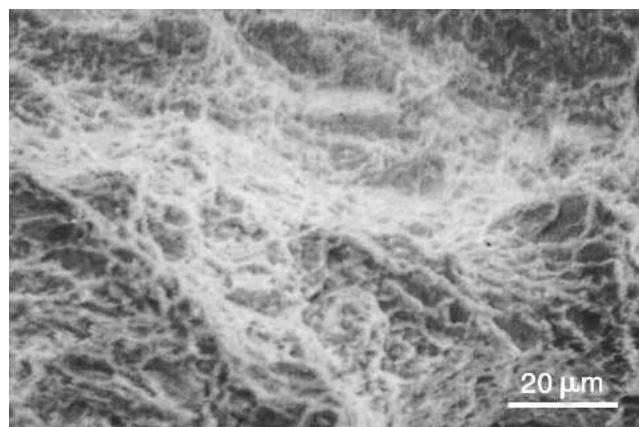
consistent with previous studies on ultrahigh-strength, low-alloy steels, indicating a depression of the impact energy for tempering between 250 and 400 °C (Ref 3, 7, 10, 11). Two main mechanisms have been suggested for TME (Ref 10, 11, 14, 16). The first is the decomposition of lath-boundary-retained austenite to cementite during tempering at critical temperatures, thereby providing a susceptible site for crack nucleation. The second is related to the segregation of impurity elements, such as phosphorus and sulfur, to austenite grain boundaries during austenitization and tempering. Remembering that the amount of impurity elements in this steel is very low (Table 1), it seems that the dominant mechanism for TME in the present case is related to the former mechanism.

As shown in Fig. 3, ductility is not improved by increasing the temperature in the range of 250-400 °C, but there is no evidence of the TME phenomena. This behavior can be related to the shape of specimens, test method, and applied stress state (Ref 18). The results shown in Fig. 3 are collected from slow-speed tensile tests using un-notched cylindrical specimens. In this condition, i.e., in the absence of stress concentration effects, those steels tempered at the TME temperatures can handle the applied stress without any signs of embrittlement (Ref 18).

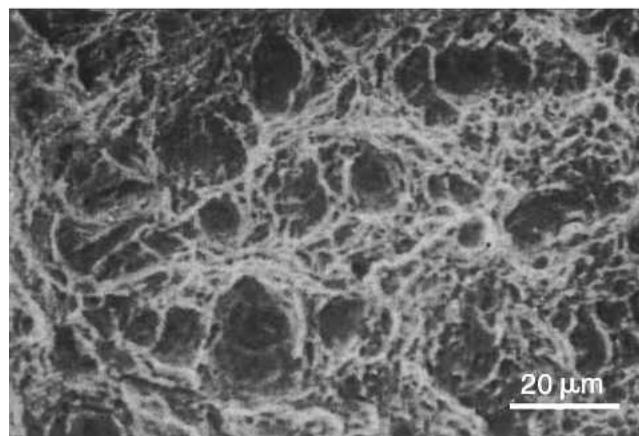
Fractography of impact specimens by SEM indicated an increase in ductility with increased tempering temperature. This was manifested by an increase in the number and size of microvoids, except for the specimen tempered at TME region (e.g., Fig. 5). The specimens treated in the TME region were less ductile and showed quasi-cleavage fracture (Fig. 6). Figure 6 shows a SEM fractograph of the specimen tempered at 450 °C showing tear ridges, which are characteristically associated with quasi-cleavage fracture. This embrittling behavior is consistent with the results of impact test for the TME region (250-400 °C) shown in Fig. 4.

The mean austenite grain size increased from 15 to 39 μm with increasing austenitization temperature from 870 to 950 °C. Generally, the heating up of alloyed steels results in the dissolution of carbides. This causes an increase in the austenite grain size as well as increasing the concentration of alloying elements in solution, which leads to higher hardenability and lower martensite start temperature (M_s). A decrease in M_s can lead to an increase in retained austenite in the martensitic at room temperature. Normally, the number of martensite packets increases with decreasing austenite grain size. This improves yield and ultimate tensile strengths. However, no significant difference in strength of the steel with different austenite grain sizes is observed (Fig. 1). Upon increasing the austenite grain size from 15 to 39 μm , the martensite packets size increased and the strengthening effect due to grain size decreased. However, the dissolution of carbides containing chromium, molybdenum, and vanadium improves the solid solution strengthening effects. Therefore, a significant decrease in strength with increasing the austenite grain size was not observed. A similar behavior has been reported for 4340 steels (Ref 4).

It can be seen from Fig. 4 that increasing the austenite grain size lowered the impact toughness for tempering temperatures above 400 °C. With the increasing austenite grain size, the size of the martensite packets increase and the grain surfaces, which act as barriers for crack propagation, decrease (Ref 19). In steels with an austenite grain size of 15 μm , in addition to the carbides and low angle grain boundaries, small martensite packets can also hinder crack propagation. Therefore, changing



(a)



(b)

Fig. 5 SEM fractographs of Charpy impact specimens austenitized at 870 °C and tempered at (a) 200 °C and (b) 600 °C

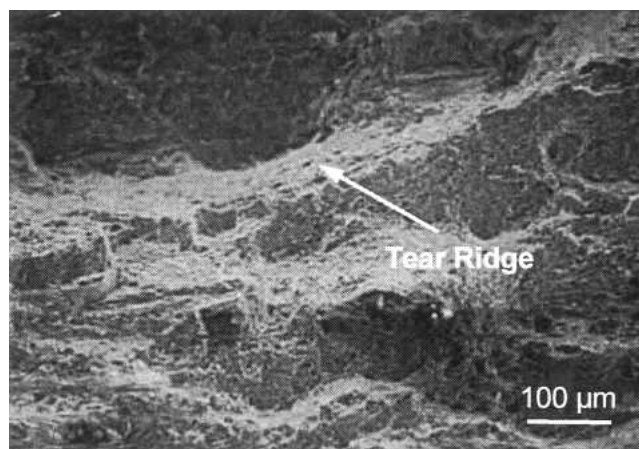


Fig. 6 SEM fractograph of Charpy impact specimen austenitized at 870 °C and tempered at 350 °C, showing the quasi-cleavage fracture

the crack path and increasing the crack distance can improve the toughness. On the other hand, increasing the austenite grain size causes the surface area of the grain boundaries to decrease, and hence, the toughness can decrease through segregation of precipitates to austenite grain boundaries. This leads to brittle steel. In addition, as the surface area of the grain boundaries decreases, the amount of precipitates on the boundaries in-

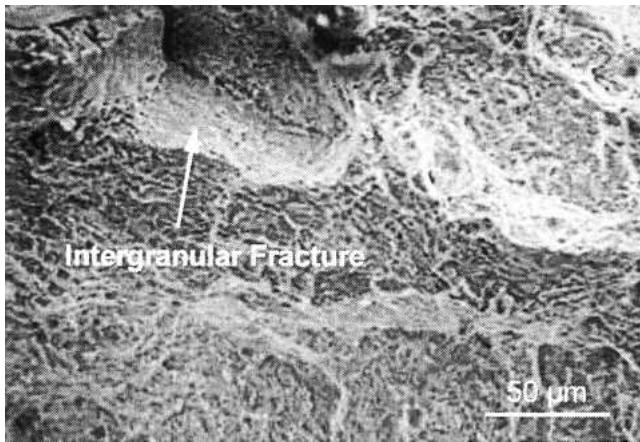


Fig. 7 SEM fractograph of Charpy impact specimen austenitized at 950 °C and tempered at 500 °C, showing an intergranular fracture

creases. The reduction in fracture energy for the specimens with the larger austenite grain size (39 μm) in the temperature range of 250-400 °C (Fig. 4) may be caused by the combined effects of grain growth and the high density of the precipitates at the grain boundaries. Banerji et al. (Ref 10) have observed similar behavior for similar condition for 4340 steel.

The higher impact energy for the large austenite grain size steel (39 μm), compared with that of the smaller one (15 μm), in the tempering range of 200-350 °C, can be attributed to the higher amount of retained austenite in the specimens with large austenite grain size. It has been shown that retained austenite can improve the toughness through crack arresting (Ref 1, 4, 8). The cracks growing through the martensite can be arrested as they reach the retained austenite region. With increased applied stress, the cracks branch and grow out of the martensite region. These cracks need higher energy to grow through the austenite compared with the straight crack growth through the martensite.

As shown in Fig. 4, the impact energy of the specimen with the small austenite grain size is lower than that for the large one for the tempering temperatures below 400 °C. This is opposite for temperatures above 400 °C. It has been stated that with increasing tempering temperature, the retained austenite loses its thermal stability and decomposes to cementite at lath-boundary-retained austenite interface, leading to a loss in toughness (Ref 8, 11, 12). Hence, with a tempering temperature above 350 °C, transformation of austenite to cementite and ferrite occurs, reducing the amount of retained austenite. The difference between impact energies for the small and large austenite grain size steel decreases. Then at 400 °C and above, the grain size effect is overcome and higher impact energy is observed for the small austenite grain size steels.

In the larger austenite grain size steels, intergranular microcracks are larger and crack coalescence is easier. Therefore, as seen in Fig. 4, the trough of impact energy for the large austenite grain size steel (39 μm) is deeper than that for small grain size steel (15 μm). Figure 7 is an example of intergranular fracture for the large austenite grain specimen. The possibility of intergranular fracture increases with increased austenite grain size. The commencement of this mode of fracture is accompanied with microcracks that act as nucleation sites for cleavage fracture (Ref 7, 12).

4. Conclusions

The mechanical behavior and fracture morphology of 32NiCrMoV125 steel treated under different austenitization and tempering conditions was studied. The results obtained can be summarized as follows:

- Increasing the tempering temperature decreased hardness, as well as yield and ultimate tensile strengths, but increased ductility.
- TME was observed in the temperature range of 250-400 °C. Considering the very low amount of impurity elements in this steel, the dominant mechanism for TME is hypothesized to be the decomposition of lath-boundary-retained austenite to cementite.
- Increasing the austenite grain size increased the impact energy for tempering temperatures above 400 °C.
- Fractography of impact specimens showed the mechanisms of intergranular and quasi-cleavage fractures in the TME temperature region (250-400 °C) and ductile fracture for the other tempering temperatures.

Acknowledgments

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