Hydrogen Embrittlement of a Ti-Strengthened 250 Grade Maraging Steel

L.W. Tsay, W.B. Huang, Y.M. Li, and C. Chen

Constant extension-rate tensile tests are performed to investigate the effects of strain rate and environmental hydrogen concentration on the tensile properties of various aged T-250 specimens. The 426 °C (800 °F) underaged specimens are very sensitive to strain rate; the 482 °C (900 °F) peak-aged specimens exhibit a reduced ductility under low strain rates; and the 593 °C (1100 °F) overaged specimens are insensitive to strain rate when tested in air. The excellent resistance to embrittlement of the overaged specimens in gaseous hydrogen could be associated with the extensive formation of reverted austenite and the incoherent Ni_3Ti precipitates. The tensile-fractured surfaces of such specimens reveal a ductile dimple fracture. However, the peak-aged specimens are susceptible to gaseous hydrogen embrittlement, and the embrittled region shows a primary fracture mode of quasi-cleavage. The least resistant to hydrogen embrittlement of the underaged specimens is characterized by a more brittle fracture appearance, that is, intergranular fracture, under a low strain rate or in the gaseous hydrogen environment.

Keywords
fracture, hydrogen embrittlement, strain rate, T-250 maraging steel

1. Introduction

MARAGING STEELS are ultra-high-strength alloys that are used frequently as structural materials for critical applications. T-250, a Ti-strengthened maraging steel, has several desirable characteristics, including high strength, high fracture toughness, and good formability. After solution treatment of the alloy, the heavily dislocated martensite can be hardened by the precipitation of $(NiFe)_3(TiMo)$ particles during aging (Ref 1-3). In addition, the reverted austenite is formed by a diffusion-controlled process (Ref 4) in the aging treatment of maraging steels, and its amount increases with increasing aging temperature (Ref 3, 5). The reverted austenite is most likely to appear at martensite lath and prior austenite grain boundaries (Ref 3, 6). The tensile properties of maraging steels in the underaged condition are very sensitive to strain rate, even if tested in air (Ref 7-10). The strain rate dependence and the brittle appearance of tensile-fractured specimens suggests that hydrogen embrittlement (HE) is involved.

Gaseous hydrogen embrittlement (GHE) is one of the most serious problems in high-strength steels (Ref 11-13). Often associated with a change in fracture mode, relative to fracture behavior without hydrogen (Ref 14-16). GHE is a time-dependent fracture process. The embrittlement appears as decreased ductility in the case of tensile tests when the steels are stressed in a hydrogen-containing environment under low strain rates. The mechanism of HE seems independent of the hydrogen source. However, the kinetics may be greatly affected by various transport processes in a particular environment (Ref 17). Repeatedly, embrittlement in high-strength steels can occur in the presence of hydrogen gas at pressures below 1 atm (Ref 12). The present study investigates the influence of aging treatments on the susceptibility to HE of the T-250 maraging steel. In particular, the effects of strain rate and testing environment on the tensile properties of aged specimens are evaluated. The correlation between microstructures and the fracture behavior of tensile specimens is also discussed.

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2. Experimental Procedure

The T-250 maraging steel, VascoMax T-250, used in this investigation has been purchased from Teledyne Vasco in the plate form, 3.4 mm thick. The chemical composition of the alloy in wt% was 18.63Ni, 3.09Mo, 1.38Ti, 0.006C, 0.002S, 0.004P, and balance Fe. All specimens are solution-treated at 816 °C (1500 °F) for 1 h and then air-cooled. The aging treatments are performed at 426 °C (800 °F), 482 °C (900 °F), and 593 °C (1100 °F) for 4 h. The dimension of tensile specimens with a gage length of 25 mm is shown in Fig. 1. An electrodischarge machine with a wire electrode is used to cut these specimens according to specifications. All tensile specimens are then ground and polished to minimize the notch effects. Cracks often initiate at a pinhole and induce premature failure for the underaged specimen if the width of tensile specimens is less than 20 mm (0.78 in.). Constant extension-rate tensile tests are

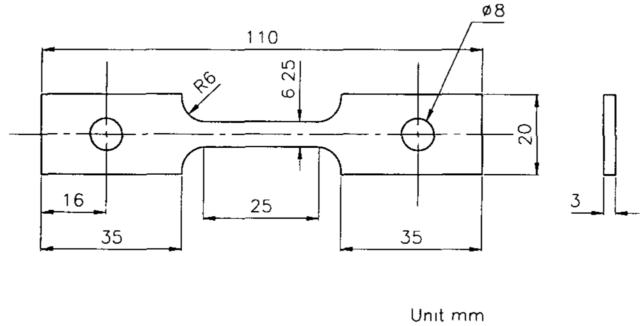

Fig. 1 Schematic showing the dimension of tensile specimens used in this work

conducted in air at room temperature with strain rates of 5×10^{-3}, 5×10^{-4}, and 5×10^{-6} s^{-1}. The corresponding crosshead speeds are 7.5 mm/min (0.3 in./min), 0.75 mm/min (0.03 in./min), and 0.0075 mm/min (0.0003 in./min), respectively. In order to investigate the environmental effect on tensile properties, the specimens are also installed in a stainless steel chamber with controlled atmospheres, that is, vacuum (~2 torr) and hydrogen gas (~2 atm), and tested at a fixed strain rate of 5×10^{-6} s^{-1}. The results of mechanical properties are the average of at least three specimens for each testing condition. The fracture surfaces of tensile specimens are carefully examined by scanning electron microscopy (SEM), particularly the effect of testing conditions, that is, strain rate and environment, on the fracture mode. For microstructural observations of aged specimens, the thin foils for transmission electron microscopy (TEM) are prepared by a standard jet-polisher and examined with a JEOL-2000EX microscope (JEOL Ltd., Medford, MA).

3. Results and Discussion

The ultimate tensile strength and ductility (in terms of elongation and reduction of area, RA) of aged T-250 specimens that are tested in air with three different strain rates are given in Fig. 2. In ordinary tensile tests with a strain rate of 5×10^{-3} s^{-1}, the peak-aged specimens have the optimum combination of strength and ductility, while the overaged specimens have the best ductility among the various aged specimens. However, the underaged specimens do not provide any advantages over the peak-aged specimens. As a result, the T-250 maraging steel is not recommended for use in the underaged condition. In addition, the underaged specimens are very sensitive to strain rate in the tensile tests. When the strain rate is decreased from 5×10^{-3} s^{-1} to 5×10^{-4} s^{-1}, the underaged specimens suffer a great loss in ductility with no apparent change in the tensile strength. As the strain rate is lowered further to 5×10^{-6} s^{-1}, not only the ductility but also the tensile strength are decreased significantly. For peak-aged specimens, the tensile strength reveals essentially no change as the strain rate is decreased from 5×10^{-3} s^{-1} to 5×10^{-6} s^{-1}. When the strain rate is decreased from 5×10^{-4} s^{-1} to 5×10^{-6} s^{-1}, a small drop in elongation and an obvious decrease in RA are observed. The decline in RA implies that HE of the peak-aged specimens still occurs in air under a low strain rate, for example, 5×10^{-6} s^{-1}. No embrittlement phenomenon or strain-rate dependence is observed for specimens in the overaged condition.

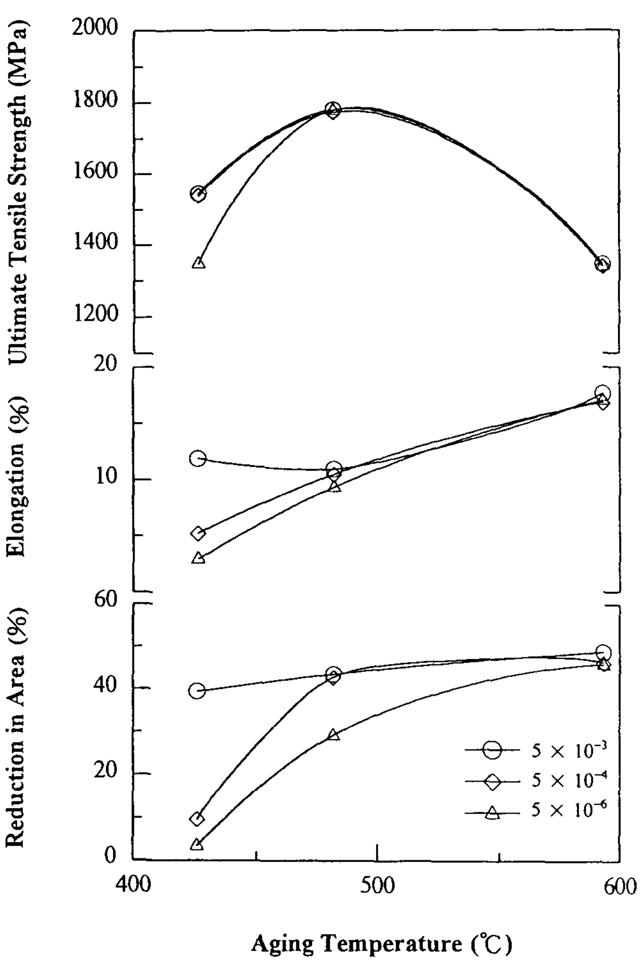

Fig. 2 Tensile strength and ductility of aged T-250 specimens tested in air with three different strain rates

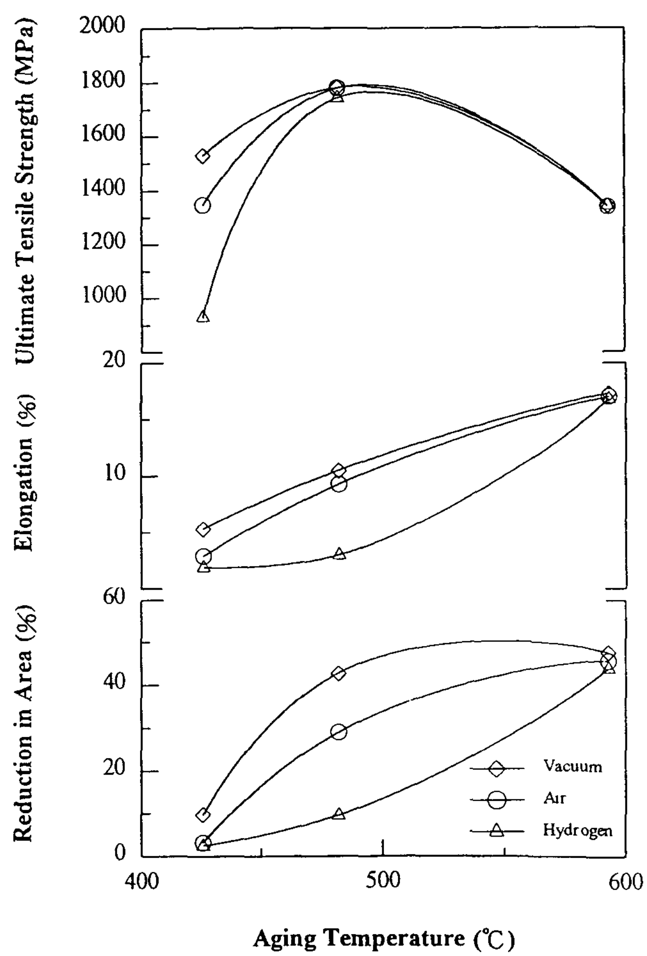

Fig. 3 Tensile results of aged specimens tested under vacuum, air, and gaseous hydrogen environments at a strain rate of 5×10^{-6} s^{-1}

The tensile results of aged specimens that are tested under various environments are shown in Fig. 3. The results indicate that the tensile strength and ductility of the underaged specimens decrease most significantly with increasing hydrogen in the environment, that is, vacuum, air, and gaseous hydrogen environments. In the case of the peak-aged specimens, the influence of hydrogen on the tensile properties becomes severe in the gaseous hydrogen environment, in which a great reduction in ductility is noticed. However, the tensile properties of the overaged specimens show basically no environmental dependence. This implies that T-250 maraging steel is not sensitive to GHE in the overaged condition.

Fractographic observations are made on tensile-fractured specimens with attention paid to the neighborhood of the crack initiation site and the near-surface region. Distinct fracture features between these locations and the interior are observed for specimens susceptible to HE, that is, the brittle appearance for the former and a dimple fracture for the latter. Recent work suggests that the transport of hydrogen to the crack initiation site results in strain-rate dependence (Ref 18). Furthermore, the decrease in strain rate causes the decline in fracture stress in hydrogen (Ref 19). The underaged specimens tested in air with a strain rate of 5×10^{-3} s^{-1} reveal a ductile fracture mode (Fig. 4a). However, the fracture appearance (Fig. 4b) is brittle with quasi-cleavage and some secondary cracks along prior austenite grain boundaries if the strain rate is reduced to 5×10^{-4} s^{-1}. By further lowering the strain rate to 5×10^{-6} s^{-1}, intergranular together with quasi-cleavage fractures (Fig. 4c) are observed on the tensile-fractured surfaces of the underaged specimens. If the same-aged specimens are tested in gaseous hydrogen at a 5×10^{-6} s^{-1} strain rate, the fracture appearance is intergranular in nature, as shown in Fig. 4(d). The greater extent of intergranular cracking also represents the longer period for subcritical crack growth and is more susceptible to HE of the specimens. The decrease in strain rate or the increase in hydrogen of the environment would greatly increase the susceptibility to HE for the underaged specimens. For the peak-aged specimens, quasi-cleavage (Fig. 5) is observed on tensile-fractured specimens tested in hydrogen gas at a 5×10^{-6} s^{-1} strain rate. In contrast, a ductile dimple fracture is found for such specimens tested in vacuum or air. Nevertheless, the overaged specimens reveal a dimple fracture even for testing in the gaseous hydrogen environment at a 5×10^{-6} s^{-1} strain rate.

Microstructure is known to be one of the most important factors that affects the HE susceptibility of high-strength steels. The resistance to HE can be related to the fracture separation process, which in turn depends on the microstructure and the hydrogen concentration (Ref 20-22). The microstructures in the 593 °C (1100 °F) aged specimens reveal coarse Ni_3Ti incoherent precipitates and a significant amount of austenite at martensite lath and grain boundaries, as shown in Fig. 6(a). The

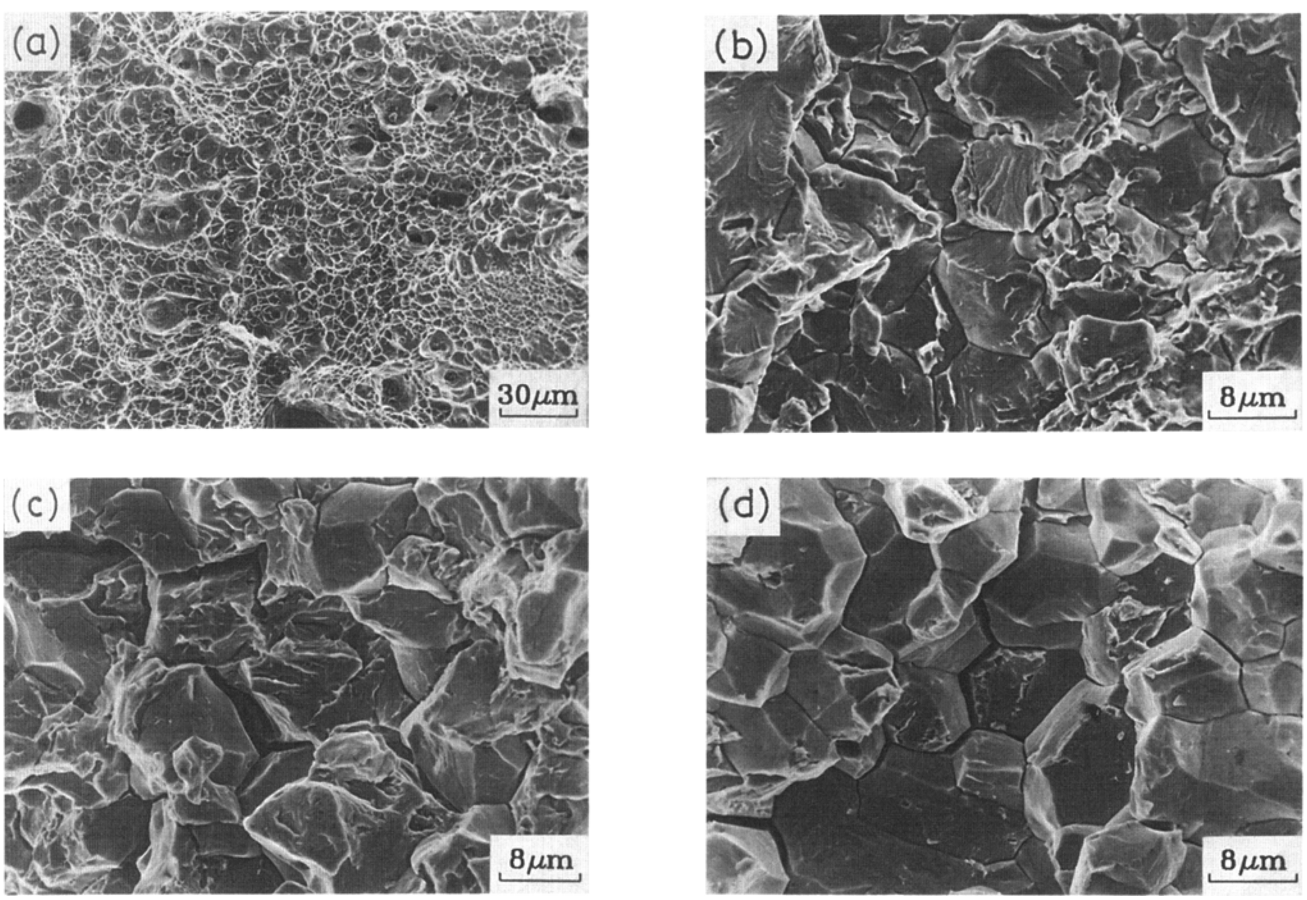

Fig. 4 Fractographs of the underaged specimens tested in various environments and strain rates: (a) air and 5×10^{-3} s^{-1}, (b) air and 5×10^{-4} s^{-1}, (c) air and 5×10^{-6} s^{-1}, and (d) gaseous hydrogen and 5×10^{-6} s^{-1}

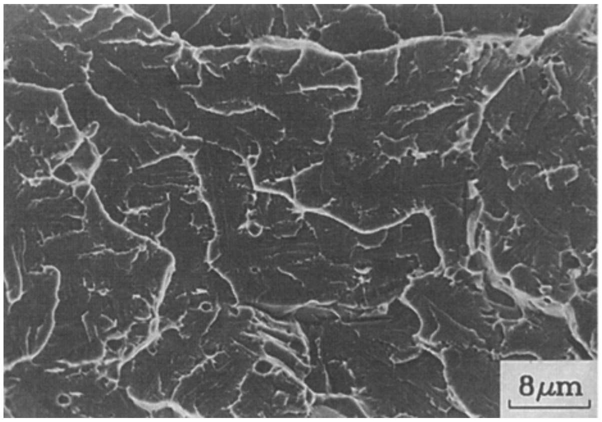

Fig. 5 Fractograph of the peak-aged specimen tested in gaseous hydrogen with a strain rate of $5 \times 10^{-6}\ s^{-1}$

extensive formation of reverted austenite of the overaged specimens not only causes the substantial change in mechanical properties but also affects the susceptibility to HE of the alloy. For the peak-aged specimens, reverted austenite is seldom observed, but the precipitation of partially coherent Ni_3Ti within the matrix as well as boundaries is evident, as shown in Fig. 6(b). In the case of underaged specimens, the presence of very fine and coherent Ni_3Ti precipitates with no reverted austenite is found, as illustrated in Fig. 6(c). These coherent precipitates are much smaller than those in the peak-aged specimens.

For precipitation-strengthening alloys, the deformation behavior could be related to the degree of coherency of the precipitates. The slip mode is often used to describe the appearance of surface slip lines, as either being planar or wavy after plastic deformation (Ref 23). A transition to wavy slip from planar slip can be made by changing the coherency of precipitates, for example, the change from coherent to incoherent precipitates. Since the coherent precipitates can be shared (Ref 24), and hydrogen atoms can be transported (Ref 25-27) by mobile dislocations, dislocation pileups could induce intense stress concentration at grain boundaries with a considerable amount of hydrogen. In addition, the transport of hydrogen to the crack-initiation site is also time-dependent (Ref 18). The fast movement of dislocations associated with high strain rates enhances the dislocation escaping from the hydrogen atmosphere, resulting in less susceptibility to HE. Under low strain rates, more time is available for hydrogen to diffuse to detrimental sites. Consequently, the underaged specimens that contain coherent precipitates are very sensitive to strain rate (Fig. 2). For example, these specimens have a high susceptibility to HE at a $5 \times 10^{-6}\ s^{-1}$ strain rate and a low susceptibility to HE at a $5 \times 10^{-3}\ s^{-1}$ strain rate. On the other hand, high stress and hydrogen concentrations at the grain boundaries promote the formation of intergranular fracture for the underaged specimens with a decreasing strain rate and/or with increasing hydrogen in the environment. The foregoing results are in agreement with the investigation of Soeno and others (Ref 7-10), in which the strain rate dependence decreases as the precipitates lose their coherency by raising the aging temperature.

The decrease in strain rate has less influence on tensile ductilities in air for the peak-aged specimens tested in Fig. 2. The

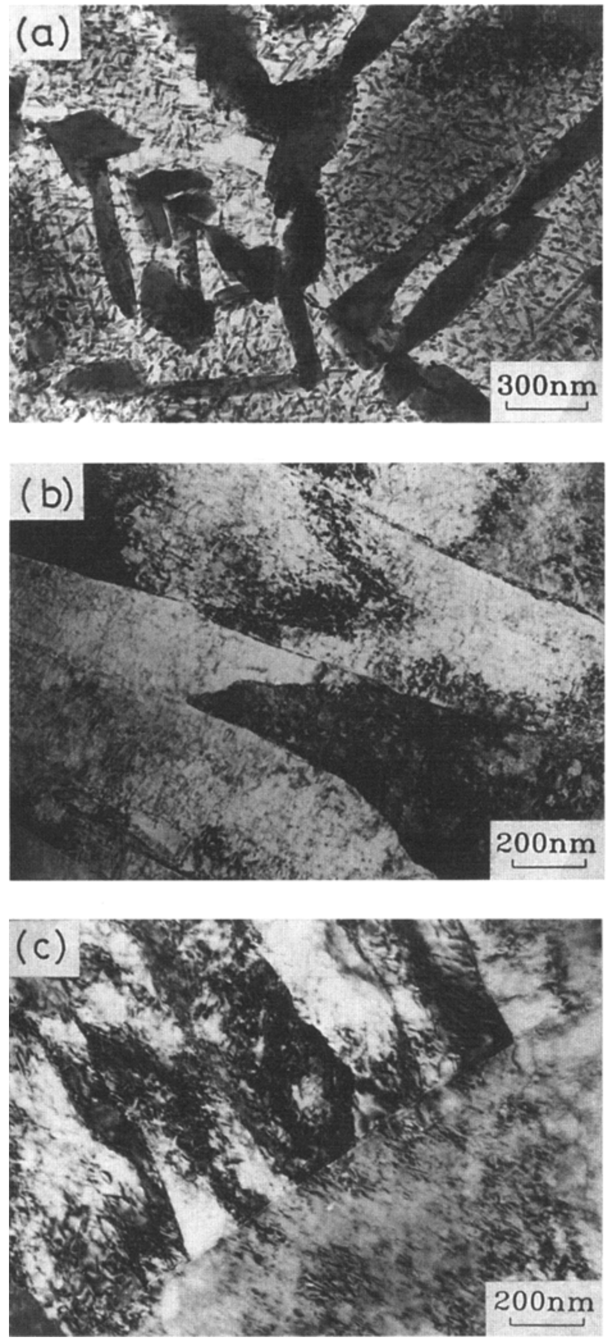

Fig. 6 TEM micrographs of (a) the 593 °C (1100 °F) overaged, (b) the 482 °C (900 °F) peak-aged, and (c) the 426 °C (800 °F) underaged specimens

reduced susceptibility to HE could be attributed to the less planar slip of partially coherent precipitates in such specimens. However, the peak-aged specimens are still susceptible to GHE and exhibit quasi-cleavage fracture (Fig. 5). The excellent resistance to embrittlement in gaseous hydrogen of the overaged specimens could be associated with the extensive formation of reverted austenite and the incoherent precipitates. The good HE resistance of reverted austenite and the wavy slip of the alloy containing incoherent precipitates in tensile tests are possible reasons why the overaged specimens are immune to GHE.

The tensile-fractured surfaces of the overaged specimens show a ductile dimple fracture even at the near-surface region where the influence of hydrogen is most severe.

4. Conclusion

- The tensile properties deteriorate most significantly with a decreasing strain rate or with increasing hydrogen in the environment for the underaged specimens. Such effects are less severe for specimens in the peak-aged condition. However, neither the embrittlement phenomenon nor the loss in tensile properties are found in the overaged T-250 specimens.
- The excellent resistance to HE of the overaged specimens is attributed to not only the presence of incoherent Ni_3Ti precipitates but also to the extensive formation of reverted austenite. The tensile-fractured surfaces of these specimens reveal a ductile dimple fracture even for testing in the gaseous hydrogen environment.
- Fine and coherent precipitates in the underaged specimens could lead to intergranular fracture, particularly for testing in gaseous hydrogen. In the case of the peak-aged specimens, the existence of partially coherent precipitates would result in less susceptibility to HE. This is consistent with the fractographic observation that a less brittle fracture mode, such as quasi-cleavage, is observed for the peak-aged specimens tested in gaseous hydrogen.

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References

1. D.M. Vanderwalker, *Metall. Trans. A*, Vol 18 (No. 7), 1987, p 1191
2. D.M. Vanderwalker, TEM Study of Precipitation in Maraging Steels, *Symposium of TMS Annual Meeting*, (Phoenix, AZ), TMS, 1988, p 255
3. V.K. Vasudevan, S.J. Kim, and C.M. Wayman, *Metall. Trans. A*, Vol 21 (No. 10), 1990, p 2655
4. A. Goldberg and D.G. O'Connor, *Nature*, Vol 213 (No. 1), 1947, p 170
5. H.J. Rack and D. Kalish, *Metall. Trans.*, Vol 2 (No. 11), 1971, p 3011
6. D.T. Peters, *Trans. ASM*, Vol 61 (No. 1), 1968, p 62
7. K. Soeno, T. Kuroda, and K. Taguchi, *Trans. Iron Steel Inst. Jpn.*, Vol 19 (No. 8), 1979, p 484
8. K. Soeno, *Trans. Iron Steel Inst. Jpn.*, Vol 21 (No. 7), 1981, p 469
9. K. Soeno and K. Taguchi, *Trans. Iron Steel Inst. Jpn.*, Vol 21 (No. 9), 1981, p 618
10. K. Soeno and M. Tsuchiya, *Trans. Iron Steel Inst. Jpn.*, Vol 22 (No. 11), 1982, p 848
11. R.P. Gangloff and R.P. Wei, *Metall. Trans. A*, Vol 8 (No. 7), 1977, p 1043
12. D.P. Williams and H.G. Nelson, *Metall. Trans.*, Vol 1 (No. 1), 1970, p 63
13. H.G. Nelson, D.P. Williams, and A.S. Tetelmen, *Metall. Trans.*, Vol 2 (No. 4), 1971, p 953
14. D. Hardie and S. Liu, *Corros. Sci.*, Vol 38 (No. 5), 1996, p 721
15. R.A. Oriani and P.H. Josephic, *Acta Metall.*, Vol 22 (No. 9), 1974, p 1065
16. I.O. Shim and J.G. Byrne, *Mater. Sci. Eng. A*, Vol 123 (No. 2), 1990, p 169
17. H.K. Birnbaum, Hydrogen-Related Failure Mechanisms in Metals, *Symposium of TMS-AIME*, TMS, 1979, p 326
18. J.R. Buckley and D. Hardie, *Corros. Sci.*, Vol 34 (No. 1), 1993, p 93
19. T.S. Sudarshan, M.R. Louthan, Jr., and R.P. McNitt, *Scr. Metall.*, Vol 12 (No. 9), 1978, p 799
20. C.D. Beachem, *Metall. Trans.*, Vol 3 (No. 2), 1972, p 437
21. C. Chen, A.W. Thompson, and I.M. Bernstein, *Metall. Trans. A*, Vol 11 (No. 10), 1980, p 1723
22. A.W. Thompson, Effect of Metallurgical Variables on Environmental Fracture of Engineering Materials, *Symposium of TMS-AIME*, TMS, 1979, p 379
23. T.L. Johnston, R.G. Davies, and N.S. Stoloff, *Philos. Mag.*, Vol 12, 1965, p 305
24. G.S. Chen and D.J. Duquette, *Metall. Trans. A*, Vol 23 (No. 5), 1992, p 1551
25. C. Hwang and I.M. Bernstein, *Scr. Metall.*, Vol 17 (No. 11), 1983, p 1299
26. G.M. Pressouyre, *Acta Metall.*, Vol 28 (No. 7), 1980, p 895
27. J.K. Tien, A.W. Thompson, I.M. Bernstein, and R.J. Richards, *Metall. Trans. A*, Vol 7 (No. 7), 1976, p 821