Hydrogen-induced Delayed Fracture Properties for Ultra-high Strength Low-alloy Steels Processed by Thermomechanical Treatments; Ausforming vs Warm Tempforming

Yuuji Kimura[1], Taku Moronaga[2] and Tadanobu Inoue[1]

[1]*Research Center for Structural Materials, National Institute for Materials Science, Tsukuba, Ibaraki 305-0047, Japan*
[2] *Electron Microscopy Unit, National Institute for Materials Science, Tsukuba, Ibaraki 305-0047, Japan*

The present study aimed to make clear the difference between the effects of warm tempforming and ausforming on the hydrogen-induced delayed fracture properties of ultra-high strength steels. Warm tempforming and ausforming treatments were applied to 0.4C-2Cr-1Mo-2Ni steel (in mass%), using multi-pass caliber rolling with a rolling reduction of 78%. The warm tempformed and ausformed samples were respectively annealed and tempered at 570 ºC for 1 h, followed by water-cooling. The hydrogen-induced delayed fracture properties were evaluated by slow-strain-rate testing for hydrogen-pre-charged notched bar specimens and hydrogen immersion testing. The warm tempformed sample exhibited higher resistance to delayed fracture than the ausformed sample at an ultra-high tensile strength of 1.6 GPa, from the balance between the critical diffusible hydrogen content (below which hydrogen embrittlement never occurs) and the maximum diffusible hydrogen content from the usage environment. The difference in the hydrogen-induced delayed fracture property between the warm tempformed and ausformed samples was discussed in association with the microstructural evolution inside the elongated austenite grains.

Keywords: *low-alloy steel, martensitic structure, thermomechanical treatment, delayed fracture*

1. Introduction

Low-alloy martensitic steels with tensile strength of 1.5 GPa or over generally have poor hydrogen-induced delayed fracture properties, limiting their structural applications. Ausforming (AF) is a thermomechanical treatment in which metastable austenite is plastically deformed before martensitic transformation. On the other hand, warm tempforming (TF) is a thermomechanical treatment that deforms tempered martensite at elevated temperatures. In both thermomechanical treatments, the hydrogen-induced delayed fracture properties were reported to be significantly improved in medium-carbon low-alloy steels, when compared with conventional quenched and tempered treatment [1), 2)]. This is thought be due to the suppression of intergranular fracture along the boundaries of prior-austenite grains through the elongation of prior-austenite grains [2)].

In this study, warm tempforming and ausforming treatments were applied to 0.4C-2Cr-1Mo-2Ni steel (in mass%), using multi-pass caliber rolling with a rolling reduction of 78%, and then annealed and tempered at 570 ºC for 1 h, respectively. Hydrogen-induced delayed fracture properties were investigated for the warm tempformed and ausformed samples with ultra-high tensile strength, σ_B of 1.6 GPa [3)].

2. Experimental

0.4C-2Cr-1Mo-2Ni steel with a chemical composition of 0.40 C, 0.27 Si, 0.20 Mn, 2.01 Cr, 1.03 Mo, 2.04 Ni, 0.001 P, <0.001 S, 0.020 Al, 0.0015 N, 0.0040 O and the balance Fe (all in mass%) was used. Figure 1 shows the outlines of thermomechanical treatments. The austenitized bars were water-quenched, tempered at 500 ºC for 1 h, and then warm tempformed by using multi-pass caliber rolling with a rolling reduction of 78% (TF sample). On the other hand, the austenitized bars were air-cooled to 600 ºC and then ausformed by using multi-pass caliber rolling with a rolling reduction of 78%, followed by air quenching (AF sample). The TF and AF samples were annealed and tempered at 570 ºC for 1 h, followed by water-cooling, respectively. Table 1 shows the tensile properties of TF and AF samples. TF and AF samples exhibited an ultra-high σ_B of 1.6 GPa.

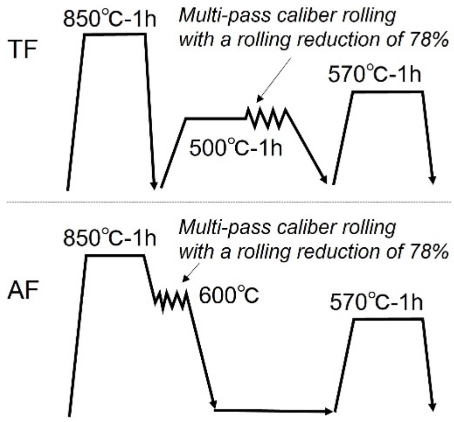

Figure 1 Outlines of thermomechanical treatments.

Table 1 Tensile properties of the TF and AF samples.

Samples	$\sigma_{0.2}$	σ_B	El (%)
TF	1530	1660	15.3
AF	1450	1600	14.5

Slow-strain-rate testing (SSRT) was carried out at a crosshead speed of 0.005 mm/min for hydrogen pre-charged notched bar specimens with a stress concentration factor of 4.9 to evaluate hydrogen embrittlement susceptibility. Hydrogen intrusion behavior was examined by hydrogen immersion testing for round bar specimens with a diameter of 7 mm and a length of 20 mm at 30 ºC in an aqueous solution containing 0.5 mol/L NaCl and 0.01 mol/L HCl (pH = 2). Thermal desorption spectrometry (TDS) analysis with a quadrupole mass spectrometer was carried out to measure the hydrogen content within the samples. Hydrogen that was desorbed up to 300 ºC was regarded as diffusible hydrogen that caused the delayed fracture at room temperature.

3. Results and Discussion

3.1 Microstructure

Figure 2 shows the microstructures of the TF and AF samples. Mean prior-austenite grain size before TF and AF treatments was measured to be 20 μm. The TF sample exhibits an ultrafine elongated grains structure with a strong <110>//RD fiber texture (a, e). On the other hand, the AF sample exhibits an ausformed martensitic structure, where elongated prior-austenite grains consisting of packets that are subdivided by fine blocks (b). Although the initial prior-austenite grain size and the rolling reduction were the same, the TF resulted in a finer, more anisotropic grain structure than the AF. Carbide particle size distribution was observed to be almost the same between the TF and AF samples (c, d). However, the carbide particles in the TF sample were spheroidized and most of the intergranular carbides were aligned along the RD. These carbide particles are considered as cementite. Furthermore, STEM observation further revealed the presence of nanoscale Mo-rich precipitates in both the TF and AF samples. The number of the Mo-rich precipitates was observed to be larger in the AF sample than in the TF sample. Such Mo-rich precipitates were considered to act as effective hydrogen trapping sites.

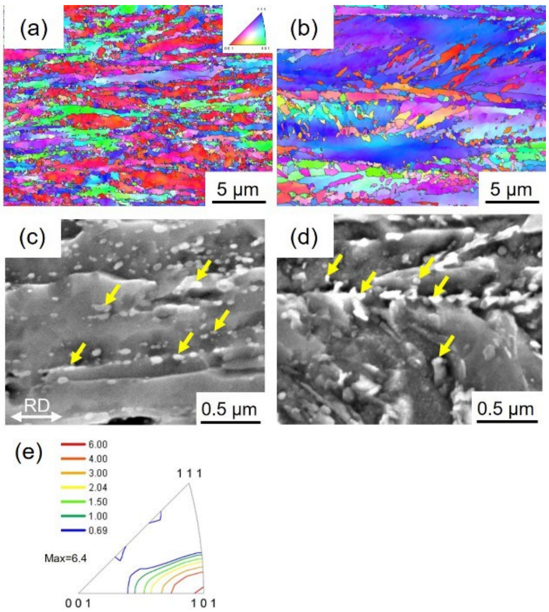

Figure 2 Microstructures of the TF (a,c) and AF(b,d) samples. Inverse pole figure for the RD in the TF sample (e) is also shown.

3.2 Delayed fracture properties

Figure 3 shows the relationship between the notch tensile strength, σ_{NB} and diffusible hydrogen content, H_D of the TF and AF samples. The σ_{NB} was calculated by dividing the maximum tensile load by the initial minimum cross-sectional area at the notch. Hydrogen charging resulted in the decrease of σ_{NB} in both the TF and AF samples, indicative of hydrogen embrittlement. The AF and TF samples exhibited similar hydrogen embrittlement susceptibility. Here, the critical hydrogen content, Hc, below which fracture does not occur at the applied stress corresponding to the σ_{NB}, can be estimated from the σ_{NB}–H_D curves. The respective values of Hc at the applied stress of 0.9σ_B were estimated be 2.2 mass ppm for the AF sample and 2.3 mass ppm for the TF sample.

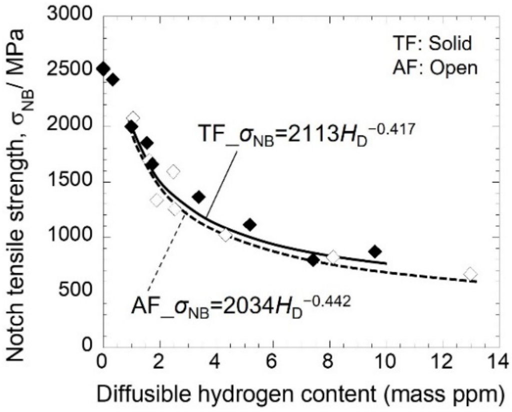

Figure 3 Variations in the notch tensile strength of the TF and AF samples as a function of diffusible hydrogen content.

The maximum content of diffusible hydrogen intrusion from an atmospheric corrosive environment, H_E was estimated by the immersion testing, which simulated the hydrogen uptake from a severe atmospheric corrosive environment on the main island of Okinawa, Japan. The respective H_E values were estimated to be 0.73 mass ppm for TF sample, and 1.48 mass ppm for AF sample. Such hydrogen intrusion behavior mainly depends on the precipitation state of the nanoscale Mo-rich precipitates.

If the Hc is less than H_E, the material might suffer from delayed fracture; the Hc /H_E value is hence used as a measure of hydrogen-induced delayed fracture resistance. The Hc /H_E values at the applied stress of 0.9σ_B can be estimated to be 1.5 for the AF sample and 3.2 for the TF sample. Therefore, the balance between the hydrogen embrittlement susceptibility and the hydrogen intrusion indicates that the TF sample has higher delayed fracture resistance than the AF sample.

4. Conclusions

TF sample demonstrated a higher delayed fracture resistance than AF sample at 1.6 GPa level, based on the balance between the hydrogen embrittlement susceptibility (Hc) and the hydrogen intrusion (H_E). A comparison of the hydrogen embrittlement behavior of TF and AF samples suggested that anisotropic grain structure with a finer transverse grain size and stronger <110>//RD fiber texture was more effective in enhancing the delayed fracture resistance when the rolling reduction and prior-austenite grain size were the same.

Acknowledgments

The study was partly supported by grants from the JSPS KAKENHI Grant Number 19H0246

References

1) S. Yamasaki, M. Kubota and T. Tarui: Nippon Steel Tech. Rep., **80** (1999) 50-55
2) Y. Kimura, T. Inoue and E. Akiyama: Mater. Sci. Eng. A, **703** (2017) 503-512.
3) Y. Kimura, T. Moronaga and T. Inoue: ISIJ Int. **62** (2022) 377-388.