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Caliber-rolled TWIP steel for high-strength wire rods with enhanced hydrogen-delayed fracture resistance

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Twinning-induced plasticity (TWIP) steels were fabricated in wire rods via cold caliber rolling (CCR), and their microstructure, mechanical properties and hydrogen-delayed fracture (HDF) resistance were evaluated. CCR TWIP steels showed a better combination of strength and ductility than conventional bolt steels as a result of CCR inducing all possible $\{111\}\langle 112\rangle$ twins. The HDF resistance of CCR TWIP steels was excellent, owing to the inherently low hydrogen diffusivity of the face-centered cubic structure and the high density of non-diffusible hydrogen trapping sites.

Keywords: Twinning-induced plasticity steel; Hydrogen embrittlement; Caliber rolling; High strength steel; Bolt

The strengthening of automotive steels has been long pursued in efforts to improve fuel efficiency. The strength of steels used for power train components, such as connecting rods and engine block fasteners, is now typically >1 GPa [1]. Such strengthening, however, accompanies counter effects. One serious effect is that steels become more susceptible to catastrophic hydrogen-related failure, often called hydrogen embrittlement or hydrogen-delayed fracture (HDF) [2]. To suppress HDF, introduction of non-diffusible hydrogen trapping sites into the steel matrix has been considered [3,4]. For example, in the case of tempered martensitic steels, which are the most widely used steel grade for power train components, the presence of incoherent TiC precipitates improves the HDF resistance, owing to their high binding energy with hydrogen [5]. However, martensite is inherently vulnerable to HDF, because the hydrogen diffusivity in the ferritic body-centered cubic (bcc) lattice is relatively high. An atomistic simulation showed that hydrogen diffusion in bcc iron is $\sim 10^6$ times faster than that in face-centered cubic (fcc) iron [6]. Several experimental studies have revealed that the hydrogen diffusivity of various ferritic steels is 100–10000 times higher than that of austenitic steels [7–9]. Accordingly, the use of austenitic steels for the power train component steel is an alternative means of suppressing HDF.

Austenitic twinning induced plasticity (TWIP) steel is an advanced high-strength steel grade exhibiting superb strain hardenability and, as a result, excellent combined strength: ductility >1 GPa, 70% [10,11]. Furthermore, its HDF resistance is reported to be superior to that of other steel grades at similar strength levels, primarily owing to the austenitic matrix [12–14]. In comparison with an austenitic stainless steel, the TWIP steel possesses a higher combination of strength–ductility, and better HDF resistance as a result of TWIP, not transformation-induced plasticity [15,16]. At present, the target components of TWIP steel are mainly autobody inner structures, and therefore the steel is manufactured in the form of a thin plate or sheet. However, most power train components are made from wire rods via forging and upsetting. In order to broaden the applicability of TWIP steel to wire rod products (mainly machinery components), it is necessary not only to understand the microstructure evolution and the corresponding change in mechanical properties of TWIP steel during wire rod processing, but also to examine its formability and HDF resistance in relation to wire rods. While there have been numerous studies on these characteristics of

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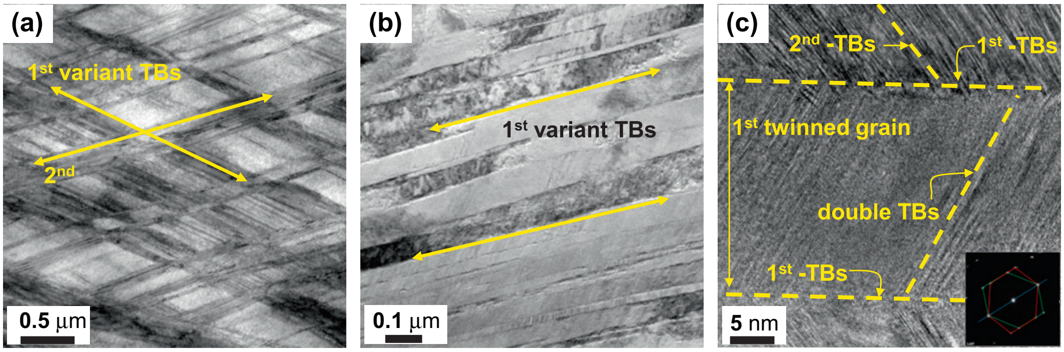

Figure 1. TEM images of TWIP steels prepared using (a) CCR (RA = 54%): multi-variant TB, and (b) cold sheet rolling (RA = 50%): primary variant TB. (c) HR-TEM image of CCR TWIP steel (RA = 54%): with various TB, but no martensite transformation.

TWIP steel sheets, little research has been conducted on TWIP steel wire rods.

In this study, a TWIP steel of Fe–18Mn–1.5Al–0.6C (in wt.%) was fabricated in the form of wire rods via cold caliber rolling (CCR), and the microstructural characteristics and mechanical properties were examined at various CCR strains. In addition, the upsettability of CCR TWIP steel was evaluated by compression tests as a measure of formability. The HDF resistance of CCR TWIP steel was examined by a series of slow strain rate tests (SSRT) and compared with that of conventional pearlitic and tempered martensitic steel specimens.

A high-Mn TWIP steel with a chemical composition of Fe–18Mn–1.5Al–0.6C (wt.%) was prepared in the form of 12-mm-thick hot-rolled plates, which were then machined to rods 11.8 mm in diameter. The rods were caliber rolled sequentially through rolls with diameters ranging from 11.8 mm to 5 mm (equivalent to a reduction of area (RA) of up to ~82%) at room temperature.

Transmission electron microscopy (TEM) images were taken on jet-polished 3-mm-disc samples by Cs-corrected high-resolution TEM (JEOL JEM-2100F) at 200 kV [17]. Tensile tests were conducted at a strain rate of $10^{-3}\ \mathrm{s}^{-1}$ using dog bone specimens with gauge dimensions 12.5 mm long and 2.5 mm in diameter. Compressive tests for evaluating upsettability were performed at a strain rate of $10^{-2}\ \mathrm{s}^{-1}$ using cylindrical specimens with gauge dimensions 4.2 mm long and 2.8 mm in diameter. Tensile and compressive tests were performed using an INSTRON 8862 with a 100 kN load cell. Compressive tests were stopped when the load reached 95 kN, although neither cracks nor abrupt load drops were detected.

The HDF resistance was examined by SSRT with notched samples in the tension mode [18]. A notch simulating a bolt screw increases the hydrogen mobility in the steel during loading [19]. A 60° notch 1 mm deep with a root radius of 0.1 mm was made at the center of the cylindrical sample 6 mm in diameter. Before SSRT, the notched samples were hydrogen-charged in an aqueous solution of 3 mass% NaCl and 0.3 mass% NH_4SCN. During electrochemical hydrogen charging, the current density was fixed at $60\ \mathrm{A\ m}^{-2}$, and the charging time was varied at 0, 48, 72, 96 and 120 h. The hydrogen-charged specimens were then coated with Cd. The Cd coating layer acts as an effective barrier against hydrogen emission during SSRT [20]. SSRT was performed at a stroke speed of $0.005\ \mathrm{mm\ min}^{-1}$. After SSRT, the Cd coating layer was removed, and the hydrogen content was analyzed by thermal desorption spectroscopy (TDS) using a gas chromatograph (Agilent GC 7890A). For the TDS analysis, the specimen was heated to 500 °C at a constant heating rate of $100\ ^\circ\mathrm{C\ h}^{-1}$. The emitted gases were sampled at 5-min intervals under a continuous 99.999% He gas flow.

Sheet-rolling of TWIP steels is typically limited to a thickness reduction of ~70% ($\bar{\varepsilon}\fallingdotseq1.4$), and the present CCR was carried out up to 82% RA ($\bar{\varepsilon}\fallingdotseq3.4$) [21]. The extended working limit of CCR compared with that of sheet rolling is associated with the characteristics that all possible variants of $\{111\}\langle112\rangle$ twins are active for CCR, while only the first and second variants are active for sheet rolling. As shown in Figure 1, at the same RA, CCR TWIP steel (Fig. 1a) exhibited multi-variant twins, but twins with a single variant were dominant in the sheet-rolled TWIP steel (Fig. 1b). Different deformation modes may occur in CCR TWIP steels under compressive stresses in all direction of a circular array. Consequently, all possible twins operate simultaneously, owing to the identical Schmid factor in all grains. In addition, twins developed by CCR were much narrower than those developed by sheet rolling, indicating that growth of the former was more limited by its multi-variant nature. A HR-TEM im-

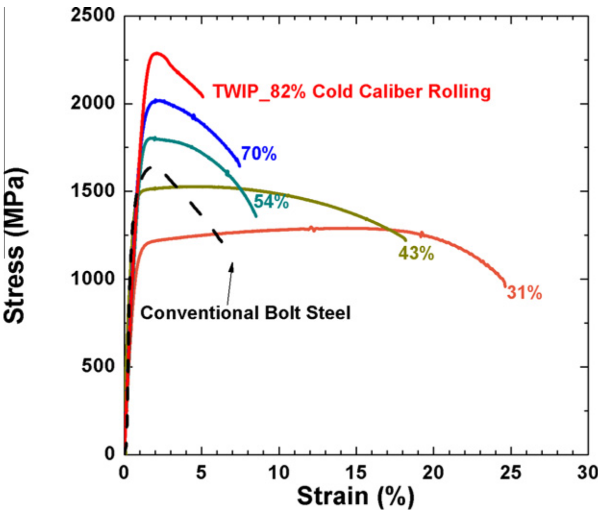

Figure 2. Nominal stress–strain curves of TWIP steels produced using CCR with various RA, and conventional 1600 MPa-grade pearlitic bolt steel.