In-situ Neutron Diffraction Study on Tensile Deformation of Hydrogen-Charged Ultra-High Strength Steel (Postprint)

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Abstract

Time-of-flight neutron diffraction was employed to comparatively investigate the tensile deformation behavior and axial lattice deformation characteristics of hydrogen-charged and uncharged 1250 MPa ultra-high strength steel, and the microstructure morphology and grain orientation features in the fracture region were observed. Under no-load conditions, the axial (110) and (200) interplanar spacings of hydrogen-charged specimens were respectively larger and smaller than those of uncharged specimens, demonstrating that the entry of H atoms into tetrahedral interstitial sites increased the axial (110) interplanar spacing, while the balancing of internal stresses decreased the axial (200) interplanar spacing. Uncharged specimens achieved a tensile strength of 1250 MPa and underwent necking with ductile fracture, whereas specimens containing 8.0$×10^{-6}$ diffusible hydrogen fractured brittlely during stepwise loading to 500 MPa. Neutron diffraction analysis revealed that uncharged specimens exhibited essentially linear elastic deformation when tensile stress was loaded to 500 MPa, but at 700 MPa, axial {200} grains preferentially exhibited non-linear elastic deformation compared to grains of other orientations, and at 800 MPa, axial {110} grains also showed non-linear elastic deformation, with axial {200} grains preferentially undergoing micro-yielding while axial {211} grains remained in the linear elastic stage; in hydrogen-charged specimens, non-linear elastic deformation appeared in axial {110} grains when stretched to 300 MPa, and axial {200} grains also exhibited non-linear elastic deformation at 400 MPa, with axial {110} grains preferentially undergoing micro-yielding while axial {211} grains remained in the linear elastic stage. Fracture cross-section observations revealed that uncharged specimens developed a pronounced axial <110> tensile fiber texture, while hydrogen-embrittled specimens exhibited not only obvious grain boundary crack initiation but also transgranular crack propagation and local crystal rotation features. Based on the concept of micro-yielding in differently oriented grains, it was explained that hydrogen charging causes preferential micro-yielding in axial {110} grains rather than in axial {200} grains, while brittle fracture occurs via hydrogen-accompanied micro-region plastic deformation.

Full Text

In Situ Neutron Diffraction Study of Tensile Deformation in Hydrogen-Charged Ultra-High Strength Steel

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Abstract

The tensile deformation behavior and axial lattice strain response of 1250 MPa ultra-high strength steels with and without hydrogen charging were comparatively investigated using time-of-flight neutron diffraction, complemented by fracture morphology and microstructure observation. Before tensile loading, the axial (110) lattice plane spacing of the hydrogen-charged specimen was larger than that of the non-charged specimen, while the axial (200) lattice plane spacing was smaller, suggesting that hydrogen atoms occupying tetrahedral sites promoted the expansion of axial (110) lattice plane spacing while balanced internal stress caused a corresponding reduction in axial (200) lattice plane spacing. The non-charged specimen underwent necking and ductile fracture after reaching 1250 MPa tensile strength, whereas the specimen containing 8.0$×10^{-6}$ diffusible hydrogen fractured brittlely at 500 MPa during stepwise loading. Neutron diffraction analysis revealed that in the non-charged specimen, deformation remained essentially linear-elastic up to 500 MPa; however, at 700 MPa, axial {200} grains exhibited preferential non-linear elastic deformation compared to other orientations, and at 800 MPa, axial {110} grains also showed non-linear elastic behavior. This indicates that axial {200} grains underwent preferential microyielding while axial {211} grains remained in the linear-elastic regime. In contrast, the hydrogen-charged specimen showed non-linear elastic deformation in axial {110} grains at 300 MPa, followed by axial {200} grains at 400 MPa, demonstrating that axial {110} grains underwent preferential microyielding while axial {211} grains remained linear-elastic. Longitudinal sectioning observations revealed a pronounced axial <110> tensile fiber texture in the non-charged specimen, while the hydrogen-embrittled specimen exhibited not only significant grain boundary crack initiation but also transgranular crack propagation and local crystal rotation characteristics. Based on the concept of orientation-dependent microyielding, we explain that hydrogen charging promotes preferential microyielding in axial {110} grains rather than axial {200} grains, leading to brittle fracture accompanied by microscale plastic deformation.

Keywords: brittle fracture, hydrogen charging, local plastic deformation, high strength low alloy steel, neutron diffraction, lattice strain

Introduction

The use of high-strength steel can significantly reduce structural weight, improve energy efficiency, and lower equipment maintenance costs. During long-term service, hydrogen embrittlement has long been recognized as a major low-stress failure mechanism in high-strength steels, prompting extensive research on hydrogen embrittlement mechanisms, material susceptibility, delayed fracture evaluation, and mitigation methods. Recently, the large neutron scattering cross-section and high penetration depth of hydrogen and its isotopes have been exploited to investigate hydrogen-material interactions and their relationship with microstructure. For instance, Nash et al. used time-of-flight neutron diffraction to study strain field evolution at crack tips in compact tension fatigue specimens under constant load, finding that hydrogen charging increased average strains across all crystallographic orientations at various distances from the crack tip. Hoelzel et al. employed angle-dispersive neutron diffraction to examine the effects of high-pressure, low-temperature (80 K) hydrogen and deuterium charging on austenitic stainless steel crystal structure, revealing that hydrogen occupies octahedral interstitial sites in austenite with increasing unit cell volume. Castellote et al. studied cold-drawn pearlitic steel under various hydrogen charging conditions using angle-dispersive neutron diffraction, also demonstrating hydrogen-induced ferrite unit cell expansion. Ishikawa et al. investigated low-temperature hydrogen-induced cracking in welded joints, showing that tensile residual stresses from post-weld cooling combined with hydrogen enrichment near joints caused grain boundary embrittlement and cracking. These studies demonstrate that neutron diffraction serves as a powerful tool for investigating delayed fracture and hydrogen embrittlement in high-strength steels.

In developing large offshore platform structures for accelerated marine resource exploitation, the safe and effective use of ultra-high strength steel is a critical technical prerequisite. Offshore platforms must withstand dynamic tensile loads from storms, waves, and ocean currents while operating year-round in harsh marine environments, requiring high strength, ductility, low-temperature toughness, and particularly low environmental fracture sensitivity combined with excellent corrosion resistance. Building upon previously published neutron diffraction results for non-charged specimens, this work compares the lattice strain characteristics of ultra-high strength mooring chain steel under tensile deformation with and without hydrogen charging, and discusses the mechanism by which diffusible hydrogen affects microyielding and plastic deformation through fracture surface microstructure observation.

Experimental Procedures

The experimental steel had a chemical composition of 0.22C-0.25Si-0.70Mn-3.40(Cr+Ni+Mo)-0.13(Nb+Ti) (mass fraction, %). After continuous casting, hot rolling, and peeling, 87 mm diameter round bars were produced. These were then formed into 84 mm diameter mooring chains through flash welding, followed by heat treatment: water quenching after heating at 1273 K for 1 h, then tempering at 873 K for 1 h. Standard cylindrical tensile specimens were extracted from the r/3 position (where r is the radius) of the straight section of chain links, with no significant microstructural or macroscopic mechanical property variations between specimens. While notched tensile specimens are typically used to accentuate hydrogen embrittlement through stress concentration, this study employed smooth specimens (8 mm diameter, 30 mm gauge length) to obtain larger diffraction sampling volumes and more uniform deformation regions, thereby reducing neutron diffraction data acquisition time while still capturing hydrogen embrittlement phenomena.

Electrochemical hydrogen charging was performed in a 3% NaCl + 0.3 g/L NH_4SCN electrolyte for 72 h at a current density of 0.05 mA/cm^2. Since Cd plating, commonly used to prevent hydrogen loss, has strong neutron absorption, this study employed an acidic NH_4Cl Zn plating solution for protective coating. Preliminary tensile tests with 10% uniform deformation confirmed that the Zn coating did not undergo premature delamination. After hydrogen charging and Zn plating, specimens were stored in a refrigerated box (5-7 °C) for 120 h before neutron diffraction testing. Additional charged and plated specimens were stored under the same conditions for Zn coating removal and subsequent thermal desorption spectrometry (TDS) analysis of diffusible hydrogen content.

Neutron diffraction experiments were conducted on the ENGIN-X time-of-flight instrument. The north detector captured spectra reflecting axial deformation characteristics, while the south detector captured radial deformation. Time-of-flight neutron diffraction spectra (time vs. intensity) were converted to interplanar spacing spectra (d vs. intensity) using pre-calibrated conversion coefficients DIFC, DIFA, and ZERO ($t = DIFC \times d + DIFA \times d^2 + ZERO$). Since radial deformation in cylindrical tensile specimens is smaller than axial deformation and more difficult to resolve for hydrogen embrittlement effects, this study focused exclusively on axial deformation comparisons between charged and non-charged specimens. An Instron extensometer monitored macroscopic strain on the specimen gauge section. To ensure proper alignment and eliminate grip clearance effects, a 10 MPa preload was applied before formal tensile loading. Single-peak fitting of neutron diffraction spectra yielded specific (hkl) interplanar spacings (d), and axial elastic strains (e) for grains with {hkl} orientation were evaluated from relative changes in d with respect to initial values ($d_{,0}$).

Fracture surface morphology was examined using an S-4300SE field emission scanning electron microscope (SEM). Longitudinal sections were prepared by wire cutting to observe microstructures near the fracture surface and crack propagation characteristics. Electron backscatter diffraction (EBSD) was employed to analyze local microstructures and secondary crack initiation near the surface layer.

2.1 Hydrogen Charging and Diffusible Hydrogen Analysis

[Figure 1: see original paper] shows the thermal desorption curve for a hydrogen-charged specimen after 120 h storage in the refrigerated box, indicating a diffusible hydrogen content of 8.0$\times 10^{-6}$. Non-charged specimens contained less than 6.0$\times 10^{-8}$ diffusible hydrogen. Literature indicates that the hydrogen diffusion coefficient in high-strength steel at ambient temperature (DFe) is 6.3$\times 10^{-5}$ mm^2/s, while in Zn plating (DZn) it is 6.9$\times 10^{-10}$ mm^2/s. Under room temperature conditions with 8 mm thick Zn plating on a 10 mm diameter steel bar, approximately 650 h is required for 50% loss of initial diffusible hydrogen content. Conversely, the high diffusion coefficient in steel ensures that 120 h storage results in macroscopically uniform hydrogen distribution in cylindrical steel specimens.

2.2 Tensile Testing

Preliminary tests at a strain rate of 5.5$\times 10^{-5}$ s^{-1} (0.1 mm/min crosshead speed) yielded a tensile strength of 1270 MPa, yield strength of 1160 MPa, total elongation of 18.5%, and reduction of area of 60%. During in-situ tensile testing of non-charged specimens, external load control was used below 1000 MPa to obtain accurate elastic strain data; above 1000 MPa, extensometer strain control was employed to obtain accurate plastic strain data, using the stress corresponding to the end of plastic stress relaxation as the average stress during neutron diffraction acquisition. After exceeding 1250 MPa and necking initiation, the extensometer was removed for safety, and crosshead displacement control was used for macroscopic plastic deformation until fracture. For hydrogen-charged specimens, [Figure 2: see original paper] shows the stepwise loading data recorded during in-situ tensile testing. The red curve represents macroscopic strain from the extensometer, while the blue curve shows crosshead displacement. Minimal plastic deformation was recorded before brittle fracture occurred at 500 MPa.

2.3 In-Situ Tensile Lattice Elastic Strain of Non-Charged Specimens

[Figure 3: see original paper] illustrates peak shifts for axial (110) and (200) reflections during tensile deformation. The black curve shows the initial peak shape before loading, the blue curve shows maximum peak shift before necking, and the red curve shows the post-fracture peak shape. The axial (110) diffraction peak exhibits residual compressive strain characteristics ($d_{110} < d_{110,0}$) after unloading. Comparison of the three curves reveals that the (200) peak becomes weaker and broader due to <110> fiber texture formation during tensile deformation. [Figure 4: see original paper] shows the relationship between lattice strain and applied stress for non-charged specimens, including macroscopic strain recorded by the extensometer. The stress corresponding to 0.2% plastic strain (intersection of the red dotted line with the macroscopic strain curve, approximately 1120 MPa) approximates the 0.2% offset yield strength. Results show linear-elastic deformation for all grain orientations up to 500 MPa. The macroscopic elastic modulus from extensometer data is E = 198 GPa, while orientation-specific elastic moduli are E_{110} = 225 GPa, E_{200} = 178 GPa, and E_{211} = 229 GPa. At 700 MPa, axial {200} grains exhibit lattice strains exceeding the linear-elastic prediction (dashed line in [Figure 4: see original paper]), indicating non-linear elastic deformation, while axial {110} and {211} grains remain linear-elastic. At 800 MPa, axial {110} grains show non-linear elastic deformation with strains exceeding the linear-elastic prediction, while axial {211} grains remain linear-elastic and axial {200} grains show reduced deviation, returning to near-linear-elastic behavior.

This non-uniform return to linear-elastic behavior warrants discussion regarding whether it relates to microyielding or merely experimental error. If the latter, it would suggest either no microyielding before macroscopic yielding, or that microyielding is independent of preferential orientation-dependent deformation, making this in-situ tensile method insufficient for characterizing microyielding. We first assume that microyielding occurs before macroscopic yielding and is associated with specific grain orientations. In fully annealed low-carbon steels, distinct upper and lower yield points are observed: the upper yield point corresponds to the maximum non-linear elastic stress at yielding onset, while the lower yield point corresponds to the minimum flow stress during initial plastic deformation. Macroscopic strain is non-uniform; when macroscopic stress reaches the upper yield point, plastic deformation initiates at stress concentrations, forming Lüders bands on the specimen surface while stress drops to the lower yield point. Generally, in ferritic stainless steel, axial {200} grains (with <200>//TD orientation) are "soft"grains with lower elastic modulus (~125 GPa), while axial {110} grains (with <110>//TD orientation) are "hard" grains with higher elastic modulus (~210 GPa). The elastic moduli in this study are higher than literature values with smaller anisotropy, attributed to differences in steel composition and microstructure. Although no distinct macroscopic upper/lower yield points or Lüders bands were observed, the elastic anisotropy suggests that microyielding should preferentially occur in <200>//TD grains, forming a "microscopic upper yield point," which explains the larger non-linear elastic strain in axial {200} grains at 700 MPa. At 800 MPa, as microyielding becomes more significant and reaches the "microscopic lower yield point," the non-linear elastic strain in axial {200} grains decreases slightly, returning to near-linear-elastic behavior (note that local plastic strain cannot be directly inferred from non-elastic strain).

To balance increased macroscopic stress, axial {110} grains bear greater elastic stress than in normal linear-elastic deformation, producing non-linear elastic behavior—i.e., axial {110} grains exhibit larger non-linear elastic strain, reaching their "microscopic upper yield point." At 900 MPa, axial {110} grains show significant yielding at the "microscopic lower yield point" with slightly reduced non-linear elastic strain, while axial {200} grains begin work hardening and can bear greater stress, exhibiting larger elastic strain. At higher stresses, both axial {110} and {200} grains enter the work-hardening regime, gradually exhibiting continuous macroscopic yielding. Thus, the phenomena in [Figure 4: see original paper] are attributed to orientation-dependent microyielding differences. In austenitic stainless steel tensile deformation studied by in-situ neutron diffraction, orientation-dependent microyielding differences (axial {200} grains yielding before axial {111} grains) were observed near 200 MPa (below the 265 MPa yield limit), though this phenomenon was not specifically discussed.

2.4 In-Situ Tensile Lattice Elastic Strain of Hydrogen-Charged Specimens

[Figure 5: see original paper] shows peak shifts for axial (110) and (200) reflections during tensile deformation of hydrogen-charged specimens. The black curve represents the initial peak shape before loading, the blue curve shows maximum shift before brittle fracture, and the red curve shows the post-fracture peak shape. The black dashed line indicates the initial (110) interplanar spacing; the post-fracture peak center shows no significant deviation from the initial $d_{110},_0$, indicating minimal residual stress after unloading—a clear difference from [Figure 3: see original paper]. Due to minimal plastic deformation, no obvious intensity or shape changes are observed in the (200) peak among the three curves. [Figure 6: see original paper] shows the relationship between lattice strain and applied stress for hydrogen-charged specimens, including macroscopic strain recorded by the extensometer. Brittle fracture occurred during holding at 500 MPa before reaching 0.2% plastic strain. Below 200 MPa, all grain orientations exhibit linear-elastic deformation with macroscopic modulus E = 197 GPa and orientation-specific moduli E_{110} = 233 GPa, E_{200} = 177 GPa, and E_{211} = 231 GPa, indicating minimal hydrogen effect on elastic modulus. However, at 300 MPa, axial {110} grains show deviation and non-linear elastic deformation, reaching the {110} "microscopic upper yield point," while axial {200} and {211} grains remain linear-elastic, creating significant heterogeneous non-linear elastic deformation. At 400 MPa, axial {200} grains exhibit obvious non-linear elastic deformation, reaching the {200} "microscopic upper yield point," while axial {110} grains show reduced non-elastic deformation. According to the orientation-dependent microyielding model discussed previously, axial {110} grains have reached the "microscopic lower yield point," while axial {211} grains remain linear-elastic. At 500 MPa, axial {110} grains begin work hardening, and axial {200} grains approach the "microscopic lower yield point" with reduced non-linear elastic deformation. However, until brittle fracture occurs, this microyielding is insufficient to induce non-elastic deformation and microyielding in axial {211} grains.

In non-charged specimens, axial {200} grains preferentially exhibit non-linear elastic deformation followed by microyielding, whereas in hydrogen-charged specimens, axial {110} grains show this behavior. To understand this difference, we must analyze hydrogen distribution in steel: some hydrogen diffuses into the lattice as interstitial solute, while some segregates to defects such as vacancies, dislocations, inclusion boundaries, and grain boundaries. Hydrogen embrittlement depends primarily on diffusible hydrogen content rather than segregated hydrogen. Hydrogen diffusing to lattice defects creates triaxial stress states, similar to crack tips or voids caused by carbides. Hydrogen atoms weaken Fe-Fe bond strength and cleavage plane cohesion, and their accumulation can cause lattice separation and crack formation. Literature predicts this weakening effect is related to low-concentration diffusible hydrogen occupying tetrahedral sites in ferritic steel and octahedral sites in austenitic steel. Additionally, dissolved hydrogen increases dislocation mobility, causing local plastic deformation that produces localized plastic fracture features during low-stress hydrogen embrittlement.

In this study, time-of-flight neutron diffraction was used to compare the effects of hydrogen charging and Zn plating on ferrite crystal structure under no-load conditions. Due to low diffusible hydrogen content, the increase in lattice parameter from full-pattern refinement was not significant (Table 1). However, single-peak fitting analysis of individual reflections (Table 1) shows that after hydrogen charging, axial (110) interplanar spacings are larger than in non-charged specimens, while axial (200) spacings are smaller, and axial (211) spacings show no significant difference. This indicates non-uniform hydrogen distribution in ferrite that depends on grain orientation. Since tetrahedral hydrogen occupancy in ferritic steel is surrounded by four nearest-neighbor (110) planes rather than (200) or (211) planes, the increased (110) spacing is attributed to hydrogen charging, while the decreased (200) spacing results from intergranular stress balancing. The increased (110) spacing reduces atomic bond strength in axial {110} grains, while the decreased (200) spacing relatively increases bond strength in axial {200} grains. Consequently, under hydrogen charging, <110>//TD oriented grains reach the "microscopic upper yield point" at lower applied stress (300 MPa) and begin microyielding. When <110>//TD grains are at the "microscopic lower yield point" with significant yielding, <200>//TD grains act as relatively hard grains and undergo pronounced non-elastic deformation to share the load (400 MPa). At 500 MPa, <200>//TD grains also microyield while <110>//TD grains begin work hardening, but axial {211} grains remain unyielded, placing the material in a microscopically overloaded stress state. Cleavage/quasi-cleavage crack propagation leads to low-stress fracture. Although macroscopic plastic deformation and work hardening are not significant, this microyielding and local plastic deformation can be confirmed by grain rotation or elongation near the brittle fracture surface. Moreover, microyielding and local plastic deformation cause hydrogen segregation at crystal defects, promoting intergranular crack initiation and propagation along grain boundaries under local stress, which combines with cleavage crack propagation from microyielding to produce overall brittle fracture.

2.5 Fracture Surface Observation and Local Grain Orientation Evaluation

[Figure 7: see original paper] shows fracture morphologies of non-charged and hydrogen-charged specimens. Figure 7a indicates that the non-charged specimen exhibits obvious necking, a wide annular shear lip, and distinct longitudinal secondary cracks. Figure 7b reveals microscopic longitudinal secondary cracks and fine dimples in the central region, confirming good ductility. Figure 7c shows that the hydrogen-charged specimen lacks obvious necking but displays 45° local shear fracture features. Figure 7d demonstrates a macroscopic Z-shaped brittle propagation region (circled) surrounded by shear lips. Detailed observation near the white dot in Figure 7d (Figure 7e) reveals significant intergranular fracture and several cleavage facets (quasi-cleavage). Since quasi-cleavage facets are typically connected by local shear, creating an uneven fracture surface, this indicates microscale plastic deformation. The presence of local plastic deformation at the specimen center and shear lips at the outer edges correlates with the locally elongated brittle fracture recorded by the extensometer during in-situ testing. On the other hand, quasi-cleavage facets indicate that the main crack propagated through grain interiors rather than solely along grain boundaries, demonstrating that investigation of intragranular microscale deformation is important for understanding hydrogen embrittlement characteristics.

[Figure 8: see original paper] presents longitudinal section microstructures near fracture surfaces of in-situ tensile specimens. Figure 8a shows that bainitic ferrite grains in the non-charged specimen are significantly elongated along the tensile direction, with most (110) planes oriented perpendicular to the loading direction—characteristic of uniaxial tensile plastic deformation. Necking creates triaxial tensile stress concentration near the fracture surface, generating longitudinal secondary cracks that beneficially enhance fracture toughness. Figure 8b shows microstructure, crack initiation, and secondary crack propagation in the hydrogen-charged specimen. The sharp triple-junction crack in the upper-right box initiated along a grain boundary, but some secondary cracks propagated into grain interiors (red circles), causing crack deflection or branching. Assuming no local plastic deformation during crack propagation, these cracks could be completely closed by rigid crystal rotation or translation. However, the white dashed boundaries cannot fully close with adjacent boundaries near the black dashed lines, indicating local tensile plastic deformation during crack opening. In axial {110} grains (light yellow-green regions within small red circles), crack tips are blunted, showing quasi-cleavage features, while in axial {200} grains (dark red-orange regions within large red circles), crack tips are sharp, showing cleavage features. These observations confirm the previous inference and agree with literature that hydrogen-induced brittle fracture exhibits not only significantly reduced fracture stress but also local plastic deformation.

Conclusions

1. Cylindrical specimens containing 8.0$×10^{-6}$ diffusible hydrogen fractured brittlely at 500 MPa during in-situ tensile loading, while non-charged specimens underwent necking and ductile fracture after reaching 1250 MPa tensile strength, demonstrating clear differences in macroscopic tensile deformation behavior.

2. Under no-load conditions, hydrogen charging increased axial (110) interplanar spacings and decreased axial (200) interplanar spacings compared to non-charged specimens, indicating non-uniform diffusible hydrogen distribution in ultra-high strength steel that depends on grain orientation.

3. The non-uniform hydrogen distribution caused preferential microyielding and accelerated grain rotation in axial {110} grains, leading to deformation behavior different from non-charged specimens where axial {200} grains yielded preferentially.

4. Longitudinal section observations revealed transgranular crack propagation and local crystal rotation in hydrogen-embrittled specimens, confirming that hydrogen-assisted brittle fracture occurs through hydrogen-accompanied microscale plastic deformation that embrittles the matrix.

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Note: Figure translations are in progress. See original paper for figures.