

## In-situ Micromechanics of Hydrogen-induced Deformation in Pearlitic Steels

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Hydrogen is a clean fuel that can facilitate the transition from a fossil-fueled society to that with a net-zero carbon emission. Using the existing natural gas pipeline system to distribute hydrogen is an important strategy to increase the economic feasibility of this transition to a hydrogen economy [1]. However, it is established that the existing pipelines, which are comprised mainly from steels, are susceptible to hydrogen embrittlement [1]. Fracture due to hydrogen embrittlement could lead to hydrogen leakage and, subsequently, serious incidents, considering the flammable nature of hydrogen. A safe implementation of hydrogen energy requires better management of the risk of hydrogen embrittlement in pipeline steels, and this requires a thorough understanding of how hydrogen leads to the failure of the materials.

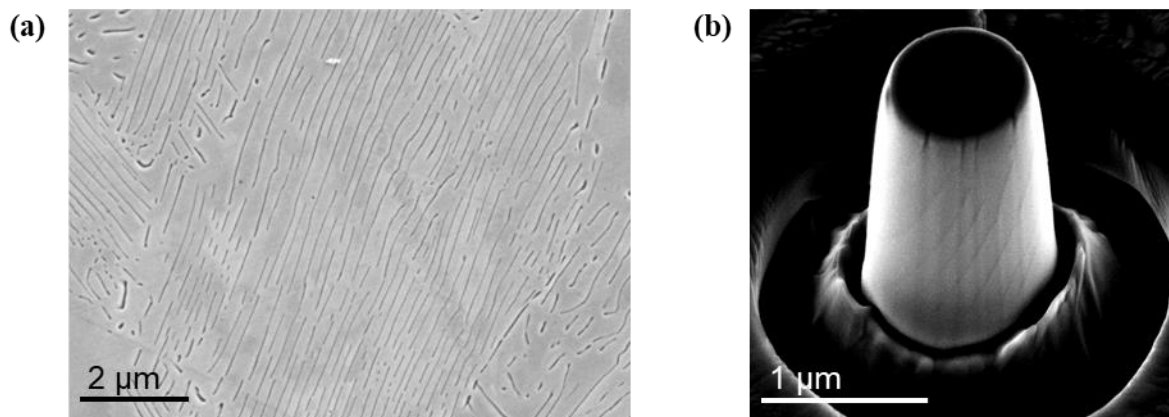
Commercial pipeline steels contain a high-volume fraction of pearlite (up to 30%), which consists of lamellar cementite ( $\text{Fe}_3\text{C}$ ) phases and their interfaces with the ferrite matrix (Figure 1a). It has been hypothesized that the ferrite–cementite interface can attract hydrogen solute atoms which can reduce the cohesive energy of the interfaces and facilitate the local deformation, leading to failure [2]. However, this hypothesis has not yet been experimentally validated. To investigate the effect of hydrogen on the interface, we used an *in-situ* micromechanical compression test in scanning electron microscope (SEM) with micropillar specimens that contain cementite lamella (dark features in Figure 1b). Our transmission electron microscopy analysis (not shown) revealed that the ferrite matrix and cementite have a specific Isaichev crystal orientation relationship of  $(112)_{\text{ferrite}}// (101)_{\text{cementite}}$  and  $[111]_{\text{ferrite}}// [010]_{\text{cementite}}$  [3]. This relationship allowed us to select the ferritic grains with the desired cementite arrangement by using electron backscatter diffraction (EBSD) indexing, and thereby fabricate the micropillars with  $45^\circ$  inclined angles of cementite lamella (as confirmed in Figure 2b and c). After measuring the mechanical properties of uncharged micropillars, we introduced hydrogen into a bulk specimen with prefabricated micropillars via *ex-situ* cathodic hydrogen charging in 0.1 M NaOH in water at 2.2 V for 1 hour. We then promptly conducted the *in-situ* compression on the micropillars within 20 minutes post-charging. The fact that hydrogen would still be present in the bulk specimens at the time of the test was confirmed by using a series of hydrogen thermal desorption analyses (data not shown) for the duration of time required for the *in-situ* mechanical tests in the SEM vacuum chamber.

Figure 2a contains the stress-strain curves of the uncharged and hydrogen-charged pillars, and represent the results of multiple measurements. Decreased yield strength was observed for the hydrogen-charged micropillars with respect to that of the uncharged specimen. The data shown here shows a reduction of the resolved interfacial strength from 0.93 GPa to 0.43 GPa. The curve of the uncharged sample (red in Figure 2a) exhibits several large strain bursts, which correspond to the shear deformation of the ferrite–cementite interface in the post-mortem TEM observation (Figure 2b). In contrast, the hydrogen-charged micropillar underwent a smoother deformation process (blue curve in Figure 2a), and the post-mortem TEM observation of the charged specimen concluded that the bulk strain is mainly the result of dislocation slip in the ferrite (Figure 2c), in parallel to the cementite lamella along the  $\{112\}$  plane [3], a

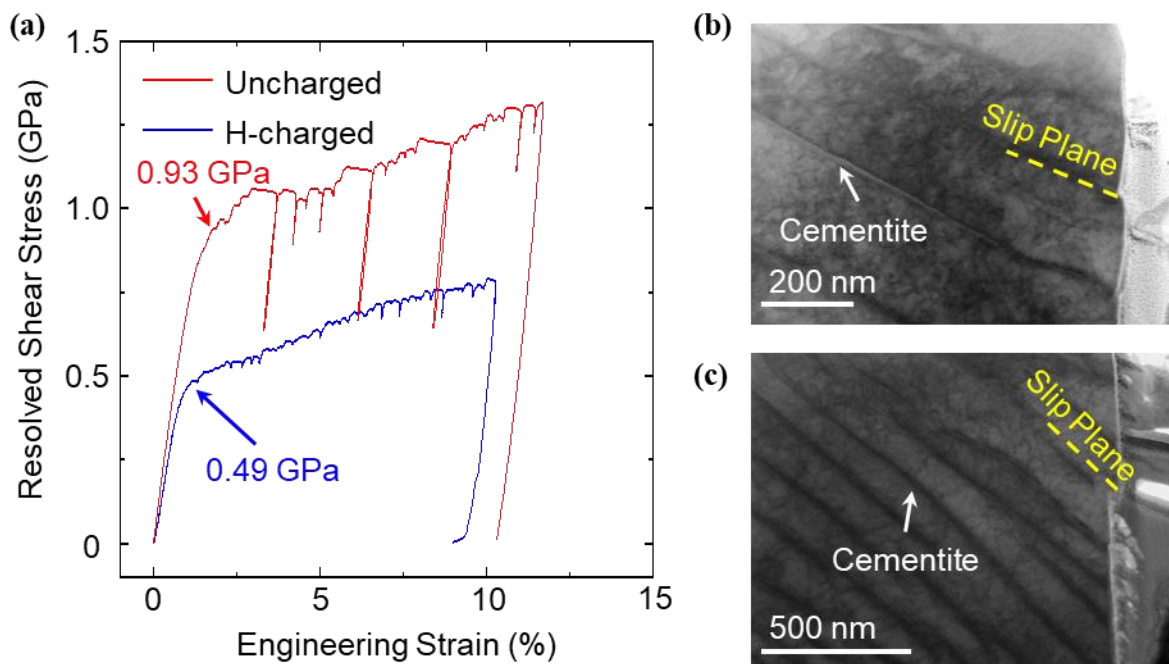
primary slip system of the body-center cubic iron lattice. These results indicate that the presence of hydrogen facilitated the dislocation movement in ferrite, rather than reducing the cohesion of the ferrite–cementite interface, providing clear evidence that hydrogen-enhanced plasticity is responsible for the hydrogen-induced deformation of pearlite, whereas hydrogen-enhanced interface decohesion is not.

### References:

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- [2] Yu, S.-H., et al., *Effects of lamellar structure on tensile properties and resistance to hydrogen embrittlement of pearlitic steel*. *Acta Materialia*, 2019. **172**: p. 92-101.
- [3] Zhou, D. and G. Shiflet, *Ferrite: cementite crystallography in pearlite*. *Metallurgical Transactions A*, 1992. **23**(4): p. 1259-1269.



**Figure 1.** SEM images of (a) the microstructure of the pearlitic specimen and (b) an example of the micropillar of the specimen for in-situ micro-compression tests.



**Figure 2.** (a) Engineering shear stress–strain curves of the uncharged (red) and hydrogen-charged (blue) specimens from the in-situ compression tests. TEM cross-section images of (b) an uncharged and (c) a hydrogen-charged pillar after in-situ compression tests, with slip planes shown by the yellow broken lines.