

# Review of metal carbide nanoprecipitate effects on hydrogen embrittlement of high strength martensitic steel

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## Abstract

**Purpose** – High-strength martensitic steels having strong hydrogen embrittlement (HE) susceptibility and the metal carbide (MC) nanoprecipitates of microalloying elements such as Nb, V, Ti and Mo in the steel matrix can effectively improve the HE resistance of steels. This paper aims to review the effect of MC nanoprecipitates on the HE resistance of high-strength martensitic steels.

**Design/methodology/approach** – In this paper, the effects of MC nanoprecipitates on the HE resistance of high-strength martensitic steels are systematically described in terms of the types of MC nanoprecipitates, the influencing factors, along with numerical simulations.

**Findings** – The MC nanoprecipitates, which are fine and semicoherent with the matrix, effectively improve the HE resistance of steel through the hydrogen trapping effects and microstructure optimization, but its effect on the HE resistance of steel is controlled by its size, number and distribution state.

**Originality/value** – This paper summarizes the effects and mechanisms of MC nanoprecipitates on HE performance of high-strength martensitic steel and provides the theoretical basis for corrosion engineers to design high-strength martensitic steels with excellent HE resistance and improve production processes.

**Keywords** Metal carbide nanoprecipitates, High-strength martensitic steels, Hydrogen embrittlement

**Paper type** Literature review

## 1. Introduction

High-strength martensitic steels are widely used in offshore engineering, petrochemical engineering and nuclear power generation because of their high strength, good ductility and corrosion resistance (Chen *et al.*, 2020; Yao *et al.*, 2008; Zhang *et al.*, 2018). However, high-strength martensitic steels are usually sensitive to hydrogen embrittlement (HE) and the HE susceptibility increases with the steel strength (Barnoush and Vehoff, 2010; Venezuela *et al.*, 2018). Therefore, a lot of research has been conducted to improve the HE resistance of high-strength martensitic steels. Based on the observed local hydrogen concentration, researchers believe that HE in high-strength martensitic steels is mainly the result of combined

effects of hydrogen-enhanced decohesion (HEDE) and hydrogen-enhanced localized plasticity (HELP) (Bechtle *et al.*, 2009; Djukic *et al.*, 2015; Li *et al.*, 2018).

Numerous studies have found that the addition of microalloying elements (Nb, Ti, V and Mo) improves the HE resistance of steel. Metal carbide (MC) nanoprecipitates not only optimize the microstructure and improve the mechanical properties but also increase the hydrogen trap density in the high-strength martensitic steels to improve HE resistance (Wei *et al.*, 2011; Wei and Tsuzaki, 2012).

Based on the existing research, this paper reviews the effects of MC nanoprecipitates on the HE resistance of high-strength martensitic steels from three aspects: the types of MC

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nanoprecipitates, the influencing factors (coherence, trapping sites and size) and numerical simulations.

## 2. Types of metal carbide nanoprecipitates

### 2.1 Influence of NbC nanoprecipitates on hydrogen embrittlement

In recent years, the research on the HE resistance of steel by NbC nanoprecipitates mainly focused on the effect of hydrogen trap status and optimizing the microstructure.

The NbC nanoprecipitates influence the effect of hydrogen traps status in two respects (Fan *et al.*, 2021; Zhang *et al.*, 2015, 2020):

- Hydrogen trapping effects: The uniformly distributed NbC nanoprecipitates act as irreversible hydrogen traps, which can effectively trap hydrogen and prevent hydrogen from the stress concentration region to prevent HE caused by high local hydrogen concentration.
- Grain refinement effects: The NbC nanoprecipitates refine the proto-austenitic grain (PAG) by pinning the grain boundaries, increasing the number of reversible hydrogen traps and making the hydrogen traps evenly distributed, further reducing the local hydrogen concentration and suppressing HEDE.

However, the two respects remain somewhat controversial. Zhang *et al.* (2015) found by quantitative calculations of the trap activation energy that the hydrogen trapping effects of the NbC nanoprecipitates contribute 85% and the grain refinement effects contribute only 15%. However, Seo *et al.* (2020) found that the undissolved NbC nanoprecipitates inhibited the growth of PAG mainly through the pinning effect and increased the amount of PAG boundaries, thus reducing the local hydrogen concentration and improving the HE resistance of tempered martensitic steel.

In term of optimizing the microstructure, Zhang *et al.* (2020) found that the NbC nanoprecipitates reduced the  $\Sigma 3$  grain boundaries, increased the proportion of low-angle grain boundaries, directly pinned hydrogen-dislocation Cottrell atmospheres and hindered dislocation movement and multiplication, which weakened HELP and improved the HE resistance of steel. However, Zhao *et al.* (2021) presented the

opposite result where the NbC nanoprecipitates could weaken the beneficial  $\{111\}$  weave in the martensitic steel, resulting in reduced HE resistance, as shown in Figure 1.

In recent years, researchers have directly observed hydrogen trapping behavior of NbC nanoprecipitates by atom probe tomography (APT). It can be observed that hydrogen is trapped at the NbC nanoprecipitates interface in Figure 2 (Zhang *et al.*, 2020), which directly demonstrates the hydrogen trapping effects at the NbC nanoprecipitates interface. However, whether the interior of the NbC nanoprecipitates trap hydrogen has not been confirmed by the experiments.

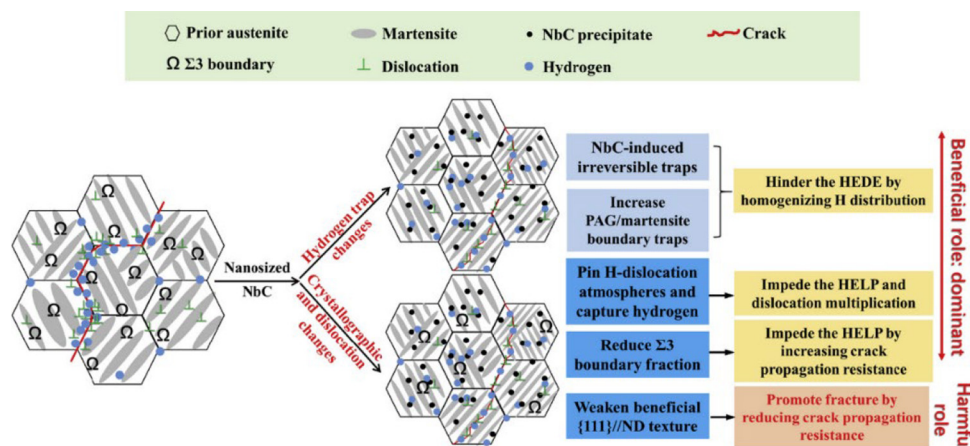
### 2.2 Influence of TiC nanoprecipitates on hydrogen embrittlement

The hydrogen trapping behavior of incoherent TiC precipitates has been investigated in depth by electrochemical permeation and thermal desorption spectroscopy (TDS) (Lee and Lee, 1984, 1987). Wei *et al.* (2003) were the first to observe the hydrogen trapping effects of (semi)coherent TiC when studying 0.42C-0.30Ti steel. Up to now, hydrogen trapping sites of different TiC nanoprecipitates have been investigated.

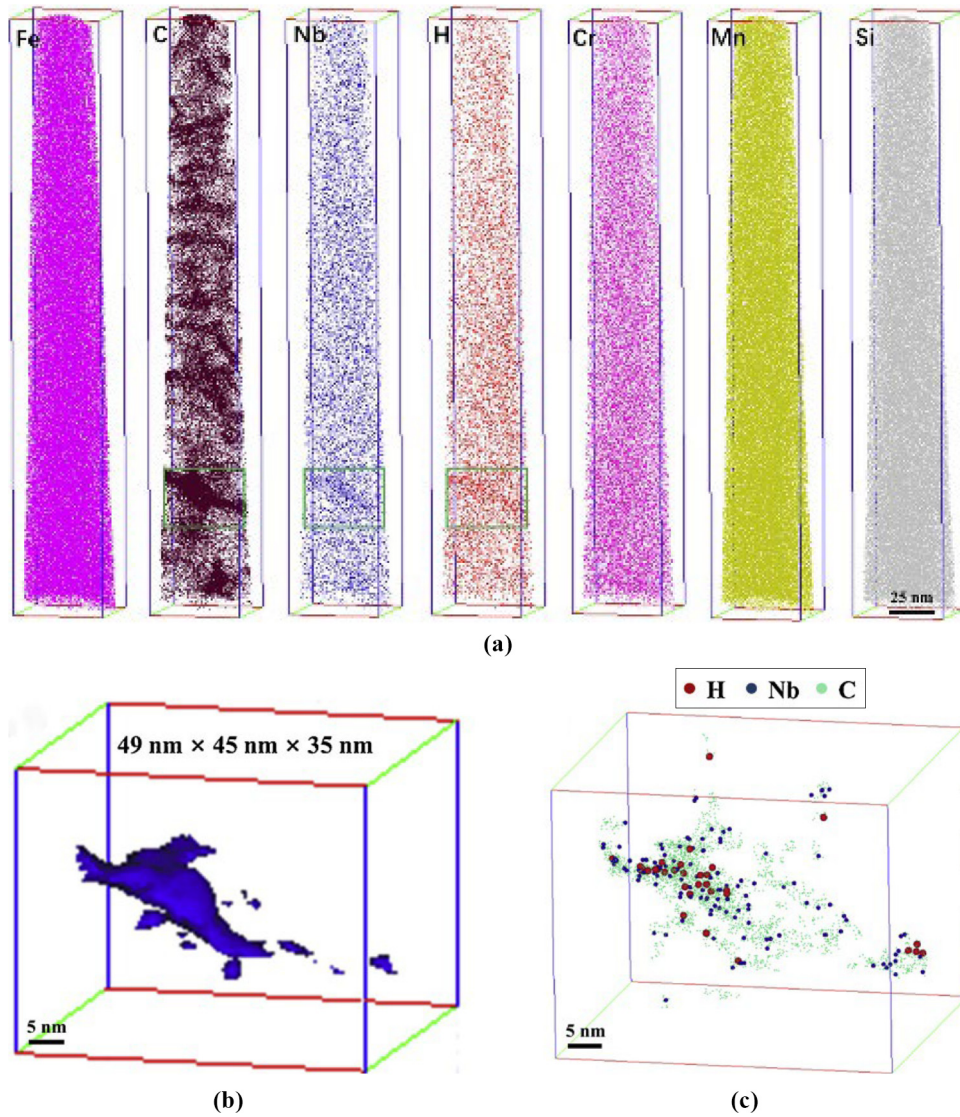
Wei *et al.* (2003, 2004) and Wei and Tsuzaki (2006), systematically investigated the hydrogen trapping behavior of TiC nanoprecipitates in high-strength martensitic steels by TDS and high-resolution transmission electron microscopy (HRTEM). When the steel is tempered at 550°C, the Baker–Nutting semicoherent relationship between the TiC nanoprecipitates and the matrix is  $(100)_{\text{TiC}} // (100)_{\alpha}$ ,  $[011]_{\text{TiC}} // [001]_{\alpha}$ . As the tempering temperature increases, it gradually turns into an incoherent relationship. When tempered at 1,000°C, TiC and the matrix are incoherent relationship and a high-temperature peak appears in the TDS curve in Figure 3(a). The shape of TiC nanoprecipitates also changes from disk-like to spherical in Figure 3(b). They also found that the semicoherent TiC nanoprecipitates trap hydrogen only at the wide interface and the incoherent TiC nanoprecipitates can trap hydrogen at the internal carbon vacancies only during high-temperature tempering.

Some researchers have also studied the effects of TiC nanoprecipitates on the HE resistance of steel from the perspective of tempering time. By charging the tempered

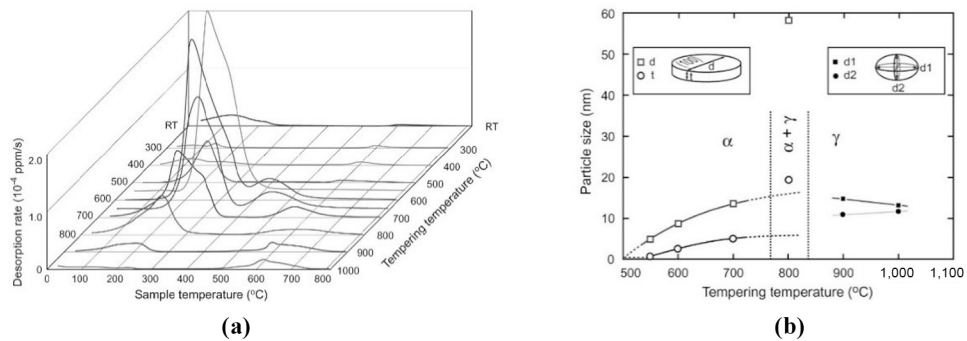
**Figure 1** Schematic illustrating the effects of the NbC nanoprecipitates on the HE in the high-strength martensitic steel (Zhang *et al.*, 2020)



**Figure 2** Element distributions in high-strength martensitic steel (Zhang et al., 2020): (a) atom maps; (b) atom fraction of 7.4% (C + Nb) isoconcentration surfaces of the region enclosed in a green box; and (c) distributions of C, Nb and H atoms inside the isoconcentration surfaces



**Figure 3** Change of TiC nanoprecipitates with increasing tempering temperature (Wei and Tsuzaki, 2012). (a) TDS spectra for the samples with tempering temperature in an argon atmosphere. (b) Change in the size and shape of the TiC nanoprecipitates with tempering temperature





specimens with the same amount of hydrogen, the specimens tempered for 1 h exhibited excellent HE resistance (Depover and Verbeke, 2016).

To visualize the hydrogen trapping sites by TiC nanoprecipitates, Takahashi *et al.* (2010) observed deuterium-filled specimens by APT and found that deuterium was enriched at the interface of TiC nanoprecipitates and this result confirmed that hydrogen was trapped at the TiC/matrix interface.

### 2.3 Influence of VC nanoprecipitates on hydrogen embrittlement

Researchers have extensively investigated the effect of VC nanoprecipitates on the HE resistance of steel in terms of hydrogen trapping and grain refinement (Cheng *et al.*, 2018; Li *et al.*, 2019; Zhao *et al.*, 2021). The content of V in steel has a great influence on the VC nanoprecipitates. With the V content increasing, the proportion of VC nanoprecipitates less than 10 nm increased significantly and the effective trap density increased accordingly (Li *et al.*, 2019). However, the HE resistance of steel and the V content are not proportional. Lee *et al.* (2016) found that as the V content in steel increases, the size and number of VC nanoprecipitates keep increasing. When the content is 0.2 Wt.%, the steel has the best HE resistance, but the large size of undissolved VC easily leads to HE when the content reaches 1.01 Wt.%.

The above discussions are regarding nanosized VC precipitates. Recently, Peral *et al.* (2020) found that submicron VC precipitates also have a strong hydrogen trapping effect. These submicron VC particles, which precipitate along dislocations during tempering, effectively trap hydrogen and improve the HE resistance of steel.

Some researchers have studied the hydrogen trapping sites of VC nanoprecipitates by APT (Takahashi *et al.*, 2012; Chen *et al.*, 2017). As shown in Figure 4, most of hydrogen (actually deuterium) was trapped in the misfit dislocation on semicoherent VC nanoprecipitates interface of ten to dozens of nanometers. Meanwhile, other studies have shown that hydrogen is enriched inside VC nanoprecipitates (Malard *et al.*, 2012).

### 2.4 Influence of Mo-containing precipitates on hydrogen embrittlement

Mo has a large effect on the other carbides precipitates. The addition of Mo to steel promotes the nucleation of MC-type

carbides such as (Ti, Mo)<sub>2</sub>C, (Nb, Mo)<sub>2</sub>C and (Mo, V)<sub>2</sub>C, which can easily form precipitates with diameters less than 20 nm because Mo can not only reduce the interfacial and Gibbs free energies of the precipitates but also reduce the size and retard the coarsening of precipitates (Nagao *et al.*, 2018; Zheng *et al.*, 2018). Therefore, the present study of Mo focuses on the role of coprecipitates with the other microalloying elements on the HE resistance of steels.

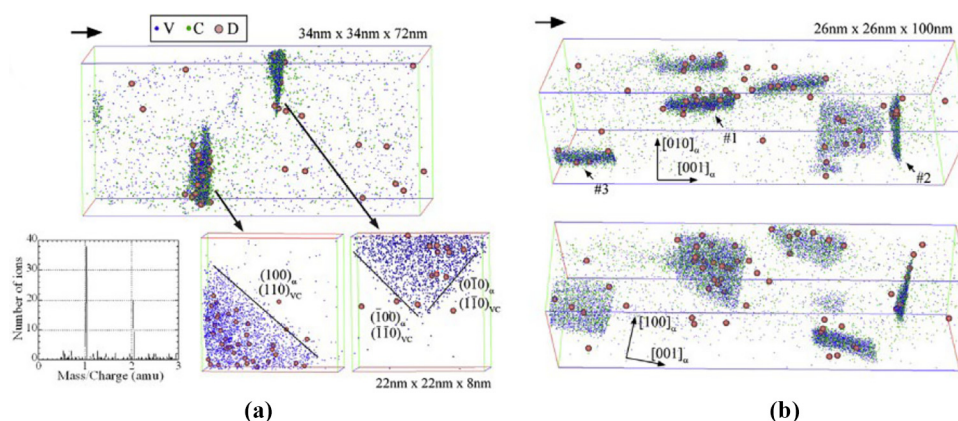
NbC, VC and TiC have the same NaCl-type crystal structure, whereas Mo<sub>2</sub>C has a hexagonal crystal structure (Wei and Tsuzaki, 2012; Lee *et al.*, 2019). It is generally believed that compared with NbC, VC and TiC nanoprecipitates, the hydrogen trapping ability of the Mo<sub>2</sub>C nanoprecipitates is relatively weak (Wei and Tsuzaki, 2012). However, Lee *et al.* (2019) compared the steel with Mo, Cr and V, 1Cr-0.4Mo steel with the Mo<sub>2</sub>C nanoprecipitates had the best HE resistance. As shown in Figure 5, the authors observed by APT that the Mo<sub>2</sub>C nanoprecipitates trapped hydrogen at the coherent interface, which effectively reduced the mean free path of diffusible hydrogen and made the hydrogen evenly distributed to prevent HE caused by high local hydrogen concentration.

## 3. Influencing factors of metal carbide precipitates

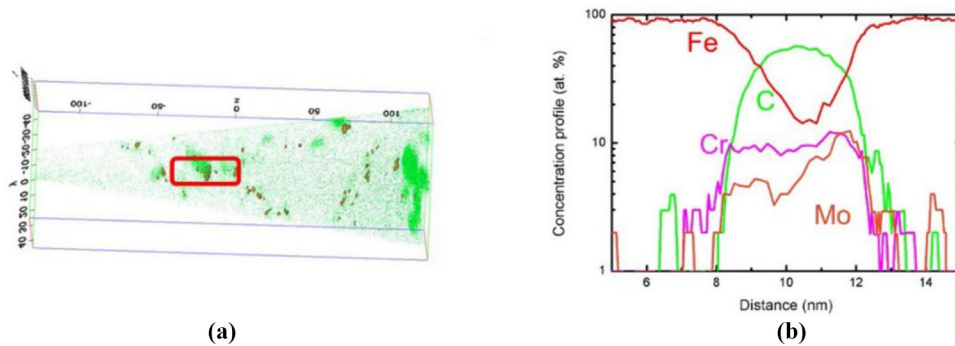
### 3.1 Effect of coherence

The coherent relationship of the MC nanoprecipitates/matrix interface is a critical factor for the hydrogen trapping capacity and HE resistance and it is also related to the binding energy magnitude of the hydrogen trap (Wei and Tsuzaki, 2006; Wei *et al.*, 2011). As listed in Table 1, the binding energy of hydrogen traps at the interface of (semi)coherent nanoprecipitates is slightly higher than grain boundaries and dislocations, which can be used as deep hydrogen traps within the grains. In general, the steel matrix near the fine-sized (semi) coherent nanoprecipitates has a strong coherent strain field, which can irreversibly adsorb a large amount of hydrogen in its lattice interstices. Especially, when defects such as vacancies or dislocations exist at the phase interface, the coherent strain field will enhance their hydrogen absorption capacity (Wei *et al.*, 2015; Shi *et al.*, 2020).

Figure 4 APT observation of hydrogen capture by VC nanoprecipitates (Takahashi *et al.*, 2012)



**Figure 5** APT analysis of 1Cr-0.4Mo steel (Lee *et al.*, 2019): (a) isosurface image of 5-at.% Mo and (b) atomic concentration profile of Fe, C, Cr and Mo in the marked area of (a)



**Table 1** Trapping sites and corresponding hydrogen-trapping activation energies in steel

Trapping sites	$E_b/(\text{kJ}\cdot\text{mol}^{-1})$	Reference
Iron lattice	5.4–7.1	Dwivedi and Vishwakarma (2019)
Cementite particle	11–18	Bhadeshia (2016)
Low-angle grain boundary	17.2–18.6	Dwivedi and Vishwakarma (2019)
Austenite/martensite interface	22	Dwivedi and Vishwakarma (2019)
Dislocation	20–35	Nagao <i>et al.</i> (2018)
Austenite/dislocation boundary	37	Dwivedi and Vishwakarma (2019)
Microvoid	35.2–40	Dwivedi and Vishwakarma (2019)
High-angle grain boundary	47.4–57.4	Nagao <i>et al.</i> (2018)
MnS interface	72	Dwivedi and Vishwakarma (2019)
$\text{Al}_2\text{O}_3$ interface	79–86.2	Dwivedi and Vishwakarma (2019)
(Semi)coherent (Nb, V, Ti)C <sub>2</sub> interface	42.6–98	Dwivedi and Vishwakarma (2019), Lee and Lee (1984); Ma <i>et al.</i> (2020), Wei <i>et al.</i> (2004)
Incoherent TiC	68–137	Wei and Tsuzaki (2006)

The incoherent MC nanoprecipitates have extremely high hydrogen trap binding energy of 60–150 kJ/mol and their hydrogen trapping sites are the internal carbon vacancies (Wei *et al.*, 2011), which will result in extremely high energy barriers for hydrogen to enter the traps and cannot be trapped at room temperature. Recently, it has also been shown that a certain amount of hydrogen can be adsorbed at the incoherent nanoprecipitates interface at low temperatures (Chen *et al.*, 2020). However, the ability of the incoherent nanoprecipitates to adsorb hydrogen at low temperature is relatively weak.

### 3.2 Effect of trapping sites

Different coherence relationships between MC nanoprecipitates and matrix lead to different hydrogen trapping sites. The hydrogen trapping sites at the coherent, semicoherent and incoherent nanoprecipitates interface are at the interfacial tetrahedral gap positions, interfacial carbon vacancies/misfit dislocations and internal carbon vacancies, respectively (Ma *et al.*, 2020; Wei and Tsuzaki, 2006; Wei *et al.*, 2011). Stefano *et al.* (2016) investigated the binding energies of hydrogen trapping sites at different interfaces in TiC nanoprecipitates by first principles calculation. The binding energy of hydrogen traps at the coherent interface is too low to trap hydrogen effectively, as shown in Figure 6(a). The researchers focused on the interfacial carbon vacancies/misfit dislocations and the internal carbon vacancies.

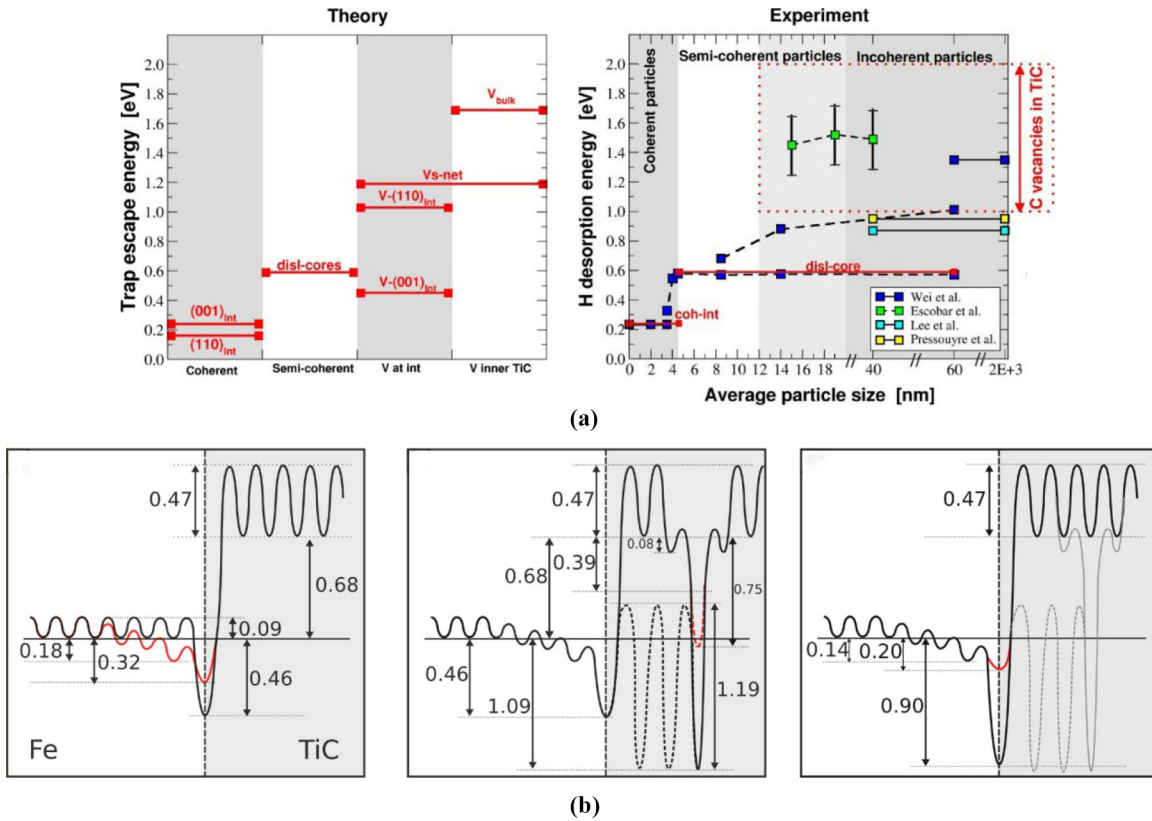
Internal carbon vacancies have higher binding energy than interfacial carbon vacancies/misfit dislocations. In terms of the energy barrier, hydrogen needs to cross a certain energy barrier before it can be trapped. As shown in Figure 6(b), the energy barrier of interfacial carbon vacancies/misfit dislocations is only 0.09 eV, so hydrogen can be trapped at room temperature. For the internal carbon vacancies, the energy barrier is as high as 1.61 eV. Therefore, the internal carbon vacancies can trap hydrogen only during the high-temperature heat treatment (Wei and Tsuzaki, 2006; Wei *et al.*, 2011). In summary, the interfacial carbon vacancies/misfit dislocations in the semicoherent nanoprecipitates can be used mainly as deep hydrogen traps to enhance the HE resistance.

### 3.3 Effect of size

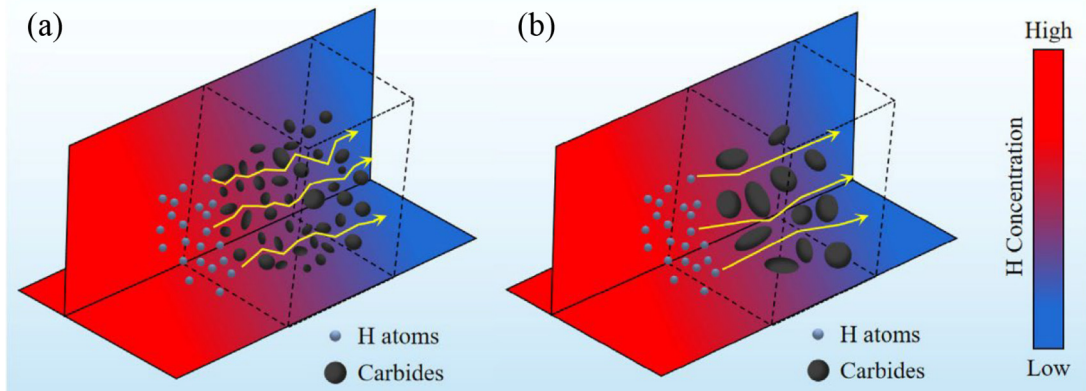
The fine nanoprecipitates in steel are maintained in a semicoherent state, which is beneficial for the HE resistance of steel (Wei and Tsuzaki, 2012). The hydrogen diffusion paths increase more in the nanoprecipitates with smaller sizes, which reduces the hydrogen diffusion coefficient and improves the HE resistance (Zhao *et al.*, 2021), as shown in Figure 7.

The content of microalloying elements in steel, tempering temperature and time can affect the size of precipitates. When the content of microalloying elements is high, the nanoprecipitates grow, coarsen or produce large undissolved particles, which can reach several hundred nanometers or even

**Figure 6** Comparison of different hydrogen trapping sites in nanoprecipitates (Stefano et al., 2016). (a) Comparison of theory and experimental values. (b) First principles investigation of hydrogen binding energies for different trapping sites



**Figure 7** Schematic diagram of hydrogen diffusion path (Zhao et al., 2021)



micrometers and these precipitates usually cause HE. Lee et al. (2016) showed that when the V content is above 0.5 Wt.%, a larger size of undissolved VC precipitates is generated, which induce quasi-cleavage cracking and increase HE susceptibility of steel. When the tempering time is long or the tempering temperature is high, the TiC nanoprecipitates coarsen and its hydrogen trapping ability decreases gradually. Studies have shown that when the tempering time is 20 h, the size of TiC nanoprecipitates  $\geq 70$  nm has lost the hydrogen trapping ability (Depover and Verbeken, 2016).

In summary, the HE resistance can be improved by controlling the addition of microalloying elements in martensitic steels and by reasonable regulation of the production process.

#### 4. Application of numerical simulations in precipitates

In recent years, numerical simulation techniques provide new research tools for the study of HE in high-strength martensitic steels, which can be used to quantify the effects of the binding



energy, coherence, trapping sites and size of MC nanoprecipitates. Through first principles calculations, Kawakami and Matsumiya (2012) suggested that the main hydrogen trapping site in the TiC nanoprecipitates is the TiC/matrix interface and the  $V_4C_3$  nanoprecipitates is carbon vacancies on the (001) broad surface. Stefano et al. (2016) calculated that the origin of the deep hydrogen trapping site in the semicoherent TiC nanoprecipitates is the core of the misfit dislocation by first principles calculations. Shi et al. (2020) showed that the misfit dislocation core is the most effective in the NbC/ $\alpha$ -Fe semicoherent interface by density flooding theory.

The hydrogen capture site of the many pending problems in the field of HE, especially at the atomic-scale, is gradually solved through a combination of numerical simulations and experiments. Researchers hope to discover the precise HE mechanism through numerical simulations.

## 5. Conclusions

MC nanoprecipitates can effectively improve the HE resistance of high-strength martensitic steel, but it is influenced by the type of precipitate, coherence, trapping sites and size of the nanoprecipitates. The main conclusions are described below:

- The fine MC nanoprecipitates semicoherent with steel matrix effectively improve the HE resistance of high-strength martensitic steel through hydrogen trapping effect and optimized microstructure. However, the coarsened or undissolved large particle precipitates can cause HE.
- The size of MC nanoprecipitates can affect the coherence relationship between the precipitates and the steel matrix, which affects the hydrogen trapping sites. The combination of numerical simulations with HRTEM and APT can study the trapping sites of MC nanoprecipitates at the atomic scale.
- The semicoherent MC nanoprecipitates, which hydrogen trapping sites are the interfacial carbon vacancy/misfit dislocations, can act as the main deep hydrogen traps.

## 6. Outlook

- It is a good way to improve the HE resistance of high-strength martensitic steel by adding an appropriate amount of microalloying elements into the steel to introduce fine semicoherent MC nanoprecipitates by controlling the heat treatment process.
- The effect of nanoprecipitates of multi-microalloying elements on the HE resistance of high-strength martensitic steel should be studied in depth.
- Researchers should make full use of numerical simulation combined with HRTEM and APT to explore the interaction mechanism between nanoprecipitates and hydrogen and reveal the nature of nanoprecipitates-enhanced HE resistance of steel.

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