



Evaluation of the effect of V_4C_3 precipitates on the hydrogen induced mechanical degradation in Fe-C-V alloys



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ABSTRACT

The impact of V_4C_3 precipitates on the sensitivity to hydrogen embrittlement is evaluated in this work. Two main conditions are compared with each other, i.e. an as-quenched and a quenched and tempered condition in which carbides have precipitated. Tensile tests are performed on both uncharged and hydrogen charged specimen and a correlation with the amount of charged hydrogen is made by hot/melt extraction. The tempered induced carbides trap a significant amount of hydrogen which explains why the degree of hydrogen embrittlement increases for the quenched and tempered condition. To verify at which trapping sites hydrogen is located, thermal desorption spectroscopy is performed. A considerable amount of hydrogen is trapped in a rather irreversibly way, which still has an effect on the degree of hydrogen embrittlement. Additionally, a slightly modified temper treatment is applied to vary the carbide size and hence evaluate the impact of the hydrogen induced ductility loss.

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1. Introduction

The mechanical degradation due to the impact of hydrogen has been reported to impede the application of newly developed high strength alloys [1]. Carbides play an important role while dealing with the hydrogen embrittlement (HE) issue since they can, on the one hand, realize a mechanical strengthening effect and, on the other hand, they are quoted to be beneficial to reduce the susceptibility to the hydrogen induced ductility loss by trapping hydrogen. Carbides such as TiC, NbC, V_4C_3 , $Cr_{23}C_6$, and Mo_2C are indeed known to cause secondary hardening [2] and some of them, TiC and VC, have been reported to be beneficial for improving the resistance to HE due to hydrogen trapping [3–7].

Thermal desorption spectroscopy (TDS) can be used to study the hydrogen trapping sites in a material. The hydrogen trapping capacity was for instance investigated by means of TDS on four steels in which Nb, Ti, V and Mo was introduced as carbide forming element [8]. Only TiC showed an additional desorption peak around 600 °C and the hydrogen trapping capacity of the alloy carbides varied in descending order of $NbC > TiC > VC > Mo_2C$. Asahi et al. [9] studied the hydrogen trapping behavior in vanadium-added steel. The VC precipitate in 0.25C–1.5Mn–V steels had a desorption activation energy (E_a) of 33–35 kJ/mol. A theoretical approach on the hydrogen interaction

with multiple traps was performed by Dadfarnia et al. [10]. Traps with binding energy > 60 kJ/mol can be categorized as very strong or irreversible ones and those < 30 kJ/mol as weak and reversible [11]. Trapping of hydrogen by VC in Fe–C–Mn steel was studied by small angle neutron scattering in [12] and it was estimated that the precipitates trapped approximately 5 wppm of hydrogen. They concluded that VC precipitates are effective irreversible hydrogen trapping sites for moderate hydrogen concentrations up to a few wppm hydrogen. It was concluded that the hydrogen trapping was homogeneously within the precipitates rather than at the precipitate/matrix interface.

Zhang et al. [13] investigated the diffusion coefficient by means of the hydrogen permeation technique and the reduction of area after hydrogen charging during a slow strain rate test (SSRT). They concluded that the diffusion coefficient of hydrogen in V or Nb added alloys was lower than without alloying. The combined effect of V and Nb on the hydrogen diffusion coefficient was not larger than when only Nb was added, which was attributed to the low density and high spacing of the VC precipitates. V or Nb addition reduced the susceptibility of hydrogen induced fracture when comparing air and hydrogen pre-charged samples in SSRT. In this case, the combined effect of V and Nb on the hydrogen induced delayed fracture resistance was better than only Nb. Additionally, intergranular fracture along prior austenitic grain boundaries and cleavage fracture of the non-alloyed material was more visible compared to the Nb-steel and Nb-V steel, where a mixture of quasi-cleavage, transgranular and intergranular was obtained.

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Micro-alloyed additions give rise to a strengthening effect due to a reduction in grain size. The resistance to delayed fracture was correlated to the tendency for intergranular fracture along prior austenitic grain boundaries. If the tendency is higher, the resistance to delayed fracture is better according to Zhang et al. [13]. These observations enhance the interest of introducing carbides into steel grades in order to reduce their susceptibility to HE.

Other investigation on the role of vanadium was performed by Spencer et al. [4]. They refer to previous work of Raymond [14] in order to motivate their goals. Raymond [14] already suggested that addition of Si or V could reduce the susceptibility to hydrogen embrittlement. Alloying elements can act as effective traps and these are able to limit hydrogen migration to stressed regions in a material, where it can initiate cracks or assist their growth. Experimental analysis by Spencer et al. [4] was done on quenched and tempered steels, which were heat treated to approximately the same yield and tensile strength and a variation of carbon and vanadium content was studied. They concluded that carbon did not cause a difference in the hydrogen embrittlement susceptibility. However, the alloy with a higher vanadium content did change significantly since a lower hydrogen induced ductility loss was obtained. Therefore, the addition of vanadium carbides was considered to be beneficial. In addition, an extra improved ductility was observed for some materials which was attributed to the presence of molybdenum and consequently the precipitation of Mo_2C in these alloys. Consequently, this is a good example of how addition of carbide-forming elements can enhance the resistance to hydrogen embrittlement. A hydrogen extraction study was done as well and the main conclusion was that vanadium carbides act as beneficial hydrogen traps for diffusible hydrogen.

Lee et al. [15] investigated the role of Mo/V carbides in hydrogen embrittlement of tempered martensitic alloys. The V containing material showed the largest peak of diffusible hydrogen, indicating that V carbides were capable of trapping a high amount of hydrogen. This was attributed to the high chemical affinity of V for hydrogen. Ideally, vanadium carbide should be present as VC, however in most cases, V_4C_3 predominates in steel [2]. The empty carbon sites of the V_4C_3 can act as a physical trap for hydrogen. V has five electrons in its outer shell, able to make a bonding with four carbon valence electrons. An extra vanadium electron is left, this may be an attractive hydrogen trap site as also reported by Pressouyre [16]. The presence of fine V_4C_3 particles of less than 50 angstroms in diameter were investigated in steels with 0.2% C and more than 0.10% V by means of transmission electron microscopy (TEM) by Aoki and Tanino [2,17]. The carbides had a coherent interface and according to Yoshino [2,18], these helped to promote the resistance to stress corrosion cracking.

An extensive study about modeling and characterization of V based precipitates was elaborated by Yamasaki and Bhadhesia [5], although they did not perform a hydrogen related experimental study. The precipitation and Ostwald ripening behavior of V_4C_3 (plate shaped) particles during the tempering of a ternary Fe–C–V martensitic steel was studied. TEM was used to validate the proposed theory. When carbides, such as V_4C_3 , are introduced into the steel, they can behave as hydrogen trapping sites in order to enhance the resistance to static fracture. The mechanical and hydrogen trapping properties depend on many parameters but the two most significant terms are the carbide size and number density according to the authors. V_4C_3 played a more important role in enhancing the resistance to hydrogen embrittlement owing to its much higher hydrogen trapping capacity than Mo_2C . Modeling of V_4C_3 precipitation was therefore a useful tool for the development of hydrogen resistant steels as it was possible to estimate the average length, volume fraction, and number density of particles in a manner consistent with experimental observations. Unfortunately, the effective correlation with their interaction with

hydrogen was not made, it is only emphasized that this carbide can enhance the resistance to hydrogen embrittlement.

The present study aims to correlate the effect of V_4C_3 precipitates on the trapping ability and consequently on the hydrogen induced ductility loss of steels with increased strength. Focus was put on generic quenched and tempered alloys in which the possible trapping sites were limited to verify the trapping capacity of the carbides.

2. Experimental procedure

2.1. Material characterization

Three laboratory Fe–C–V alloys with increasing carbon content were produced with a stoichiometric amount of the ternary alloying element V. Al was added to bind the present nitrogen avoiding it from forming nitrides [5]. The carbon increase allows a reliable estimation of the impact of the carbides with variable strength level and to confirm their role in different Fe–C–V alloys. The chemical compositions are given in Table 1.

The alloys were cast in a Pfeiffer VSG100 incremental vacuum melting and casting unit under an argon gas atmosphere. The materials were hot rolled till 1.5 mm. An appropriate heat treatment was applied in order to obtain two main conditions; one as quenched (as-Q) state with as little precipitates as possible and one quenched and tempered (Q&T) state where free carbon is enabled to precipitate with V during tempering. Temperature vs. time graph of the used heat treatment is presented in Fig. 1. All materials were austenitized at 1250 °C for 10 min to obtain a fully austenitic microstructure and to dissolve the carbides from the processing. These materials were then quenched in brine (7 wt% NaCl) to obtain a fully martensitic structure. This condition will further be referred to as as-Q (Fig. 1(a)). A fast brine quench was chosen to guarantee that no retained austenite was present in the microstructures since this would complicate the interpretation of the experimental results, as discussed in more detail in [36]. A similar approach was applied in previous work [20,41,42] and no retained austenite was observed.

Next to the as-Q condition, a second condition was prepared by tempering the quenched material at a certain temperature for one hour to generate, in a controlled way, V carbides, followed again by brine quenching (Fig. 1(b)). Tempering was performed at different temperatures to determine at which temperature the secondary hardening effect, due to the generation of small V_4C_3 , was most potent.

All materials were further ground and tensile samples were machined with their tensile axis parallel to the rolling direction and the specimen geometry is shown in Fig. 2. Finally, the surface of the samples was sandblasted to remove possible oxides remaining from processing.

Vickers hardness measurements were performed to determine the as-Q hardness level and the tempering temperature at which secondary hardening was most effective. A weight of 2 kg and a pyramidal diamond tip was used.

The microstructure was first investigated by light optical microscopy (LOM), for which grinding, polishing and etching with 4%

Table 1
Chemical composition of the used materials in wt%.

Material/Element	C	V	Other
Alloy A	0.100	0.570	
Alloy B	0.190	1.090	200–300 ppm Al
Alloy C	0.286	1.670	

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