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Removal of hydrogen from 304 austenitic stainless steel at room temperature by the electro-oxidation method

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Abstract

Hydrogen embrittlement effect can negatively affect properties of metal parts in energy industries. Recently the electro-oxidation (EO) method has been developed to restore hydrogen-induced mechanical degradation, showing good efficiency to remove hydrogen in ferritic and martensitic steels and recover their mechanical properties. However, the understanding about the hydrogen trapping sites and the type of removed hydrogen by the electro-oxidation (EO) method, and its universal applicability is still unclear. Here, as the first example to assess the effectiveness of the EO method to remove hydrogen in austenitic steels, widely used 304 stainless steel was investigated to evaluate removal of hydrogen and reveal hydrogen trapping states and removal ratio through thermal desorption analysis (TDA), in which hydrogen was replaced by its deuterium (D) isotope to eliminate intrinsic hydrogen interference. It was found that for gaseous charged deuterium, deuterium can be removed by the EO method at room temperature with the efficiency higher than that of desorption in the ambient condition, and the removal ratio of deuterium was linked with the charging temperature. The results demonstrate the effectiveness of the EO method and help to reveal

its mechanism to remove deuterium in 304 austenitic stainless steel specimens, and provide insights on its applicability to mitigate hydrogen embrittlement effect in energy industries.

Keywords: Hydrogen embrittlement; electro-oxidation; deuterium; thermal desorption, hydrogen removal; 304 austenitic stainless steel

1. Introduction

High-strength ferritic and martensitic steels are known to be susceptible to hydrogen embrittlement to affect their mechanical properties[1–3]. Recently, the electro-oxidation (EO) method has been reported to remove electrochemically charged hydrogen in low-carbon steel、AISI 430 ferritic (430SS) and AISI 420 martensitic stainless steel (420SS) and restore their mechanical properties from hydrogen-induced degradation [4,5], therefore it raises the prospect to alleviate or completely offset negative issues associated with hydrogen embrittlement effect. Nevertheless, the understanding about the trapping states and the type of removed hydrogen by the EO method is still unclear, and its applicability for other categories of stainless steels remains unexplored[4].

Austenitic steel is a type of widely used stainless steels with lower diffusivity of hydrogen than ferritic stainless steels[1]. 304 austenitic stainless steel is applied in the hydrogen energy field due to its many characteristics, such as high corrosion resistance and relatively strong resistance to hydrogen embrittlement[6]. There are reports on hydrogen embrittlement behaviour and hydrogen traps of 304 austenitic stainless steel specimens[7–9], but there is no study on removing hydrogen in austenitic stainless steel, whether the EO method is effective to remove hydrogen in 304 austenitic stainless steel is not confirmed yet.

On the other hand, thermal desorption analysis (TDA) is a common way to analyze hydrogen

trapping states in the steel specimens and work out the activation energy of hydrogen trapped in various sites like grain boundary and interstitial lattice defects[10,11,1,12,13]. It is noted that hydrogen may be introduced to the stainless steel material during its preparation process to cause intrinsic interference in the thermal desorption analysis[14], while deuterium, as isotope of hydrogen, is not involved in the preparation process of stainless steels to have advantage of avoiding the interference[4]. Furthermore, previous studies show that the solubilities of hydrogen and deuterium in iron (Fe) are equal in the temperature range of 9-506ºC, and many factors, including the diffusion activation energy and the isotope effect on the effective diffusivity, are well studied[15,16]; in particularly, the permeability, diffusivity and solubility of hydrogen/deuterium in 304 austenitic stainless steel are well investigated[17], which can provide good guidance on the hydrogen/deuterium transport. Consequently, it is advantageous to use deuterium replacing hydrogen for the study of charging, electrochemical removal and thermal desorption analysis[18,19].

Herein, 304 austenitic stainless steel specimens were charged with deuterium through gaseous precharging at different temperatures, and the resulted deuterium-charged specimens were evaluated for deuterium removal by the EO method, combined with thermal desorption analysis to analyze the desorption and trapping states of deuterium. The removal performance of deuterium in 304 austenitic stainless steel was compared with that of hydrogen in ferritic and martensitic stainless steel.

2. Experimental

A commercial 304 austenitic stainless steel block was cut into square pieces of 10 mm×10 mm×1 mm, which were subjected to a meticulous polishing process with sandpaper ranging from 800 mesh to 1500 mesh to eradicate the surface oxidized layer, and ultrasonically cleaned with ethanol. The information about the specimens is shown in the Table 1,Figure 1 and Figure 2.

Table 1 Chemical composition of 304 austenitic stainless steel

Elements[wt.%]	C	Mn	Si	Cu	Ni	P	S	Cr	Fe
304	0.059	1.08	0.36	/	8.05	0.032	0.0102	18.13	Balance

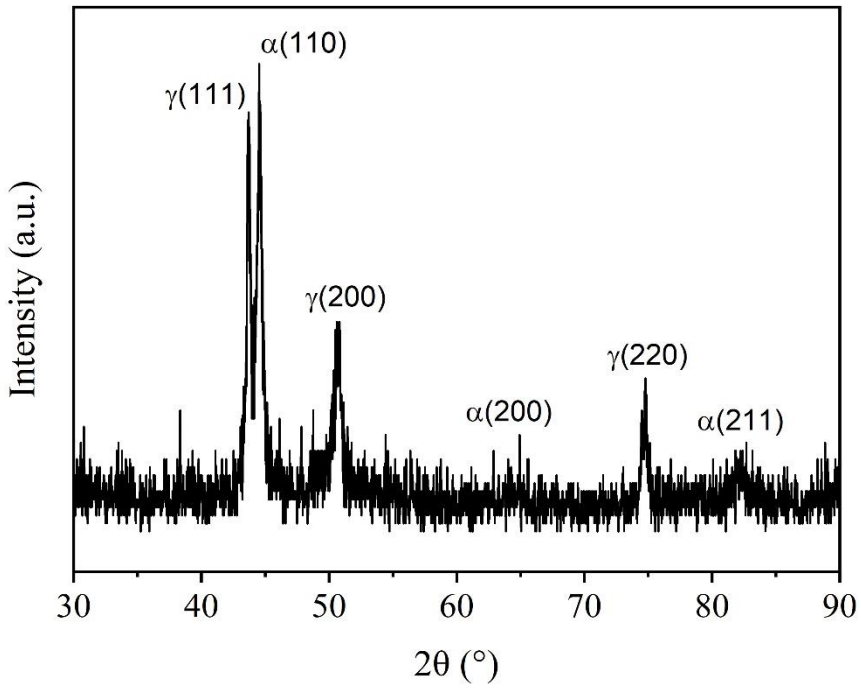

Figure 1 XRD patterns of a blank 304 stainless steel specimen

Figure 1 shows XRD patterns of the blank 304 austenitic stainless steel specimen, in which two phases are present: the austenite phase (γ-Fe, with a face-centered cubic structure) and the ferrite phase (α-Fe, with a body-centered cubic structure).

Figure 2 optical image of a blank 304 stainless steel specimen

Figure 2 is the optical image of a blank 304 stainless steel specimen, showing austenitic structure and ferrite, together with the obvious grain boundaries.

The specimens were charged through a gaseous deuterium charging process of 500 kPa and 24 h at the chosen temperature in a home-made apparatus[18], which was intended with uniformly distributed deuterium inside the specimen, and the resulted specimen charged with deuterium was named as 304-D. The specimen charged at specific temperature was designated as 304-D-T°C, where T was the charging temperature of 22°C、100°C or 400°C. The EO deuterium removal process was performed with a three-electrode set-up[4], in which a 304-D specimen functioned as the working electrode, using Pt foil, Hg/HgO as the counter electrode, reference electrode, respectively, while 0.1 M KOH solution was used as the electrolyte.

Thermal desorption analysis was conducted by heating from room temperature to 880°C at a rate of 10°C/min using a quadrupole mass spectrometer to analyse the deuterium. The trap activation energy for deuterium can be determined following the formula[11],

$$\frac{\partial \ln\left(\frac{\Phi}{T_p^2}\right)}{\partial\left(\frac{1}{T_p}\right)} = -\frac{E_a}{R}$$

where Φ is the heating rate, K/h; T_P is the peak temperature, K; E_a is the trap activation energy, J/mol; and R is the gas constant, J/(K·mol).

3. Results and discussion

Figure 3 gives the thermal desorption profiles of 304-D specimens charged at 22°C、100°C and 400°C, apparently, the profile area increases with increasing charging temperature[20], this can result from the increase of deuterium content[12], because of faster diffusion and higher solubility of deuterium in 304 austenitic stainless steels with the increase of gaseous charging temperature while

other conditions were kept constant[18]. In comparison, there is no desorption peak in the thermal desorption profile of blank 304 specimen shown in Figure 4 and Figure 5, indicating the absence of deuterium. Furthermore, 304-D-22ºC and 304-D-400ºC specimens exhibit single peak with the highest peak temperature of 556ºC and 475ºC, respectively, while 304-D-100ºC shows two peaks of peak 1# (208ºC) and peak 2# (536ºC). In the thermal desorption profiles, the desorption peak links with the trapping sites, and the peak at a higher temperature corresponds to the relatively stronger trapping sites. Figure 3 means that deuterium in the 304-D-100ºC specimen is trapped in weak (peak 1#) and strong (peak 2#) sites, and in the 304-D-22ºC and 304-D-400ºC specimens, deuterium is trapped in strong trapping sites[7,21,22]. It is reported that charged hydrogen always occupy the strong trapping sites of stainless steel[12], moreover, in this work, the charged conditions of pressure and duration is milder than the reported gaseous charging conditions in the literatures[7,12], it is likely that the content of charged deuterium is low relative to that of trapping sites in the specimens[23]. Therefore, in the 304-D-22ºC specimen, low content of deuterium is almost trapped in the strong sites; in the 304-D-100ºC specimen, deuterium is trapped in both the weak sites (peak 1#) and the strong sites (peak 2#)，while in the 304-D-400ºC specimen, the temperature of 400ºC can result in the release of deuterium trapped in the weak sites or affect the status of the weak sites during the gaseous charging process, so the deuterium is trapped in the strong sites and there is no desorption peak 1# in the thermal desorption profile[22]. As the content of charged deuterium is low relative to that of trapping sites, it is possible for the released deuterium to be trapped again increases[23], this may contribute to the variation of the highest peak temperature of 556ºC, 536ºC and 475ºC for 304-D-22ºC, 304-D-100ºC and 304-D-400ºC in the Figure 3, respectively.

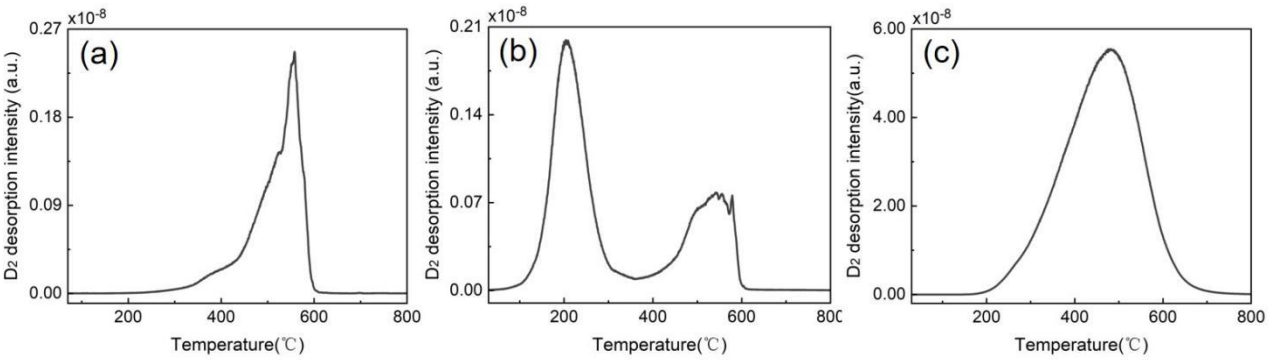

Fig. 3. Thermal desorption profiles of 304-D-22ºC (a), 304-D-100ºC (b) and 304-D-400ºC (c).

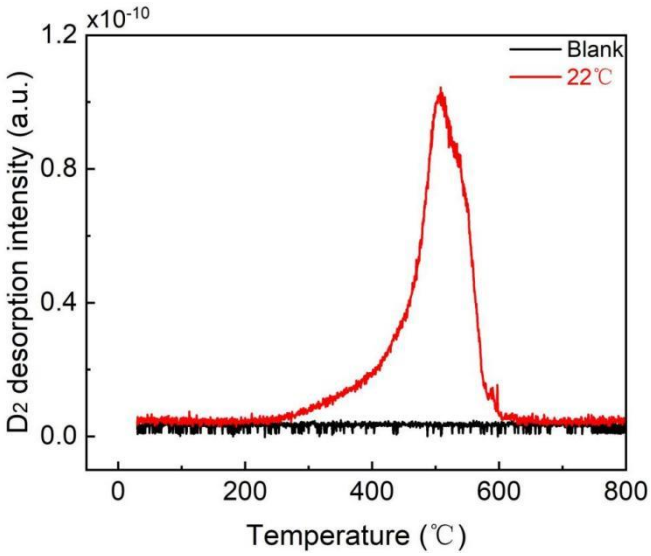

Figure 4 TDA plot of 304-D-22°C and a blank 304 specimen

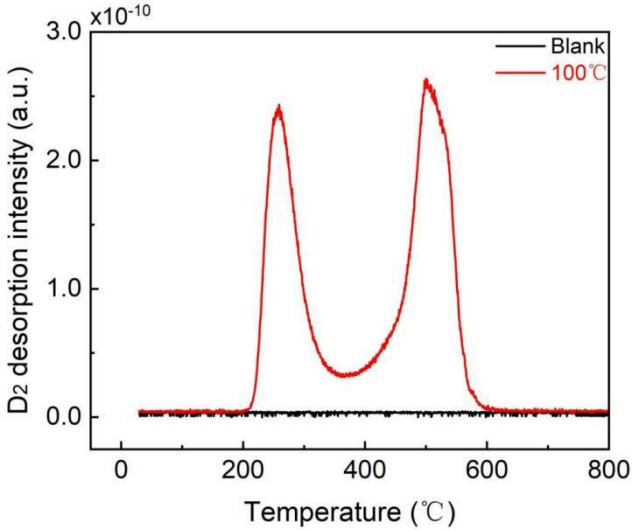

Figure 5 TDA plot of 304-D-100°C and a blank 304 specimen

To understand the deuterium desorption behavior, 304-D specimens charged at different temperatures were held at room temperature (22ºC) for different periods of time[13]. As the

experiment processes including transferring the sample from the charging chamber to the TDA chamber and then getting it the vacuum ready at room temperature before thermal desorption analysis took about 0.5 h, which is treated as the holding time of 0.5 h and the specimen was used as the reference for the overall charged deuterium.

Figure 6 shows that for all specimens, the peak intensity decreases with the increase of holding time before stabilizing, indicating the release of deuterium at room temperature. Comparatively, the content of remained deuterium in the specimen increases with increasing charging temperature in the descending order of 304-D-22ºC、304-D-100ºC and 304-D-400ºC [1].

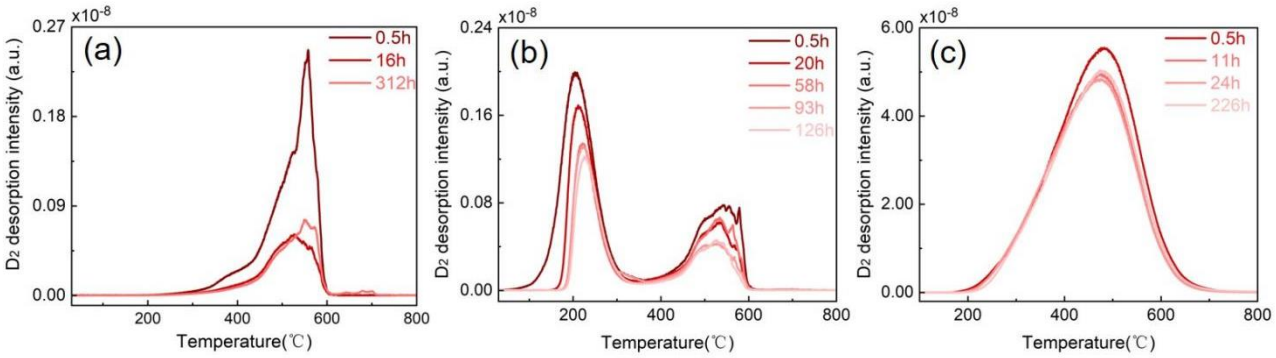

Fig. 6. Thermal desorption profiles for the 304-D specimens with different holding times: 304-D-22ºC (a), 304-D-100ºC (b) and 304-D-400ºC (c).

In order to verify the effect of the EO process to remove deuterium in 304 specimens at room temperature, two deuterium-charged specimens were chosen for each charging temperature, for comparison, one 304-D specimen without EO treatment was used as a control reference, the other used for the EO process was treated at 0.6 V for 4 h.

Figure 7(a) shows the thermal desorption profile of the control reference and the EO-treated 304-D-22ºC specimen, comparatively, the peak height of EO-treated 304-D-22ºC specimen is much lower, the removal ratio of the removed deuterium to the overall charged deuterium is calculated as about

70%[20]. Similarly, the removal ratio for 304-D-100ºC (Figure 7(b)) and 304-D-400°C (Figure 7(c)) is 52% and 11%, respectively[20].

It is noted that the comparison of Figure 6 and Figure 7 can suggest that the EO process has higher efficiency to remove deuterium than the desorption process of exposing the specimens to air in the ambient conditions. For example, the removal ration of 304-D-22ºC by the 4 h-EO process in Figure 6 is comparable with that of 304-D-22ºC by 16 h-exposure to air in Figure 7, similar phenomena are observed for the 304-D-100ºC and 304-D-400ºC specimens. This kind of removal acceleration effect is consistent with that reported in the ferritic and martensitic steel specimens prepared through electrochemical charging[4], indicating the generality of the EO process for the accelerating removal of hydrogen/deuterium in different types of stainless steels.

On the other hand, Figure 7 exhibits the decrease of the removal ratio with increasing charging temperature, for which the following reasons are: (i) the difference in the trapping states of deuterium; (ii) the variation of deuterium diffusivity at different temperature: the diffusivity coefficient of deuterium in 304 austenitic steels is 1.6 $X10^{-12}$ cm^2/s, 7.0 $X10^{-10}$ cm^2/s, 5.0 $X10^{-7}$ cm^2/s for 22ºC, 100ºC and 400ºC, respectively[17,25], the roughly calculated depth ($L=2(Dt)^{1/2}$) of 24 h-charging for 304-D-22ºC and 304-D-100ºC specimen is about 10 μm and 200 μm, while it is full depth for the 304-D-100ºC specimen, and the diffusion length of 4 h at 22ºC is about 5 μm [4], and (iii) the difference in the amount of charged deuterium in the specimens, therefore, the 4 h-EO process at room temperature in this work can have different impact on the removal of deuterium in these specimens, particularly for the low removal ratio for 304-D-400ºC specimen.

Comparatively, the EO-process of 5 h can remove 68% charged hydrogen in the 430 ferric stainless steel specimen, which can lead to the recovery of its tensile properties, while the removal

ratio of hydrogen in 420 martensitic stainless steel depended on the hydrogen charging conditions[5]. This means that the removal ratio of the EO process is also linked the type of stainless steels, in addition to the charging conditions.

The mechanism for the removal of deuterium in 304 austenitic stainless steel by the EO method can be described as previously reported[4], during the EO process the deuterium on the surface of 304 stainless steel specimen in contact with the KOH electrolyte was oxidized to D^+, and subsequently removed from the surface of 304 stainless specimen. The resulted D concentration gradient between the internal part and surface of 304 stainless steel can promote the diffusion of D to the surface and the successive removal of D.

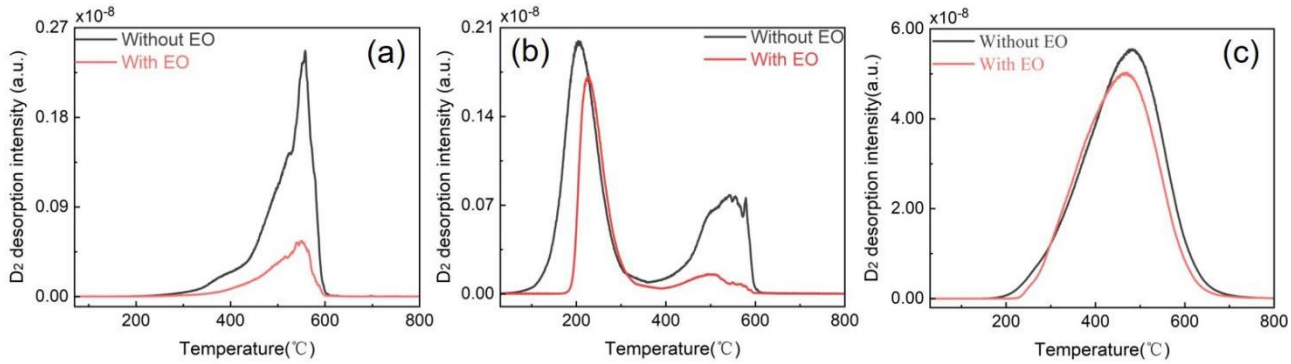

Fig.7.Thermal desorption profiles of 304-D specimens with and without EO-treatment for 304-D-22°C (a), and 304-D-100°C (b), and 304-D-400°C (c).

Based on the profile characteristics of all 304-D specimens in Figure 3, the Ea of each desorption peak for 304-D-100ºC is determined as the representative. Figure 8(a) shows the profiles of different heating rates, in which the intensity of the deuterium desorption peaks increases with the increase of heating rate[1]. The fitting curves are shown in Figure 8(b) and 8(c), and the activation energies is 32.8 kJ/mol, 369.3 kJ/mol for the peak 1# and peak 2#, respectively[11]. Notably, as the diffusion rate of deuterium is relatively low for 304 austenitic stainless steel[25], its desorption need consider detrapping and diffusion, the calculated activation energy by the Choo-Lee method is the

sum of the trap binding energy and diffusion activation energy[11]. The 304-D specimens consist of mainly γ austenitic phase and some α phase as shown in Figure 1, deuterium desorption can be affected primarily by the diffusion process[7]. This means that the calculated activation energy in this work may not perfectly match with the real case, as the activation energies are relatively lower in comparison with the reported values of activation energy for deuterium in austenitic stainless steels[17]. .

In 304 stainless steel specimens, there are grain boundaries, vacancy-type defects, dislocations, and interstitial lattice sites[7–9]. The thermal desorption peak around 200ºC is related to the possible trapping sites of grain boundaries, and the thermal desorption peak around 536ºC infers possible trapping sites of dislocations and vacancy-type defects based on the desoprtion peak around 200-400ºC and 200-600ºC of thermal desorption profiles obtained from gaseous charged 304 austenitic steels at the similar heating rate[7].

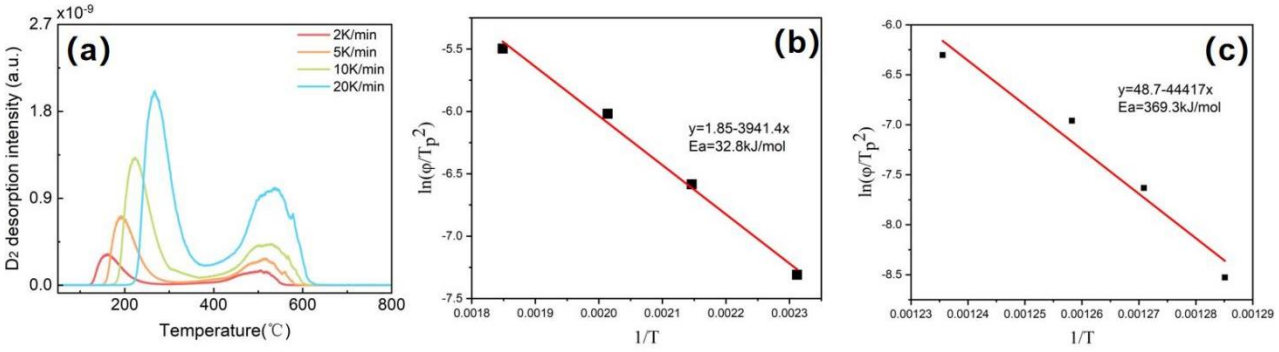

Fig.8. Thermal desorption curves at different heating rates for 304-D-100ºC (a)，the fitting curve for the peak 1 (b) and peak 2 (c) of 304-D-100ºC.

As a result, the combination of Figure 3 and Figure 4 indicates that the EO method can remove deuterium from the vacancy-type defects, dislocations, grain boundaries and lattices of 304 austenitic stainless steel specimens, and the removal ratio decreases with the increase of deuterium charging

temperature. Although the EO process at room temperature can not completely remove deuterium in 304 stainless steel under the conditions reported, the thermal desorption analysis can advise the trapping sites of remained deuterium in the specimen.

4. Conclusions

In conclusion, as the first example to investigate the EO method to remove hydrogen in austenitic steels, widely used 304 stainless steel was studied to evaluate hydrogen removal and reveal hydrogen trapping states and removal ratio through thermal desorption analysis, for which deuterium replaced hydrogen to be charged by gaseous precharging at 22ºC、100ºC、400ºC to avoid intrinsic interference of hydrogen. It was found that the EO method can enhance the removal of deuterium compared with the desorption in the ambient conditions, and the removal ratio depended on trapping state of deuterium, which is associated with the charging temperature. This work verifies the hydrogen removal behaviour in austenitic stainless steel, and provides good supplement to the understanding on the EO process with electrochemical charged ferritic and martensitic steels, and its applicability to mitigate hydrogen embrittlement in the energy industries.

Acknowledgement

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