

# In-situ Investigation on Deuterium Induced Cracking of Titanium

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**Abstract:** Deuterium induced cracking (DIC) of Ti was in-situ investigated by the hot stage microscope (HSM) technique combined with the pressure-volume-temperature (PVT) method. Results show that only several cracks are observed on the surface of Ti exposed to D<sub>2</sub> at 550 °C, while more unique circular cracks appear when Ti is heated from room temperature to 550 °C in the D<sub>2</sub> atmosphere. The morphology change of Ti induced by deuterium during heating is consistent with the characteristics described by the preferred edge-attack model. X-ray photoelectron spectroscopy (XPS) measurements indicate that the surface passivation layer on Ti, consisting mainly of titanium oxides, carbides and nitrides, plays an important role in the formation of circular cracks.

**Key words:** hydrogen storage materials; titanium; deuterium induced cracking; kinetics

Research and development of neutron production targets through nuclear reactions can be used as powerful tools in numerous fields requiring neutron beams. In particular, when the  $D(d, n)$  reaction is considered, the development of TiD<sub>2</sub> targets is an important matter in the nuclear materials development and in neutron production<sup>[1-3]</sup>. However, Ti is potentially susceptible to hydrogen-induced cracking (HIC) as a consequence of hydrogen absorption<sup>[4-9]</sup>.

Clarke et al<sup>[4]</sup> assessed the likelihood of failure due to hydrogen pick-up in titanium containers for nuclear fuel waste disposal. Slow crack growth occurred at low hydrogen concentrations, while fast propagation of a brittle crack was observed only at the hydrogen concentrations above critical value which depended upon the material involved and the orientation of the crack relative to the manufactured microstructure. Liu et al<sup>[3]</sup> found that the additive hydrogen reduces the grain size, leading to the formation of lamellar or needle-like structure with an increasing volume fraction of titanium hydride (fcc). HIC can be qualitatively explained by the hydride formation-rupture event at crack tips and localized deformation.

On the other hand, HIC is also closely related to the surface

passivation layer (SPL) on Ti, which spontaneously forms in oxidized environments. Hadjixenophontos et al<sup>[10]</sup> investigated the role of surface oxides in hydrogen sorption kinetics of titanium thin films (TiO<sub>2</sub>/Ti), and concluded that hydrogen transport across the oxide layer acts as the decisive kinetic barrier, and the hydride nucleates from the substrate to the surface to form the layered structure. However, the effect of surface passivation layer on HIC of Ti is still not fully understood, especially during the heating process in gaseous hydrogen, since this kind of SPL is very sensitive to the temperature<sup>[11]</sup>. Mizuno et al<sup>[11]</sup> found that no oxygen is detected on the titanium surface in ultrahigh vacuum (UHV) above 450 °C, which is due to the enhancement of diffusion of oxygen from the surface into the bulk.

In order to meet requirements of the preparation of titanium deuteride targets with few or no cracks, it is important to understand how deuterium interacts with Ti in different D<sub>2</sub> charging processes. So its properties can be controlled and reliably predicted. In the present work, deuterium induced cracking (DIC) of Ti was in-situ investigated through the pressure-volume-temperature (PVT) method combined with the hot stage microscope (HSM) technique. The effect of the

Received date: February 14, 2020

Foundation item: National Natural Science Foundation of China (21401173, 11775194); National Magnetic Confinement Fusion Energy Research Project (2015GB109002)

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passive layer on the deuterium absorption and deuterium induced cracking of Ti was discussed as well.

## 1 Experiment

Titanium foil of purity 99.99% with dimensions of  $\Phi 10$  mm $\times$ 1 mm (purchased from General Research Institute for Nonferrous Metals) was mirror-polished using silicon carbide abrasive papers (400#~2000#). Then the polished titanium foil was rinsed ultrasonically in distilled water and dried by strong argon gas current.

Hydrogen exposure experiments were performed in a high-vacuum ( $\leq 10^{-4}$  Pa) Sievert's apparatus with a digital microscope above the sample reactor (Fig.1). The reactor can work at high temperatures ( $\leq 850$  °C). A thermocouple is installed inside the reactor, which is near the sample less than one millimeter. A quartz glass window is assembled as a part of the reactor for in-situ observation during gas-solid reactions. This method is also known as HSM techniques combined with the PVT method<sup>[12]</sup>.

Two kinds of D<sub>2</sub> charging processes were considered. (1) Charge D<sub>2</sub> during heating. D<sub>2</sub> was introduced into the reactor at room temperature (RT) and maintained for sufficient time to approach equilibrium, and then the sample was heated to 600 °C at a heating speed of 10 °C /min in D<sub>2</sub> atmosphere. (2) Charge D<sub>2</sub> at a given temperature. D<sub>2</sub> was introduced into the reactor at a given temperature (around 550 °C) and maintained for sufficient time to approach equilibrium. High purity D<sub>2</sub> (99.999%) was supplied by a ZrCo bed.

X-ray photoelectron spectroscopy (XPS, ESCALAB 250) was used to analyze the surface chemical states of Ti during heat treatment in UHV, so a proper degassing temperature could be determined before D<sub>2</sub> charging. The vacuum pressure remained below  $3.0 \times 10^{-6}$  Pa during specimen heating. 300 W of Al K $\alpha$  X-ray with 1486.6 eV radiation was used for XPS. The analyzer was operated at a pass energy of 30 eV. The

reference used for binding energy calibration was the Ag 3d<sub>5/2</sub> line at the binding energy of 368.2 eV. Besides the in-situ observations, characterization of microstructures and phases of the sample after D<sub>2</sub> charging were performed by scanning electron microscopy (SEM, Sirion 200) and X-ray diffraction (XRD, X'Pert PRO) respectively.

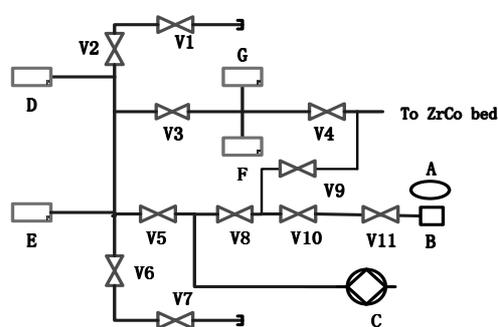
## 2 Results and Discussion

### 2.1 Surface states of Ti during heating before deuteration

Table 1 shows the reference values of XPS binding energies of Ti 2p, O 1s, C 1s and N 1s in different chemical states. Fig.2 is the XPS spectra of Ti foil during heat treatment at different temperatures in UHV.

The initial surface of Ti foil is mainly composed of TiO<sub>2</sub> (characteristic peak at 458.90 eV for Ti<sup>4+</sup> 2p<sub>3/2</sub>) and metal Ti (453.95 eV for Ti 2p<sub>3/2</sub>), as shown in the XPS spectrum of Ti 2p at RT in Fig.2a. The O 1s spectrum at RT in Fig.2b further confirms that the surface of the sample is mainly TiO<sub>2</sub> (O 1s with the binding energy value of 530.00 eV at RT). The C 1s spectrum in Fig.2c shows that there are carbon contaminants (C 1s peak of 285.30 eV at RT) and a small amount of TiC (C 1s peak of 281.80 eV at RT) on the initial surface of Ti. The N 1s spectrum in Fig.2d shows that a small amount of TiN<sub>2</sub> (N 1s peak of 397.20 eV at RT) is formed on the surface of Ti. Therefore, the initial mirror-finished surface of Ti foil mainly consists of titanium dioxide, metallic Ti, carbon contaminations and titanium nitride.

When heated to 200 °C, TiO<sub>2</sub> converts to the low-valent oxides (mainly TiO, with the characteristic peak at 460.35 eV for Ti 2p<sub>1/2</sub> and 454.15 eV for Ti 2p<sub>3/2</sub>) and the surface oxygen content increases as shown in Fig.2b. When heated to 600 °C, titanium oxides decompose according to the rapidly increasing intensity of the peak of metallic Ti (453.80 eV for Ti 2p<sub>3/2</sub>) and



A: digital microscope; B: high-vacuum reactor; C: pumping system; D: 133 kPa manometer; E: 13.3 kPa manometer; F: 1.33 kPa manometer; G: combination gauge ( $10^{-8}$  Pa~ $10^5$  Pa); V1~V11: all-metal angle valves

Fig.1 All-metal high vacuum experimental system

Table 1 Reference values of the binding energies for Ti 2p, O 1s, C 1s and N 1s in different chemical states (eV)

Specimen	Ti 2p <sub>3/2</sub>	Ti 2p <sub>1/2</sub>	O 1s
Ti metal	453.80 <sup>[13]</sup>	459.95 <sup>[13]</sup>	-
TiO	454.80 <sup>[14]</sup>	460.50 <sup>[14]</sup>	531.20 <sup>[15]</sup>
Ti <sub>2</sub> O <sub>3</sub>	457.15 <sup>[16]</sup>	464.00 <sup>[16]</sup>	531.50 <sup>[17]</sup>
TiO <sub>2</sub>	458.80 <sup>[18]</sup>	464.30 <sup>[13]</sup>	529.90 <sup>[18]</sup>
Solid solution of O in Ti	454.30 <sup>[17]</sup>	-	531.40 <sup>[17]</sup>
Adsorbed oxygen specimens	-	-	534.30 <sup>[19]</sup>
Hydroxide	-	-	532.50 <sup>[19]</sup>
Specimen	Ti 2p <sub>3/2</sub>	Ti 2p <sub>1/2</sub>	C 1s
TiC	455.00 <sup>[20]</sup>	461.00 <sup>[20]</sup>	281.6 <sup>[18]</sup>
Graphite	-	-	284.5 <sup>[18]</sup>
Specimen	Ti 2p <sub>3/2</sub>	Ti 2p <sub>1/2</sub>	N 1s
TiN	456.30 <sup>[21]</sup>	462.30 <sup>[21]</sup>	396.9 <sup>[18]</sup>
TiN <sub>2</sub>	454.80 <sup>[22]</sup>	460.50 <sup>[22]</sup>	397.2 <sup>[17]</sup>
Chemisorbed N <sub>2</sub>	-	-	399.9 <sup>[23]</sup>
Solid solution N <sub>2</sub>	-	-	398.8 <sup>[23]</sup>
Doped N <sup>3-</sup>	-	-	396.9 <sup>[23]</sup>

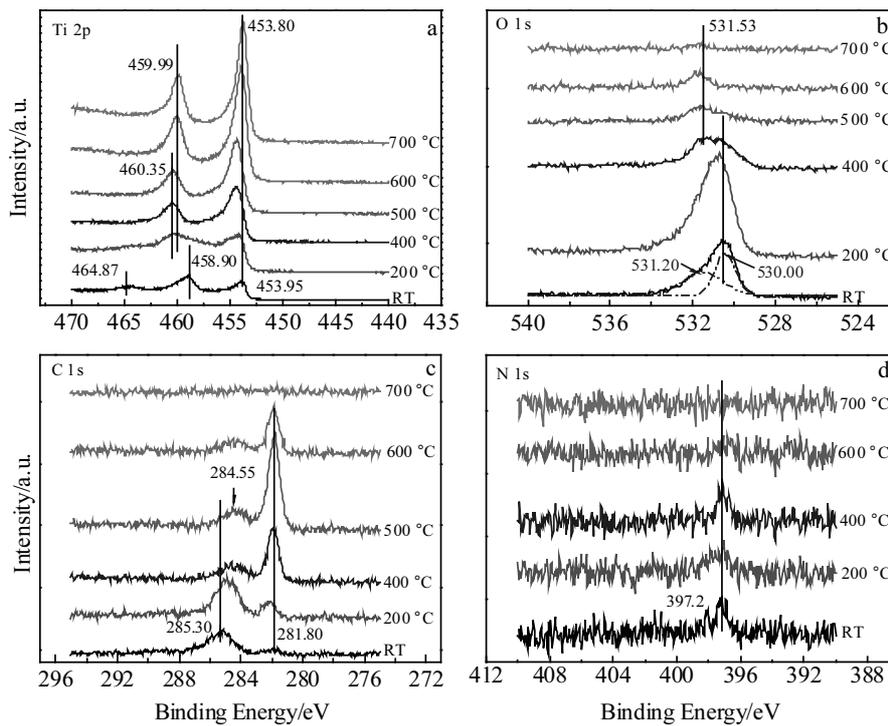


Fig.2 XPS spectra of Ti foil annealing at different temperatures in UHV: (a) Ti 2p, (b) O 1s, (c) C 1s, and (d) N 1s

the decreasing intensity of the peaks of O 1s. Therefore, it is clear that oxygen can be removed from the initial surface of Ti by heating up to 700 °C in vacuum.

C and N compounds have the similar results with oxides. Above 200 °C, the surface carbon contamination reacts with the metallic Ti to form TiC, since the C 1s peak shifts from 284.5 eV to 281.8 eV, as shown in Fig.2c. TiC is stable from 200 °C to 600 °C, and its amount even increases at 500 °C. Titanium nitride cannot be observed at 600 °C, as shown in Fig.2d.

The “active” surface of the sample was also characterized

when cooled down to RT in UHV after degassing at 700 °C. The results shown in Fig.3 reveal that the “active” surfaces of Ti are contaminated again by oxygen, and the surface oxide is mainly TiO (460.35 eV for Ti 2p<sub>1/2</sub> and 531.20 eV for O 1s). No carbon or nitrogen contaminations are found on the “active” surfaces of Ti.

The above spectra are in agreement with the XPS analysis of Ti in the range of 200~450 °C by Mizuno et al<sup>[11]</sup>. They found that TiO<sub>2</sub> decomposes into suboxides at 180 °C, and the remaining TiO<sub>2</sub> continuously decreases until the temperature is heated to 420 °C. TiC decomposes above 420 °C. In this

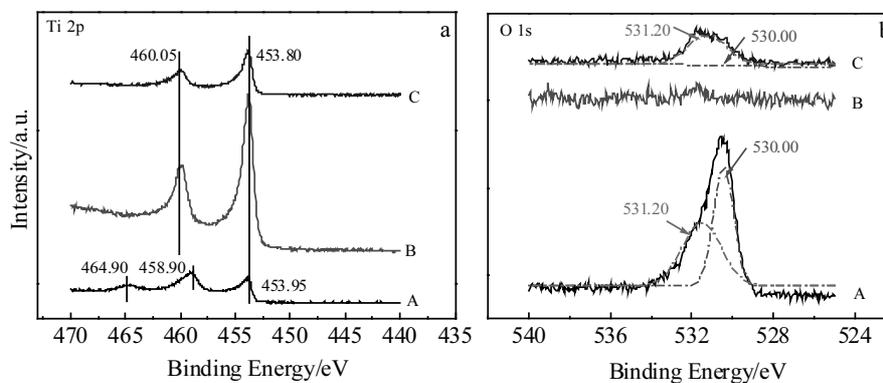


Fig.3 XPS spectra of titanium foil in UHV: (a) Ti 2p and (b) O 1s (A: the initial surface; B: the surface kept at 700 °C for 35 min; C: the surface cooled down to RT)

research, the surface passivation layer (mainly consisting of titanium oxides, carbides and nitrides) entirely diminishes at 700 °C in UHV, showing an access to the “active and clean” Ti surface. In the following DIC investigations, Ti was annealed at 750 °C in vacuum to obtain a higher quality surface.

**2.2 Kinetic curves of D<sub>2</sub> absorption and crack growth**

Fig.4 shows the kinetics of D<sub>2</sub> absorption by heating Ti from RT to 600 °C. The absorption proceeds gently at RT while D/Ti atomic ratio reaches to 0.13 in 60 min, and speeds up as the temperature increases as shown in Fig.4. The deuterium absorption rate is very high when D/Ti atomic ratio reaches 0.4 in 3 min at 550 °C (Fig.5). Additionally, there is no obvious incubation period in the initial stage as reported by Fernández et al<sup>[24]</sup>, indicating that the activation degassing process of the sample at 750 °C before deuteration is sufficient to destroy SPL, and the surface still keeps “active” at 550 °C after the degassing process.

Different kinds of D<sub>2</sub> charging processes lead to morphology changes of Ti: concentric cracking and edge cracking occur when D<sub>2</sub> charging is conducted in the heating process and at the given temperature, respectively. As shown in Fig.6, the morphology of Ti changes abruptly when the temperature increases to 550 °C: circular cracks firstly form at the edge of the sample, then progress to the central part. However, when charging D<sub>2</sub> at the fixed temperature of 550 °C, there are no circular cracks at the first stage. Only several cracks appear after 100 min as shown in Fig.7.

The average D/Ti atomic ratio is 1.55 at the end of each experiment. Fig.8 shows that circular cracks form from the edge to the center under D<sub>2</sub> charging during heating process. Fig.9 suggests that the charged sample consists of δ phase TiD<sub>2</sub> (fcc) when cooled to RT in each experiment.

**2.3 Two models**

The different morphologies of Ti during D<sub>2</sub> charging indicate different kinetics of D<sub>2</sub> absorption. The unique mor-

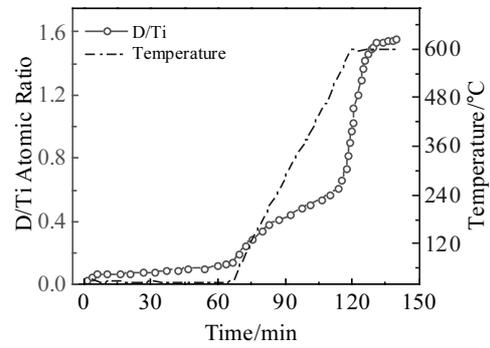


Fig.4 Kinetic curves of D<sub>2</sub> absorption by heating Ti foil from RT to 600 °C (initial deuterium pressure of 13.38 kPa)

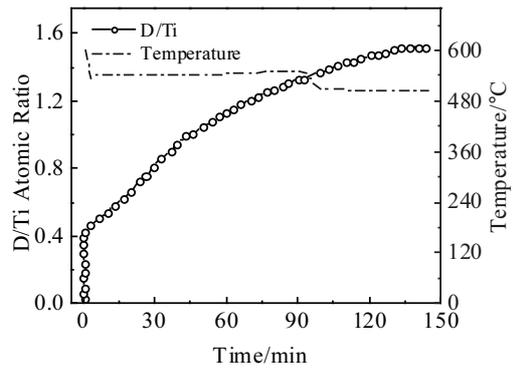


Fig.5 Kinetic curves of D<sub>2</sub> absorption by heating Ti foil at 550 °C and then 500 °C (initial deuterium pressure of 13.38 kPa)

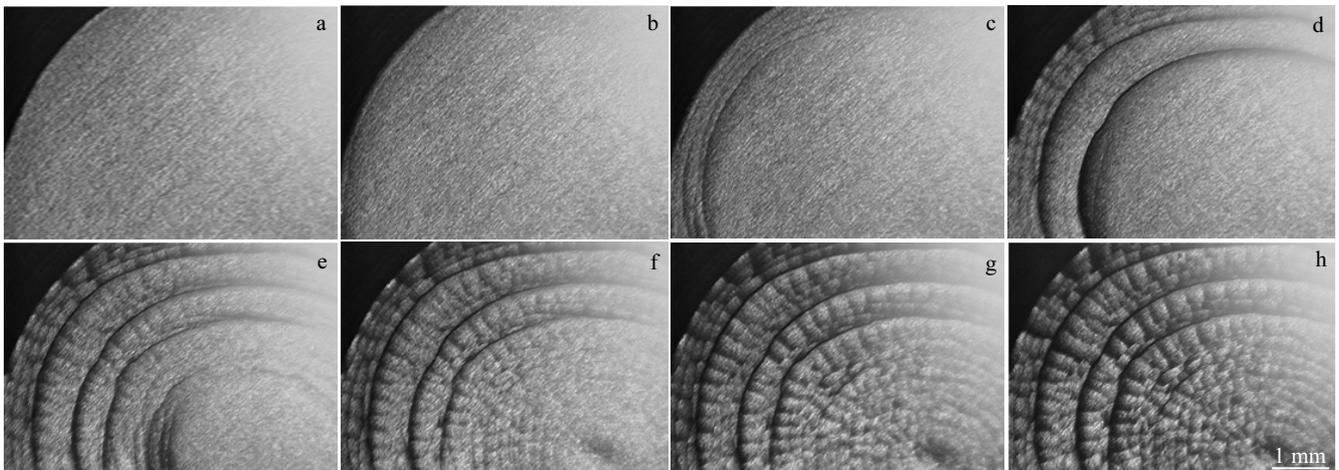


Fig.6 Morphologies of Ti foil during heating process from RT to 600 °C for different time with the initial deuterium pressure of 13.38 kPa: (a) 120 min, (b) 125 min, (c) 127 min, (d) 129 min, (e) 131 min, (f) 133 min, (g) 140 min, and (h) 165 min

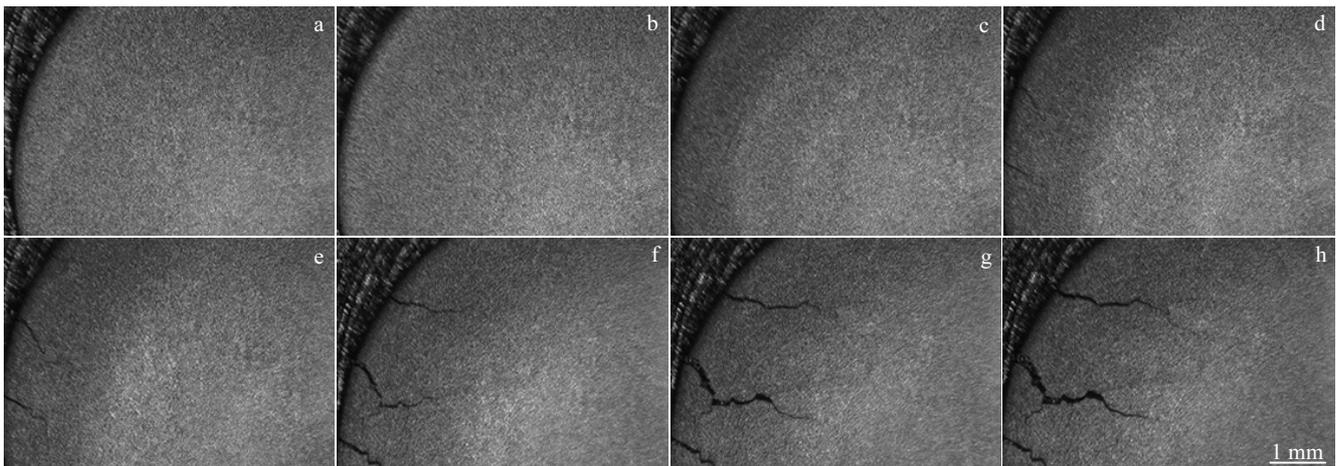


Fig.7 Morphologies of Ti foil at a given temperature of  $\sim 550\text{ }^{\circ}\text{C}$  for different time with the initial deuterium pressure of 13.38 kPa: (a) 0 min, (b) 10 min, (c) 40 min, (d) 60 min, (e) 70 min, (f) 100 min, (g) 120 min, and (h) 145 min

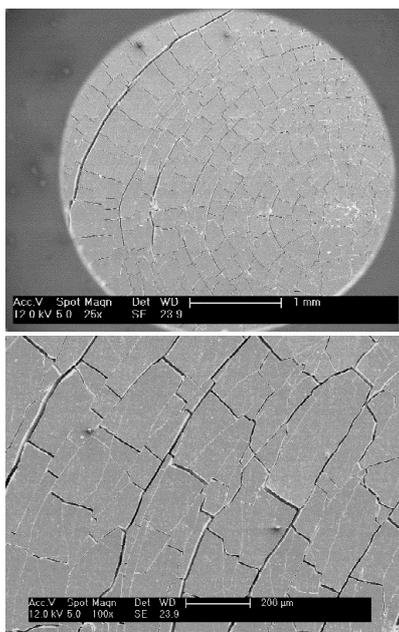


Fig.8 SEM images of Ti foil at the end of the heating process from RT to  $600\text{ }^{\circ}\text{C}$  with the initial deuterium pressure of 13.38 kPa

phology change of Ti during heating process from RT to  $600\text{ }^{\circ}\text{C}$  is consistent with the preferred edge-attack model<sup>[25,26]</sup> (Fig.10a), which is caused by the inhomogeneity of thickness of the surface passivation layer at the edge locations.

The  $\text{D}_2$  absorption process of Ti foil at a given temperature can be explained by the contracting-envelope progression model<sup>[12,27]</sup>, as shown in Fig.10b. The titanium deuteride product firstly forms on the outer surface of the sample, and then progresses into the bulk, retaining constant thickness and velocity under proper steady state conditions as a result of

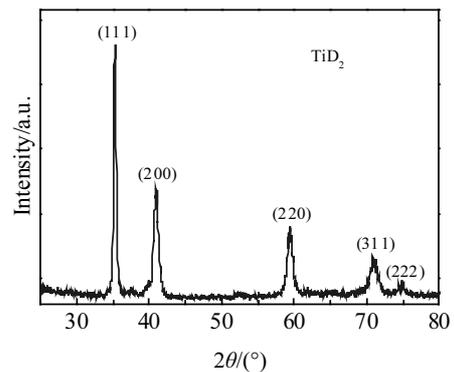


Fig.9 XRD pattern of titanium at the end of the  $\text{D}_2$  absorption experiments with the initial deuterium pressure of 13.38 kPa

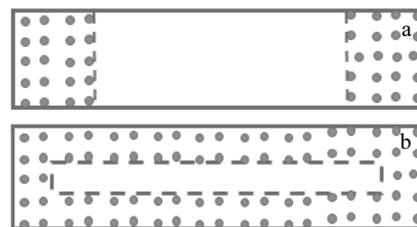


Fig.10 Schematic diagrams of preferred edge-attack model (a) and contracting envelope model (b)<sup>[12,27]</sup> (cross-section; dotted areas refer to the hydride phase)

cracking on the outer surface. The rate of hydrogenation reaction is controlled by the diffusion of hydrogen through the product layer, and can be expressed by the rate of the reaction front (deuteride-metal interface) movement<sup>[12,27]</sup>.

### 2.3.1 $\text{D}_2$ absorption by heating Ti at a given temperature of $550\text{ }^{\circ}\text{C}$

According to the Ti-H phase diagram<sup>[28]</sup>, the process of D<sub>2</sub> absorption of Ti at 550 °C generates three phases successively:  $\alpha$  (solid solution, hcp)→ $\beta$  (solid solution, bcc)→ $\delta$  (deuteride, fcc)<sup>[29]</sup>. Assuming that the deuteride layer formed on the surface of Ti is continuous and has the average composition of D/Ti≈1.0 ( $\beta$  phase, TiD) or D/Ti≈2.0 ( $\delta$  phase, TiD<sub>2</sub>), the D<sub>2</sub> absorption kinetic curves (Fig.5) can be transformed into the curves of the deuteride layer thickness versus time (Fig.11). There is successive formation of two deuteride phases during the same deuteriding process.

As expected, the initial acceleration stage is the result of nucleation and growth of hydride phase ( $\beta$ ) on the metallic surface. The average thickness of the  $\beta$ -phase deuteride layer increases sharply to 175  $\mu\text{m}$  within the first two minutes, and increases smoothly as the reaction proceeds, as shown in the inset of Fig.11.

Qualitatively, the reaction rate is controlled by the diffusion of deuterium through the continuously growing deuteride layer<sup>[30]</sup> until the thickness (~175  $\mu\text{m}$ ) is stable and the reaction begins to speed up as a result of cracking on the outer surface. In the second stage, the  $\beta$  phase deuterium layer propagates from the outside to the inside of the sample.

After the whole sample converts to  $\beta$  phase, the  $\delta$  phase deuteride layer begins to appear on the surface of the sample. The control step of D<sub>2</sub> absorption thus changes to the D diffusion through the  $\delta$  phase. When the reaction reaches the steady state, the  $\delta$  deuterium layer propagates into the sample at a constant rate, as shown in Fig.11. According to the slopes of the D<sub>2</sub> absorption kinetic curves in Fig.11, the diffusion rate of D atoms in the  $\delta$  phase is lower than that in the  $\beta$  phase at the same temperature (1.32  $\mu\text{m}\cdot\text{min}^{-1}$  and 4.62  $\mu\text{m}\cdot\text{min}^{-1}$  at 550 °C), and decreases as the temperature decreases to 500 °C.

### 2.3.2 D<sub>2</sub> absorption by heating Ti from RT to 600 °C

In Fig.4, only a single phase transformation ( $\alpha$ → $\delta$ ) exists while charging D<sub>2</sub> at RT. At the steady state, the diffusion of D

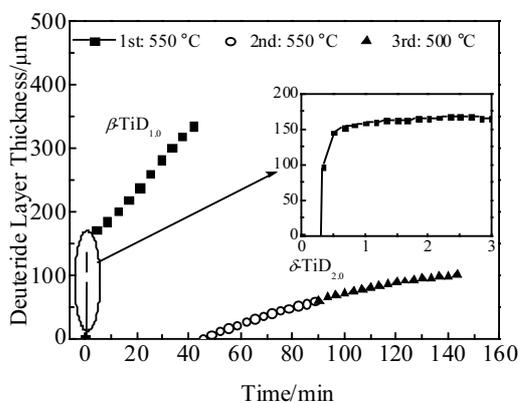


Fig.11 Variation of the deuteride layer thickness on Ti with time when charging D<sub>2</sub> at 550 °C and then 500 °C with the initial deuterium pressure of 13.38 kPa

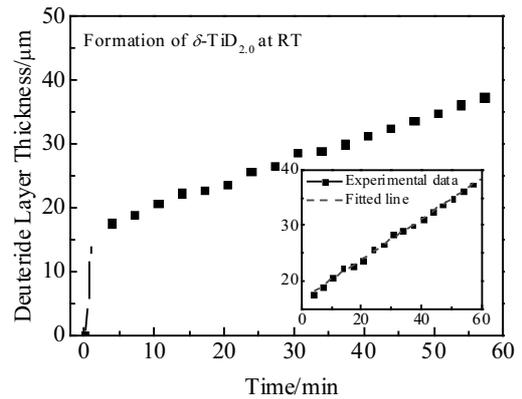


Fig.12 Variation of deuterium layer thickness on Ti foil and fitting curve with time when charging D<sub>2</sub> at RT with the initial deuterium pressure of 13.38 kPa

atoms in the  $\delta$  phase is very slow at RT, the thickness of  $\delta$  deuterium layer increases from ~20  $\mu\text{m}$  to 40  $\mu\text{m}$  in 60 min (diffusion rate: 0.36  $\mu\text{m}\cdot\text{min}^{-1}$ ), as shown in Fig.12.

After D<sub>2</sub> absorption at RT for 65 min, the sample is heated to 600 °C with the heating rate of 10 °C/min, and the diffusion of D atoms in the  $\delta$  phase accelerates, as well as the thickness increase of the deuterium layer. Therefore, there is still a nearly linear increase of deuterium absorption with time, as shown in Fig.4.

The rapid D<sub>2</sub> absorption of Ti occurs at 550 °C during heating process. Circular cracks firstly form on the edge of the sample, and then progress to the central part. Mintz et al<sup>[26]</sup> attributed this preferred edge-attack phenomenon to the inhomogeneity of the thickness of SPL at the edge. XPS results in this work also support it with the fact that “active” surfaces of Ti obtained at 700 °C can be contaminated again by the residual oxygen in the UHV when cooled down to RT (Fig.3).

It is worth mentioning that the inhomogeneity of the thickness of the  $\delta$  phase Ti deuteride (formed at RT) at the edge, possibly leading to microcracks at the surface, may also play an important role in the formation of circular cracks. In this case, as the diffusion rate of D atoms in the  $\delta$  phase is much lower than that in the  $\beta$  phase at the same temperature, the thinner  $\delta$  phase zones at the edge favor the diffusion of D atoms, resulting in the propagation of circular cracks from the edge to the center.

## 3 Conclusions

1) The surface passivation layer of the mirror-finished Ti, mainly consisting of titanium oxides, carbides and nitrides, diminishes when heated up to 700 °C in ultrahigh vacuum. However, the obtained active surface of Ti is contaminated again by the residual oxygen in ultrahigh vacuum when cooled down to RT.

2) Deuterium induced cracking of Ti is sensitive to the D<sub>2</sub>

charging process. Only several cracks are observed on the surface of Ti when charging D<sub>2</sub> at a given temperature of 550 °C, while circular cracks appear when Ti is heated up to 550 °C in the D<sub>2</sub> atmosphere.

3) Combined with the kinetics investigation, the morphology changes are consistent with the characteristics described by the contracting envelope model or preferred edge-attack model. Surface passivation layer as well as deuteride layer are suggested to play important roles in the formation of cracks.

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## 钛中氘致裂纹的原位研究

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**摘要:** 采用热台显微镜 (HSM) 和压力-体积-温度 (PVT) 相结合的方法原位研究了钛在恒定温度及升温过程中的氘致裂纹 (DIC) 现象。结果表明: 在 550 °C 恒温吸氘 (D<sub>2</sub>) 过程中, 钛片表面只出现很少的裂纹; 而在由室温升温至 550 °C 吸氘过程中钛表面出现了由边缘向中心部分扩展的环状裂纹。钛在升温过程中的形貌变化特征与“边缘进攻”模型符合; X 射线光电子能谱 (XPS) 测试显示, 由钛氧化物、碳化物和氮化物组成的钛表面钝化层在环状裂纹形成过程中发挥了重要作用。

**关键词:** 储氢材料; 钛; 氘致裂纹; 动力学

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