



# Disruption tests on repaired tungsten by CVD coating

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## Abstract

The chemical vapor deposition (CVD) coating is considered as one of the possible methods for in situ repairing of the tungsten armor. In the present work, CVD coatings on eroded tungsten specimens were prepared to investigate the applicability of this method to repair the eroded tungsten surface. To simulate the damaged surface relevant to the disruption erosion, specimens were irradiated by an electron beam at a heat flux of 1250 MW/m<sup>2</sup> before the CVD repairing procedure. From metallographic results, no pores or cracks were observed at the interface between the CVD layer and the eroded layer seemed to be successfully repaired by the CVD coating. However, the CVD layer was delaminated by thermal shock tests which simulate disruption heat loads. It was found that complete removal of the resolidified area before CVD coating is effective to repair the eroded surface.

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## 1. Introduction

Tungsten has a high threshold energy for physical sputtering, good thermal conductivity and the highest melting point of all metals. These physical properties make it attractive to use tungsten as an armor material for the next step fusion experimental reactor [1]. However, the erosion of tungsten due to the plasma–wall interactions, especially during disruptions, is severe and a method which allows to repair the damaged surface by remote handling is strongly required. The chemical vapor deposition (CVD) coating is proposed as one of the possible methods for in situ repair of the armor.

In the present work, CVD coating tests on damaged tungsten surfaces were performed to investigate the durability of the repaired surface. To simulate the damaged surface, disruption heat loads were subjected to the tungsten sample by the electron beam irradiation facility. During the electron beam irradiation, melting and evaporation occur on the surface, and a resolidified layer is produced [1]. The CVD coating was deposited on this

eroded surface. The interfaces between CVD layer and the base material were observed by scanning electron microscopy. After repairing the damaged area, thermal shock tests were performed in order to elucidate the durability of the coated layer. The effect of the resolidified layer of the damaged surface on the adhesion of CVD coating was examined.

## 2. Experimental

In this experiment, pure sintered tungsten (P–W) and 1% La<sub>2</sub>O<sub>3</sub> containing tungsten (La–W) were used as a substrate material. Specimens used in this work were supplied from the Tokyo Tungsten Corporation. Both tungsten grades (P–W and La–W) were fabricated by sintering of the powdered W and La<sub>2</sub>O<sub>3</sub>. The purity for 1% La<sub>2</sub>O<sub>3</sub> and pure W was 99.9% and 99.999%, respectively. The size of the specimens was 20 mm × 20 mm × 5 mm. To simulate the damage caused by the disruptions, the substrate materials were irradiated by an electron beam irradiation facility [2,3]. The typical electron beam profile used in this work is shown in Fig. 1. The adsorbed power density at the peak position was 1250 MW/m<sup>2</sup> and the pulse duration was 2 ms. The temperature of the specimens before irradiation was kept at room temperature.

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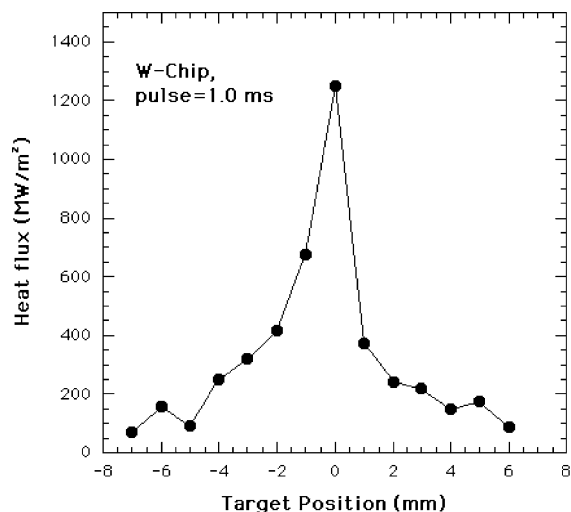


Fig. 1. Typical beam profile used in this work. The heat flux was measured by calorimetric methods using a tungsten cylinder.

The CVD layer was produced by the chemical reaction between  $WF_6$  and  $H_2$  [4]. Substrate temperature and gas flow rate are the key parameters, which determine the quality of the CVD coating. In this work, they were kept at 1000 K and 1.2 m/s, respectively. Under these conditions, deposition rate was about 0.2 mm/h and the CVD layer was grown up to 1 mm thickness. Table 1 summarizes the impurity contents of the CVD layer. The impurity level of the CVD is very low and the purity is 99.9999%. One test specimen which was not subjected to the electron beam was also prepared for comparison.

After repairing the damaged area by CVD coating, thermal shock tests were performed in order to elucidate the durability of the coated layer. In this test, CVD coated samples were irradiated by the electron beam irradiation facility. Acceleration voltage of the electron

Table 1  
Impurity content of the CVD layer

Impurity	Concentration (ppm)
O	0.065
F	0.31
Na	0.011
Al	<0.001
Si	<0.001
Ca	<0.04
Cr	<0.001
Fe	0.047
Ni	<0.001
Mo	<0.004
Purity	99.9999%

beam, absorbed heat flux at the peak position, pulse duration, pressure of the vacuum chamber and temperature of the specimen are 65 kV, 1250  $W/m^2$ , 2 ms,  $3.0 \times 10^{-2}$  Pa and room temperature, respectively.

### 3. Experimental results and discussions

#### 3.1. Repairing of damaged surface by CVD coating

Before the CVD coating, La–W and the P–W were irradiated by the electron beam facility at the heat flux of 1250  $W/m^2$  in order to simulate the surface damage by the disruption erosion. Fig. 2 shows the typical surface damage of P–W and La–W after the irradiation of 1250  $W/m^2$  [5]. As can be seen from Fig. 2, the resolidified layer is observed on the surface. The diameter and the depth of the crater were 6 mm and 0.3 mm, respectively. The diameter of the resolidified area was almost the same for P–W and La–W, however, the structure of the eroded area showed completely different features. Splashing of the melted layer was observed in the case of La–W, whereas a relatively smooth surface was seen for

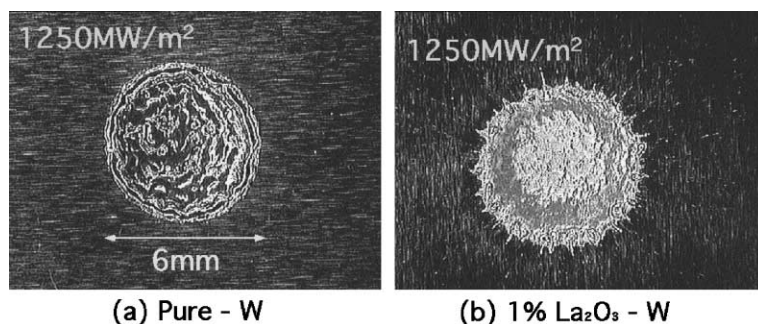


Fig. 2. Optical microscope images of a resolidified area after electron beam irradiation (2  $\mu$ s): (a) pure tungsten and (b)  $La_2O_3$  containing tungsten [4]. Absorbed power density was 1250  $W/m^2$ .

P–W. In addition, it was found that the weight loss due to the evaporation and/or droplet formation for La–W (1.80 mg) was three times larger than that for P–W (0.65 mg) [5].

The CVD coating was deposited on these damaged specimens without any pre-treatment of the surface. Fig. 3 shows the micrograph of the interface between CVD layer and substrate material (P–W). Surface defects such as cracks or deformations were not observed on the CVD layer. On the surface of the substrate, a melted and resolidified layer due to the electron beam irradiation is observed. In addition, at the edge of the resolidified area, crack formation has been detected. These defects are due to the thermal stress during resolidification of the melted surface after the electron beam irradiation. The grain size of the resolidified layer seemed to be larger than that of the substrate and the CVD coating layer (1–2  $\mu\text{m}$ ). However, from the result of microscopy observation, no pores or cracks were observed at the interface between the CVD layer and the substrate and the eroded layer seemed to be successfully repaired by the CVD coating.

### 3.2. Thermal shock tests on repaired surface

#### 3.2.1. Durability of CVD coating without pre-treatment of the damaged area

In order to investigate the durability of the repaired CVD layer against the disruption heat load, another thermal shock test was performed on the coated test samples. The specimens repaired by CVD coating were again irradiated by the electron beam irradiation facility at a absorbed heat flux of 1250 MW/m<sup>2</sup> for 2 ms. P–W and La–W specimens with and without damage before CVD coatings were prepared for comparison. Table 2 summarizes the results of the thermal shock tests. In the case of La–W which was pre-damaged before the CVD coating, the CVD layer was delaminated by a single shot. In the case of P–W, the CVD layer was delaminated after several shots. For both specimens, the delamination occurred at the interface between CVD and substrate. On the other hand, CVD layers which were

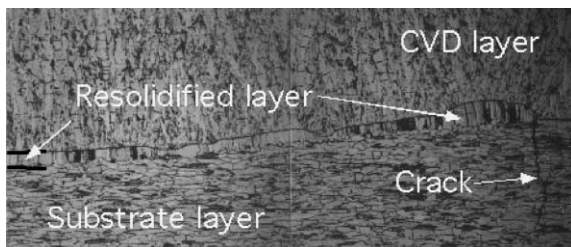


Fig. 3. Photograph of the interface between CVD and the substrate material (P–W).

Table 2  
Result of the thermal shock tests on the repaired surface

No.	Material	Predamaged area	Results
1	La–W	1250 MW/m <sup>2</sup> , 2 ms	Delaminated by 1 shot
2	La–W	1250 MW/m <sup>2</sup> , 2 ms	Delaminated by 1 shot
3	La–W	1250 MW/m <sup>2</sup> , 2 ms	Delaminated by 1 shot
4	La–W	1250 MW/m <sup>2</sup> , 2 ms	Delaminated by 2 shots
5	La–W	None	Delamination did not occur up to 5 shots
6	P–W	1250 MW/m <sup>2</sup> , 2 ms	Delaminated by 2 shots
7	P–W	1250 MW/m <sup>2</sup> , 2 ms	Delaminated by 3 shots
8	P–W	1250 MW/m <sup>2</sup> , 2 ms	Delaminated by 2 shots
9	P–W	1250 MW/m <sup>2</sup> , 2 ms	Delaminated by 2 shots
10	P–W	None	Delamination did not occur up to 5 shots
11	P–W	1250 MW/m <sup>2</sup> , 2 ms + Cleaning	Delamination did not occur up to 4 shots
12	P–W	1250 MW/m <sup>2</sup> , 2 ms + Cleaning	Delamination did not occur up to 4 shots

not subjected to the electron beam loading before the CVD coating, were not delaminated after multiple irradiations up to 5 shots (nos. 5 and 10 in Table 2). This fact indicates that the existence of the resolidified layer at the interface of the CVD layer plays an important role for the adhesion of the CVD layer. During the resolidification process, the surface of the melted layer is considered to be oxidized due to the oxygen impurity in the vacuum chamber. Although the oxygen content level in the chamber is unclear, oxygen is considered to exist to some extent due to the low vacuum level (0.03 Pa) at the sample position. Therefore, the adhesion at the damaged area is considered to be weak due to the oxidization of the resolidified layer. This might cause the initial crack at the interface, which leads to the delamination of the CVD layer during the irradiation. The adhesion of P–W was slightly better than that of La–W. This is considered to be due to the lower impurity level of P–W. The oxygen impurity contained in La<sub>2</sub>O<sub>3</sub> might influence the adhesion characteristic of La–W.

#### 3.2.2. The CVD coating after removing the eroded area

To elucidate the effect of the resolidified layer on the durability of the CVD coating, the CVD coating was also performed on P–W after removal of the damaged area. In this specimen, the resolidified layer of the

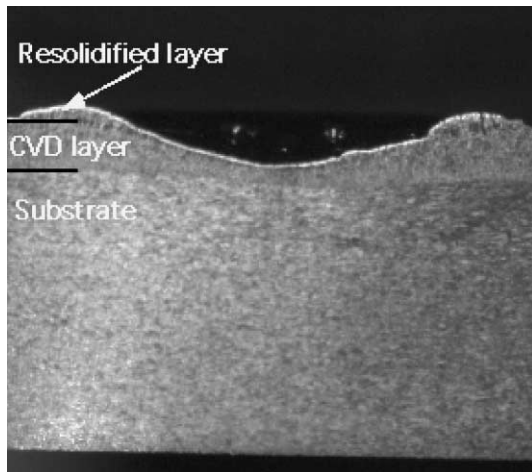


Fig. 4. Cross-sectional view of the irradiated area of the CVD coated specimen with surface cleaning before CVD coating.

substrate material was removed by machining before the CVD coating. In this case, the CVD coated layer was not delaminated after the multiple thermal shocks up to four shots at the same heat flux ( $1250 \text{ MW/m}^2$ ). Fig. 4 shows the metallographic section of the irradiated area of a CVD coated specimen after removing of the resolidified layer. After four irradiations, the surface of the CVD layer is severely eroded but the CVD layer was not delaminated. This result suggests that the adhesion between CVD coating and substrate is considered to be suitable to withstand heat loads of  $1250 \text{ MW/m}^2$ . In addition, it should be noted that no crack formation was observed at the edge of the melted layer although a large crack was observed in case of P–W or La–W (Fig. 2). From these results, it can be concluded that the CVD coating is effective to repair the damaged surface when melted and resolidified layers on damaged surface are removed before the CVD coating.

#### 4. Conclusions

In order to elucidate the applicability of the CVD coating technique to repair damaged surfaces of tungsten armor, CVD coating tests were performed on the specimens with simulated disruption erosion. The durability of the CVD layer was investigated by the thermal shock test. The CVD coatings on the damaged surface without the pre-treatment were delaminated by the thermal shock of  $1250 \text{ MW/m}^2$  for 2 ms. However, the delamination did not occur on the CVD coatings after removal of the resolidified layer. This fact indicates that the repair of the damaged surface by CVD is effective by removing the resolidified layer on the eroded surface.

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