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# Temperature Dependence of the Crystal Growth of Diamonds Deposited on Stainless Steel Substrates by the Combustion Flame Method<sup>†</sup>

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## Abstract

*In order to obtain some useful information for the high rate combustion flame diamond deposition process using various substrates, as a basic study, diamond depositions on surface cooled stainless steels with varying acetylene/ oxygen flow ratios during operation and with twin gas torches were carried out. As a result, it was proved that the diamond deposition rate was promoted by varying the gas flow ratio and/ or using the twin gas welding torch.*

**KEY WORDS:** (Combustion flame), (Diamond), (CVD), (Gas welding), (Thermal plasma)

## 1. Introduction

Since the combustion flame method, which is a diamond deposition process using an acetylene/ oxygen welding torch, has some merits, for example, high deposition rate (approximately 40 $\mu\text{m}/\text{h}$ ), atmospheric process and so on, it is expected to be utilized as a low cost diamond deposition process. However, the combustion flame method has some disadvantages such as small deposition area (5mm in diameter) in comparison with those of conventional thermal and plasma CVD, together with thermal damage of the substrate during diamond deposition due to direct irradiation of the combustion flame<sup>1)-3)</sup>.

As protection processes from the thermal damages of the substrate, techniques using substrate surface cooling<sup>4)</sup> and insertion of a thermal stress buffer layer between substrate and top coating<sup>5)</sup> were developed and some benefits could be confirmed. On the other hand, for the large area diamond deposition process, though the combustion flame methods using parallel equipped gas welding torches and/ or traversing substrates have been successfully conducted, thermal damage of the substrate is inevitable in the case of parallel equipped gas welding torch use and a higher deposition rate is required in the case of use of the traversing substrate.

In this study, in order to increase the deposition rate in the case of a traversing substrate use, the diamond deposition using the combustion flame method with variation of the temperature and with twin gas welding

torches was carried out.

## 2. Experimental

**Fig.1** shows the schematic diagram of the conventional single torch type combustion flame diamond deposition equipment. This equipment consists of acetylene/ oxygen combustion flame welding torch, gas supply system including mass flow controller and substrate holder. A sample was deposited on the stage of the substrate holder. Inlet pressures of  $\text{C}_2\text{H}_2$  and  $\text{O}_2$  were 2.5kg/cm<sup>2</sup> and 3.5kg/cm<sup>2</sup>, respectively. Mass flow of  $\text{O}_2$  was fixed at 1.25 SLM, while mass flow ratio of  $\text{C}_2\text{H}_2/\text{O}_2$  was also fixed at 1.15. Deposition distance was fixed at 10mm to make the acetylene feather of the combustion flame irradiate the surface of the substrate. Deposition time was 20 min. As a substrate, 12 mm x 10 mm x 1mm molybdenum plate was used. The substrate was polished by #400 water resist sand paper before operation in the case of diamond deposition on polished Mo coating. The deposition temperature during operation ( $T_d$ ) was observed by a radiation thermometer.  $T_d$  was controlled by varying the contact condition between the substrate and the cooling pipes located on the substrate surface. Therefore, the temperature was controlled without varying the conditions of combustion flame. **Fig.2** shows the twin torch type combustion flame diamond deposition equipment. In this case, in order to protect the substrate from thermal damages during diamond deposition, a substrate surface cooling system was added. After

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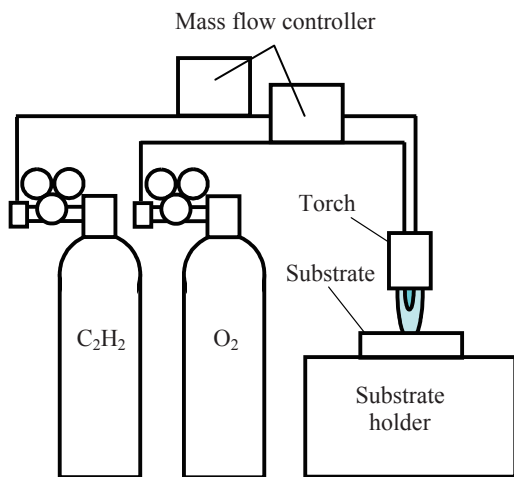
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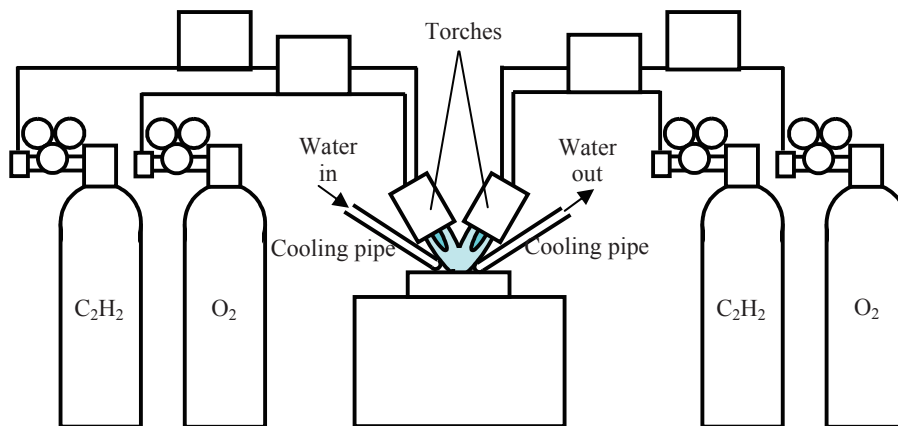
**Fig.1** Schematic diagram of the single torch type combustion flame diamond deposition equipment.

diamond deposition, investigation of the microstructure of the diamond deposited substrate was carried out using optical microscope and X-ray diffraction. Table I shows the experimental conditions.

### 3. Results and Discussion

#### 3.1 Diamond deposition without surface cooling

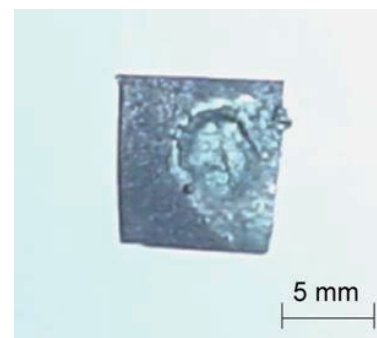
**Fig.3** shows the appearance of the diamond deposited on the stainless steel substrate without substrate surface cooling. In this case, though we do not need to take account of the fracture and peeling off of the coating as shown in the case of thermal sprayed substrate, diamonds could not be deposited due to the melt down of the substrate which had occurred during diamond deposition. This phenomenon was thought to occur due to its low melting point compared to that of the molybdenum coating. **Fig.4** shows the phase diagram of the Fe-Mo binary system. According to the phase diagram, though the melting point of Mo is 2893 K, the melting point of Fe is 1733 K. So, in order to prevent the melt down during the diamond deposition, the promotion of heat



**Fig.2** Schematic diagram of the twin torch type combustion flame diamond deposition equipment.

**Table 1** Experimental conditions in the case of the single torch use.

Working gas	C <sub>2</sub> H <sub>2</sub> /O <sub>2</sub>
C <sub>2</sub> H <sub>2</sub> flow rate	1.4 SLM
O <sub>2</sub> flow rate	1.25 SLM
Deposition distance	10 mm
Deposition time	5, 10 min
Deposition temperature	1200-1723K
Substrate	Mo



**Fig.3** Appearance of the stainless steel sample after operation.

conductivity between the substrate and the substrate holder was achieved by the infiltration of silver between them. However, the melt down could not be eliminated even in this case.

**3.2 Diamond deposition with surface cooling**

Therefore, diamond deposition with substrate surface cooling was conducted to protect the substrate from the heat from the combustion flame shown in Fig.5.

Fig 6 shows the appearances and optical micrographs of the diamonds deposited on Fe coating and 304 stainless steel substrate (deposition temperature: 1423 K). By cooling the substrate surface, diamonds could be deposited on the stainless steel substrate without melt

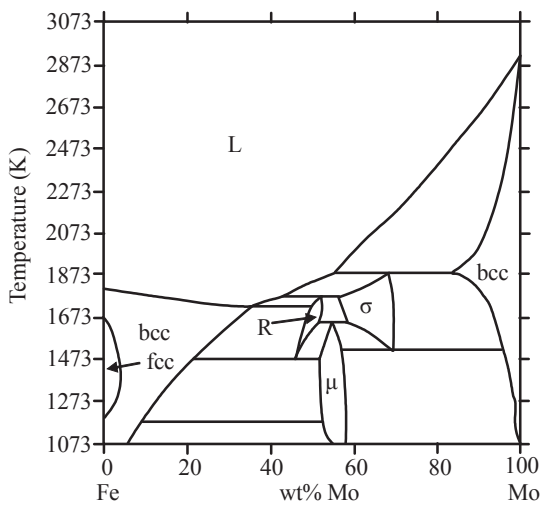


Fig.4 Phase-diagram of Fe-Mo binary system.

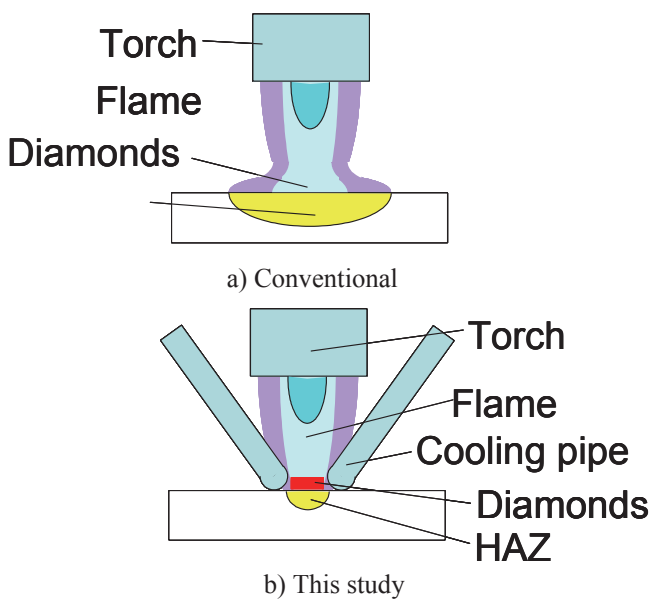


Fig.5 Schematic diagram of the substrates during diamond deposition.

down of the substrate during operation. From these results, it was proved that this technique was effective for diamond deposition on the relatively low melting point substrate and stainless steel was available as the substrate for diamond deposition in the case of the combustion flame method. The reason why diamond could be deposited even on the stainless steel substrate is thought to be as follows;

- a) An intermediate layer is created on the substrate by chemical reaction between substrate and combustion flame during diamond deposition.
- b) Nucleation occurs during flight in the combustion flame.

Fig.7 shows the XRD pattern of the stainless steel substrate after diamond deposition. As shown in this figure, iron carbide as well as diamond and pure iron was confirmed.

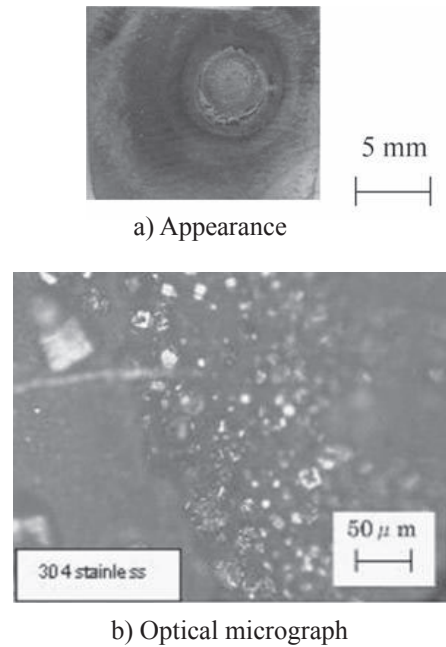


Fig.6 Appearance and surface micrograph of the diamond deposited on 304 stainless steel substrate.

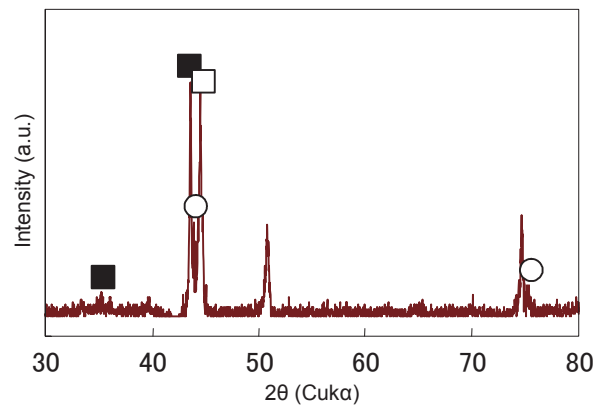


Fig.7 XRD patterns of the surfaces of the sample. (○ : Diamond, ■ : Fe<sub>3</sub>C, □ : Fe)

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### 3.3 Diamond deposition on stainless steel substrates with varying $C_2H_2/O_2$ flow ratio during operation

Using the combustion flame method, it is difficult to deposit diamond particles densely due to the increased deposition temperature, diamond particle density is decreased though diamond growth rate is promoted. Therefore, in our previous study<sup>4)</sup>, diamond deposition on Mo substrates with varying the  $C_2H_2/O_2$  flow ratios during operation was conducted in order to deposit diamond particle densely. Consequently, it was proved that diamond particle density could be promoted by varying the working gas flow ratio of  $C_2H_2/O_2$  as shown in Fig.8. In this study, to confirm whether this technique is dependent or not on the condition of 304 stainless steel substrate, diamond deposition on stainless steel substrates with varying  $C_2H_2/O_2$  flow ratios during operation was conducted. Fig.9 shows the appearance and surface micrograph of the diamond deposited on 304 stainless steel substrate. As shown in this figure in comparison with Fig.6, diamond particle density was promoted and diamond deposition with varying  $C_2H_2/O_2$  flow ratio was proved to be effective for dense diamond particle deposition.

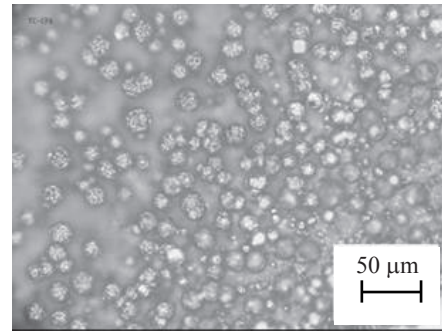
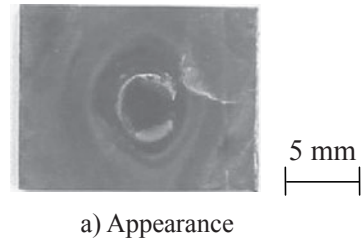


Fig.9 Appearance and surface micrograph of the diamond deposited on 304 stainless steel substrate with varying  $C_2H_2/O_2$  flow ratio during operation.

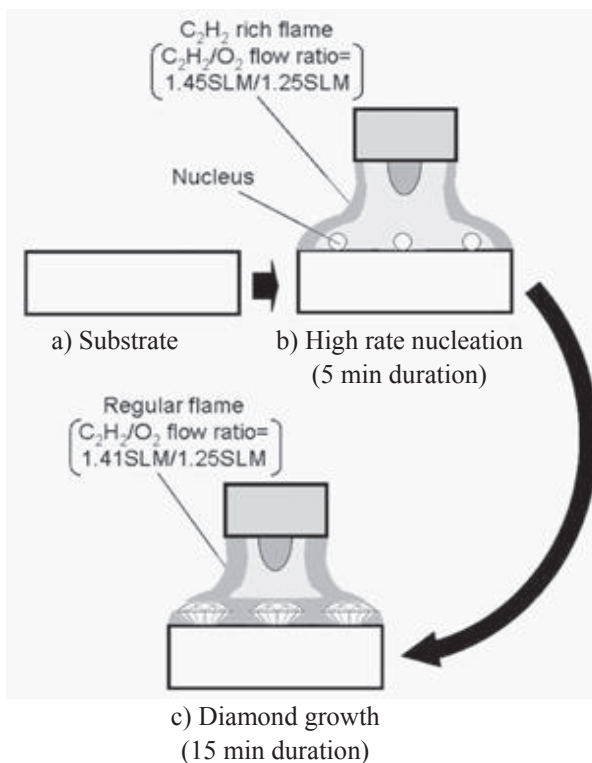


Fig.8 Procedure of the diamond deposition on stainless steel substrate with varying  $C_2H_2/O_2$  flow ratio during operation.

### 3.4 Diamond deposition using twin torch

In the previous section, it was proved that by varying the  $C_2H_2/O_2$  flow ratio during operation, diamond deposition rate was promoted. In this section, as another diamond deposition rate promotion method, the results in the case of the diamond deposition using a twin torch is introduced. Fig. 10 shows the appearance of the diamond deposition using twin gas welding torch. The combustion flames were thought to be mixed into a high density flame according to the appearance.

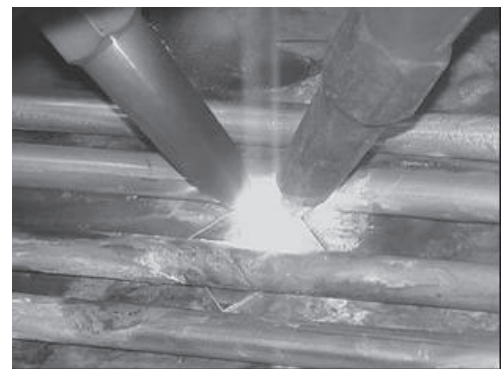
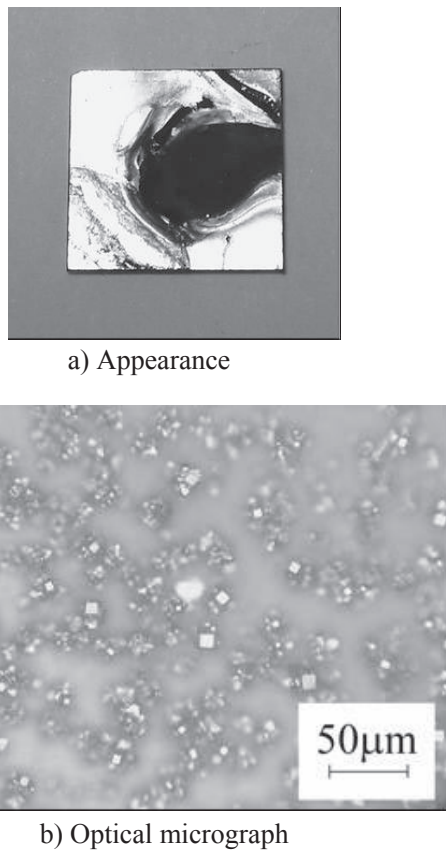
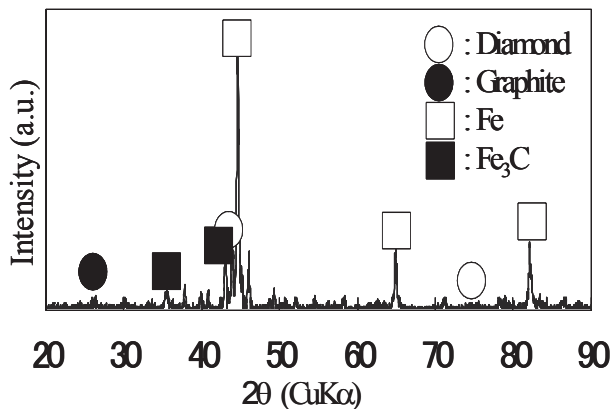


Fig.10 Appearance of the substrate during diamond deposition using twin gas welding torch.



**Fig.11** Appearance and optical micrograph of the 304 stainless substrate surface after 15 min. diamond deposition in the case of the twin torch use.



**Fig.12** XRD pattern of the stainless steel substrate after diamond deposition using the twin gas welding torch.

**Fig.11** shows the appearance and the optical micrograph of the stainless steel substrate after 15 min. diamond deposition using the twin torch. Although the heat transfer from the combustion flame to the substrate combustion flames, diamond could be deposited without melt down of the substrate. In addition, it takes only 15min. to deposit 10  $\mu\text{m}$  large diamond particles in contrast to 20 min. to deposit the same size diamond particles in the case of the single torch use. From this result, this technique using the twin torch is thought to be effective for high rate diamond deposition in the combustion flame method. As for the crystal structure, as shown in the XRD pattern (**Fig.12**), iron carbide was also confirmed.

#### 4. Conclusion

Diamond deposition on the 304 stainless steel substrate was carried out by combustion flame CVD. Consequently, the following results were obtained.

- (1) By cooling the substrate surface, diamond particles could be deposited on the stainless steel substrate without melt down of the substrate.
- (2) By varying the working gas flow ratio of  $\text{C}_2\text{H}_2/\text{O}_2$ , diamond deposition rates could be promoted.
- (3) By using a twin gas welding torch, diamond deposition rates could be improved.

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