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Citation	Transactions of JWRI. 2011, 40(2), p. 37-40
Version Type	VoR
URL	https://doi.org/10.18910/10559
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High Rate Diamond Deposition by Cooled Acetylene/ Oxygen Combustion Flame†

SASO Katsumasa*, ANDO Yasutaka*, KOBAYASHI Akira**

Abstract

Since the combustion flame method using acetylene/oxygen is a high rate diamond synthesis process can be conducted in open air, this process is thought to be useful for low cost diamond production. In this study, to develop a method for diamond synthesis on a thick and/or complicated shape article, diamond depositions by water-cooled and air-cooled combustion flames were carried out. It was proved that diamonds could be deposited on 3 and 5 mm thick 304 stainless steel substrates without melt down of the substrate. Besides, in the case of 15 l/min in air flow rate, diamond like hexahedron and octahedron particles could be synthesized on the substrate without meltdown of the substrate even in the condition without substrate bottom cooling.

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

1. Introduction

The combustion flame method is a diamond deposition process using an acetylene/ oxygen welding torch [1)-3)]. This process is hoped to be a useful method for low cost diamond deposition because of its excellent features such as high deposition rate, deposition which can be conducted in open air, simple constitution of the equipment and so on. However, since the combustion flame contacts the substrate surface during deposition, it is very difficult to deposit diamonds on the thick or complicated shape substrates without thermal damages.

As methods to eliminate the thermal damages, the following techniques are thought to be effective.

(1) Cooling the substrate surface by water cooled pipes (Cooled substrate surface method (CS method))

(2) Cooling the combustion flame by water cooled pipes (Water cooled combustion flame method (WC method, AC method))

(3) Cooling the combustion flame by air (Air cooled combustion flame method)

In the case of the CS method, according to our previous study [4)], since it is not sufficient for diamond deposition on the substrate without thermal damages of the substrate to cool the substrate surface, cooling of the substrate bottom is still demanded. Therefore, the WC and AC methods are thought to be better than the method (1). However, in cases of (2) and (3), some disadvantages such as energy loss, deactivation of the activated particles and recombination of radicals due to cooling combustion flame should be taken into account.

In this study, in order to develop the high rate diamond deposition process for thick or complicated shaped articles, as a basic study, diamond deposition using cooled combustion flame was carried out.

2. Experimental

Fig.1 shows the schematic diagram of the conventional combustion flame diamond deposition equipment. This equipment consists of acetylene/ oxygen welding machine, mass flow controller, substrate holder. In this study, diamond depositions using above mentioned “Water cooled combustion flame method (WC method)” and “Air cooled combustion flame method (AC method)” were carried out. **Fig.2** shows the illustration of the situations around the substrates during diamond deposition in cases of these methods. Although the water cooled substrate holder was used in the case of WC methods, the non-cooled substrate holder was used in the case of AC method. As the substrate, 3 and 5 mm thick 304 stainless steel plates (with 15mm*15mm in the other dimensions) in the case of WC method and 1 mm thick Mo plate (with 12mm*10mm in the other dimensions) were used in AC method. In order to confirm the deposition temperature during deposition, an infrared thermometer was used. After the diamond deposition, investigation of the microstructure of the diamond deposited substrate was carried out using optical

† Received on Dec. 26, 2011
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** JWRI

Transactions of JWRI is published by Joining and Welding Research Institute, Osaka University, Ibaraki, Osaka 567-0047, Japan

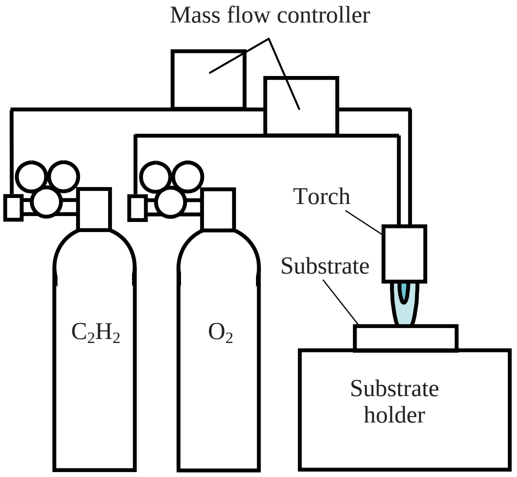
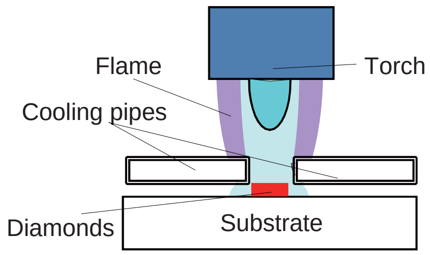
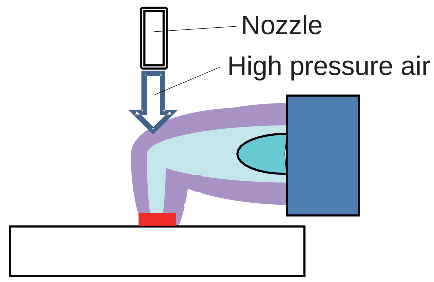


Fig.1 Schematic diagram of the single torch type combustion flame diamond deposition equipment.



a) Water cooled combustion flame method



b) Air cooled combustion flame method

Fig.2 Illustrations of the substrate protection systems.

Table I Diamond synthesis condition

Atmosphere	Open air
Ambient pressure	1 atm
Working gas	C_2H_2/O_2
C_2H_2 flow rate	1.45SLM
C_2H_2 pressure	0.06kg/cm^2
O_2 flow rate	1.25SLM
O_2 pressure	0.4kg/cm^2
Air flow rate	15～16L/min
Air pressure*	0.1～0.3kg/cm^2
Synthesis time	20～300min
Synthesis temperature	598～1001℃
Substrate	Mo, SUS304

* Only in the case of air cooled combustion flame method

microscope and X-ray diffraction. Table I shows the experimental conditions.

3. Results and Discussion

3.1 Diamond deposition using water cooled combustion flame method

Figs.3-4 show the appearances of the diamond deposited samples and optical micrograph of the diamonds on the conditions of 3 and 5mm thick 304 stainless steel substrates. In the conventional method without cooling combustion flame, even in the case of "Cooled substrate surface method (CS method)", the substrate was meltdown during deposition. However, in

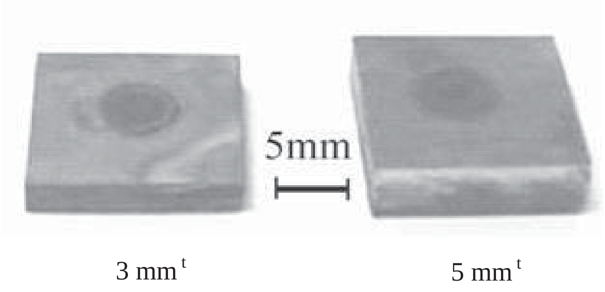
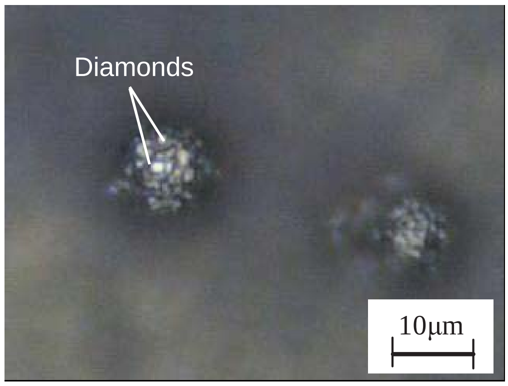
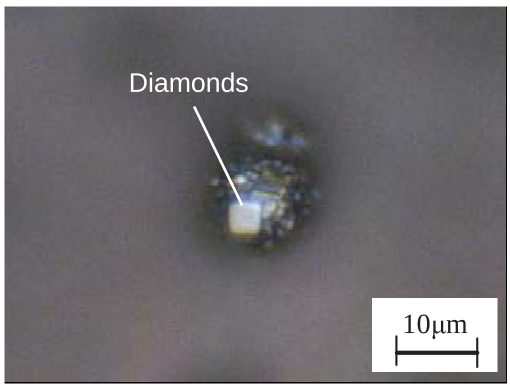


Fig.3 Appearances of the 304 stainless steel substrates after diamond deposition.



a) 3 mm^t



b) 5 mm^t

Fig.4 Optical micrographs of the surfaces of the diamond deposited 304 stainless steel samples.

the case of the WC method, as shown in Figs.3-4, diamond particles with (100) crystal plate could be deposited without melt down of the substrate though diamond deposition rate was deteriorated compared to that in conventional cases. From this result, it was proved that the combustion flame still had enough reactivity to create diamond particles even in the case of the WC method. **Fig.5** shows the XRD patterns of the diamond deposited samples. Also from this result, existence of diamonds could be confirmed.

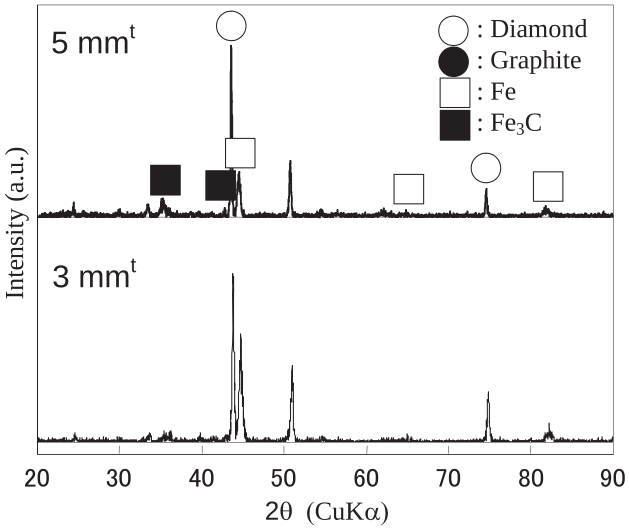
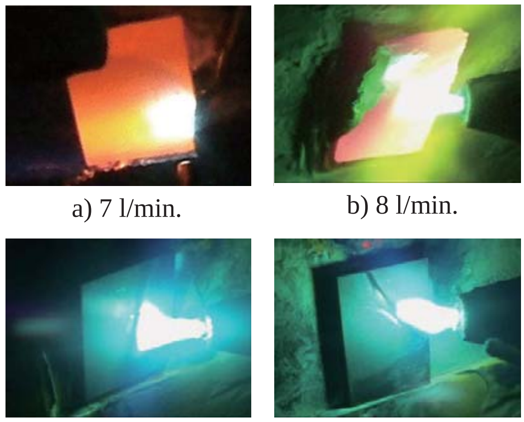


Fig.5 XRD pattern of the diamond deposited 304 stainless steel sample.

3.3 Diamond deposition using air cooled combustion flame method

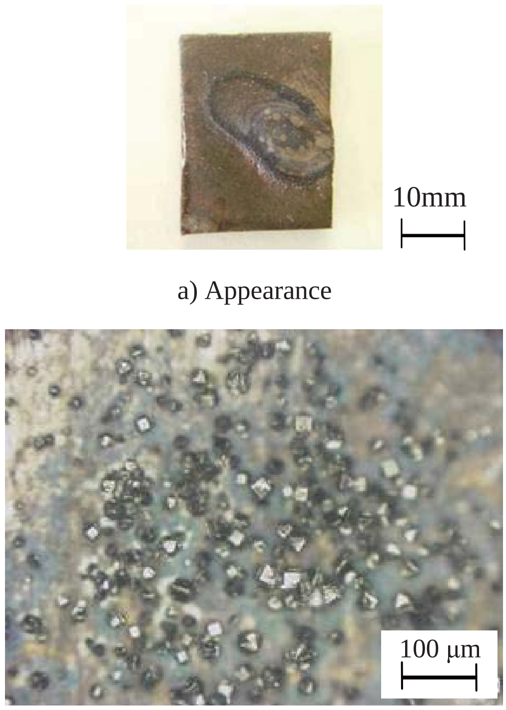
In the case of WC method, though diamonds could be deposited, diamond deposition ability of the combustion flame was drastically deteriorated. Therefore, in this experiment, the combustion flame was cooled by air as a low heat capacity coolant. **Fig.6** shows the photographs of the air cooled combustion flames during diamond deposition. In the case of below 7 l/min. in air flow rate (Q_{Air}< 7 l/min.), the cooling air flux was not sufficient due to its low dynamic pressure, as meltdown of the substrate occurred during deposition though the acetylene feather, which included radicals contributed to diamond deposition, could not reach the substrate surface due to the insufficient deformation of the combustion flame. In the case of Q_{Air}=7 l/min., though the acetylene feather could reach the substrate surface, meltdown of the substrate occurred due to the insufficient cooling air flux. On the other hand, in the case of Q_{Air}=8 l/min., the acetylene feather could reach the substrate surface and the substrate could stand 20 min. continuous combustion flame irradiation without meltdown of the substrate deposition even on the condition of the substrate without cooling its bottom. In the cases of Q_{Air}>8 l/min., since deposition temperature was decreased due to sufficient cooling air flux, diamonds could not be deposited. **Fig.6** shows the appearance and optical micrograph of the Mo substrate after diamond deposition using combustion flame in the case of Q_{Air}=8 l/min. As shown in this figure, 10 μm large diamond particles with (100) or (111) crystal plane could be deposited on the Mo substrate. From this result, air-cooled combustion flame had enough ability to create diamonds on the non-cooled substrate without



a) 7 l/min. b) 8 l/min.

c) 10 l/min. d) 12 l/min.

Fig.6 Photographs of the air cooled plasma jets during diamond synthesis.



a) Appearance

b) Optical micrograph

Fig.7 Appearance and optical micrograph of the the diamond deposited Mo sample in the case of air cooled combustion flame method.

meltdown of the substrate during deposition. Besides, it was proved that the diamond number density in this experiment was almost the same as that in our previous study by conventional combustion flame method.

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 C_2H_2/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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