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Nanoparticle Generation by Confront Electrode Type Plasma Jet†

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Abstract

Nanotechnology is expected to improve the performance of products in many fields. Mass production of carbon nanoparticles by arc discharge has recently been attempted. The carbon nanotube (CNT) has remarkable properties such as high bend and pull strength, low electric resistance, and very small sharp figures are expected to be used in many industrial fields. In this study, a confront electrode type plasma jet has been applied to produce CNTs and MWCNTs (Multi wall CNT).

KEY WORDS: (Plasma Jet) (Carbon Nanotube) (Microwave Discharge) (Confront Electrode Type) (MWCNT)

1. Introduction

Nanotechnology is expected to improve the performance of products in many fields such as ultrahigh-speed transistors and high-capacity memory chips in the IT field, solar cells and fuel batteries in the environment and the energy field, and carbon nanotubes in the material field. Use of nanoparticles is one of the nanotechnologies and, for example, stiffness and/or wear resistance of materials increase by adding nanoparticles to them.

A plasma arc jet is expected to be used in carbon nanotubes (CNTs) production because generation of high purity CNTs with arc discharge method was reported [1)]. One of the authors had already developed a high power plasma jet, was called a gas tunnel type plasma jet, and its performance was described in previous studies [2-4)]. For example, the plasma jet produced by a 200kW Class gas tunnel type plasma jet produces a high temperature of more than 20,000K, high energy density, and high thermal density of 80%. These properties are superior to those of other conventional type plasma jets [5)]. Therefore this plasma has a great potential for various applications in thermal processing [6)]: high quality ceramic coatings by the gas tunnel type plasma spraying method [7, 8)], and alumina coatings with Vickers hardness of Hv = 1200 - 1600 [9)]. As another application, the gas tunnel type plasma jet was applied to the surface nitridation of titanium. Also, the speedy formation of a high functional thick TiN coating [10, 11)] was investigated. On the basis of the knowledge of this device, we have been developing a confront electrode type plasma generator.

In this work, carbon nanotubes were generated with a confront electrode type plasma generator by arc discharge method. A movable confront electrode type plasma generator for effective production of CNT was developed and tested and the results were also reported.

2. Experimental Description

2.1 Experimental apparatus

A schematic diagram of the confront electrode type plasma jet is shown in **Fig.1**. This plasma source has a pair of confront electrodes and the upper electrode is made of Cu and the lower one is made of C (graphite) or C with Ni/YC. The electrodes are connected to a high frequency power supply and a DC power supply (maximum current of 450A). Working gas is introduced into center of the electrodes and cover gas is introduced into both sides of the upper electrode to keep arc discharge stable. A DC arc discharge is generated between the confront electrodes after the first dielectric breakdown by applying high frequency voltage.

2.2 Nanoparticle generation experiment

The nanoparticle generation in 5 experiments were carried out with Ar or N_2 gas as working gas at the electrodes separation of 1.2mm, discharge current of 40~120A, and several discharges (discharge duration of

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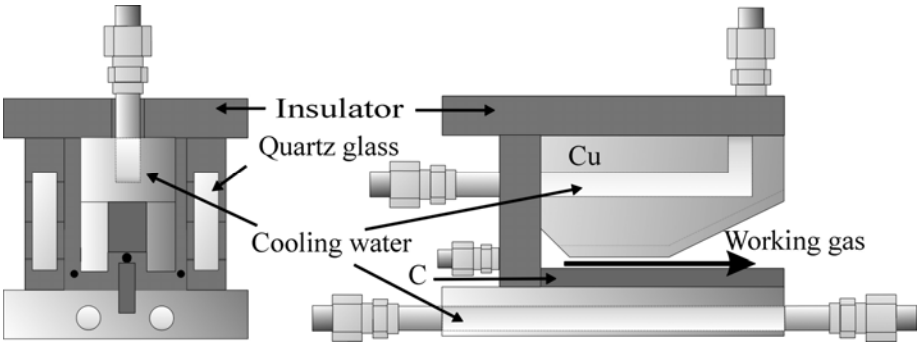

Fig.1 Schematic diagram of the confront electrode type plasma jet.

Table1 Experimental conditions for multi walled carbon nanotube (MWCNT) production.

Experiment No.		1	2	3	4	5
Current (A)		40	40	80	120	100
Discharge duration and number of times		1(s):3	1(s):3 3(s):3	1(s):3	1(s):4	1(s):3
Material and polarity of electrodes	upper	Cu:＋	Cu:＋	Cu:－	Cu:＋	Cu:＋
	lower	C:－	Ni/Y:－	Ni/Y:＋	C:－	Ni/Y:－
Working gas		Ar	Ar	N_2	N_2	N_2
Gas flow rate (l/min)	Center	10	10	6	3	3

1~3 seconds). The details of experimental conditions are listed in **Table 1**. The surface of the lower electrode was observed to investigate nanoparticle generation with a FE SEM at Toyohashi University of Technology.

3. Results and Discussion

The surface observations by a FE SEM were carried out at the center of cathode or anode spots. **Figure 2** shows the electrode of the experiment 1 and its observed surface. The observation spot of the electrode is indicated by an arrow.

Figure 3 shows the electrode of the experiment 2. CNTs were not observed on the both surfaces of C and C with a catalyst of Ni/Y electrodes for the experiment 1 and 2. Spherical features were found on the both sample surfaces. These were caused by insufficient DC arc discharge because of the low discharge current of 40A in these experimental conditions although dielectric breakdown occurred by applying high voltage.

Figure 4 ~ 6 were observations of the sample 3~5 surfaces respectively. MWCNTs (Multi Walled CNT) were observed on the surfaces of the lower electrodes at experiment 3, 4, and 5. MWCNTs were also observed around the cathode spot, however, C particles were intermingled with them. The high-purity MWCNTs were observed in the cathode spot of the experiment 4 because the arrangement of C atoms was changed to suit the discharge at the spot where electrons were emitted mainly. MWCNTs observed on the surface of C electrodes were purer than those of C with a catalyst of Ni/Y.

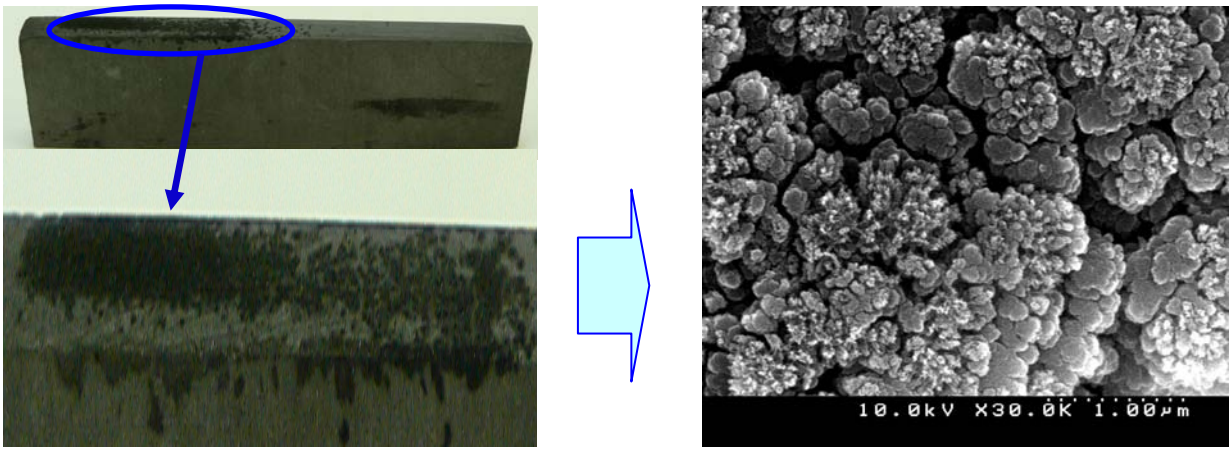

Fig.2 Surface of the experiment 1 sample.

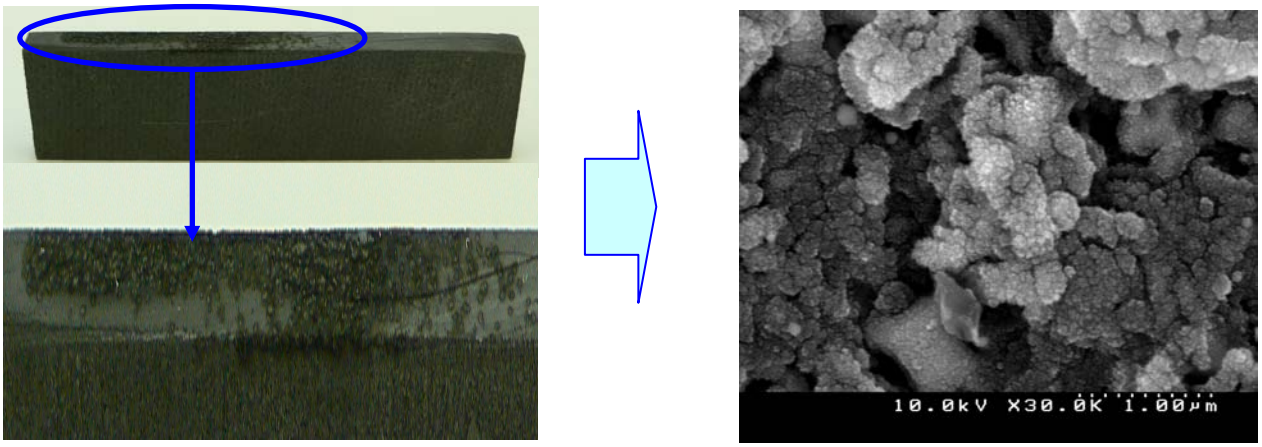

Fig.3 Surface of the experiment 2 sample.

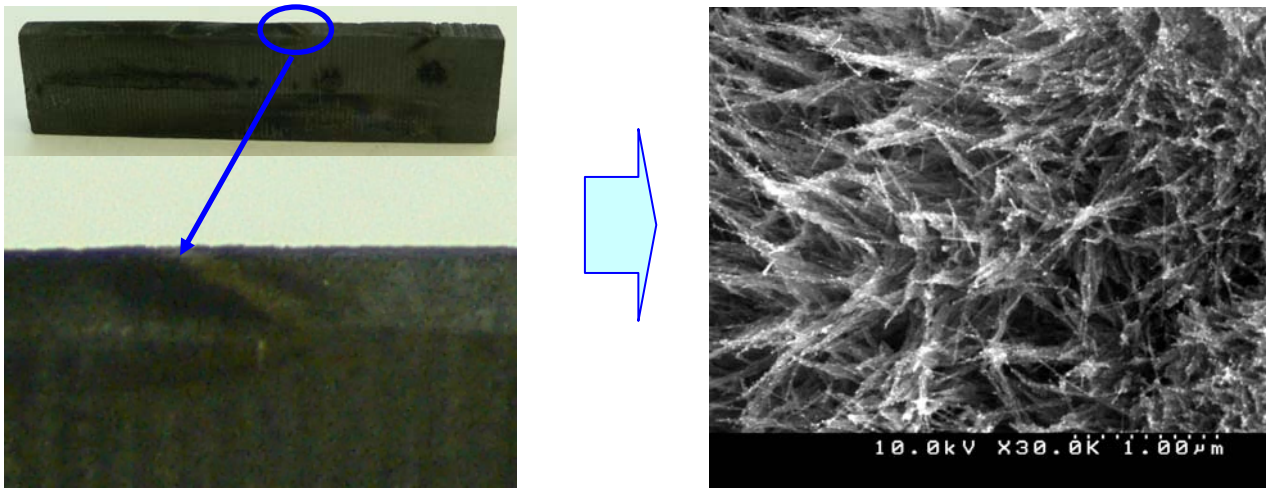

Fig.4 MWCNT observed at the surface of the experiment 3 sample.

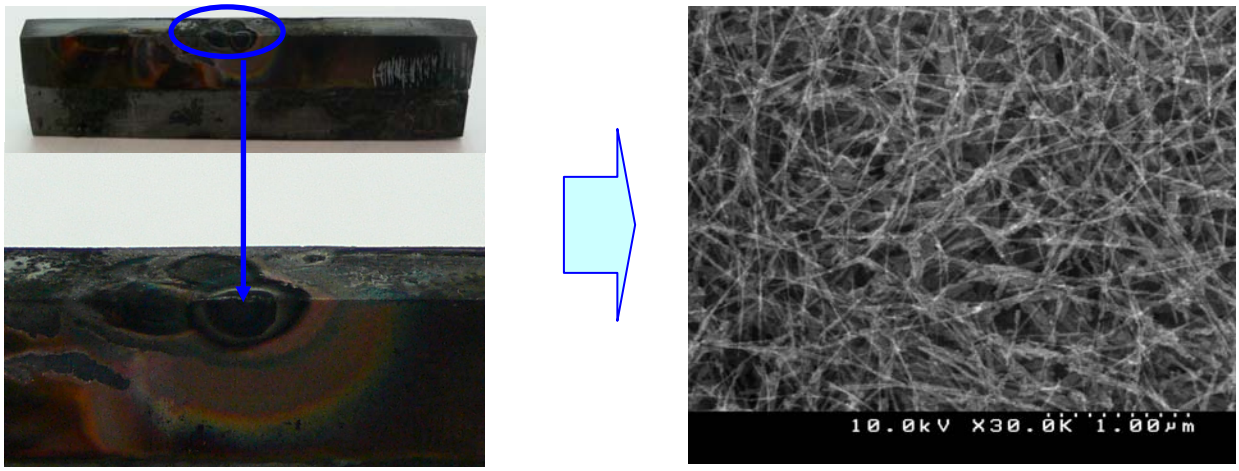

Fig.5 MWCNT observed at the surface of the experiment 4 sample.

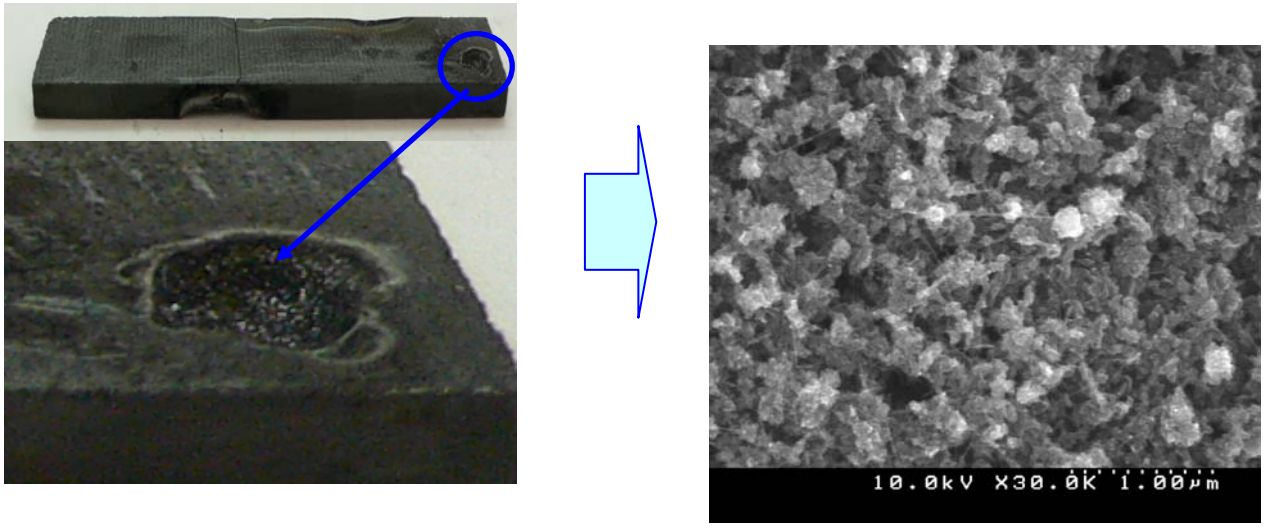

Fig.6 MWCNT observed at the surface of the experiment 5 sample.

4. Improvement of Confront Electrode Type Plasma Generator

4.1 Movable confront electrode type plasma generator

As MWCNTs were generated in the cathode spot in the nanoparticle generation experiments described above, and wear of the lower electrode was found, so a movable confront electrode type plasma generator which can supply a lower electrode successively by a lift was fabricated and is shown in **Fig.7**. To reduce wear of the upper electrode by heating, the upper electrode is covered with a C plate. The passage of a working gas flow is formed by using an upper electrode of a concave shape type by itself to obtain stable arc discharge.

4.2 Current-voltage characteristics of movable confront electrode type plasma generator

I-V curve measurements of the movable confront electrode type plasma generator were carried out at several experimental conditions listed in **Table 2**. By using a pair of confront electrodes of C, wear of the electrodes decreased. I-V curve measurement results of this plasma generator are shown in **Fig.8**.

The most stable plasma jet was obtained at the electrodes separation of 0.5mm, working gas flow rate of 8 l/min, and discharging current of 100A. A drooping characteristic of arc discharge was observed in the experiment 1. Arc discharge was not

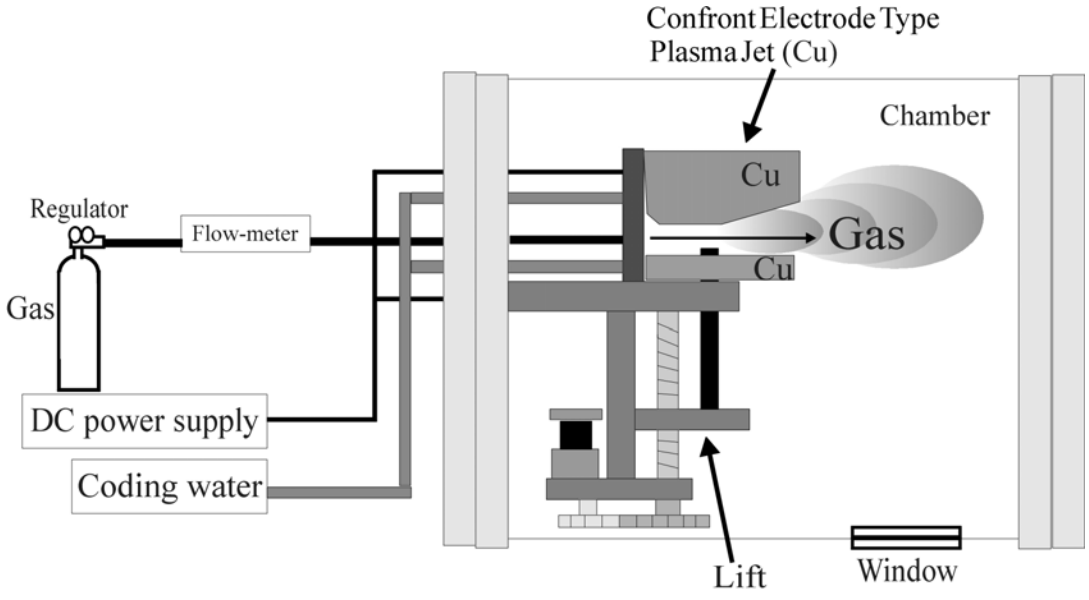

Fig.7 Schematic diagram of the movable confront electrode type plasma jet.

Table2 Experimental conditions for I-V characteristic measurements.

Experiment number		1	2	3	4
Discharge current(A)		10~130			
Discharge duration		continuous			
Electrode interval (mm)		0.5	0.5	1	0.5
Material and polarity of electrodes	upper	C:－	C:－	C:－	C:＋
	lower	C:＋	C:＋	C:＋	C:－
Working gas		He			
Working gas flow rate (l/min)		8	4	4	8

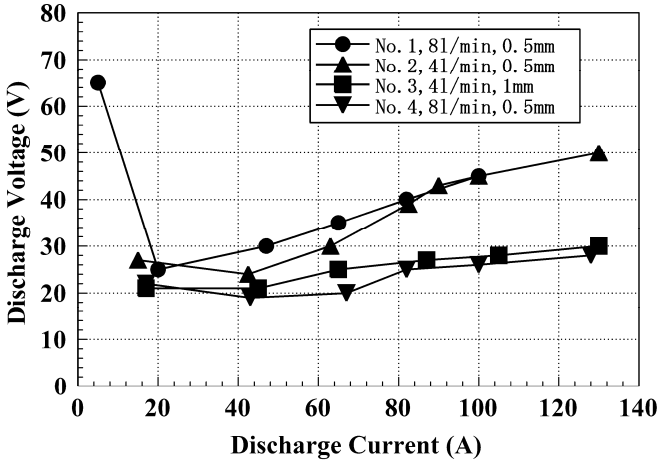

Fig.8 Current-voltage characteristics measurement results.

ignited at other experimental conditions below a discharge current of 15A because of the voltage limit (66V) of the DC power supply.

5. Conclusion

The confront electrode type plasma generator was fabricated and the nanoparticle generation was carried out. The following results were obtained.

(1) MWCNTs were generated by the confront electrode type plasma generator.
(2) High purity MWCNTs were obtained at the electrodes distance of 1.2mm, discharging current of 120A, and N_2 (as working gas) flow rate of 3 (l/min).
(3) The movable confront electrode type plasma generator was developed and its current-voltage characteristics were measured.

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