## An underwater arc discharge method of CNT production using carbon electrode physical vibration\*

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We examined a method of arc discharge in water involving physical vibration as a more cost-effective means of achieving a high yield of highly crystalline CNTs. A device vibrating constantly at 50 Hz was used to apply physical vibration to the carbon electrodes, resulting in the ability to produce stable arc discharge over long periods in water. A powder consisting of a large quantity of fine particles was generated. Observation of this powder under an electron microscope showed that there was little amorphous carbon and that the purity of the CNTs produced was high.

Keywords: CNTs, arc discharge, water, physical vibration

### 1. Introduction

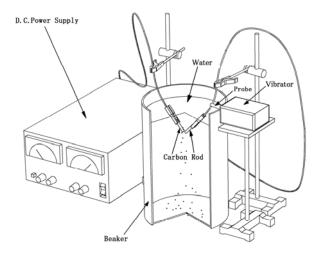
Generally, high-quality crystalline carbon nanotubes (CNTs) are generated using the arc discharge method in a vacuum<sup>1</sup>. This means that arc discharge is carried out in a low-pressure gas-phase medium, necessitating the use of sealed reaction chambers and vacuum equipment. To eliminate this requirement, some researchers have investigated arc discharge in a water-phase medium as an effective method of CNT formation<sup>2-4</sup>.

The underwater arc discharge method is an economical and easy way of producing CNTs. However, because it is difficult to continue stable arc discharge underwater, the purity of the CNTs produced is low, and large quantities of amorphous carbon are also generated. We conducted a study from the unique point of view of developing a new method of CNT synthesis using arc discharge with the aim of producing large amounts of CNTs at low cost.

We examined an arc discharge method in water that involves the application of physical vibration to the carbon electrodes. This technique enabled stable arc discharge for prolonged periods in the water, and generated a large quantity of fine particles including high-purity CNTs.

## 2. Experiment

Figure 1 shows a schematic view of the apparatus used for arc discharge in water with physical vibration. The environment for arc discharge was simple water, and the equipment consisted of a beaker, a DC power supply, a 50-Hz vibrating device and carbon rods as the electrodes for arc discharge. These electrodes were placed in the center of the beaker after connecting them to the DC current source, and the probe of the vibrator was made to come into contact with one of the electrodes.



# Fig. 1 Schematic view of the apparatus for arc discharge in water with physical vibration

Next, an arc discharge was initiated by applying a voltage of 20 V and a DC current of 20 A to the electrodes, and vibration at 50 Hz was applied to one of the electrodes. This made the electrode vibrate at 50 Hz, meaning that the distance between the electrodes constantly changed. As a result, it was possible to continue arc discharge in the water for two hours.

After the application of arc discharge in the water, a large number of black particles appeared at the bottom of the beaker. A sample of these particles was placed on a microscope specimen grid and examined under a high-resolution transmission electron microscopy system operating at 200 kV (HR-TEM model H9500; Hitachi Co., Ltd.).

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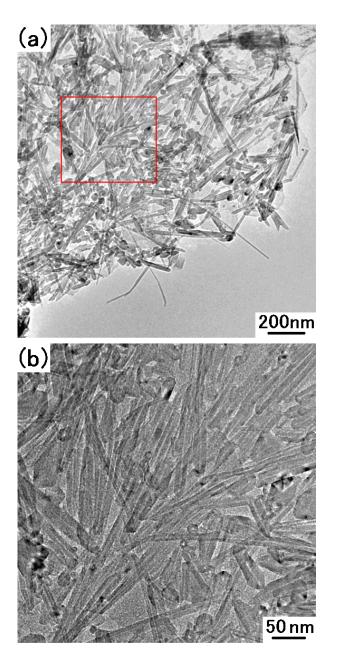


Fig. 2 TEM images of CNTs: (b) is an enlarged view of (a).

### 3. Results and Discussion

In the TEM observations, many high-purity CNTs were found among the fine particles at the bottom of the beaker. These CNTs also contained onion-like carbon particles, as shown in Fig. 2.

Figure 2 (b) is an enlarged view of Fig. 2 (a). The nanotubes produced were very straight and clean, and the TEM images revealed that they were in fact MWNTs with 40 graphite layers and a diameter of 20 nm, as shown in Fig. 3.

We compared the method of arc discharge in water using physical vibration to the regular method of arc discharge in water.

Table 1 shows the amounts (or yields) of fine particles collected at the bottom of the beaker. Ten samples were

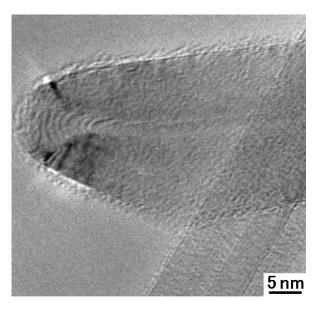


Fig. 3 High-magnification TEM image of a CNT tip

taken, and were divided into CNTs and amorphous carbon (such as graphite particles). The samples were then defined by the percentage of CNTs among the fine particles using TEM observation. Additionally, we estimated the CNT yield among all matter produced from the amounts of fine particles and the percentages of CNTs they contained.

The cathode deposit amounts per hour were 0.76 g with the physical vibration method and 0.28 g with the regular method. Furthermore, the physical vibration method resulted in a CNT percentage of 29%, while the usual method produced just 4%.

Compared with the regular water method (without vibration), the amounts of fine particles generated increased by 250%, and the purity of the CNTs produced improved by 700% under the method of arc discharge in water with electrode vibration.

It is assumed that arc discharge can continue for long periods automatically since the distance between the electrodes is constantly changed due to the vibration. As this addition causes the electrode to repeat contact and separation, it is supposed that CNT forms easily as a result of electric tension.

Table 1. CNT purity

DC arc discharge in water with physical vibration

Yield per hour	Amount	CNT percentage
Amount of fine particles generated	0.760 g	29 %
CNT yield	0.220 g	—

#### DC arc discharge in water without physical vibration

Amount of fine particles generated 0.280	g 4 %
CNT yield 0.011	g –

This simple method of arc discharge in water also makes it easy to collect high-purity CNTs at the bottom of the beaker.

As a result, arc discharge in water with carbon electrode vibration enabled stable arc discharge for long periods in water. A powder consisting of a large quantity of fine particles including CNTs was generated.

Observation of this powder under an electron microscope showed that there was little amorphous carbon and that the purity of the CNTs produced was high. The CNTs could also be easily collected from the bottom of the beaker. Furthermore, the system does not use a vacuum environment, thereby eliminating almost all the complex and expensive machinery usually required for performance of the arc discharge method.

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