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Measurement of anode surface temperature in carbon nanomaterial production by arc discharge method

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ABSTRACT

Nano-graphite particles, multi-wall carbon nanotube (MWNT), and pyrolytic graphite were prepared at different positions of the anode surface in an arc discharge. Graphite electrodes were employed for the arc discharge under helium environment at atmospheric pressure. Nano-sized carbon products were characterized by scanning electron microscopy and transmission electron microscopy. During the arc discharge, two-color pyrometry combined with a high-speed camera was conducted to measure the temperature distribution of the anode surface. The growth temperature of pyrolytic graphite, MWNT, and nano-graphite particles were in the ranges of 2400–2600 K, 2600–2700 K, and 2700–3500 K, respectively. The local temperature of anode surface is a critical parameter to determine the products with different morphologies. The formation mechanism of these carbon nanomaterials is suggested based on the local temperature of anode surface and their thermodynamic stability.

1. Introduction

There has been dramatic interest in carbon nanomaterials since the discovery of fullerenes in 1985 [1]. Since carbon nanomaterials have remarkable physical, chemical, and electronic properties, they show great promise for many potential applications in a variety of technological fields ranging from conductive and high-strength composites to nanoelectronics [2–5]. Therefore, much effort has been made to prepare carbon nanomaterials. For instance, laser vaporization of carbon was introduced to synthesize fullerenes [6]; chemical vapor deposition (CVD) method was used to prepare carbon nanotube and graphene [7,8]; and mechanical exfoliation method was employed to prepare graphene [9].

The arc discharge method has also been employed to prepare lots of carbon nanomaterials as an environmentally benign technique. Fullerene was scraped from the chamber wall in helium arc discharge at low pressure [10]; multi-wall carbon nanotube (MWNT) was collected from cathode deposit at low pressure argon arc [11]; single-wall carbon nanotube (SWNT) was found in a “collaret” around the cathode deposit, the web-like structure was suspended between cathode and chamber wall, and cloth-like soot was suspended in the chamber wall by using the catalyst in helium arc [12]; and graphene flakes were produced in the inner wall of the chamber under hydrogen atmosphere [13]. In particular, many investigations have been carried out on the preparation of carbon nanomaterials on anode surface in the arc discharge method. For example, bamboo-like carbon was prepared by using a catalyst under hydrogen atmosphere [14]. Carbon nanofiber, microfiber, and pillars were obtained on different positions of the anode surface under hydrogen atmosphere [15]. Moreover, vertically-aligned carbon nanotube (CNT) was prepared by a catalyst-free hydrogen arc discharge [16]. According to these works, hydrogen atmosphere is considered as the necessary condition for the growth of tube structured carbon materials on the anode surface. In contrast, Jones et al. [17] prepared MWNT from the anode deposit by arc discharge method without hydrogen. They speculated that negative carbon ion contributes to the formation of MWNT. However, the equilibrium composition of carbon at high temperature arc plasma indicates a relatively low concentration of negative carbon ion, in which the growth of MWNT is difficult. Therefore, the growth condition of tube structured carbon materials on the anode surface is still unclear.

The local temperature seems to be responsible for explaining the formation of different products on the anode surface [15]. However, a narrow electrode gap distance and the strong radiation from the arc plasma in the typical arc discharge obstruct the temperature measurement of electrode surface [11]. Therefore, report about the temperature of anode surface is limited. Although the temperature of carbon anode was measured by an optical

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pyrometer [18], the relationship between temperature and product was not explained.

In the present work, nano-graphite particles, MWNT, and pyrolytic graphite were obtained on the anode surface by an atmospheric pressure helium arc without catalyst. The temperature distribution of anode surface was measured by two-color pyrometry. Based on measured temperature distribution, the formation mechanisms of those carbon nanomaterials were explained.

2. Experimental methods

2.1. Experimental setup

Fig. 1 indicates the schematic illustration of an experimental apparatus for the preparation of carbon nanomaterials and the two-color pyrometry combined with a high-speed camera for temperature measurement. A graphite anode rod of 30 mm in diameter (99.99%, Toyo Tanso Co., Ltd.) with an inclined top plane was put on a water-cooling copper plate. A graphite cathode rod of 6 mm in diameter (99.99%, Toyo Tanso Co., Ltd.) was placed at an oblique angle from the anode. The arc was generated for 5 min in helium environment at atmospheric pressure. The arc current was controlled at 100, 150, and 200 A at the fixed electrode gap distance of 1 mm. In addition, different electrode gap distances of 1, 3, and 5 mm were used at the fix arc current of 200 A. Due to the formation of cathode deposit and the short discharging time, the variation of electrode gap distance is negligible within 5 min. As shown in Fig. 1, the anode surface was observed through a viewport of the chamber by the high-speed camera.

The anode was evaporated by the high heat flux from the arc, leaving a hole at the arc attaching region on the anode surface. Some of the evaporated carbon species were deposited on the peripheral area of the hole to form the anode deposit. Since the evaporated carbon species adsorbed and diffused at the hot anode surface easily, the anode deposit attached to the anode surface tightly. Meanwhile, some of the evaporated carbon species formed the cathode deposit on the cathode tip.

2.2. Sample characterization

The morphological and structural characteristics of as prepared anode deposit were examined by scanning electron microscopy (SEM: JSM-6610LA, JEOL). In addition, the microstructures of the samples were investigated by transmission electron microscopy (TEM: JEM-2010F, JEOL) at the acceleration voltage of 200 kV.

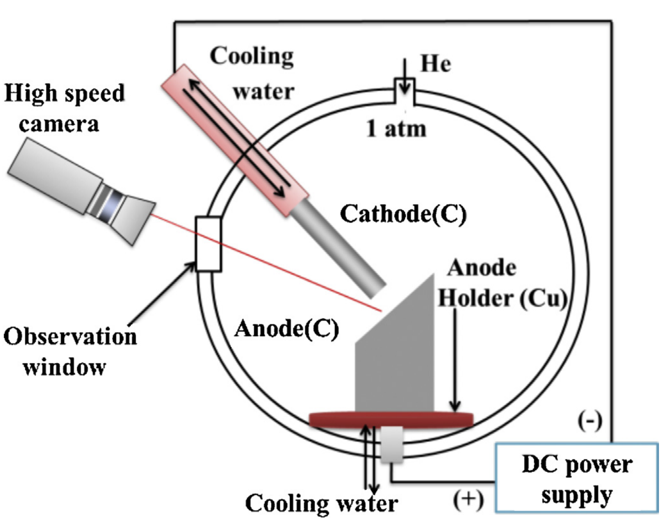

Fig. 1. Schematic diagram of an arc discharge system for the preparation of carbon nanomaterials by arc discharge method.

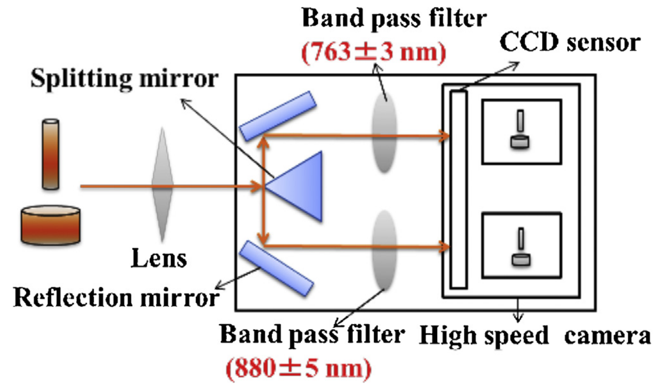

Fig. 2. Schematic illustration of temperature measurement system by two-color pyrometry.

2.3. Temperature measurement

Fig. 2 shows the schematic illustration of two-color pyrometry for the temperature measurement of anode surface during arc discharge. The radiation emitted from the anode surface was divided by a splitting mirror, and then it passed through two band-pass filters. In order to eliminate the influence of line emission from the arc, two band-pass filters of 763 ± 3 and 880 ± 5 nm were employed. Band-pass filters were selected with no line emission in the bandwidth of them. As shown in Fig. 3(a) and (b), images of the electrode at wavelengths of 880 ± 5 and 763 ± 3 nm were simultaneously recorded by the high-speed camera (FASTCAM-SA WTI, Photron). Measurement conditions of

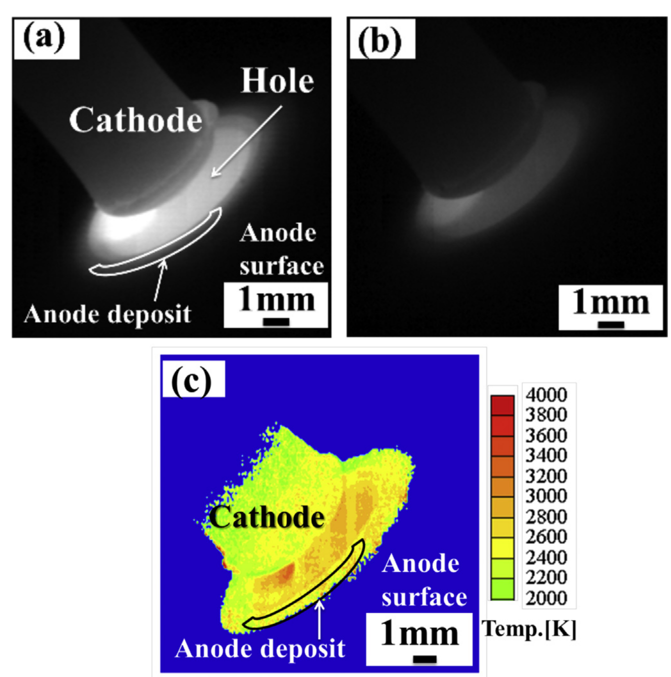

Fig. 3. Images of the electrode at the wavelength of (a) 880 ± 5 nm and (b) 763 ± 3 nm recorded by the high-speed camera at the arc discharge condition of 200 A for arc current and 1 mm for electrode gap distance, and (c) a corresponding temperature distribution map calculated by Plank's law based on the radiation intensity from images (a) and (b).