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# Nanotube deposition in a continuous arc reactor for varying arc gap and substrate temperature

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**Abstract**

A new continuous method for producing mounted carbon nanotubes (CNT) has been developed using the arc discharge method, in which a woven carbon substrate is used as a carbon source. In the process, carbon nanotubes grow on the fibres of the carbon substrate during the arc discharge. The method used differs from the conventional arc discharge method in that it deposits on the anode using low current (less than 20 A), with inter-electrode gaps of more than 5 mm and is run at atmospheric pressure, so that the substrate can be continuously fed and recovered.

The aim of this work was to study the effect of the physical parameters of the arc on substrate surface temperature and on the CNT growth there. The effects of arc gap and buffer gas flow through the anodic substrate were investigated. An optical pyrometric technique was used to determine the substrate surface temperature. It was found that carbon nanotube growth was favoured over the temperature range 3600–3700 ± 50 K and not favoured at higher temperatures of 3800–4000 ± 50 K. This indicates that CNT growth is unlikely to be due to vaporization/condensation of small molecular carbon species.


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*Keywords:* Arc discharge; Continuous method; Nanotube; Surface temperature

## 1. Introduction

In 1978 Abrahamson et al. reported finding nanoscale hollow fibrils or tubular fibers on anodes and cathodes after an arc discharge in nitrogen [1]. However, in 1991, Iijima published a paper which brought such "nanotubes" (CNT) to the world's attention, and researchers began to realize the future potential of the products [2]. The best methods presently available to produce high quality CNT are based on the electrical arc and on laser ablation [3]. However, in their batch mode, neither of these techniques are scalable to fulfil industrial needs for high quality CNT applications (e.g. in the composite materials industry), and this has been a bottleneck in nanotube research and development.

A modified version of the conventional arc apparatus was developed to improve the carbon nanotubes synthesis process [4]. The method invented enables continuous production of carbon nanotubes. In this method, a carbon tape (carbon substrate) was fed between the electrodes, touching the anode support. The carbon nanotubes are grown on the surface of this carbon tape as it is passed through the arc.

This article reports on the novel approach developed for measuring the surface temperature. To understand the growth mechanism of CNTs in the arc discharge process, it is crucial to study the effect of physical parameters such as arc gap and buffer gas flow on the anode surface temperature (in this case the carbon substrate acts as an anode). Nanotubes are grown in a very small distance from the anode surface (less than an electron mean free path) and within the estimated anode fall region. The temperature and composition of the body of the arc may be misleading

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when assessing the growth conditions. In contrast, the condition of the anode surface and species colliding with it should be important, and should be strongly affected by the surface temperature. In recent studies, temperature measurements have been made only of the arc between the two electrodes [5–7]. No one appears to have measured the surface temperature of the anode or cathode in an arc producing nanotubes at either of these electrode surfaces. This deficiency is apparently due to the narrow inter-electrode gap (<1 mm) in the conventional arc discharge, making the electrode surface difficult to see. In this work, however, a frontal view of the anode was able to be viewed and studied due to a larger than usual gap (2–8 mm) between the electrodes.

## 2. Experimental set-up

### 2.1. Materials and equipment

Woven carbon tapes (Carbonics UVIS-3/2-22, specific weight 770 g/m$^2$ and purity 99.9%) were used most often as the anode substrate on which to grow the CNT. The tapes used were approximately 30 mm wide, 2 mm thick and generally 2 m long (cut from a wider roll). Two graphite rods (Union Carbide graphite SPK, >99% pure) with diameters of 12 and 3 mm were used as anode support and cathode, respectively. The anode support was built from the rod hollowed to a tube containing at its end a 6 mm diameter porous carbon disc which rubbed against the moving substrate. This is shown in Fig. 1(a). The anode support was mounted on a hollow stainless steel tube that was connected to a buffer gas supply and to the 120 V DC 20 A power supply. Nitrogen (British Oxygen Corp., zero grade, purity > 99.998%) was used as a buffer gas which flowed through the tape from behind via the porous disc with a flow rate ranging from 0 to 0.6 L/min. Adjustment of a ballast resistor set the current, and the peak-to-peak AC component was limited to 0.4 A.

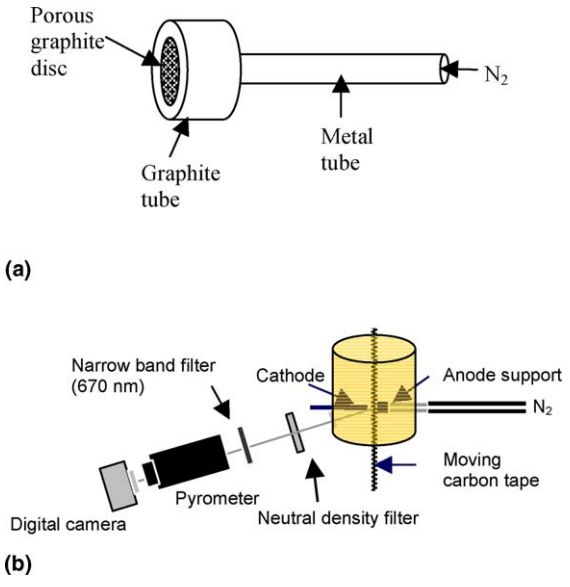


Fig. 1. Schematic diagram of apparatus for surface temperature measurement. (a) Anode support and (b) side view of the experimental set-up.

### 2.2. Experimental

The carbon tape substrate was passed between the two electrodes, touching the positive contact so that the substrate functioned as the anode surface. Thus we call the positive contact an "anode support". CNT were then grown on the surface of the carbon substrate where the arc discharge contacted the tape. During the process, the cathode slowly sublimed. Its position was manually adjusted from outside the chamber to maintain a constant arc gap. The plasma was observed via a window of the chamber. A magnified image of the plasma and the electrodes was projected onto a screen using a lens; a grid on the screen was used as a scale. This projected image allowed easy manual adjustment of the distance between the cathode and the substrate.

The reactor was operated at atmospheric pressure using the DC power supply (with a current of 16 A and 70 V voltage for a gap of 5 mm). The inter-electrode gap was varied over the range of 2–7.7 mm but no larger because at higher inter-electrode gaps the arc became unstable. As noted above, the flow of the anode buffer gas was set in the range 0–0.6 L/min. A larger flow (10 L/min) of the same gas was introduced into the arc reactor away from the arc, to flush the arc environment. The speed of the tape was maintained at 3 mm/s. Two replications were done for each condition to observe the repeatability of the process.

The surface temperature of the substrate was measured as follows. A Sony digital camera (DSC-W1—5 Mpixels CCD sensor) and an optical pyrometer (OPTIX Ox 4001) were used to capture the image of the substrate including a built-in reference intensity. The set-up is shown in Fig. 1(b). The pyrometer operates with light filtered by its own filter to pass red at an average wavelength of 660 nm. As normally used it detects a surface temperature by viewing the surface through an adjustable filter, and the user compares the surface with a dot of calibrated intensity (the dot intensity remaining constant for all observations). Rotation of the graduated filter until the dot and background intensities are equal gives a temperature reading on a scale attached to the filter. However, the temperature range for this pyrometer was only 2073–3273 K. In order to extend the temperature range, first a neutral-density filter (Roland ND1.0, 10% transmission) and then a narrow-band filter (Ealing electro-optics wavelength 670 nm, band width 665–675 nm) were placed alternatively in front of the pyrometer while observing a constant high intensity radiant source (the anode of a "standard carbon arc" [8]). Noting the temperature reading on the scale for each filter in place and using Planck's law for the red wavelength, allowed the second filter to be calibrated in place. By using both the filters together, a new temperature range