[Materials]

Applied Technologies for Carbon Nanostructure

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Abstract

Carbon nanostructures represented by a Carbon Nanotube (CNT) have outstanding features, such as a high aspect ratio, high thermal conductivity, and high electrical conductivity. These materials are expected to be applied in a variety of industrial fields like a display and electron source.

We applied these carbon nanostructures to emitters (electron sources) for cold cathode X-ray tubes. The cold cathode X-ray tube we developed is compact and can be easily mass produced. It is suitable for portable X-ray sources to be applied to security, medical care, and diagnosis of aged infrastructures. The emitter is a core component for cold cathode X-ray tubes. We are working on developing unique emitters

1 Preface

There is a recent growing demand for compact portable X-ray sources in the fields of security, medicine, and diagnosis of aged infrastructures. Our newly developed cold cathode X-ray tube has a construction design that is suitable for mass production and for application to compact portable X-ray sources(1). For the material of the emitter (source of electrons), the core component of the cold cathode X-ray tube, a carbon nanostructure is used. We are working on the development of our unique emitters. This paper introduces our application technology of the carbon nanostructures for cold cathode X-ray tubes.

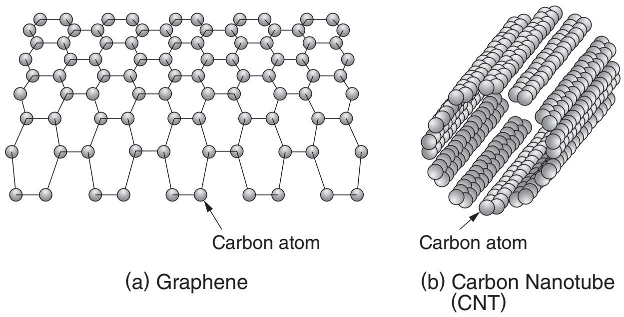


Fig. 1 Example of Carbon Nanostructure

Carbon nanostructure is a microstructure where carbon atoms are geometrically joined. Graphene in (a) has a two-dimensional structure and CNT in (b) comes in a tubular structure where a graphene sheet is rolled up. There are two types: a single-wall and a multi-wall.

2 Carbon Nanostructure

Fig. 1 shows an example of the carbon nanostructure. It comes in a microstructure where carbon atoms are bonded in a hexagonal network. The carbon nanostructure has various features such as a high aspect ratio, high thermal conductivity, and high electrical conductivity. This material is expected to be applied to a variety of industrial products such as a display and electron source.

It is a well-known fact that electrons are discharged from a solid metal to the vacuum space by a tunnel effect when a specific amount of energy, like an electric field, is given to a solid metal under the vacuum. The electron source is a component for which the aforementioned phenomenon is utilized.

In particular, the electron source working under the operating principle of electron emission by electric field is called an "emitter." The emitter is generally continuously exposed to a strong electric field or impact of residual gas ions. Consequently, its material requires mechanical and physical robustness. In addition, such a material is required to have a high melting point in order to withstand the heat generation attributable to high current density. As a material that satisfies these rigorous requirements, the carbon nanostructure material is regarded as one of the most suitable(3). The CNT is one of the carbon nanostructures has a diameter ranging from several nm to 100 nm, a length of more than µm, and a high aspect ratio. One of its features is that the electric field tends to be concentrated at its tip, and therefore, has a suitable structure for an emitter.

The surface structure of the emitter is an essential element that influences the performance of electron emission. **Fig. 2** shows the relationship between parallel CNT intervals and current density(4). Patterns (a) to (c) show the schematics of CNT intervals and electric field contour lines. Pattern (d) shows a curve indicating a change in current density caused by the effect of CNT intervals. When the same electric field is applied to the respective cases, the concentration of electric field to the CNT tip is blocked if the CNT interval is narrow. As a result, current density is lowered. On the contrary, if the CNT interval is wide, the concentration of electric field to the CNT tip is intensified and the current density in the CNT becomes high. When the current density is high, vaporization of the CNT is accelerated and the lifetime may be shortened. For this reason, the emitter is required to have an optimal surface structure in terms of CNT interval and CNT height.

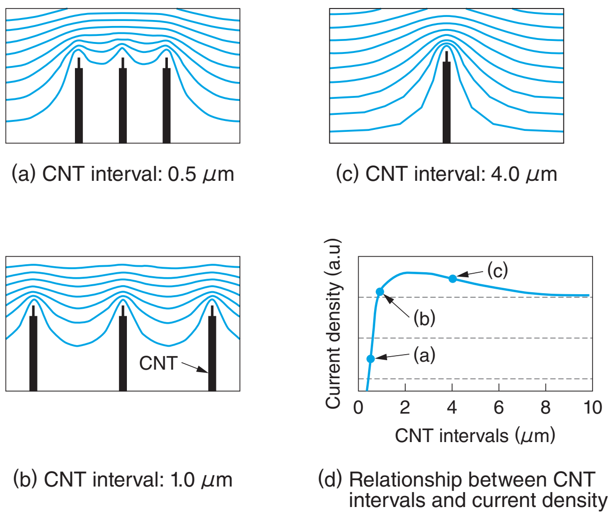


Fig. 2 **Relationship between Parallel CNT Intervals and Current Density**

Patterns (a) to (c): When the same electric field is applied to the respective cases, a concentration of electric field to the CNT tip is blocked if the CNT interval is narrow. As a result, the current density is lowered. On the contrary, if the CNT interval is wide, a concentration of electric field to the CNT tip is intensified and current density in the CNT becomes high.

3 Cold Cathode X-Ray Tube

Fig. 3 shows an external appearance of the cold cathode X-ray tube. The cold cathode X-ray tube has a construction where the target metal (anode) and the emitter (cathode) are arranged at both ends of the insulated vessel inside maintained under vacuum. **Fig. 4** shows the outline diagram of the cold cathode X-ray tube. When a high voltage is applied to the section between the cathode and the anode, electrons are accelerated toward the target metal by the high voltage and collides with this metal. At that time, an X-ray is generated. Since the emitter does not require any heating during the generation of electrons, an X-ray tube provided with an emitter is called a “cold cathode X-ray tube.” On the other hand, an X-ray tube provided with an electron source that requires heating during the generation of electrons is called a “hot cathode X-ray tube.”

Fig. 3 **Cold Cathode X-Ray Tube**

An external appearance of the cold cathode X-ray tube is shown. Specifications are 150 kV – 2 mA. Total length is 95 mm.

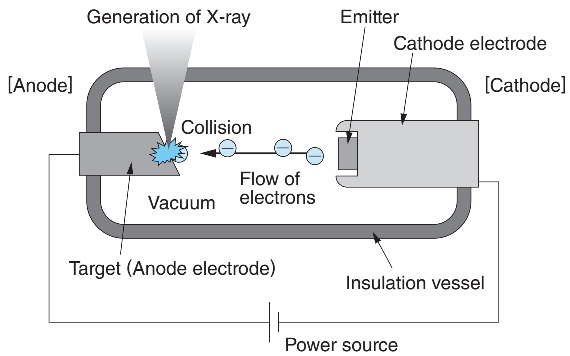


Fig. 4 **Outline Diagram of the Cold Cathode X-Ray Tube**

An X-ray is generated by electrons emitted from the emitter. Since heating is not required for the emission of electrons, this type of device is called a “cold cathode X-ray tube.” Since heating is not required, no power source for heating nor cooling system is required.

Compared with the hot cathode X-ray tube, the cold cathode X-ray tube offers some superiority such as low power consumption, compactness, fast response speed, and high electron density. The cold cathode X-ray tube, however, is assembled through a process of brazing and the emitter is required to withstand exposure to high temperatures. In addition, the manufacturing processes are complicated and not suitable for mass production. To solve these problems, we developed a cold cathode X-ray tube suitable for mass production.

4 Emitter

The emitter, using a CNT, is currently being developed and offers the following features:

(1) The surface structure is made suitable for the concentration of electrons.

(2) This emitter is suitable for incorporating the cold cathode X-ray tube and withstands hard manufacturing processes.

(3) It assures productivity that does not harm the feasibility of the mass production process of the cold cathode X-ray tubes we developed.

Fig. 5 shows the emitter manufacturing method under development. Fine asperity is fabricated on the surface of Silicon (Si) substrates by etching to be used as a CNT mold to form up an emitter surface structure. Since the condition of this asperity determines the surface structure of the emitters, it is an essential factor for emitter production. In the subsequent process, film forming is conducted on the Si substrate with asperity in the order of Aluminum (Al) and iron (Fe). These metals function as a catalyst for the growth of the CNT. Using hydrocarbon gas as a feedstock, thermal Chemical Vapor Deposition (CVD)※1 is applied to the Fe catalyst film during the process of CNT growth. Subsequently, silver (Ag) and Copper (Cu) films are formed on the as-grown CNT. These Ag・Cu films braze the CNT to a Cu pedestal which works as a built-in base for incorporation into the cold cathode X-ray tube. A Cu pedestal is placed on the Ag・Cu film and brazing is carried out. Since high-temperature process is performed during the production of emitters, each emitter can withstand a high temperature during the brazing process to incorporate into the X-ray tube. After brazing, the emitter is completed by peeling the CNT with Cu pedestal from the Si substrate. In the case of this manufacturing process, the time for forming each material film and the time for growing the CNT are very short. In addition, the production volume of emitters can be increased easily if the size of Si substrate is increased.

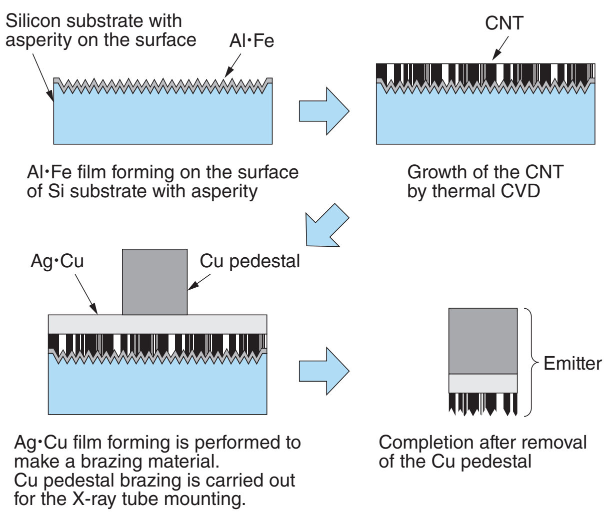


Fig. 5 **Emitter Manufacturing Method under Development**

A method of emitter production under development with the use of the CNT is shown. This method of production features a short manufacturing time and the number of products can be easily increased.

We manufactured emitters by this method and confirmed the CNT structure and emitter characteristics. Firstly, analysis was carried out by the Raman spectroscopy. **Fig. 6** shows the result of analysis by Raman spectroscopy. The G-band peak is particular to graphene or CNT and the D-band peak is mainly attributable to structural defect. Since disruption of the G-band peak peculiar to the single-wall CNT cannot be observed, it was found that a multi-wall CNT grew on the emitter. Since the single-wall CNT is sharp, it efficiently concentrates the electric field and hence, is suitable for electron emission at a lower voltage. On the other hand, the

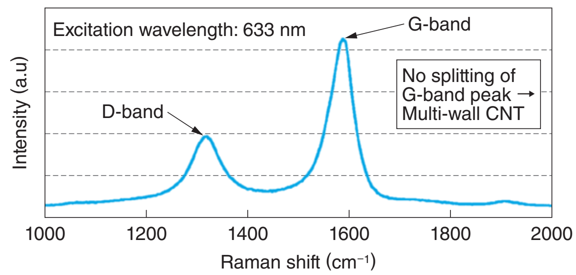
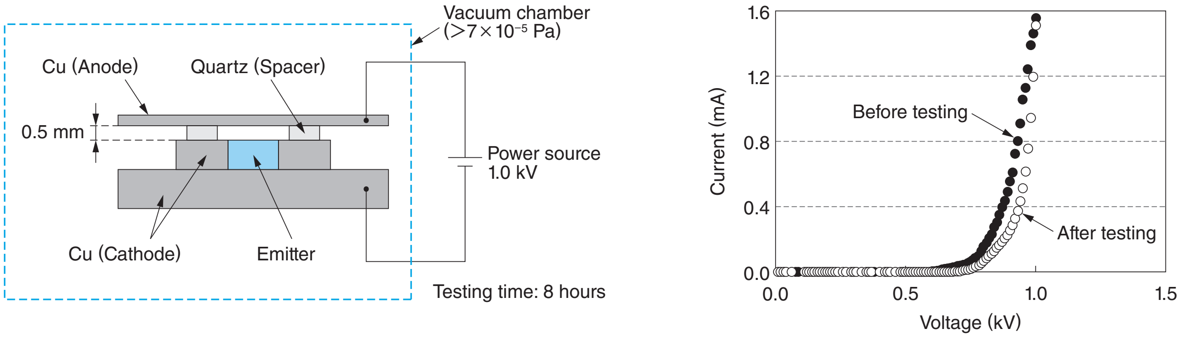


Fig. 6 **Result of Analysis by Raman Spectroscopy**

The G-band is a peak that is singular to graphene or the CNT. The D-band is a peak mainly attributable to structural defect. Since no G-band peak splitting unique to a single-wall CNT can be seen, growth of a multi-wall CNT is predicted on the emitter.



(a) Test system

(b) Test result

Fig. 7 Outline of Continuous Electron Emission Test

The distance between the cathode and anode is 0.5 mm. Voltage 1.0 kVdc was applied for 8 hours. Even a high voltage was applied for 8 hours, but the current value at 1.0 kVdc barely changed. An increase in current in a high-voltage range can be predicted by finding out a state of exponential function in the characteristic curve.

multi-wall CNT has high structural stability and current is carried through multiple walls, thus it offers outstanding long-life characteristics.

Fig. 7 shows an outline of a continuous electron emission test. In order to examine how the continuous emission of electrons by the manufactured emitter influences the electron emission characteristics, a test was carried out with a test system shown in (a). The testing conditions comprised of the cathode to anode distance was 0.5 mm and a DC voltage of 1.0 kV was applied for 8 hours. The obtained current – voltage characteristics before and after the test are shown in (b). Even though a high voltage was applied for 8 hours, the current value at 1.0 kVdc scarcely changed. An increase in current in a high-voltage range can be predicted by finding out a sharp gradient in the characteristic curve. This is due to some protruded CNTs with a relatively smaller diameter where an electric field tends to be concentrated, are removed by vaporization and as a result, electrons begin to be emitted from the entire surface of the emitter. There is still an individual difference in the current – voltage characteristics after testing. Our current challenge going forward is to reduce such individual differences.

Meanwhile, we succeeded in the X-ray photography of a mobile phone using a cold cathode X-ray tube with our emitter. The result shows a clear image of detailed wiring inside the mobile phone.

5 Postscript

As one of the applied technologies of the carbon nanostructures, this paper introduced an emitter to be used for cold cathode X-ray tubes. We will continue to develop related technologies to improve the feasibility of mass production of emitters and improve performance characteristics.

These R&D activities have been maintained as part of a joint research program with Waseda University, Tokyo. We would like to express our deepest gratitude to the program-related people at Waseda University: Professor Suguru Noda, Assistant Professor Hisashi Sugime, and Ms. Sae Kitagawa for their valuable advice and guidance.

(Note)

※1. Thermal CVD: As one of CVD methods relating to film forming techniques, this approach is realized under the control of chemical reaction by heat.

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