X-ray generation using carbon-nanofiber-based flexible field emitters

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Carbon nanofibers were grown on flexible polyimide substrates using an ion-beam sputtering technique. Field emission measurement showed a fairly low threshold voltage of 1.5 V/μm with a current density of 1 μA/cm^2. The field enhancement factor was determined to be 4400. The emitter showed resilience when exploited as a high voltage electron source for x-ray generation. The x-ray generated by the flexible emitter is capable of delivering fine images of biological samples with superior sharpness, resolution, and contrast. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2182022]

Carbon nanostructures such as carbon nanotube (CNT) and carbon nanofiber (CNF) have been very promising for field emission (FE) electron sources owing to their high aspect ratio and small tip radius. While the potential applications of these materials appears to be limited only by imagination, those better known include cold cathodes for FE displays and other vacuum microelectronic devices.[1,2] Various articles have documented the excellent field emission properties of these nanostructures.[3] Apart from these low voltage applications, CNT and CNF have also found applications in high voltage devices such as an electron source for x-ray generation.[4–6] In this letter, we investigated the field emission performance of CNFs grown on flexible polyimide substrates and demonstrated its ability to be used as a high voltage electron source for x-ray production. The advantages of the flexible substrate, among many, are lower cost of producing the emitters and also it allows emitters of any geometry and shapes to be made. The ability to grow CNFs on low cost flexible substrates can open up numerous possible fields of applications such as flexible FE displays and even x-ray radiotherapy and radiography.

The technique used to produce the CNFs on the flexible substrate has been described in detail elsewhere[7–9] except in this case the substrate used for the deposition was commercial polyimide films, measuring 25×25 mm^2 and approximately 100 μm in thickness. The sources of carbon needed to grow the CNF were a thin film of carbon first deposited on the substrates before ion beam bombardment. These samples were bombarded with Ar$^+$ ions using a Kaufman-type ion gun (Iontech. Inc. Ltd., model MPS 3000 FC). The incidence angle of the ion beam was set at 45° from the normal to the surface. This is because oblique Ar$^+$ bombardment is more suitable for ion-induced CNF growth than sputtering at normal incidence.[9] The diameter of the ion beam was 6 cm while the energy of the beam employed was 1 kV. The process was carried out at room temperature for 60 min. The x-ray tube used for this experiment was similar to that re-

ported in our early publication.[10] The experimental system is an open type x-ray tube with a beryllium window directing the x ray in a vertically upward direction. The location of the window is at 7 cm away from the target. The x-ray tube housing consists of a stainless steel structure connected directly to a turbomolecular pump. A high voltage feedthrough was used to allow electrical connection to the flexible emitter cathode, which was connected to a pulse voltage generator. The pulse power supply was operated at 600 Hz with output voltage ranging from 10 to 25 kV. The anode used in this experiment was a copper block connected to an electrical ground.

Figure 1(a) shows the scanning electron microscope (SEM) planar view of the CNF flexible emitters. The CNFs appear conical in shape and angled. The CNFs are densely distributed on the substrates with a number density of around 4.6×10^7 cm^{-2} as estimated from the micrograph. The length of the CNF ranged between 0.5 and 1 μm while the diameter is estimated to be between 0.2 and 0.5 μm. Carbon is the only element detected by energy-dispersive x-ray spectroscopy. From Fig. 1(b), it can be observed that the tips of the CNFs are capped with an ultrafine long carbon nanowire. The diameter of the capping nanowire is approximately 10 nm while the length of these nanowires is about 1 μm. The presence of these ultrafine nanowires capping the CNFs could account for the excellent field emission properties observed. Figure 1(c) shows a transmission electron microscope (TEM) micrograph of the tip of the CNF, which measured approximately 15 nm. The CNF is amorphous in nature as the electron diffraction pattern only showed diffused rings (not shown).

Field emission was carried out at a base pressure of 10^{-6} Torr using a polytetrafluoroethylene spacer of 100 μm to maintain a constant spacing. Figure 2 shows the j (A/cm^2) vsrsus E (V/μm) plot of the CNF coated flexible emitter. The inset shows a Fowler–Nordheim (FN) plot [i.e., ln(j/E^2) versus 1/E] to determine the field enhancement factor, β, assuming a work function of 5 eV. As evident from the inset, the current increases with the applied voltage in an exponential fashion in line with the FN equation. The emitter exhib-
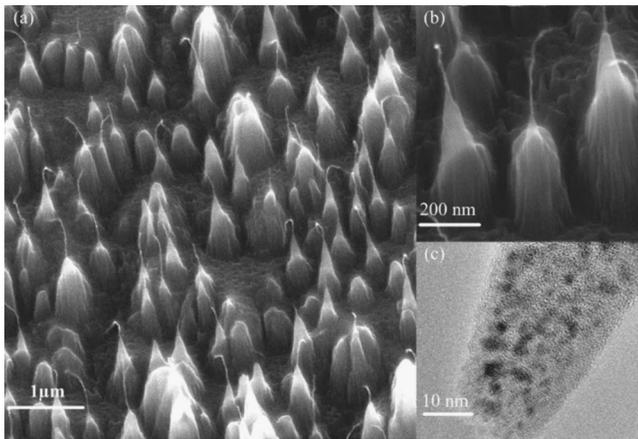
FIG. 1. Electron micrograph of the ion-beam sputter-induced grown CNF: (a) SEM micrograph of the CNFs showing dense fibers estimated to be $\sim 4.5 \times 10^7$ cm^{-2}; (b) the enlargement of the tip region showing ultrafine carbon nanowire capping; and (c) TEM micrograph of the CNF tip.

ited an initial threshold field of about 1.5 V/μm with a current density of 0.1 μA/cm^2, which is comparable to CNTs on Si and CNFs on a graphite plate.[9,10] The value of β thus obtained was about 4400. Figure 3 is a photograph showing the mockup arrangement of the flexible emitter and the copper anode when inside the x-ray tube. The surface of the copper anode was angled approximately 45° to assist x-ray emission. The radius of the emitter curl was approximately 3 mm. Figures 4(a) and 4(b) show the x-ray images of a *Bougainvillea* flower obtained from the flexible emitter exposed for about 45 s using 13 kV. From the images, it can be clearly seen that the flexible x-ray source produces a high quality picture with high resolution on the biological sample.
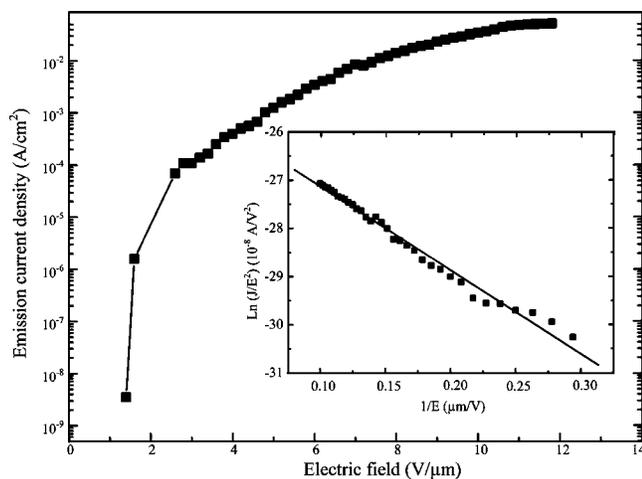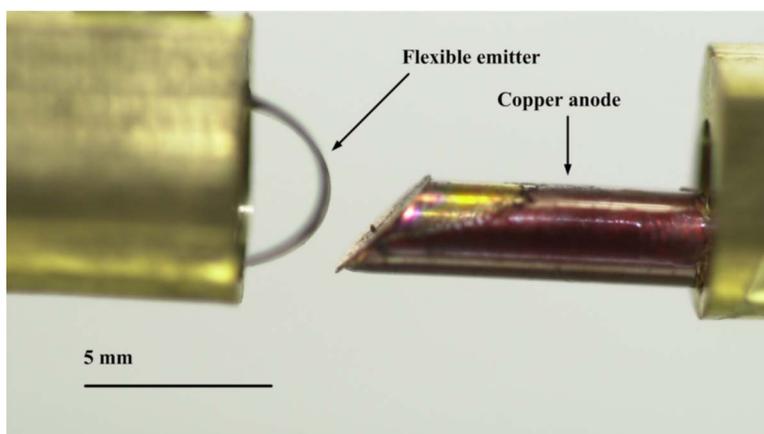
The flexible emitter x ray was able to resolve the fine details in the flower as enlarged in Fig. 4(b), in addition to the veins on the petals as indicated by the two white arrows in Fig. 4(a). The thickness of the vein was $\sim$250 μm. This shows that the flexible emitter is capable of emitting sufficient current to generate the x-ray flux necessary for a high signal to noise ratio even at such a low voltage.

Figure 5 shows the stability measurement of the flexible x-ray source operating at 16 kV as measured by a dosimeter (Ludlum 2241). From the graph, it can be seen that the fluctuation of the x-ray output was low. This suggests that the flexible polyimide based CNFs can have excellent durability for use as high power electron emitter sources. Although the stability is highly dependent on the emission stability of the pulsed power generator, the graph is a good demonstration that the emitter did not degrade even under such a high voltage and current. The same emitter has been tested for x-ray generation for several months and showed no sign of failure. There are no obvious differences in terms of x-ray stability and image quality of a biological sample when the CNFs flexible emitter is used as compared to a CNT emitter (on a tungsten tip)[10] under identical x-ray generation conditions.

This work has demonstrated the viability of using a flexible emitter as an electron source for x-ray application. There could be many possible applications of the flexible emitter, one such potential would be in radiotherapy. Most miniature x-ray tubes for radiotherapy applications used a thin layer of target material coated on the x-ray window.[11,12] In this transmission mode arrangement, x-ray efficiency is severely lowered as radiation is required to transverse through a thick layer of target material before hitting the targeted organ. The

FIG. 2. Field emission measurement of the CNF-based flexible emitter.

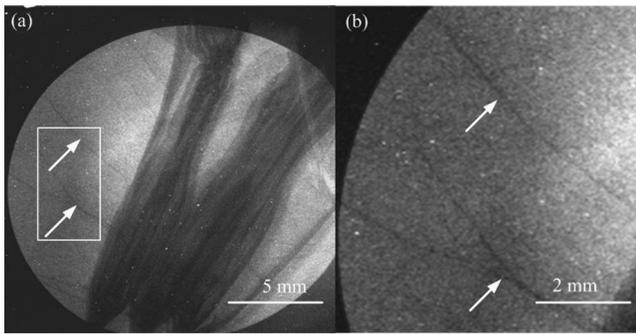FIG. 3. (Color online) Photograph of the x-ray cathode–anode.

FIG. 4. Radiograph of Bougainvillea flower. (a) The flower was imaged at 13 kV using the flexible x-ray source, showing clearly good contrast and resolution. The two arrows indicated the trace of the flower veins on the petals. (b) An enlargement of the stigma area as indicated by the white square, showing clearly the details captured.

use of beryllium as the window material is not favorable as beryllium is harmful to human beings. Using a flexible emitter would allow us to circumvent these limitations and improve the efficiency of the x ray emitted from the system. As polymers generally have a lower absorption coefficient of the x ray as compared to metals, they made an excellent candidate for this application.

As shown in Fig. 3, the emitter demonstrates sufficient flexibility for it to be curled into a radius of $\sim$3 mm while
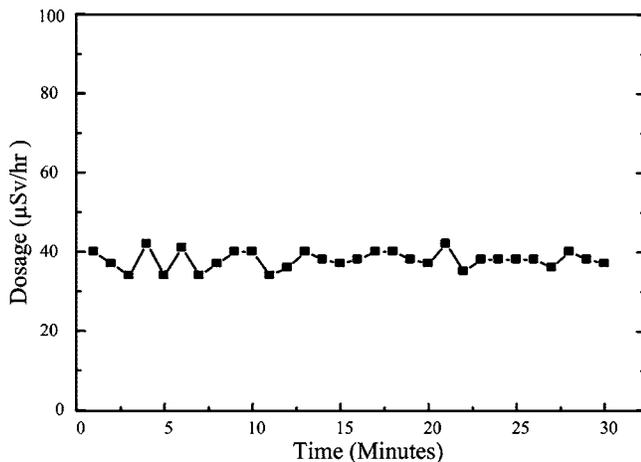
still retaining its field emission ability. The use of such a flexible emitter would allow for greater choices of x-ray tube design previously not possible in medical radiotherapy. It would be possible to make an x-ray transparent emitter as the window with a concave target surface to achieve a focused x ray, thereby reducing the spot size of the x-ray spot. This would allow for more precise radiotherapy treatment rather than the usual diverging x-ray source.

In summary, we have demonstrated an x-ray source using a flexible emitter based on CNFs. The x ray generated at the voltages of 13 keV was sufficient to produce the radiograph image of a *Bougainvillea* with superior sharpness, resolution, and contrast. The flexible emitter has the potential to be applied in x-ray radiotherapy and radiography applications.

FIG. 5. X-ray stability as a function of time.

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