Carbon nanotube field emitters

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Abstract:

In this review we give an overview of the present status of research on carbon nanotube (CNT) field emitters and their applications. The field emission is introduced and an overview is given of the measured emission properties. We give also examples of field–emission devices with CNT electron emitters that have been investigated so far. The most promising applications of CNT field emitters are the field–emission display and high–resolution electron–beam instruments. But several issues remain that still need to be studied in academic research.

Keywords:

Field emission , CNT film emitter, CNT point emitter.

Contents

1. Introduction

From the beginning of its discovery, the carbon nanotube has been regarded as an ideal material to make field emitters because of its unusually high aspect ratio as well as high chemical stability, large thermal conductivity, flexibility and high current [1].

It was realized in late 1990s that materials other than carbon cannot be used as stable field emitters. Carbon nanotubes are the most promising materials for use in field emission displays (FEDs) and high resolution electron beam instruments [2].

2. Field emission theory

Field emission is emission of electrons induced by an electrostatic field. The most common emission is field emission from a solid surface into vacuum. We will give a brief introduction to a common field emission theory.

2.1. Fowler-Nordheim Theory

Fowler-Nordheim is a semiclassical theory in which the system of the field emitter is considered to be a one dimensional structure along the direction of the external field. In this model the emission tip is a semi-infinite quantum well with the work function of Ø, and the local electric field (F) is approximated as a linear potential (Figure 1).

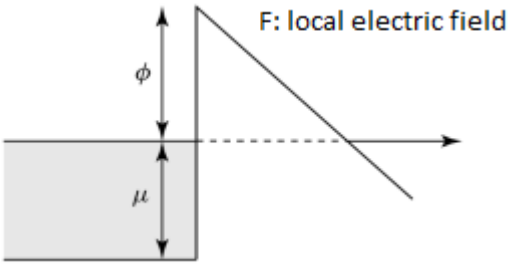

Fig. 1. The simplified model in the Fowler-Nordheim theory. The dashed line shows the tunneling region [3].

The simplified FN equation is as follow:

$$\log\left(\frac{J}{V^2}\right) = a - \frac{b}{V} \qquad (1)$$

Where J is in amperes per square centimeter of the surface and V is the applied bias voltage which is proportional to the local electric field F. The emission tip is geometrically sharp and the electric field is intensified at the tip end which produces a higher local electric field than the macroscopic applied field. The ratio between applied and local electric fields is called field enhancement ratio (β). For the nanotubes, β ranges between hundreds and thousands [4].

The above FN equation assumes a sharp interface between surface and vacuum. But in reality, the electron clouds from the metal do not terminate that sharp. Furthermore the escaping electrons from the emitter feel the image potential which is exerted from the electrons in metal (V_{im}) and can be regarded as a constant in most applications. In the classical expression, $V_{im}= -e^2/4x$, where x is the distance from the tip surface.

When the radius of the emitter tip is of the order of nanometers, the nonplanar shape of the tip should be taken into account. The image potential depends on the tip geometry so it would be a deviation from linear FN behavior mentioned above. The result is shown in [5].

2.2. The Field Enhancement Effect and the Aspect Ratio of CNTs

As noted in previous section, the geometrical shape of the tip intensifies the electric field felt by electron which is called field enhancement. The shape of the distribution of the electrical field potential in the vicinity of a sharp tip is shown in figure 2. This distribution has been calculated by solving of the Laplace equation for a region exposed to the applied potential [6,7].

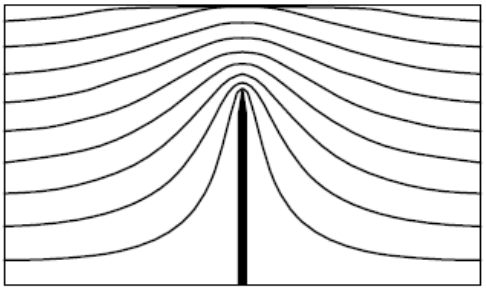

Fig. 2. The typical spatial distribution of the electrical field potential in the vicinity of a
sharp tip [7].

A geometrical parameter determining the field enhancement factor of a single nanotube is its aspect ratio $\alpha = h/d$, where h and d are the height and diameter of the nanotube, respectively. There is a linear relationship between the field enhancement factor and the aspect ratio:

$$\beta \approx h/d \quad (2)$$

Since the aspect ratio for CNTs may reach 1000 or more, the field emission from nanotubes may take place at a much lower applied voltage than conventional field emitters.

3. preparation of CNT emitters

Carbon nanotubes may be used as electron emitters in two forms: one is pointed electron source, which is suitable for electron microscopes to form a finely focused electron beam. The superior mechanical properties and unique geometry make individual carbon nanotubes ideal for scanning probe microscopy tips. Atomic force microscopy (AFM) with carbon nanotube tips allows us to image relatively deep features of the sample surface at nanometer resolution. CNT emitters are also applicable in electron beam lithography, micro X-ray sources and so on [8,9].

The other form of CNT emitters consists of a CNT film or a patterned film, which is used in many areas such as display devices, light sources, high power vacuum microwave amplifiers, industrial and clinical e-beam apparatus for surface treatment, and so on.

3.1. CNT film emitters

Several methods for preparing CNT films have been reported. Spray coating, screen printing, electrophoresis and CVD are of these methods. In spray coating a CNT suspension is sprayed onto a substrate with a conventional air brush [10]. In screen printing CNTs are mixed with binders and surfactants to prepare pastes. After printing, CNTs are heated at 450-500˚C in air to vaporize the organic binders and to form electrical and mechanical contact between CNTs and the metal electrode on the substrate. A surface treatment such as laser irradiation is needed to activate the film surfaces to expose CNT tips from the surface [11].

The first reported electrophoresis method to prepare CNT films comes as follows. First CNTs produced by methods like arc discharge were ultrasonically dispersed in isopropyl alcohol (IPA), then the suspension was droped on coplanar aluminum electrodes with a gap of 0.4 mm on a glass substrate, and an AC was applied until the IPA evaporated completely at room temperature [12].

CVD method is based on decomposition of a hydrocarbon gas over a catalytic metal to grow CNTs. Catalytic CVD allows to fabricate patterns of CNTs by preparing patterned catalyst layer and growing CNTs on the pattern. This method is suitable to prepare patterned CNT emitters which are incorporated in FEDs or electron beam lithography systems. Here we give an example of a FED using CNT field emission source reported by Samsung Display Innovation (SDI), a division of

Samsung Corporation [13]. The schematic of this device is shown in figure. 3.

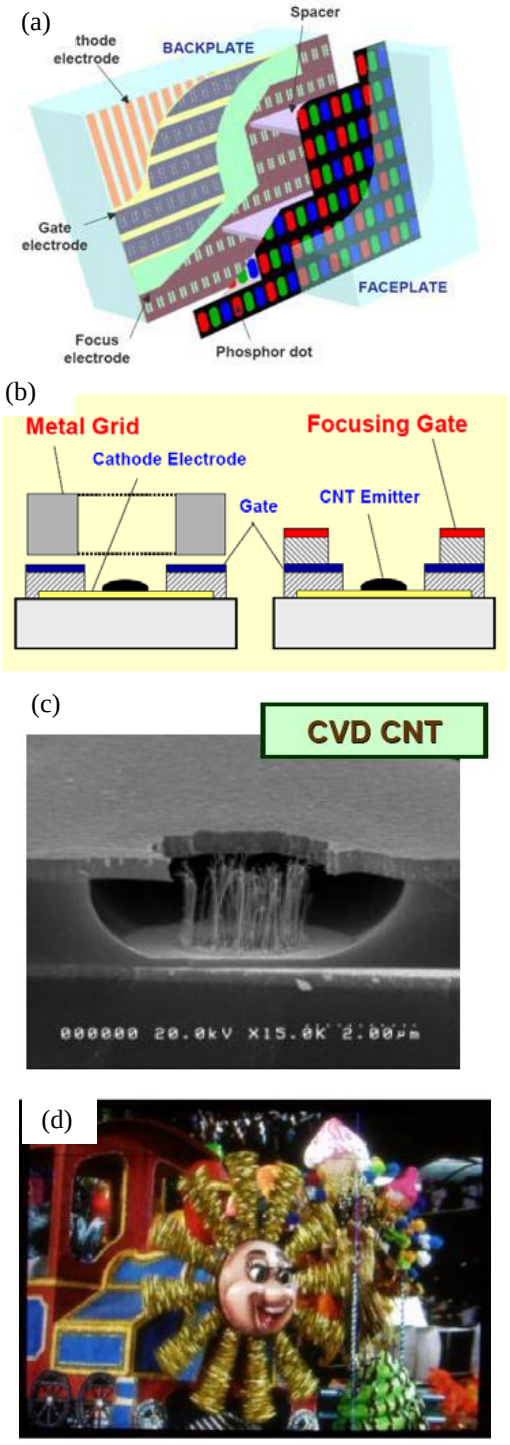

Fig. 3. CNT field emission display (a,b) schematics of display structure and triode pixel architecture; (c) SEM image of individual pixel with aligned CNTs grown directly on substrate, d) image of prototype 15" diagonal display [13].

The Samsung CNT FED is micro-fabricated on display glass and features a triode structure, where a bias voltage is applied between the bottom (cathode, ITO) and middle (gate) electrodes, and the emission is focused by the top electrode (Figure 3.b). Each pixel contains many CNT emitters. The CNT emitters are applied to the device by screen-printing of a custom-made CNT paste, or by direct growth of CNTs by CVD. In the former case, commercially available CNTs are mixed with many ingredients, and the paste is wiped through a screen mask that is placed over the device [14]. In the latter case, a catalyst layer is deposited on the device, and the CNTs are subsequently grown by thermal CVD of CO/H_2 . For screen printing of the paste, the maximum (and therefore limiting) process temperature is that of firing the paste after printing (<450°C); for CNT growth, the CVD temperature is limiting (lowest 420°C so far; must be <500°C to prevent melting of the glass). While isolated vertically aligned CNTs as grown by the CVD process are generally desired for field emission, the CNTs in the paste are generally tangled and parallel to the substrate. Therefore, CNTs as-deposited by the paste method are tipped upward by applying and peeling a tape from the CNT substrate surface, or they are raised under applied electric field [15].

Due to its scalability, the paste method is preferred and is being pursued for current prototype development. Many types of CNTs (e.g., single wall carbon nanotubes (SWCNTs), multi-wall carbon nanotubes (MWCNTs), double wall carbon

nanotubes (DWCNTs) with various diameters and lengths), have been evaluated by this method. It is believed that SWCNTs generally exhibit a higher field emission density, yet lower lifetime, while MWCNTs exhibit lower emission density, yet longer lifetime. Therefore, a careful tradeoff must be made to optimize device performance. Currently, uniform-length, high-quality, small-diameter MWCNTs are preferred (MWCNTs are also easier to disperse than SWCNTs).

A main challenge related to use of CNTs in the display is the uniformity of emission. This is improved by adding a resistive layer under the CNT paste and may be further managed by adjusting the voltages over the array to give uniform emission. Also, as the display size increases, maintaining good vacuum sealing is an increasing challenge, and inadequate vacuum decreases the lifetime as the emitters decay more quickly.

3.2. CNT point emmiters

There have been many reports on point-typed CNT field emitters, which are desirable to be applied to micro-focus X-ray sources and microwave amplifiers. Point-typed CNT field emitters typically have a 1D shape which concentrates the electric field at the tip of a field emitter effectively [16-20]. Furthermore, to achieve high-resolution in electron microscopes and other electron beam instrument, it is helpful to start with an electron source that is small, optically bright and stable. This makes CNTs a promising candidate to be used as electron-gun field emission source in high resolution electron microscopes.

Various methods have been employed to prepare CNT point emitters. CNTs are either mounted or directly grown on the apex of a supporting substrate which is electrochemically etched needle of a metal, hairpin-shaped filaments of a refractory metal, and so on.

3.2.1. Manual attachment of a CNT bundle

The easiest method is to glue a thread of CNTs on tip of a supporting substrate on which a tiny amount of conductive adhesive is applied [21]. Since this type of emitter is a bundle consisting of numerous CNTs, many individual and bundled CNTs come out from a bunch of CNTs. In an emission experiment, only few CNTs emit electrons because due to the shielding effect the electric field strong enough for electron tunneling appears only on a small amount of CNT tips.

To obtain very thin bundle of MWNTs The gluing procedure can be done under an optical microscope using dark field illumination and typical magnifications of 600-1250 [22,23]. However, due to the low resolution of optical microscope, there is not sufficient control over the length and diameter of the nanotube sample.

3.2.2. Mounting inside an SEM

In an improved preparation method, a single CNT can be mounted on a support material in a scanning electron microscope (SEM) in which two

independent piezo-driven nanomanipulators are located. First aligned or bundled CNTs and a support material are mounted on the stages of manipulators [24,25]. When a single CNT projecting out of the bundle is attached to the sidewall of supporting tip, the nanotube stuck to the substrate surface by van der waals force. In order to fix the CNT firmly, a metal or amorphous carbon is spotted at a few points along the contact between CNT and substrate by using electron beam-induced deposition. A typical example of MWNT emitters prepared by this method is shown in figure 4.

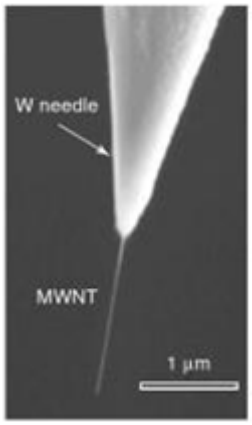

Fig. 4. SEM image of a single MWNT mounted by nanomanipulation inside an SEM.

In another attempt to produce CNT point emitters, a nanotube was selected from an ensemble of multiwalled CNTs (grown using the arc discharge method [26]. This nanotube was attached to a sharp tungsten tip using the glue of conducting carbon tape. To remove the nanotube from the ensemble, the nanotube was either pulled from the ensemble by retracting the tip, or cut by applying a current of over 30 mA: The latter method was found to cut the CNT at the position where it was relatively thin. This allows for control of the length of the nanotube sticking out from the tungsten tip. Finally, the occurrence of field emission from the liberated nanotube was tested by moving it close to a conductive plane (inside the SEM). Fig. 5 shows transmission electron microscope (TEM) images of a thin and long nanotube. Fig. 6 shows the TEM image of another nanotube sample, were a short and very thin nanotube was chosen [27].

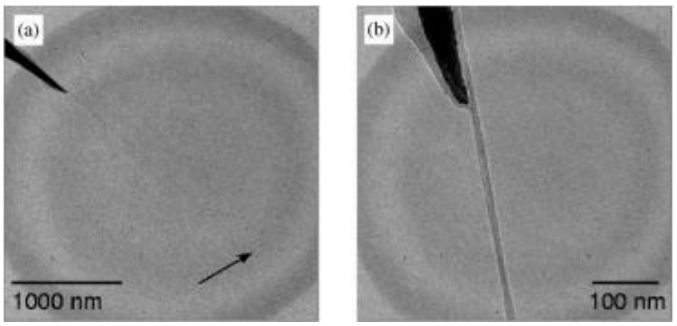

Fig. 5. TEM images of CNT emitters for several magnifications. The full length of the tube is visible in (a). The glue and the tungsten tip are visible in (b) [27].

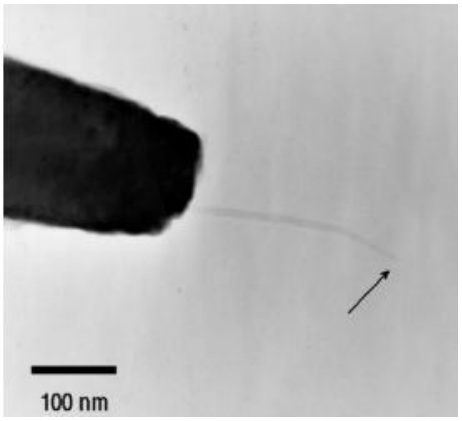

Fig. 6. TEM image of a very thin and short nanotube sample with a tube diameter of 8 nm [27].

3.2.3. Electrophoretic and magnetophortic methods

The procedure of electrophoresis attachment of single CNT or very thin bundle of CNTs on tip of a metal needle (Typically a W needle) by van der waals forces is as follows. First, CNTs are suspended in distilled water by sonication for 10-15 min, followed by placing a droplet containing CNTs on a metal plate (copper) as an electrode. The needle is dipped into the droplet on the electrode. As shown in figure 7, an alternating current (AC) is applied between the needle and the electrode to induce the electrophoresis. Due to the anisotropic properties of CNTs, polarized CNTs are aligned along the electric field and are attached to the W tip where the electric field is strongest [28].

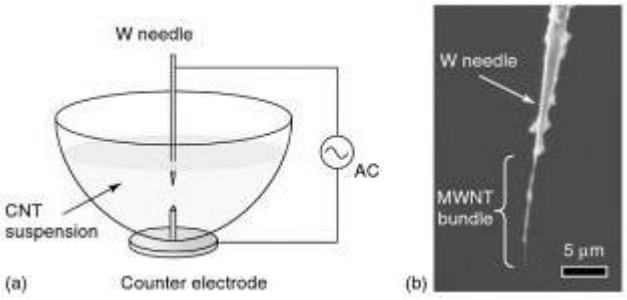

Fig. 7. (a) schematic illustrating electrophoresis to stick oriented CNTs on W tip. (b) SEM image of a thin bundle of MWNTs extending from W tip [28].

CNTs can also be aligned along the tip axis by magnetic field lines. In magnetophoretic method, an alternating magnetic field is applied to the MWNTs suspended in dichloromethane to attach aligned CNTs onto the tip of an atomic force microscope (AFM) has been reported [29].

3.2.4. Direct growth on the apex of a tip

The superior mechanical properties and unique geometry make individual carbon nanotubes ideal for scanning probe microscopy tips. Atomic force microscopy with carbon nanotube tips allows us to image relatively deep features of the sample surface at nanometer resolution.

For growth of CNTs on the AFM tip two processes have been introduced. The first one is pore growth, in which a commercial silicon (Si) tip is first anodized in hydrogen fluoride to create nanopores of 50-100 nm diameter along the tip axis [30]. Then Iron catalyst is deposited into the pores and MWNTs are grown by chemical vapor deposition (CVD) with ethylene and hydrogen at 750 ˚C.

The second method is called surface growth, in which anodization process is omitted and CNTs grow on Si pyramidal surface, guided along the edges toward the tip apex.

One important application of CNT point emitters is to make X-ray emission sources. The application of CNTs as x-ray emission sources has been led by Otto Zhou's group at the University of North Carolina, and is being commercialized by Xintex, Inc. In a CNT-based x-ray source (Figure 8), CNTs are placed in a triode configuration as a field electron emitter; the emitted electrons are incident upon a target (typically Mo) which, in turn, emits x-rays and is aimed toward the sample [31]. Overall, imaging systems using CNT x-ray emitters offer promise over current technology due to their small size, fast response time, and capability for

multiplexing and array imaging. These seek to enable rapid, high-resolution imaging without the need for moving the source and/or detector as is typical in many current x-ray and CT imaging systems. It has been reported that stationary DBT (s-DBT) for imaging human breast cancer, utilizing an array of carbon nanotube (CNT) field emission x-ray sources, provides increased spatial resolution and potentially faster imaging than current DBT systems [32].

Similar to field emission displays, CNT x-ray emitters can be arrayed as "pixels," and imaging is facilitated by the rapid response time of the emitters along with frequency division multiplexing using a single flat detector [31]. CNTs can be integrated in x-ray emitters by direct growth on the emitter substrate, or by post-growth deposition, and therefore, face similar manufacturing challenges as field emission displays. However, displays require further integration with circuit architectures and immediately need finer pixel resolution.

In a prototype device, purified SWCNTs were electrophoretically deposited in a tangled morphology on a metal substrate, which was then incorporated in a sealed emitter tube (Figure 8) [33]. It has been explained that a CNT emitter tube is stable for over 100 hours of operation (36 million cycles), with a constant voltage pulse 10 μs width at 100 Hz. The emitter current exceeds 1 A.

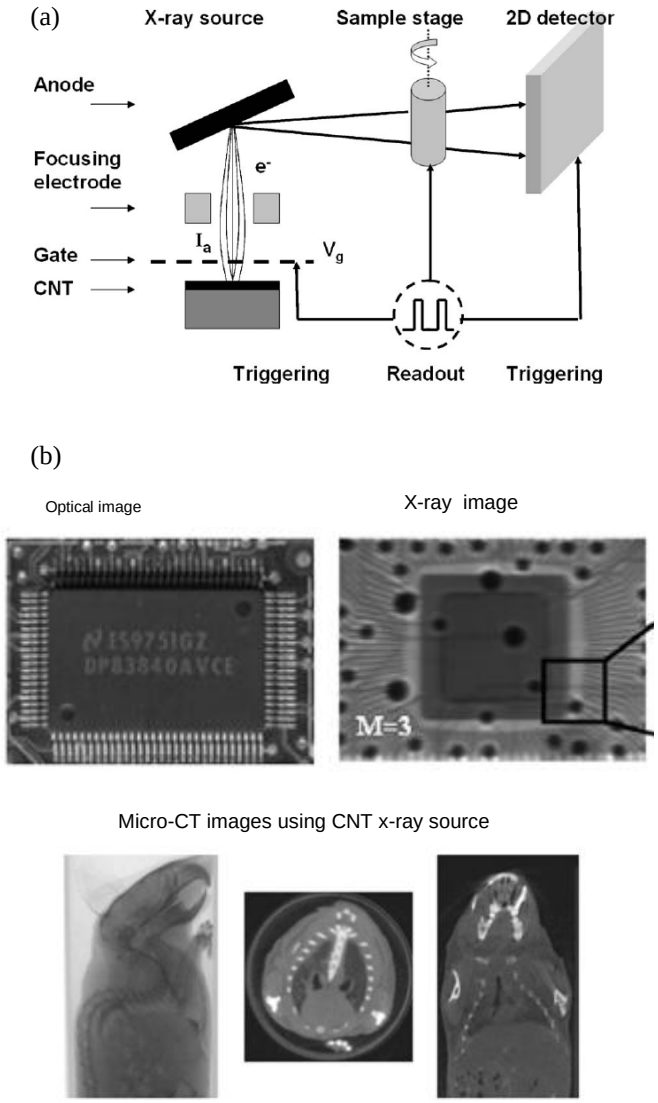

Fig. 8. (a) Schematic of CNT x-ray emission source. (b) x-ray and computed tomography images of a computer chip and a mouse carcass, obtained using CNT-based x-ray source [33].

4. Challenges

Lifetime and stability of the CNT emitters are the key issues to the application of the CNT emitter devices. Point-typed CNT field emitters typically have a 1D shape which concentrates the electric field at the tip of a field emitter effectively [34,35]. However, a 1D shape feels larger stress than a 2D or a 3D shape because stress is defined as force per unit area. This can prevent CNT point emitters from

achieving a stable and high emission current. Thus, reliable and robust point-typed CNT emitters are great challenge until now.

Several researchers have reported the failure and degradation of CNT point emitters by in situ observations inside scanning electron microscope (SEM) or TEM. The reported cases of failure involve an abrupt failure at the contact [36], a gradual peeling [37], shortening [38] or field evaporation [39] process of the wall of the CNT.

3.1. Field emission failure

A direct observation of both abrupt failure and evaporation during field emission inside TEM has been presented by M. S. Wang et al [40]. The CNT field emission tips were prepared inside TEM with a vacuum condition of around 10^{-8} Torr. Two metal tips were used that one of them was a microscopically flat Pt tip which was kept stationary and was used to assemble the CNTs to the tip by rubbing the tip in nanotube powder. The second tip was a sharp W tip connected to a piezotube which was used to select individual CNTs.

First, to prepare the CNT tip they bring W tip to contact with CNT on the Pt tip and apply a voltage ramp. After several I-V sweeps, the resistance of the whole system reduces to tens or several hundred kΩ. The CNT then breaks into two parts by passing through a large current (Figure 9). The W tip is then moved to another place for field emission measurement.

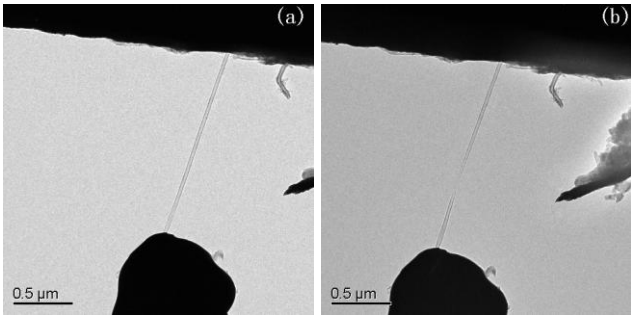

Fig. 9. TEM images showing the CNT (a) before and (b) after breaking into two parts [40].

At a bias of 80 volts the current passing through the CNT reaches several μA and the whole CNT aligns itself with the electric field direction and later the CNT is removed from the W tip (Figure 10). This kind of failure has been attributed to tensile loading of the emitter under the applied electric field.

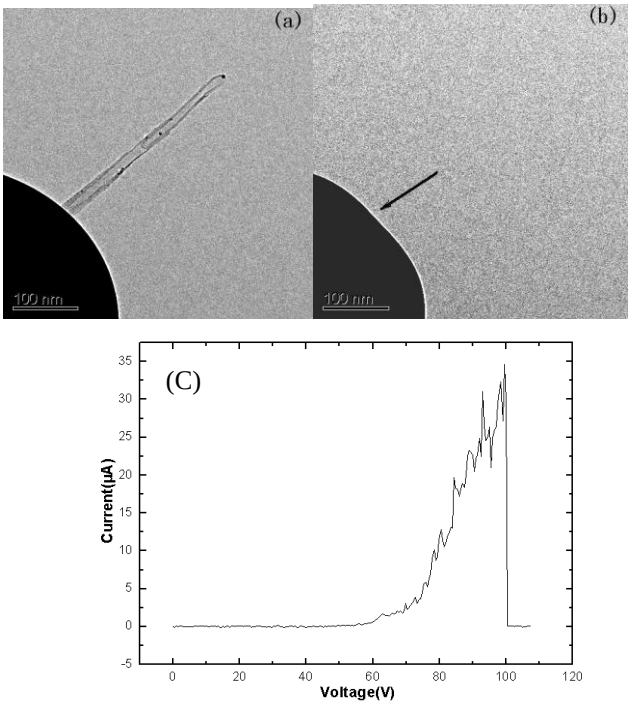

Fig. 10. TEM images showing the CNT (a) before and (b) after field emission failure at an emission current of about 35 μA. (c) the corresponding I-V curve [40].

In more recent works, extremely high field emission currents of 22.4 mA and long term emission stability of 1mA for 20h of CNT point emitters is reported [41,42].

They have prepared 2D shaped CNT point emitters which had been fabricated by formation of a freestanding CNT film into a triangular shape. The height and the thickness of the CNT point emitters were 1 mm and 5 µm, respectively.

Field emission behaviors of the field emitters as a function of their tip angles have been investigated with the gap between anode and emitter set to 1mm. Fig. 9 shows the emission properties of the 2D shaped CNT point emitters with the various tip angles.

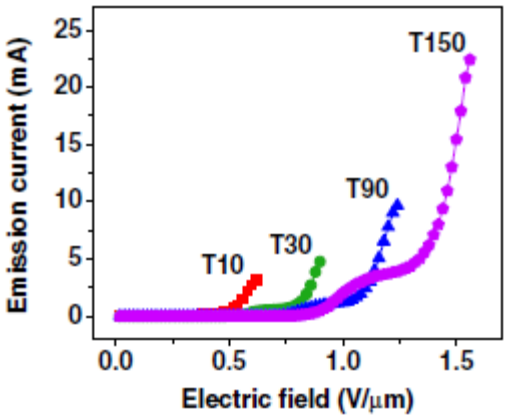

Fig. 11. Field emission J-E curves of the CNT point emitters with the tip angles of 10º and 120º [42].

The CNT point emitter with a sharper tip angle is more effective in concentration of the electric field at the emitter tip compared to the CNT point emitter with a wider tip angle. On the other hand, the emitter tip with a wide angle can tolerate higher mechanical stress during the field emission process, resulting in a higher emission current.

Several groups have reported very high field enhancement factors due to their unique emitter shapes [43,44].

In another attempt to produce point-typed CNT emitters with high emission current and good stability, they have fabricated CNT emitters using a CNT film on a graphite rod with very small area and investigate their field emission properties according to edge polishing process. The CNT emitters with edge polishing indicated a very high emission performance, which presents the high emission current and good long-term emission stability when compared to the CNT emitters without edge polishing. The turn-on electric field of the CNT emitters was 1.56 V/µm at an emission current density of 0.1 µA/cm^2 [45]. Very long term stability of CNT point emitters and higher emission currents are still issues to be solved.

5. Conclusion and outlook

A lot of research has been done in the last decade on carbon nanotube field emitters in order to incorporate them into display devices, x-ray emission sources and electron beam instruments. Despite valuable findings in the academic research in this area and in some cases commercialization of the devices using CNT emitters, there are still plenty of challenges remaining, such as the manufacture of an electron source or an array of electron sources with exactly the desired properties in a reproducible manner.

Furtheremore, new insights into this area could lead to potentially new applications of CNT electron emitters. For example they can be tested as electron emitters to develop the cold field emission gun in high voltage TEM. Due to their high aspect ratio and high field enhancement factor, the field emission will start at much lower extraction voltages.

Further improvement of CNT emitters performances is critical for this field and hopefully industry.

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