# 5 CARBON NANOTUBE ELECTRON SOURCES: IN THE MICROSCOPE

Many measurements of the electron-optical properties of CNTs have been made, but there is little in the literature which shows how the CNT actually operates in an electron microscope. To the author's knowledge, there are currently only three papers that detail field emission from CNTs in a Scanning Electron Microscope (SEM) [1, 2, 3]. All three papers employ CNTs which have been attached to field emission sources with the same method used by de Jonge et al [4] as outlined in chapter 2. The CNTs operate at very high pressures for SEM columns ($10^{-7}$ mBar) and consequently exhibit a huge amount of instability (23-32% applying the definition from chapter 4). However, upon the application of a ballast resistor, the instability was reduced by a factor of ten, which enabled images of a resolution of 30 nm to be taken (as shown in figure 5.1). Thus, these groups can claim to have published the first SEM image with a CNT source as there are no other such pictures in the literature. However, they have not determined the performance of the CNT in the electron column and the first such work will be outlined in this chapter.

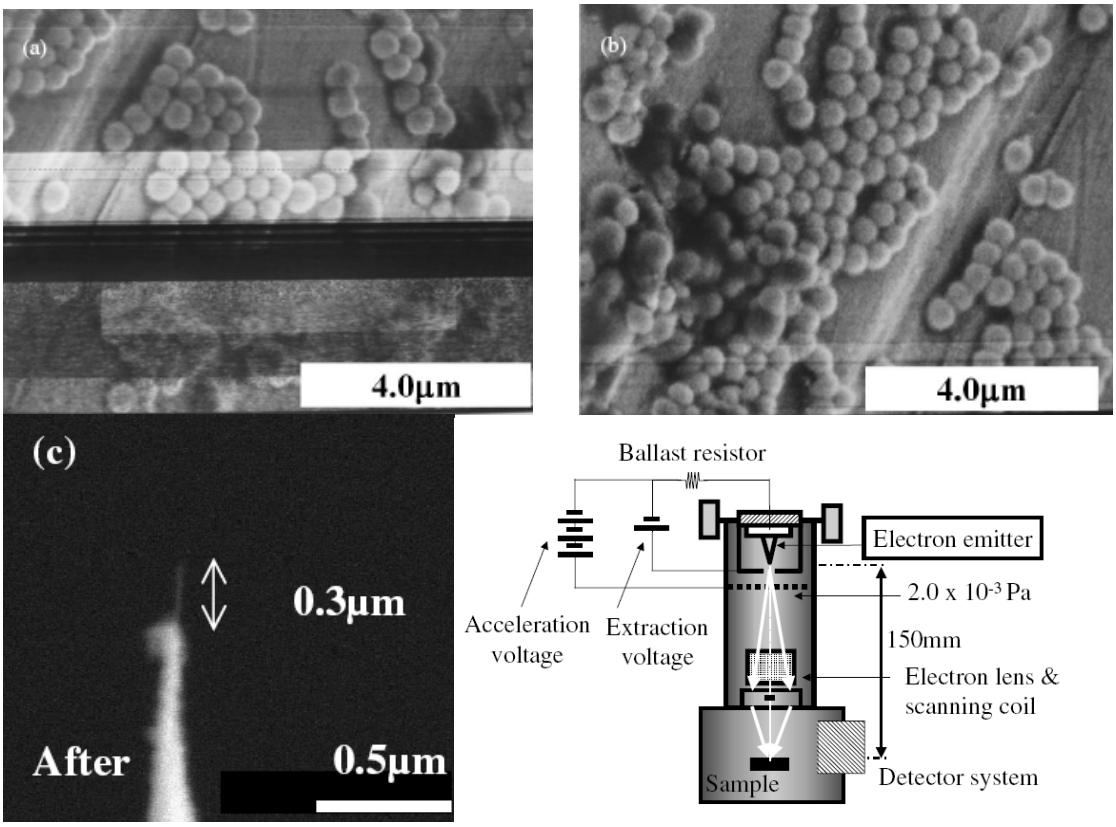


Figure 5.1: (a) shows an SEM image taken by Tetsuo Shimizu [1] with a CNT SEM source operating at $10^{-7}$ mBar without a ballast resistor to reduce instability. (b) shows the same surface with the correction, though banding can still be seen at the bottom of the image. (c) shows the CNT that was used for the field emission experimentation, and (d) gives a schematic representation of the SEM column used.

## 5.1 THE FIELD ELECTRON EMISSION VACUUM SYSTEM

The system used to make SEM images is typically used to characterize Schottky sources, so it again opens up the opportunity to directly compare the CNT electron source with the Schottky. However, it is important to first characterize the source, which was done in a Field Electron Emission Vacuum System (FEM). Figure 5.1.1 shows a schematic of the experimental set-up used in the FEM.

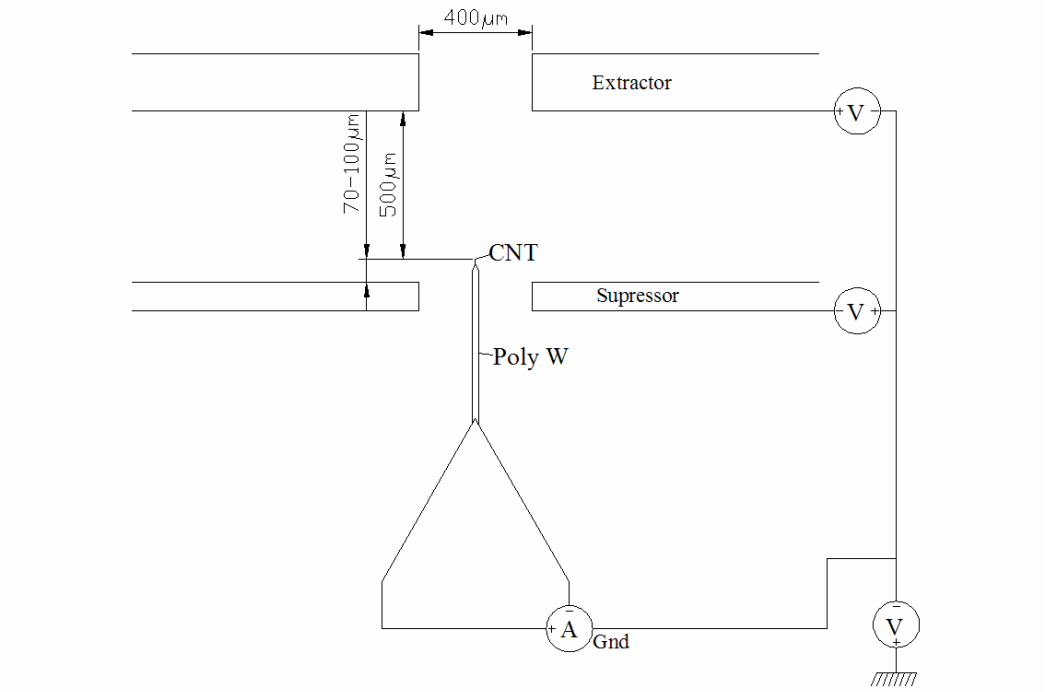


Figure 5.1.1: Schematic of the experimental geometry of the field emission test system.

The CNTs used for imaging were grown by the York method described in chapter 3, where a pre-aligned electron source module, with a readily etched polycrystalline tungsten emitter is placed into a PECVD chamber in its entirety. This, in theory, should increase the degree of alignment, with the optical axis. Following removal of the source from the chamber, the suppressor was replaced with an identical clean one, since CNTs grow all over the suppressor as well as on the tip. The source was then placed into the FEM for characterization.

Figure 5.1.2 shows the source used for the measurements. It is clear that this is a multi-CNT tip. It will be shown later that the field emission comes from one CNT at the tip even though the secondary CNTs extend to a height two thirds the height of the primary.

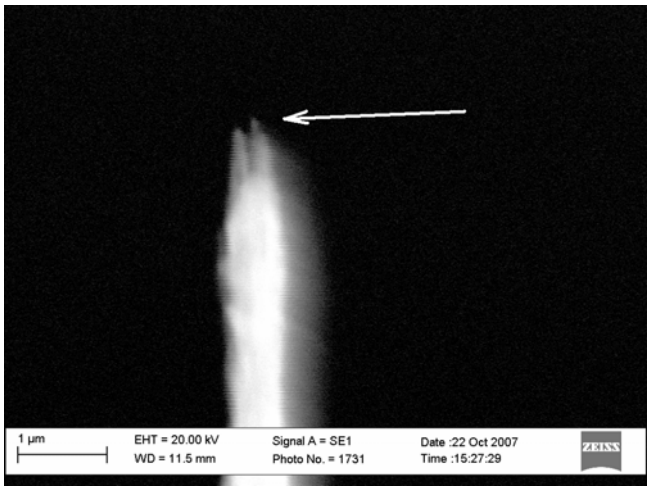


Figure 5.1.2: SEM micrograph of the source used for this study. There appear to be at least five CNTs at the apex. The topmost CNT, indicated by the white arrow, extends significantly higher than the rest. Blurring is due to charging.

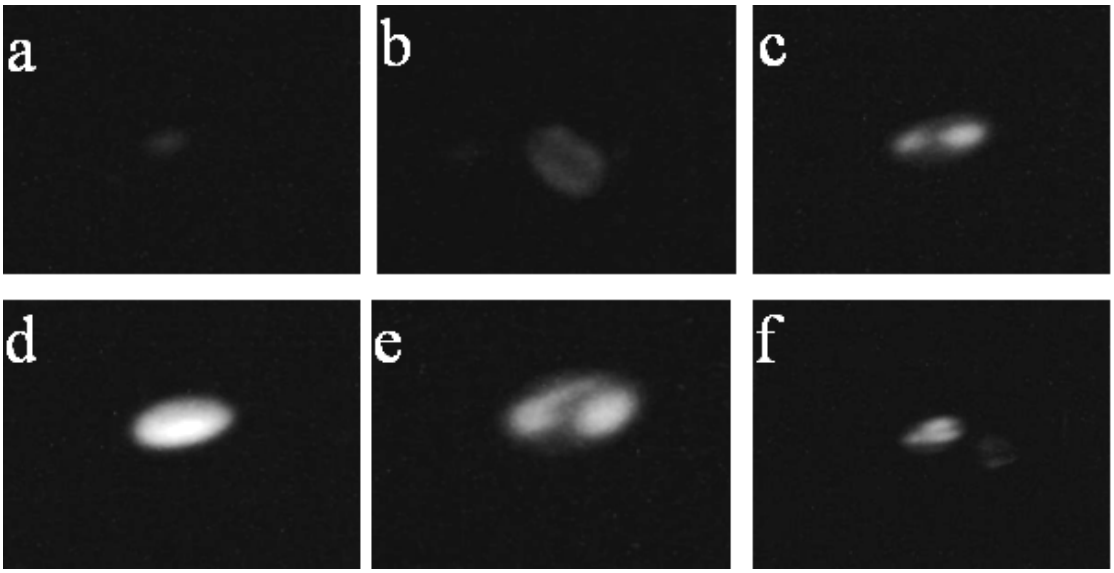


Figure 5.1.3: A sequence of field emission patterns obtained from the CNT electron source in figure C. (a) to (c) show the pattern in early stages of the rapid thermal anneal cycle where the cap structure had not fully formed. Though (d) would make an ideal electron source with a large, bright central spot, this was not a stable configuration; (e) is another intermediate. The most stable pattern was that obtained in (f). The operational conditions used to obtain this pattern were an extractor voltage of 1500 V giving a total current of between 1 and 10 µm.

In the FEM, the operational conditions needed for a stable field emission pattern and field emission current were established. The vacuum conditions were in the ultra high

vacuum (UHV) range of 5-8×$10^{-10}$ mBar. The system was baked for 15 hours at 140 ºC. After baking, the emitter was heated in-situ at temperatures greater than 800 ºC (measured with a pyrometer) as part of the rapid thermal anneal process required to complete the graphitic cap structure required for stable field emission. Whilst the drift stabilized to less than 10%, a series of field emission patterns were collected to illustrate how the tip changed during the process and this is shown in figure 5.1.3.

Because the system used to anneal the tips was different from that used at FEI, and because of the greater expense involved in growing CNTs in electron sources, great care was taken whilst annealing the tips, which explains the length of time it took to achieve this. Slightly too much heat and the CNT vaporizes. Care was also taken to nurse a central on-axis field emission spot. Another aspect of CNT electron sources is that the field emission patterns tend to be concentric about the optical axis. This means that there is not always a spot on the central axis; the alignment can be, in effect, too good. A way of moving the CNT assembly slightly so that one of the stable concentric bright spots can be used for cup current instead should be a consideration for the future. Unfortunately, the central emission spot obtained was quite weak, thus requiring a larger than desired total current of 1-10 µA which meant that the source was quite unstable during operation.

Once the central spot was optimized and stabilized, I-V characteristics were obtained and are shown in figure 5.1.4.

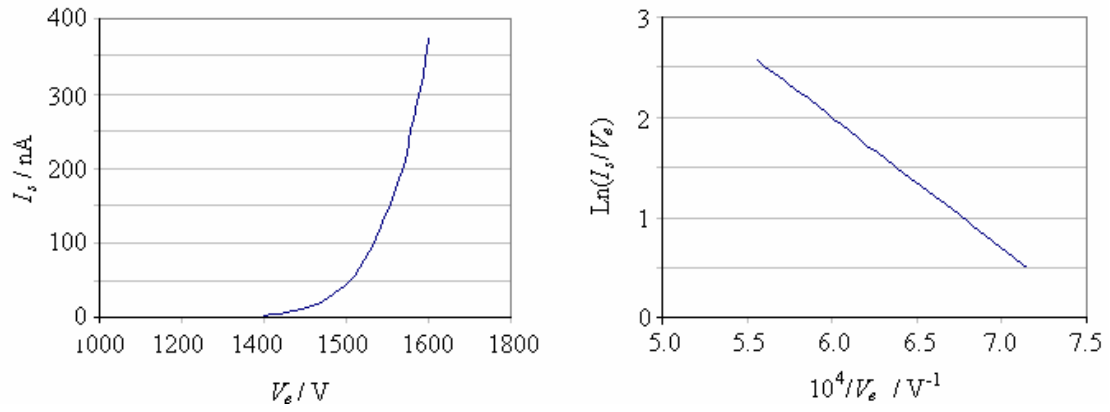


Figure 5.1.4: Left, screen current, $I_s$, as a function of extraction voltage, $V_e$. Re-plotted as a Fowler-Nordheim curve right, the straight line again indicates field emission.

The emitter was cycled in the FEM many times, switched off for several hours, evacuated to atmosphere and then pumped down to pressure again and the same I-V characteristic was observed each time. As outlined in the previous chapter, flashing at 1200 K was required to stabilize the emission current.

## 5.2 IMAGING WITH A CNT ELECTRON SOURCE

Following characterisation of the CNT, the source was transferred to an electron cathode assembly with all the lenses operating electrostatically. This work was carried out at York Probe Sources Ltd. The lens system consisted of three parts, the extractor, a lens electrode and a ground electrode. The electrode can be operated over a range of voltages (100 V to 30 kV). With a similar assembly, it is possible to obtain resolutions of 2 nm in commercial systems, but this microscope testing rig could not achieve such high resolutions because it was designed only to test whether or not a lens system is fit for operation on SEMs. Therefore, only a comparison between a Schottky emitter and a CNT emitter can be made with the same testing rig geometry. An improvement should still be visible, however.

### 5.2.1 SCHOTTKY FIELD EMITTER (SFE)

When a Schottky Field Emitter (SFE) is placed in this arrangement, a typical total emission current would be of the order of 150 μA. This would translate to a specimen current of 150-200 nA with accelerating voltages of between 5 keV and 30 keV. Its energy spread would typically be 0.6-0.7 eV with a brightness level of approximately $5\times10^{8}$ $Acm^{-2}sr^{-1}$. High resolutions are not possible because the rig is not fitted with anti-vibration equipment, but figure 5.2.1 shows an electron micrograph of a thin film deposited on a copper electron microscope grid. The smallest grid bars are 10 μm across.

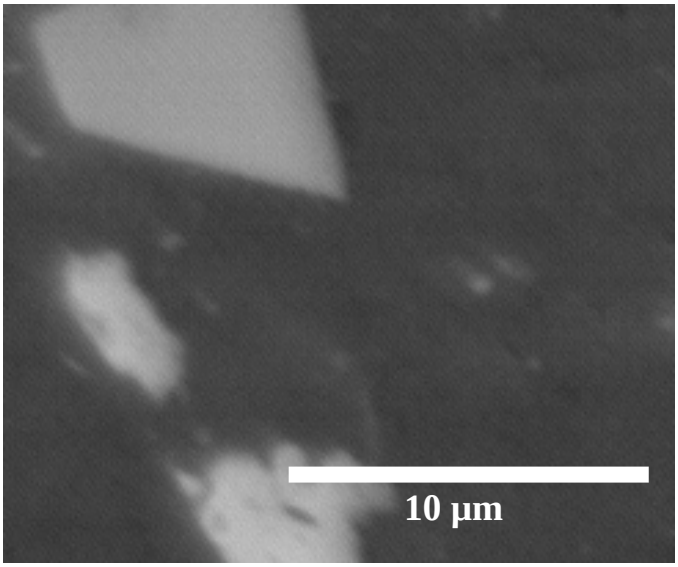


Figure 5.2.1: Scanning electron micrograph of a grid containing various objects with a scalebar included. The beam energy is 7.5 keV at 200 nA beam current.

### 5.2.2 CARBON NANOTUBE EMITTER

When the CNT ensemble was placed in the microscope, the operational parameters, as expected, were different from that of the SFE. A total emission current of 2-5 µA was used, unfortunately in the more unstable regime. The extractor voltage was 1000 V and this resulted in a specimen current of 10 nA. The first scanning electron micrograph with these parameters is shown in figure 5.2.2.1.

It can be seen that the emission current is unsteady which has caused banding in the image. This was undoubtedly due to the high current used to obtain the image. Figure 5.2.2.2 uses the same source to image the artefacts shown in figure 5.2.1 with the SFE micrograph included for comparison. Again, the same horizontal banding can be seen in the image, but the artefacts in the image are actually more clearly resolved.

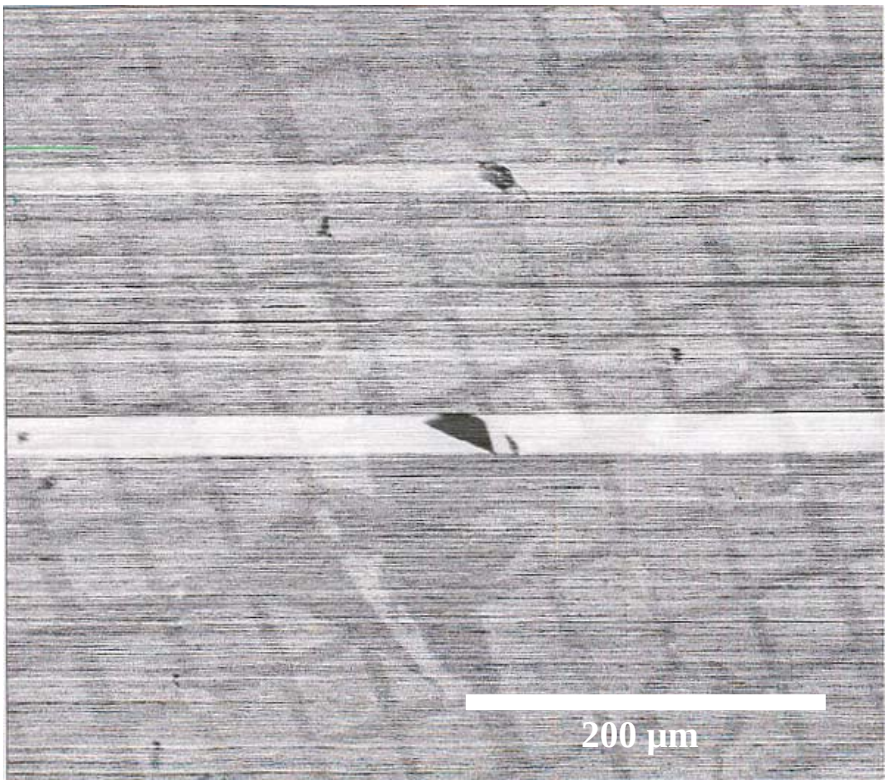


Figure 5.2.2.1: Low magnification scanning electron micrograph of the testing grid inside the electron microscope with the CNT acting as the source. The field of view is 500µm from left to right. This is the first SEM image ever to be made with a CNT grown directly onto the apex of a tungsten tip.

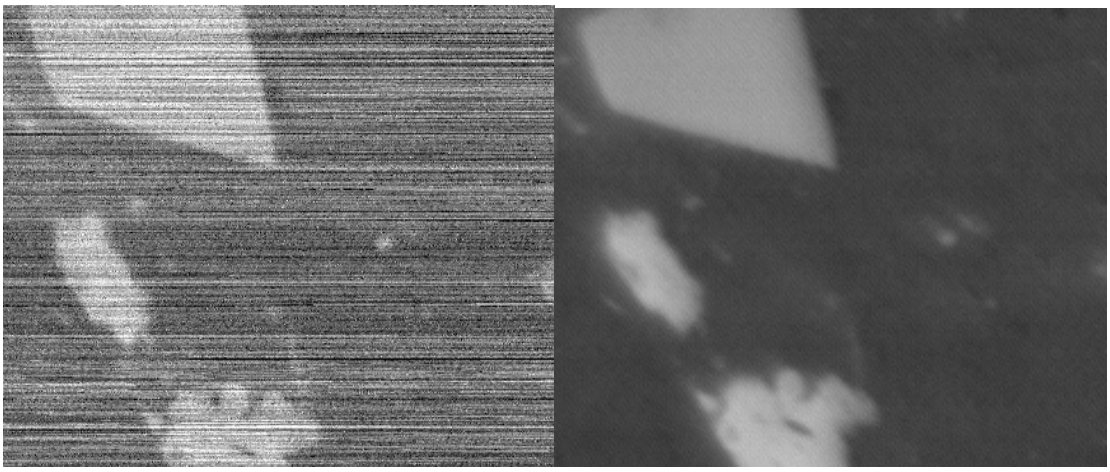

Figure 5.2.2.2: Left, a scanning electron micrograph using a CNT source which can be used to compare the resolution and stability of a typical SFE micrograph shown right. Whilst the CNT image is much noisier, the objects in the image are sharper. Pay particular attention to the improved resolution in the edges of the object with straight sides at the top left of the image. It is otherwise very difficult to see a difference. Figure 5.2.2.3 gives a clearer indication of increased resolution.

It is difficult to see in figure 5.2.2.2 that there is greater resolution with the CNT source. Figure 5.2.2.3 shows a line profile of each image in which the brightness of the object is plotted against displacement along a horizontal line. It is clear that the edges are more discreet with the CNT source because the width of the brightness drop-off at an edge is shorter. In fact, it is more than twice as sharp. This simple comparison indicates a smaller beam diameter and therefore a smaller virtual source size. It can therefore be concluded that the source size is less than 30 nm. De Jonge states that the virtual source size is approximately equal to the CNT radius [5]. Since the radius of the topmost CNT in figure C is 20 nm, increased resolution would fit in with this argument.

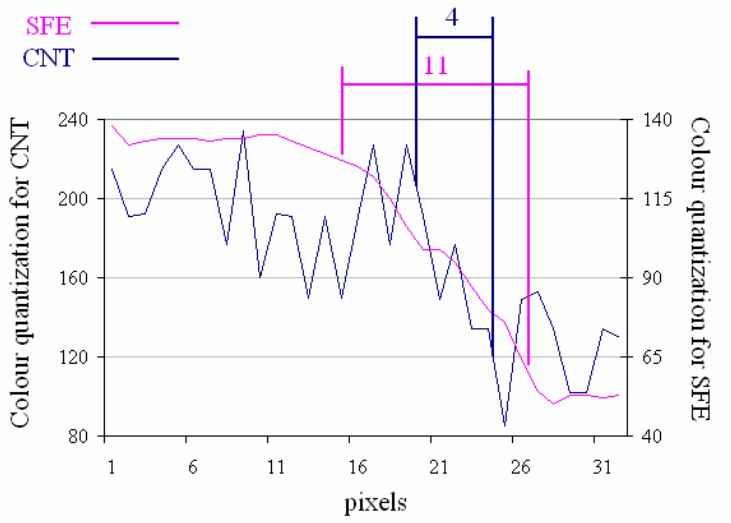


Figure 5.2.2.3: Line profile of objects in the electron micrographs shown in figure 5.2.2.2. Though noiser, the CNT profile is observed to have a much sharper fall-off. The resolution is defined as the distance it takes for the intensity to fall from 90% to 10% of the difference in colour quantization values.

Given that the beam current used was an order of magnitude less than that of the SFE, that the beam kinetic energy used was 5 keV rather than the 7.5 keV used for the SFE and that the beam defining aperture subtended an angle of 27 mrad rather than the 8 mrad used for the SFE, the energy spread must be at least of the order of 0.3 eV, which fits in well with data presented in chapter 4. By geometrical arguments, it can also be concluded that the brightness of the CNT source is a factor of 10 times the brightness of the SFE. This means that the CNT brightness is of the order of $5\times10^{9}$

$Acm^{-2}sr^{-1}$, which is greater than the minimum estimate outlined in the previous chapter.

## 5.3 SUMMARY

Unfortunately, it has not been possible to measure the virtual source or the brightness directly. The system used at Philips Research Laboratories by De Jonge has been dismantled and is now being reassembled at FEI in the US. However, these measurements are based on simple geometrical arguments with appropriate assumptions. To strengthen the case for the CNT source, further experimentation should determine from where the electron beam emanates and to measure a precise value for the source brightness.

## 5.4 REFERENCES

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