A fully sealed luminescent tube based on carbon nanotube field emission

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Abstract

A fully sealed luminescent tube of 40 cm length and 4 cm diameter based on carbon nanotube field emission is demonstrated. The device shows a homogeneous illumination over the whole length and circumference of the tube and reaches the luminance of conventional fluorescent tubes while being mercury-free, continuously dimmable and with a high illuminance capability. The realization has been made possible with the development of a chemical vapor deposition method to grow nanotubes homogeneously on long metallic wires, which provides an additional possibility to control the mean length and density of the emitters. This control has proven to be of utmost importance as it makes possible to adjust the emission voltage and emission site density needed to reach the target intensity and specifications of the device.

Keywords: Chemical vapor deposition method; Nanotubes; Field emission

1. Introduction

Carbon-based materials, and especially carbon nanotubes [1], have been used recently as cold electron emitters [2] in a variety of devices aiming at lighting applications [3–6]. In such applications, the electrons are accelerated towards a layer of phosphorescent material, which converts the kinetic energy of the electrons into visible light. Most of these devices, which are similar in shape to the traditional vacuum tubes, consist in a field emission diode or triode where a film emitter illuminates a flat phosphor layer of ∼20 mm diameter. Such elements are usually designed to be used as pixel elements in giant (outdoor) screen displays, instead of existing solutions based on thermoelectronic emission or on semi-conductor diodes. Several industrial companies have demonstrated that such elements can be realized in the three basic colors with carbon-based materials (e.g. FEPET, a subsidiary of SI Diamond Technologies [3]) or carbon nanotubes (e.g. Ise Electronics Corp. of Mie, Japan). In particular, the high brightness luminescent elements of Ise Electronics Corp. [7] have received the silver medal of the ‘Displays of the Year Awards 2000’ of the Society for Information Display, for ‘the first commercial product that uses field emission from carbon nanotubes’. In these elements, the phosphor-coated glass anode is flat and shows a surface of a few cm², which means that a large number of elements is needed for a practical application. While this constraint is usual in giant displays, it poses a severe drawback for general lighting devices.

Our approach is different and aims specifically at providing an alternative to the usual fluorescent tubes. Fluorescent tubes are based on an electric discharge in a rare gas with the addition of a small amount of mercury. Excitation of the mercury vapor produces UV photons that are converted to visible light by a phosphor layer deposited on the inner surface of the glass tube [8]. The main drawbacks of such tubes are that the light intensity is limited to ∼10,000 cd/m², that their dimming capability is poor, and that they contain mercury, a toxic substance which will be forbidden in some electronic devices in the European Union from 2008 on. An alternative solution to usual fluorescent tubes would therefore have a high market potential, provided that it corrects the drawbacks of the usual tubes while remaining cheap, of simple technology and energetically efficient. We demonstrate here a luminescent tube of 40 cm length based on carbon nanotube field emission that is mercury-free, continuously dimmable and has a high illuminance capability.

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To realize a luminescent tube based on electron emission, the phosphor-coated inner surface of a glass tube has to be bombarded homogeneously with electrons, as schematized in Fig. 1(a). This implies that a conductive layer has to be deposited below or on the phosphor to efficiently remove the electrons, and that the cathode has to be cylindrical, as it is not possible to provide a current density that is constant over the whole circumference of a cylindrical anode with emitters deposited on a flat surface. One solution would be to use a thin wire, where the electrons are extracted either by heating or by field emission [9], but this leads to extremely poor homogeneity. Furthermore, field emitters like Spindt tips and carbon nanotubes are traditionally deposited on flat Si substrates which has prevented up-to-now the realization of such tubular elements. The only possibility is therefore to deposit field emitters on a non-planar substrate.

We have shown recently that carbon nanotubes can be deposited on wires and rods by thermal chemical vapor deposition (CVD) of C2H2 at 720 °C on a variety of materials [10,11]. In thermal CVD, a hydrocarbon gas is injected in a flow tube that crosses the hot zone of a tubular oven. The nanotube growth is activated by a transition metal catalyst which decomposes the C2H2 and promotes the growth of well-graphitized tubular nanostructures, and Fig. 1(b) shows carbon nanotubes grown by this technique on a thin wire. We use Kanthal (a Fe–Al–Cr alloy) as support material, as it allows (after annealing in air at 1000 °C over 12 h) a selective control of the growth by the deposition of a Fe-based catalyst (20 mM of Fe(NO3)3·9H2O in ethanol) that is delivered onto the metallic wire by dipping [11]. This control permits to tune the nanotube density [12] and to grow nanotubes only on precise locations on the cathode. We could also demonstrate with such structures that field emission in the cylindrical geometry of Fig. 1(a) is not only possible, but very efficient [10] (to our knowledge, this was the second demonstration after the one by Millikan and Eyring in 1926 [9]), which makes possible to realize luminescent tubes. Fig. 1(c) shows an element of 10 cm length yielding 10,000 cd/m² at 7.5 kV. The next step after this achievement was of course to realize a fully sealed element with the specifications summarized in Table 1: in particular, the length had to be increased to at least 40 cm.

Table 1

<table>
<thead>
<tr>
<th>Specifications of the cold cathode luminescent tube prototype</th>
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<tr>
<td><strong>Dimensions</strong></td>
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<td>Length</td>
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<td>Diameter</td>
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<td><strong>Power consumption</strong></td>
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<td>Half nominal intensity at</td>
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<td><strong>Power supply</strong></td>
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<td>Input</td>
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Fig. 1. (a) Schematic principle of a luminescent tube based on field emission. (b) Scanning electron micrograph (SEM) of a carbon nanotube film grown directly by CVD on a metallic (Kanthal) wire. (c) Luminescent tube of 10 cm length realized with a cathode similar to the one shown in (b), at 7.5 kV applied voltage and 0.25 mA/cm² emitted current density on the cathode.
2. Up-scaling of the nanotube growth: cold atmosphere CVD

Fig. 1 shows that carbon nanotube field emitters can be grown directly on metallic wires by CVD. The only problem with traditional CVD methods is that the length of the cathode is limited, as the whole support has to be homogeneously heated in order to ensure a homogeneous growth of nanotubes. While this is easily done for lengths below 10 cm, the cost and technical problems become rapidly prohibitive for larger lengths—especially if one wants to replace the usual, 150 cm long fluorescent tubes.

In nearly all embodiments of CVD, both substrate and gas atmosphere are heated, the latter either in the flow tube of an oven (thermal CVD [13,14]), at a 2000 °C hot filament (hot filament CVD [15,16]) or in a DC or rf plasma (plasma-enhanced CVD [17,18]), while the substrate holder is only a secondary source of heat for the reaction atmosphere. To grow nanotubes on long wires, we developed a variation of CVD where the gas is heated only through the support, without the usual external heat source (oven, hot filament, plasma).

As shown in Fig. 2(a), the concept of our experimental setup is to perform the CVD growth and the field emission in the same configuration: the vacuum enclosure formed by the glass tube and the endpieces is used as the CVD reactor for the growth, as well as for the operation of the device once the reaction atmosphere has been evacuated and the required vacuum of $10^{-6}$ mbar has been reached.

As required by the specifications of Table 1, the glass tube has an outer diameter of ~40 mm. As the conductive layer, we use a transparent SnO$_2$F layer deposited by CVD [19] with a resistivity better than 300 $\Omega$ over the whole length of the tube. The phosphor is either a NP1045 from Nichia for white light or a P22 from Osram for high intensity green light, and is deposited on the SnO$_2$:F layer on inside of the glass tube by standard techniques. The Kanthal wire of 0.3 mm diameter is placed in the center of the tube after oxidation in air at 1000 °C for 12 h and the application of the 20 mM Fe(NO$_3$)$_3$·9H$_2$O ethanolic catalyst solution with both ends connected to an electrical circuit for heating.

The heating circuit has to provide a stable current during the whole duration of the growth. The heating current is provided by the secondary loop of a transformer with the primary loop connected to a variable AC source. This allows to have the support at a floating potential with respect to the electrical ground, which is essential if the cathode has to be heated during field emission. It permits furthermore to reach a stable temperature between 650 and 1200 °C as measured with an optical pyrometer. The actual growth of the emitting structures is carried out under a partial pressure of acetylene, and the deposition is stopped by cooling the wire and/or removing the partial pressure of hydrocarbons through the vacuum pumps. We use typically a 75 mbar exposure during 5 min, but nanotubes are also obtained with pressures as low as $10^{-4}$ mbar, which permitted us to follow the growth of nanotubes in real-time for the first time [20,21].

The fact that the only heat source in the reactor is the metallic wire is a very important difference with respect to conventional CVD techniques. The glass tube of radius $r_o$ acts as a heat sink at temperature $T_o$, while the support wire of radius $r_i$ is uniformly heated at a temperature $T_i$. Convection effects are largely absent as temperature and gravity gradients are perpendicular, and the conductivity equation can be solved by taking acetylene as a perfect gas with a thermal conductivity $\lambda$ that varies as $\lambda \propto \sqrt{T}$ [22]. The resulting solution of the differential equation for the temperature is shown in Fig. 2(b) (with $T_i = 950$ °C and $T_o = 20$ °C): the temperature drops very rapidly with the distance to the filament, and is lower than 650 °C at

![Fig. 2. (a) Experimental setup used for the growth of carbon nanotubes on long metallic wires by CACVD. (b) Calculated temperature profile of the gas as a function of the radius. The wire ($r_i = 0.15$ mm) is at 950 °C, the anode ($r_o = 21$ mm) is at 20 °C.](image-url)
The volume of gas heated to the temperature needed for nanotube growth represents only 0.2% of the total volume: we have hence named this method ‘Cold Atmosphere Chemical Vapor Deposition’, or CACVD [23].

We have investigated the influence of the deposition parameters (C$_2$H$_2$ pressure, wire temperature and deposition time) on the film morphology and on the field emission properties. We found that CACVD produces a homogeneous film of well-graphitized nanotubes over the whole length and circumference of the support [22]. Fig. 3 shows scanning electron micrographs (SEM) of the cathode surface as a function of deposition time. Nanotubes are found on the support surface after 1 min of deposition time already. When the time is increased to 5 and 30 min, it appears that the surface density of nanotubes increases (decreasing spacing between the nanotubes), but that the mean length of the grown structures remains approximately constant [24]. It indicates that the growth of the nanotubes proceeds very rapidly, which has been confirmed by direct estimations of the growth rate in a set-up similar to the one of Fig. 2(a) [20]. The pressure has an effect similar to the one displayed in Fig. 3, as an increase of the pressure between 1 and 400 mbar resulted in an increase of the nanotube coverage (not shown here). As for the temperature, we found an increase of the length of the nanotubes with the temperature (see Ref. [22] for the corresponding SEM micrographs), which is in contrast with conventional CVD techniques where an increase of the temperature provokes an increase of the diameter [25]. To our knowledge, this behavior is quite unique to CACVD: as the catalysis reaction is thermally activated, the rate of growth increases with temperature. In usual CVD setups, however, higher temperatures lead to increased dissociation of the hydrocarbon gas in the gas phase and hence to larger tube diameters [25]. In CACVD, only the gas in the immediate vicinity of the catalyst is heated at the reaction temperature, which allows us to control the length of the tubes. This control of the length is very important for field emission applications, as will be shown in Fig. 5.

Fig. 4 shows field emission $I–V$ curves of a CACVD cathode just after deposition: the emission sets in at a voltage of 580 V and the cathode reaches 10 $\mu$A/cm$^2$ at 900 V (for a discussion of field emission in cylindrical geometry and of the very low resulting emission fields, see Refs. [10,26]). It is now well known that the field emission properties of nanotube films are degraded as the emitted current is ramped up [12]. The extraction of increasingly higher currents results invariably in a degradation of
the emission that shows as a shift of the $I-V$ curve towards higher fields and a decrease of the field enhancement. This phenomenon involves some kind of 'training' process where the best but mechanically or electrically fragile emitters are damaged or destroyed. To ensure reproducible and stable characteristics, we systematically operate the emitters slightly above the target current density ($10 \mu A/cm^2$ for these experiments) during at least 30 min. The outcome of this break-in is shown in the inset of Fig. 4: the emitted current at 900 V applied voltage decreases at first and stabilizes then around $7 \mu A/cm^2$. The $I-V$ curve acquired after the break-in is also shown in Fig. 4, and shows an increase of the onset voltage.

We analyze the influence of the deposition parameters on the field emission by considering the voltage necessary to reach a current density of $2 \mu A/cm^2$, as well as the emitter density at an applied voltage of 1000 V as counted directly on the phosphor-covered anode. The latter property is seldom considered in field emission studies, but it is crucial for all lighting and display applications. For the pressure and time dependence, we note in Fig. 5(a) and (b) that the emission voltage decreases and then increases with increasing pressure and time, while the dependence is inverted for the emitter density. We interpret this result as an effect of shielding. The field enhancement factor for a nanotube is essentially given by its length over radius ratio as long as the nanotube is isolated [27]. When several nanotubes are assembled to form an array, the electrostatic screening between the structures decreases the effective field enhancement. While this decrease is not significant for large distances, it becomes quite noticeable as soon as the spacing is less than twice the typical length of the emitters [12,28]. As the emission current varies in a highly non-linear way with the applied field, even small variations in the field enhancement have a huge impact on the emitted current per emitter. Furthermore, the density of emitters increases with decreasing spacing, so that the combination of both effects (decrease of the field enhancement, and hence emitted current per emitter, and increase of the emission site density) results in a maximal current density for an optimal distance between emitters of $1-2$ times their height. In the case of Fig. 5(a) and (b), an increase of pressure or time results in an increase of the nanotube density, i.e. in a decrease of the distance between the nanotubes, which provokes in the first phase a decrease of the voltage when the emitter density increases, followed by an increase of the voltage when shielding becomes significant.

As for the influence of the wire temperature in Fig. 5(c), we note that the voltage and the emission site density decrease with increasing temperature. In this case, we recall that an increase of the temperature results in an increase of the nanotube length, and hence in more efficient cathodes in terms of voltage. As field emission is a very selective process, the emission of the cathode will be dominated by the longest tubes, even if their density is very low. In our case, an increasing length results in fewer emitters and hence in a lower homogeneity.

For our application, the emission voltage is not the critical parameter, and we have chosen the deposition...
conditions in order to maximize the emission site density (75 mbar of C\textsubscript{2}H\textsubscript{2} at 720 °C for 5 min). As our results show that an optimization of the emitted current can be detrimental to the emission site density, and therefore to the homogeneity of the final device, it is indeed more favorable to have a slightly less efficient cathode in terms of emission voltage, but with a more homogeneous emission [22].

CACVD allows therefore to grow high quality nanotube films on non-planar supports over lengths over 40 cm, and we do not see a conceptual limitation to the length of the cathode with this technique. It should be also possible to apply the same concept to other configurations, which could offer a high degree of control of the growth, either by influencing the properties of the nanotubes (spacing [12], diameter [29], length [22]) through the catalyst and the deposition conditions and/or by following directly the growth through field emission measurements [20], along with a simplified experimental setup as compared to existing CVD methods.

3. Design and assembly of the fully sealed element

The next step, after the up-scaling of the nanotube deposition to the length specified in Table 1, was of course to realize an independent device. There are two main problems in realizing this step: the first one is to find and choose suitable means of maintaining the vacuum through so-called getters, and the second one is to devise an assembly procedure that incorporates all the steps necessary for the preparation of the tube, namely the realization of the anode and cathode, followed by the assembly and finally the sealing of the device, while maintaining in a first stage an easy fabrication that can be realized in-house.

Gettering is a technique that is routinely used in the industry, from vacuum tubes to cathode-ray tubes and field emission flat panel displays. It involves the evaporation and/or activation of a material that will trap gas molecules and form chemical complexes to bind them. Most getters appear in the form of evaporable materials that are released from a source through microwave or direct current heating and sputtered on a surface. This is the case for all getters found in the old-days vacuum tubes. In our case, this solution is somewhat impractical for the first stage, and we therefore opted for a more expensive, but more easily implemented technology, the so-called non-evaporable getters. Such getters consist of a porous sintered Zr–V–Fe structure and are activated by heating with an electrical current to temperatures of 500 °C [30].

The realization of a tube necessitates the assembly of different parts, namely the glass tube with the phosphor and conductive layers that form the anode and two endpieces on which the cathode and getters are mounted. Care has also to be taken that cathode, anode and getters can be independently contacted, and that all parts are electrically isolated. This calls for careful design and for a simple, yet robust sealing. Usually, glass-on-glass welding is regarded as the only practical solution, but poses severe constraints to the design and realization of the parts. For the first devices, we choose to use a high vacuum glue, which allows sealing not only of glass-on-glass but also of glass-on-metal. This greatly simplifies the design and makes the assembly easier. We are of course aware that any industrial production of such luminescent tubes will have to rely on glass-to-glass welding and evaporable getters, as well as on classical metal-on-glass electrical feedthroughs instead of metallic rings and endpieces. However, our goal for this first generation prototype is to demonstrate the feasibility of a 40 cm cold cathode luminescent tube based on carbon nanotubes.

A drawing of the luminescent tube is given in Fig. 6. The central part is the same as in Fig. 1(a) and consists in a glass tube with the conductive SnO\textsubscript{2}:F layer and the phosphor layer on the inner surface. On both ends, a conductive annular ring is fixed to the tube and serves as electrical feedthrough for the anode (contact to the conductive and phosphor layers) on one end and the getter activation on the other end. Two 2 cm long glass sections isolate the rings from the metallic endpieces. The left endpiece carries the getter and one end of the cathode; the other end of the cathode is fixed to the right endpiece through a spring that tightens the wire and compensates for length variations during nanotube deposition. The right

![Fig. 6. Schematics of the first generation fully sealed luminescent tube. (a) Detail of the two endpieces, (b) overall view of the device.](image-url)
endpiece also carries the pumping stem that is sealed after evacuation.

The assembly is done as follows. The two annular metal rings are glued to the anode once the conductive and phosphor layers have been deposited on its inner side, followed by the connecting glass sections, and finally the endpiece supporting the getter with the prepared and catalyzed metallic wire already fixed to the endpiece. The free end of the cathode is then attached to the spring on the other endpiece, and the final joint is realized. The assembled tube is then connected to a pump stand with a primary pump and a turbomolecular pump, bringing the vacuum down to 10⁻⁷ mbar. The cathode is heated and degassed at 1000 °C for 15 min, and the heating current is adjusted to the calibrated value for the growth. After isolation of the turbomolecular pump, C₂H₂ is introduced via a leak valve to a pressure of 400 mbar under dynamic flow. The CACVD growth is stopped after typically 5 min by cooling the wire and evacuating the hydrocarbon partial pressure. The lamp is then tested in field emission at 10⁻⁷ mbar. If the specifications are not reached, especially regarding homogeneity (emission site density) and voltage necessary to reach the target intensity, a second (or third) 5 min growth step is realized. The whole procedure can even be reset without dismantling the tube, as the carbon deposit can be removed by heating the wire to 800 °C in air for 15 min [31] without altering significantly the catalyst. This flexibility is of course a unique advantage of CACVD. If the field emission properties are satisfactory, the whole tube is heated to 300 °C to degas the device, and especially the phosphor, under continuous pumping for 12 h. The getter is activated during cooling, and the tube is then sealed when room temperature has been reached.

The fully sealed tube is shown in Fig. 7(a) on its portable high voltage power supply, and Fig. 7(b) displays the device under operation. The power supply delivers a fixed 20 W of power at variable current and voltage, which means that the optimum operating conditions are in the upper third of the 0–7.5 kV range of the power supply (typically 5 kV). The tube starts instantly and can be continuously dimmed, with good homogeneity and high stability. The cathode emits ~1 mA/cm² under the conditions of Fig. 7(b) while carbon nanotube cathodes can sustain values that are at least one order of magnitude higher [2]. Our device can therefore reach far higher emitted intensities than the one shown in Fig. 7(b).

Does the device shown in Fig. 7 meet the required specifications of Table 1? The lifetime is yet an unknown, and lifetime tests will be performed in the future. However, the high brightness luminescent elements of Ise Electronics Corp. reach lifetimes of 8000 h [5], and their elements are similar in construction to our device. The second problem, which is of a more fundamental nature, is the overall efficiency of the device. The power consumption is at the moment higher than for a commercial luminescent tube of similar dimensions, which arises from the far lower efficiency of cathodoluminescent phosphors as compared to the UV phosphors used in fluorescent tubes [8]. At the moment, our device yields 18 lm/W at 5 kV for the white phosphor NP1045 from Nichia, and 40 lm/W at 5 kV for the green high intensity phosphor P22 from Osram. Note, however, that these phosphors are optimized for high voltage applications and that phosphors for low voltage applications are under intense investigations.

4. Conclusion

We have demonstrated the feasibility of cold cathode luminescent tubes based on carbon nanotube field emitters. We have successfully upscaled the deposition by developing CACVD, which allows us to grow nanotubes homogeneously on long metallic wires. CACVD permits also an additional control of the mean length and density of the emitters, and provides a powerful possibility to adjust the emission voltage and emission site density needed to reach the target intensity and specifications. We have also designed a simple assembly procedure that permits the realization of fully sealed elements in-house. While we are aware that the current fabrication process is artisanal and that it will have to be modified in the case of an industrialization, it represents a significant advance towards
a commercial application of these mercury-free, continuously dimmable and high-intensity luminescent tubes.

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