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► **To cite this version:**

P. Vincent, F. Panciera, I. Florea, M. Ezzedine, M.-R. Zamfir, et al.. Direct in situ Electron Microscope synthesis of CNTs with applied electric Field and Field Emission. 2021 34th International Vacuum Nanoelectronics Conference (IVNC), Jul 2021, Lyon, France. pp.1-2, 10.1109/IVNC52431.2021.9600790 . hal-03440298

HAL Id: hal-03440298

<https://hal.science/hal-03440298>

Submitted on 17 Nov 2022

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Direct in situ Electron Microscope synthesis of CNTs with applied electric Field and Field Emission

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Abstract— This work reports experiments on the oriented growth by electric field of free-standing carbon nanotubes (CNTs) by chemical vapor deposition (CVD) for field emission (FE) applications. The growths were observed in a scanning electron microscope (SEM) and in an environmental transmission electron microscope (ETEM) with the ETEM growths observed in real time. The effects of various applied voltage during growth will be presented. For high voltages, videos show that the maximum length of CNTs are limited either by the mechanical breakdown or FE induced thermal evaporation. Finally, the I(V) curves and current densities obtained will be presented with a focus on the different destruction mechanisms.

I. INTRODUCTION

The principal hurdle for the successful deployment of carbon nanotubes (CNTs) into many of the numerous industrial applications for which this quintessential nanoscience species has been proposed has always been, and remains, the mastering of selected CNT growth of types specific to each application. This is in no small part because of the difficulty in observing time-resolved growth at the necessary nanometer and atomic scales and conjointly of disposing of useful tools for controlling such growth. This article describes our recently achieved breakthrough in the dynamical observation of individual CNT growth on micro-machined chip heaters in Environmental Transmission and Scanning Electron Microscopes (ETEM and SEM). A key innovative aspect is that applied electric fields directs the systematic fabrication of extremely straight and preferentially single-wall nanotubes. This breakthrough allows an unprecedented access to the growth processes of highest quality CNTs, in situ post growth modification and eventually to the metrology of a wide spectrum of their physical properties.

II. EXPERIMENTS

In situ –real time growths (C_2H_2 , 10^{-4} – 10^{-3} mbar, $700^\circ C$) were carried out across a polarizable capacitor gap of specifically-designed micro-chip sample holders. The growth

rate varies linearly with the C_2H_2 partial pressure and the growth temperature is very critical. The CNTs are extremely straight over the $2 \mu m$ gap used to apply the electric field which we take as an indication of an absence of defects. Preliminary high resolution TEM images of the CNTs near their bases show them to be predominantly single wall carbon nanotubes (SWCNTs).

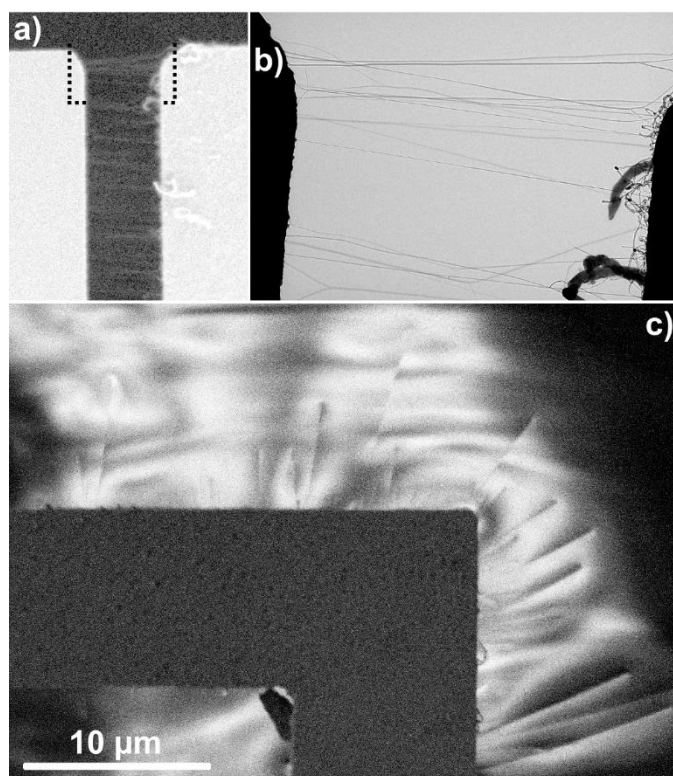


Fig. 1 a) Observation of SWNTs grown in SEM at low voltage between electrodes (4 Volt). (the SWNT appear as bright lines perpendicular to the electrodes). The growth has been directed by the electric field as TEM videos will show b) Post synthesis TEM observation of the upper part in a) (delimited by the black square). c) Growth on another part of the sample. Carbon nanotubes are visible as straight lines with contrast inversion in the image.

Nanotube are still oriented by the electric field and length up to 10 μm are obtained.

Depending the voltage applied during the growth we can distinguish two regimes. At low voltage (typically 4 Volt between the 2-3 μm gap) FE cannot occur and the electrostatic forces are relatively weak. Interestingly we observed that growth of SWNTs is directed by the electric field even at these low voltage values and the nanotube grow until they reach and stick to the opposite electrode (see figure 1).

At higher voltages (between 10 and 50 Volt) the electrostatic forces are stronger and FE can occur for the SWCNTs in the gap. In this configuration the nanotube length is limited to around (1- 1.5 μm) due to mechanical breakdown or thermally activated FE evaporation and we did not observe nanotubes crossing the gap.

After the growth we carried out I(V) measurements of the SWCNTs in order to measure the emitted current and we observe d a maximum field emission current of about 20 μA .

III. CONCLUSIONS

In this talk we will present results and videos on the growth and FE characterization of these cathodes. These experiments show the potentialities of such micro-machined heater in ETEM to better understand the growth and FE properties of CNT cathodes

ACKNOWLEDGMENT

The authors acknowledge financial support from the French state managed by the National Research Agency under the Investments for the Future program under the references ANR-10-EQPX-50 pole NanoMax and the 3DRX-online project. The authors would also like to acknowledge the Centre Interdisciplinaire de Microscopie électronique de l'X (CIMEX) and the Plateforme Nanofils et Nanotubes Lyonnaise of the University Lyon1.