Visualizing Carbon Nanotubes with LV FE-SEM

Application Note

Craig Wall, Ph.D.
Agilent Technologies

Introduction

Carbon nanotubes (CNTs) are allotropes of carbon with a cylindrical nanostructure. These cylindrical carbon molecules have unusual properties which are valuable for electronics, optics and other fields of materials science. In particular, owing to their extraordinary thermal conductivity and mechanical and electrical properties, carbon nanotubes may find applications as additives to various materials.

Carbon nanotubes are members of the fullerene structural family, which also includes the spherical Buckyballs, and the ends of a nanotube may be capped with a hemisphere of the Buckyball structure. Their name is derived from their hollow, tube-like, structure with the walls formed by one-atom-thick sheets of hexagonal carbon, called graphene. Conceptually, these sheets are "rolled" at specific, discrete chiral angles. The combination of the rolling angle and tube radius dictates the nanotube properties; for example, whether the individual nanotube behaves as a metal or semiconductor. The chemical bonding in nanotubes is composed almost entirely of sp^2 bonds, similar to those in graphite. These bonds, which are stronger than the sp^3 bonds found in alkanes, provide nanotubes with their unique strength and electrical properties. Nanotubes are categorized as single-walled nanotubes (SWNTs) and multi-walled nanotubes (MWNTs). Individual nanotubes naturally align themselves into rope-like strands held together by van der Waals forces.

The fields of potential application for CNT materials are quite broad, including structural applications, electromagnetic, electroacoustic, chemical, mechanical, electronic devices and circuits, and medicine. Some of the critical factors for nanotubes are purity, dispersability, electrical and mechanical properties, and morphology. Because of their nanoscale size, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and scanning probe microscopies have dominated the techniques used to visualize CNTs.

Agilent's 8500 FE-SEM is a low voltage, field emission SEM which employs a novel electrostatic lens design. This innovative design allows for high resolution imaging of CNTs and CNT composites, typically without the need for metal coating. The 8500 FE-SEM was used to image CNT dispersions on titanium coated silicon[1] substrate and as a microelectrode coating.[2]

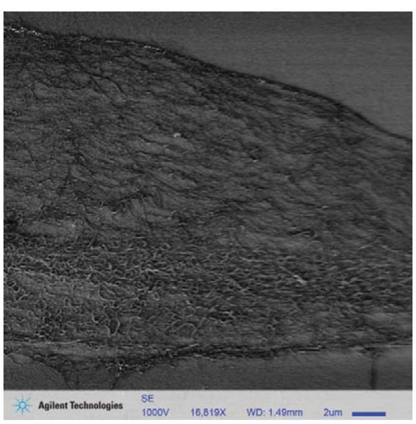

Figure 1. CNT dispersed on titanium coated silicon wafer.

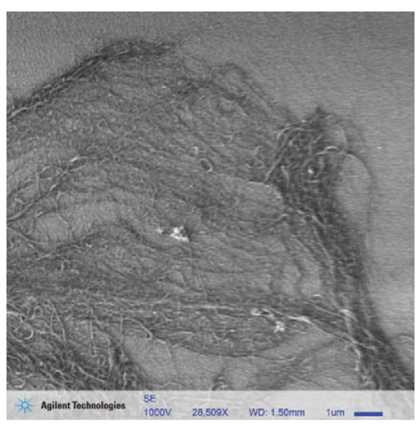

Figure 2. CNT dispersed on titanium coated silicon wafer.

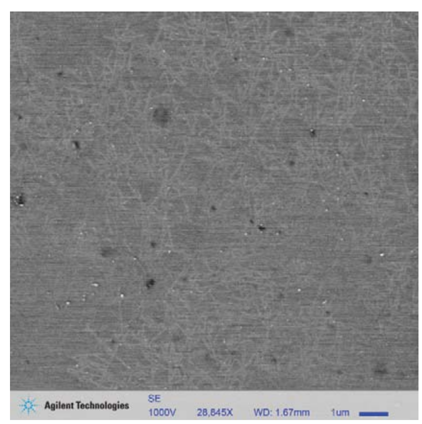

Figure 3. CNT dispersed on titanium coated silicon wafer.

Agilent Technologies

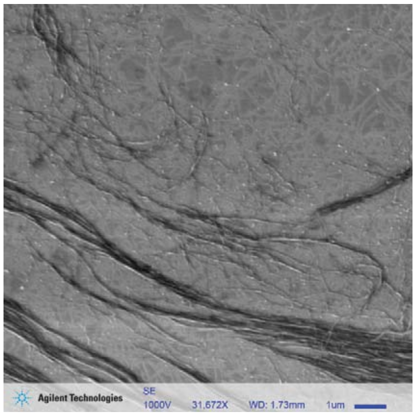

Figure 4A. CNT dispersed on titanium coated silicon wafer.

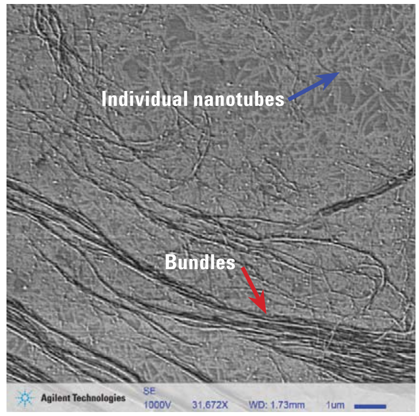

Figure 4B. CNT dispersed on titanium coated silicon wafer.

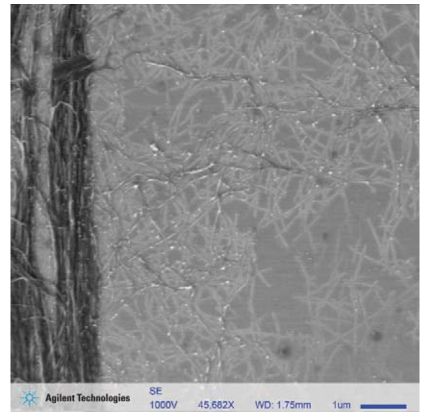

Figure 5. CNT dispersed on titanium coated silicon wafer.

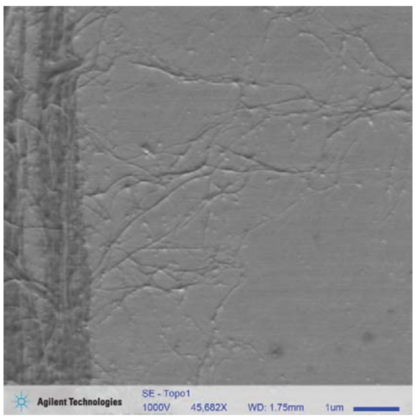

Figure 6. CNT dispersed on titanium coated silicon wafer.

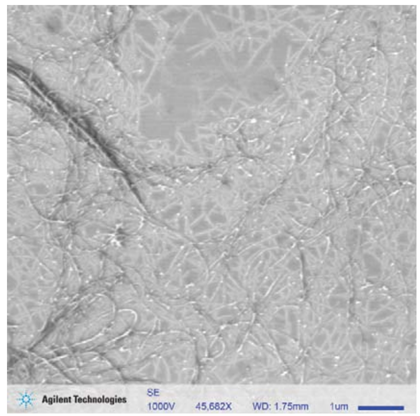

Figure 7. CNT dispersed on titanium coated silicon wafer.

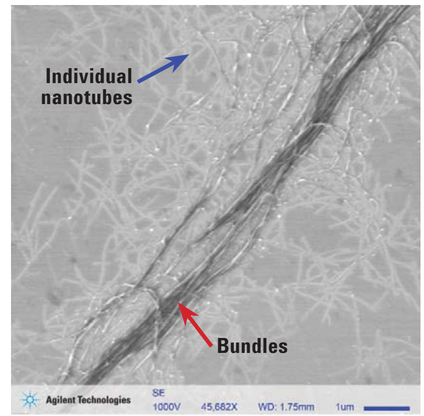

Figure 8. CNT dispersed on titanium coated silicon wafer.

CNT Dispersed on Titanium Coated Silicon Substrate

SWNTs have unique electrical properties that could be beneficial in a wide variety of electronic devices. Catalytic growth of individual nanotubes to form devices is a time-consuming tedious process that is prone to defects. Therefore the manufacturing scale production of SWNT based devices will likely rely on the purification and dispersion of nanotubes onto preformed electrical contacts.[3] LV FE-SEM is a fast, convenient way to examine these structures. However, recent work has shown the measured diameter of CNTs using FE-SEM is over-estimated.[4–7] There are two theories that attempt to explain the discrepancy in the diameter measurement; both are related to localized sample-substrate-electron beam interactions. The first theory is that electron beam induced charge contrast based on the nanotube substrate dielectric mismatch results in the observed increase in diameter.[4, 5] The second theory is that electron beam induced emission from the nanotubes results in the observed increase in diameter.[6, 7]

LV FE-SEM with an accelerating voltage of 1 kV was used to examine SWNTs dispersed on titanium coated silicon substrate, see Figures 1–13. The observed nanotube bundles yield images with the expected morphology and dimensions. Images that contain both bundles and individual nanotubes show the individual nanotubes have a different contrast mechanism as well as an exaggerated diameter, which is phenomenologically consistent with both proposed theories in the literature. The presence and morphology of the nanotube bundles does not appear to influence the morphology and dispersion of the individual SWNTs (Figures 4, 5, 7, 8, and 12). Figures 3 and 4A show typical images for individual SWNTs, however Figure 4B shows the improved visualization of individual nanotubes by applying a contrast-limited adaptive histogram expansion filter.

Although LV FE-SEM does not provide accurate measurement of the SWNT's very small diameter, it does provide a fast, convenient way to evaluate the location, length, density, and morphology of the nanotubes.

Electrodeposited CNT on Microelectrode

Implanting microelectrodes in the nervous system to study and treat neural diseases is becoming commonplace. Neurophysiologists have used wire

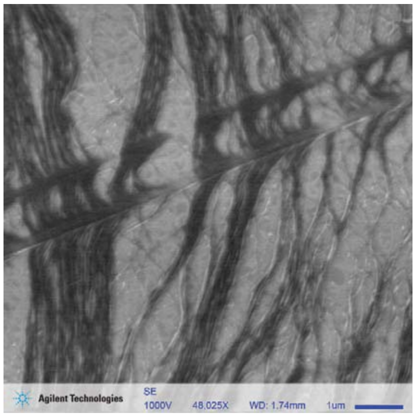

Figure 9. CNT dispersed on titanium coated silicon wafer.

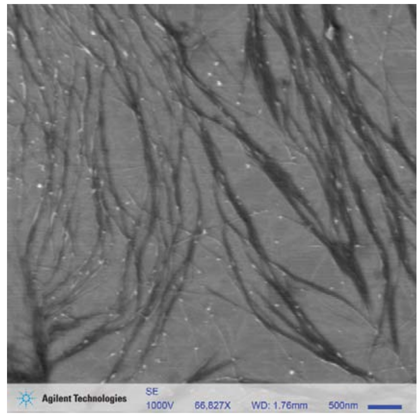

Figure 10. CNT dispersed on titanium coated silicon wafer.

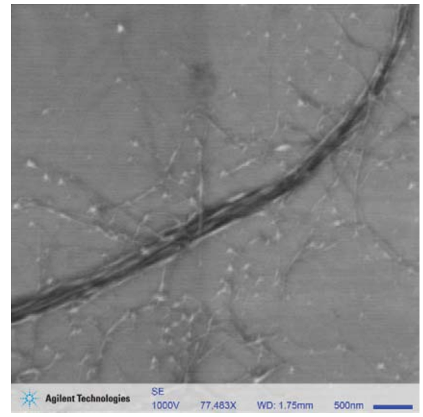

Figure 11. CNT dispersed on titanium coated silicon wafer.

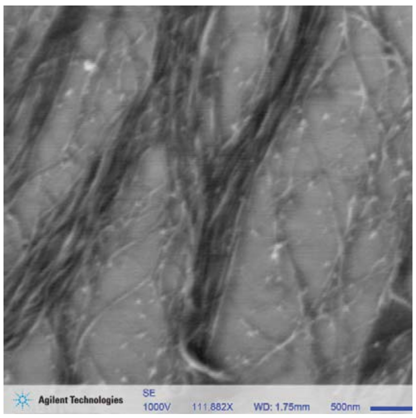

Figure 12. CNT dispersed on titanium coated silicon wafer.

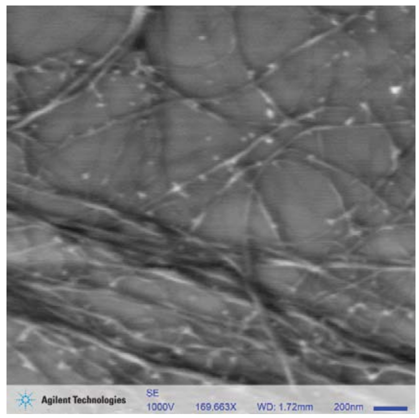

Figure 13. CNT dispersed on titanium coated silicon wafer.

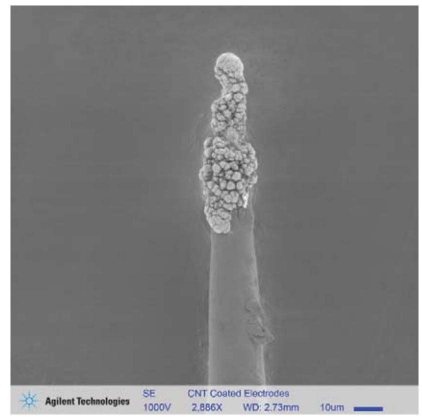

Figure 14A. CNT coated microelectrode.

based microelectrodes to study brain function and treat disease for the last 5 decades. However, the ultimate success of these experiments depends on the microelectrode-brain tissue interface. The high surface area and unique electrical properties of CNTs make them an interesting candidate to improve the microelectrode interface.

LV FE-SEM with 1 kV accelerating voltage was used to examine the surface structure and morphology of CNT coated microelectrodes used in neurophysiology, see Figures 14–19. The images, progressing from Figure 14 to Figure 19, show an increasing level of detail in the CNT coating as the field size (scan area) is reduced. Figure 14 shows the comparison of a secondary electron image to a topographic mode image of a CNT coated microelectrode. The topographic view is generated by

Figure 14B. CNT coated microelectrode, topographic view of figure 14A.

Figure 15. CNT coated microelectrode.

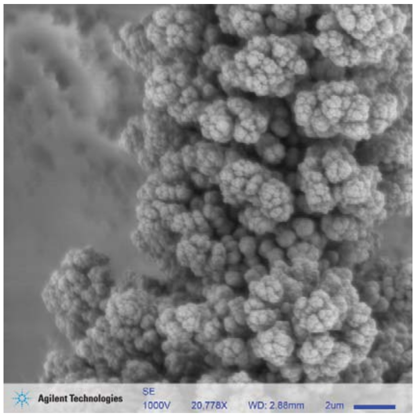

Figure 16. CNT coated microelectrode.

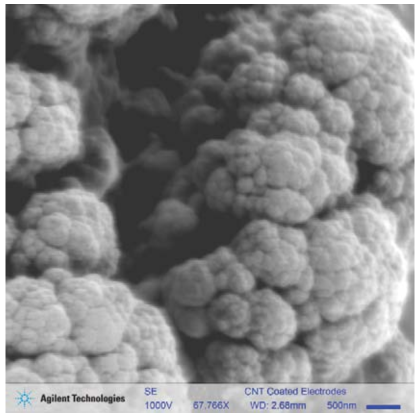

Figure 17. CNT coated microelectrode.

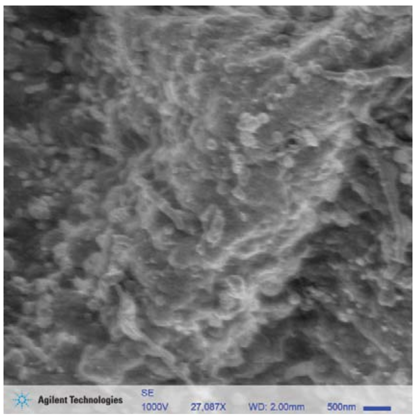

Figure 18. CNT coated microelectrode.

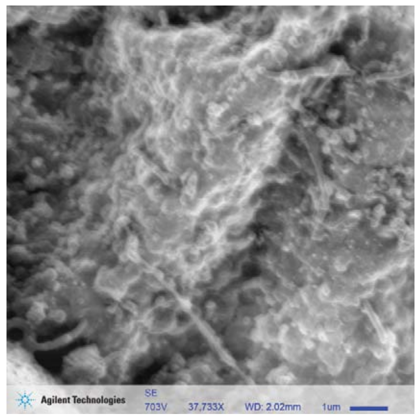

Figure 19. CNT coated microelectrode.

the four quadrant multichannel plate detector in the Agilent 8500 FE-SEM by subtracting the right two quadrants from the left two quadrants. Topographic mode highlights fine surface details by effectively generating a real-time shadowing similar to sunlight shining on mountains. Figures 16–19 show the morphology of the coating as well as the increased surface area and at the highest resolution, individual nanotubes in the coating matrix.

Conclusions

LV FE-SEM provides a straightforward technique for high resolution imaging of carbon nanotubes and carbon nanotube composites, typically without the need for metal coating. Although the application range of nanotubes examined spans many levels of sophistication in materials and processing, the morphological features of interest could easily be investigated with the Agilent 8500 FE-SEM.

References

1. CNT on Titanium courtesy of Professor Marcus Lay University of Georgia.
2. CNT on microelectrodes courtesy of Plexon Corporation.
3. L.D. Lipscomb, P. Vichchulada, N.P. Bhatt, Q. Zhang, M.D. Lay, *J. Mater. Sci.* **46,** 6812 (2011)
4. Y. Homma, S. Suzuki, Y. Kobayashi, M. Nagase, D. Takagi, *Appl. Phys. Lett.* **84,** 1750 (2004).
5. R.Y. Zhang, Y. Wei, L.A. Nagahara, I. Amlani, R.K. Tsui, *Nanotechnol.* **17,** 272 (2006).
6. A. Nojeh, B. Shan, K. Cho, and R.F.W. Pease, *Phys. Rev. Lett.* **96,** 056802 (2006)
7. S. Suzuki, *J. Surf. Sci. Nanotech.* **9,** 103 (2011)

Nanomeasurement Systems from Agilent Technologies

Agilent Technologies, the premier measurement company, offers high precision instruments for nanoscience research in academia and industry. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Agilent's leading-edge R&D laboratories ensure the continued, timely introduction and optimization of innovative, easy-to-use nanomeasurement system technologies.

www.agilent.com/find/nano

Americas

Canada	(877) 894 4414
Latin America	305 269 7500
United States	(800) 829 4444

Asia Pacific

Australia	1 800 629 485
China	800 810 0189
Hong Kong	800 938 693
India	1 800 112 929
Japan	0120 (421) 345
Korea	080 769 0800
Malaysia	1 800 888 848
Singapore	1 800 375 8100
Taiwan	0800 047 866
Thailand	1 800 226 008

Europe & Middle East

Austria	43 (0) 1 360 277 1571
Belgium	32 (0) 2 404 93 40
Denmark	45 70 13 15 15
Finland	358 (0) 10 855 2100
France	0825 010 700* *0.125 €/minute
Germany	49 (0) 7031 464 6333
Ireland	1890 924 204
Israel	972-3-9288-504/544
Italy	39 02 92 60 8484
Netherlands	31 (0) 20 547 2111
Spain	34 (91) 631 3300
Sweden	0200-88 22 55
Switzerland	0800 80 53 53
United Kingdom	44 (0) 118 9276201

Other European Countries:
www.agilent.com/find/contactus

Printed in USA, September 6, 2011
5990-9039EN

Agilent Technologies