



Fakultät II – Informatik, Wirtschafts- und Rechtswissenschaften  
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# **Nanorobotic handling and characterization of carbon nanotubes inside the scanning electron microscope**

Dissertation zur Erlangung des Grades eines  
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von

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# Übersicht

Kohlenstoff-basierte Nanomaterialien, wie z.B. einlagige Schichten aus Kohlenstoff, so genanntes Graphen und vor allem Kohlenstoffnanoröhren (carbon nanotubes, CNTs), haben sich zu einem der vielversprechendsten Materialien der Nanotechnologie entwickelt. Aufgrund ihrer herausragenden physikalischen Eigenschaften wurden CNTs bereits zahlreiche Anwendungsmöglichkeiten in unterschiedlichsten Bereichen vorhergesagt. Mit Hilfe der aktuellen Herstellungsverfahren lassen sich jedoch die geometrischen und physikalischen Eigenschaften der CNTs nicht vollständig kontrollieren. Die auf einer Abscheidung aus der chemischen Gasphase beruhenden CVD-Verfahren (chemical vapour deposition, CVD) könnten in naher Zukunft mit herkömmlichen Herstellungsverfahren der Mikrosystemtechnik vereinbar sein, so dass eine direkte Herstellung von CNTs in zukünftigen Mikrosystemen möglich wäre. Bis dahin stellt jedoch gerade die Mikro-Nano-Integration von CNTs in bestehende Mikrosysteme eine der größten Herausforderungen dar. Um einerseits die Herstellungsverfahren weiter optimieren und andererseits den Aufbau erster CNT-basierter prototypischer Bauteile ermöglichen zu können, ist die Handhabung und Charakterisierung einzelner CNTs von zentraler Bedeutung. Die Grundidee dieser Arbeit ist deshalb die Erforschung neuartiger, roboterbasierter Methoden für die Handhabung und Charakterisierung von einzelnen CNTs. Dazu wurde ein nanorobotisches System im Rasterelektronenmikroskop aufgebaut, mit dessen Hilfe neue direkte und zerstörungsfreie Verfahren zur elektrischen und mechanischen Charakterisierung von gewachsenen CNTs erforscht wurden. Außerdem wurden Strategien für die kontrollierte Mikrogreifer-basierte Handhabung von CNTs erforscht, die eine reproduzierbare Herstellung von prototypischen CNT-basierten Komponenten erlauben. Die beschriebenen nanorobotischen Methoden und Strategien schaffen die Voraussetzung für eine zukünftige Automatisierung dieser Prozessabläufe auf der Nanoskala.



# Abstract

Carbon-based nanomaterials such as mono-layered sheets of carbon, so-called graphene, and especially carbon nanotubes (CNTs) have become promising materials in nanotechnology. The extraordinary physical properties of CNTs are the reason for a multitude of potential applications that are foreseen in different areas. Using current fabrication techniques the exact geometric properties of CNTs, and thus their physical characteristics, are not completely controllable. Chemical vapour deposition (CVD)-based techniques might become completely compatible with standard micro fabrication techniques in the near future and seem to be the best approach to realize the direct synthesis of CNTs in future devices. However, the micro-nano-integration of CNTs into existing micro systems is one of the main challenges. In order to optimize the fabrication techniques and to allow the assembly of CNT-based prototypic devices, reliable handling and characterization of CNTs is required. The main idea of this work, therefore, is the development of novel nanorobotic methods for the handling and characterization of individual CNTs. For this purpose, a nanorobotic system is integrated into a scanning electron microscope facilitating the development of direct and nondestructive methods for mechanical and electrical characterization of as-grown CNTs that are coming directly from its CVD-based fabrication without any further treatment. In addition, novel strategies for the reproducible microgripper-based pick-and-place handling of CNTs are developed that enable the assembly of prototypic CNT-based devices. The presented methods and strategies provide the basis for a future automation of nanohandling sequences.



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# 1. List of abbreviations

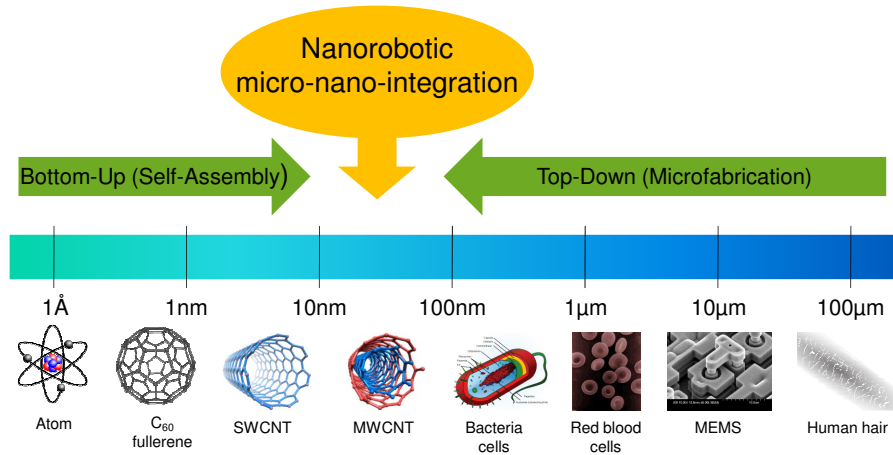
AFM	atomic force microscope
ARC	asymmetric ribcage
BSE	backscattered electron
CMOS	complementary metal oxide semiconductor
CNT	carbon nanotube
Cu	copper
CVD	chemical vapor deposition
DFP	depth from focus
DEP	dielectrophoresis
DOF	degree of freedom
EBiD	electron beam-induced deposition
EDX	energy dispersive X-ray spectroscopy
FIB	focused ion beam
GIS	gas injection system
HRSEM	high resolution scanning electron microscope
HRTEM	high resolution transmission electron microscope
ITRS	international technology roadmap for semiconductors

MEMS	micro-electro-mechanical systems
MWCNT	multi-walled carbon nanotube
NEMS	nano-electro-mechanical systems
PECVD	plasma-enhanced chemical vapor deposition
SE	secondary electron
SEM	scanning electron microscope
Si	silicon
SPM	scanning probe microscope
STEM	scanning transmission electron microscope
STM	scanning tunneling microscope
SWCNT	single-walled carbon nanotube
TEM	transmission electron microscope
VIA	vertical interconnect
4PP	four point probe

## 2. Introduction and motivation

Within the last years, nanotechnology has become increasingly important with many applications in different areas of research and life. The criterion for nanotechnology is the size of the considered components. Nanoscale objects have a characteristic size of up to 100 nm in at least one dimension. Carbon-based nanomaterials such as mono-layered sheets of carbon, so-called graphene [55], and especially carbon nanotubes (CNTs) [65] have become promising materials in nanotechnology. CNTs have extraordinary physical properties that enable the improvement of existing microsystems or even the production of novel products. For example: CNTs and especially bundles of single-walled carbon nanotubes (SWCNTs) are up-and-coming materials to replace and outperform classical copper interconnects [141, 130]. Furthermore, carbon nanotubes have a huge potential in micro- and nanoelectronics [5, 62] and nanosensor applications [99, 138]. CNTs and especially multi-walled carbon nanotubes (MWCNTs) can be used to realize improved tips for atomic force microscopes (AFMs) [59]. Such so-called CNT-enhanced AFM supertips can overcome the limitations of classical micro-machined silicon tips in scanning micro- and nanostructures with a high aspect ratio.

The main challenge therefore is the so-called micro-nano-integration of nanoscale objects into existing microsystems in order to exploit the nanotechnology-based effects. Micro-nano-integration can be seen as the continuation of the classical packaging of integrated circuits to connect the nanostructures to the micro- and macroworld. Micro-nano-integration will be a key issue for the future realization of complex nanoobject-enhanced systems. In general, two different strategies are being pursued to reach this goal: The top-down and the bottom-up approach. Figure 2.1 illustrates both approaches but also shows the existing gap that can be closed by robotic micro-nano-integration.



**Figure 2.1.:** Illustration of bottom-up and top-down approach.

The top-down approach is based on the further downscaling of conventional microfabrication techniques leading to smaller and smaller functional structures. For example: The ongoing miniaturization of integrated circuits is reaching the limit of structures that are realizable by conventional lithographic methods. Intel introduced 32 nm transistors in 2009. However, the international technology roadmap for semiconductors (ITRS) predicts that silicon-based complementary metal oxide semiconductor (CMOS) transistors will run out of the long-term trend given by Moore's law within the next ten years. The main problem is that smaller and faster chips generate more and more heat. Nanomaterials that show quantum effects can reverse this trend by consuming less power. In addition, the ongoing miniaturization and increase of efficiency and sensitivity in microsystem technologies is reaching the limits of classical materials. For this reason, novel nanomaterials such as CNTs have to be developed and explored to facilitate the further miniaturization of microstructures and -systems.

The bottom-up approach uses physical or chemical self-organizing techniques to realize self-assembly on the molecular or even atomic scale. For example: Fabrication techniques of CNTs are based on self-organizing processes and allow the catalytic growth of nanotubes at well-defined positions with properties in a specific spectrum. Such a direct integration of CNTs into microsystems by in-situ growing techniques is the desired long-term goal providing a micro-nano-integration



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technique that is compatible with CMOS-manufacturing processes. However, the catalytic growth of CNTs by chemical vapor deposition (CVD)-based techniques still encounters problems that remain to be solved. For this reason, it is essential to provide systematic characterization techniques for individual CNTs in order to optimize the CVD-based fabrication process parameters for future mass production of CNT-based devices.

Nanorobotic manipulation systems that can be integrated into the vacuum chamber of scanning electron microscopes (SEMs) are one of the most promising approaches [52, 29] to quickly close this gap and realize the micro-nano-integration of nanoscale objects having diameters between some and hundreds of nanometers. Besides the possibility of imaging objects with sizes down to several nanometers, the SEM offers a spacious vacuum chamber for installing nanorobotic systems. The nanorobotic technology can bring CNTs from basic research to their foreseen applications by providing reliable handling and systematic characterization strategies.

The main idea of this work, therefore, is the development of novel nanorobotic methods for the handling and characterization of individual CNTs. For this purpose, a nanorobotic system is integrated into an SEM allowing the development of direct and nondestructive methods for the mechanical and electrical characterization of as-grown CNTs. As-grown means that the nanotubes come directly from its CVD-based fabrication without any further treatment. In addition, novel strategies for the reproducible microgripper-based pick-and-place handling of CNTs are developed that enable the assembly of prototypic CNT-based devices. The presented methods and strategies provide the basis for a future automation of nanohandling sequences.

The work is structured as follows: In Chapter 3, the CNT basics including structure, fabrication techniques, and application areas are presented. The state-of-the-art in the research field of nanorobotic CNT handling and characterization inside the SEM is discussed in Chapter 4. In Chapter 5, the realization and integration of the nanorobotic system is described which is used to develop novel nanorobotic strategies for reliable handling and nondestructive characterization of individual CNTs that are presented in Chapter 6. Experimental results of the pick-and-place handling, mechanical and electrical characterization are evaluated in Chapter 7. Finally, a summary and outlook is given in Chapter 8.



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