7 AFM Sensors in Scanning Electron and Ion Microscopes: Tools for Nanomechanics, Nanoanalytics, and Nanofabrication

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Abstract. In this chapter the synergies upon the integration of atomic force microscope sensors in scanning electron and ion microscopes are outlined and applications are presented. Combining the capabilities of the standalone techniques opens the world to nanoscale measurements and process control. The high-resolution microscopy imaging provides direct visual feedback for the analysis of specific sample features and of individual nanostructures. Fundamental static and dynamic mechanics of cantilever beams are reviewed with an emphasis on the usage of the beams as force and mass sensors in a vacuum. Static force sensing is applied to probe the mechanical properties of nanowires in tensile, bending and compression experiments and dynamic force sensing is used for AFM in SEM applications. Cantilever-based dynamic sensing is discussed to measure the mass of material deposited or etched using the electron or ion beam inherent to the microscope.

Key words: Cantilever-based force/mass sensor, Piezoresistive cantilever, Vibration, Resonance, Scanning electron/ion microscope, Focused electron/ion beam induced deposition and etching

Symbols

α_{th}	Thermal expansion coefficient
A_c	Deflection amplitude at cantilever end
A_d	Excitation amplitude
A_s	Area of sample cross section
β_n	Vibration eigenmode of the nth mode
β_{th}	Temperature coefficient of Young's modulus
B	Measurement bandwidth
Δf	Frequency shift
Δf_{min}	Minimum resolvable frequency shift
Δm	Mass shift
Δm_{min}	Minimum resolvable mass shift
ΔW	Total energy lost per vibration cycle
δf	Frequency noise
δm	Mass noise
E	Young's modulus
f	Frequency
f_0	Resonance frequency (point-mass model)
f_d	Driving frequency
f_n	Resonance frequency of the nth mode
F_a	Applied force

h	Cantilever height (thickness)
I	Geometrical (area) moment of inertia
J	Molecular flux
k	Spring/force constant
l	Cantilever length
L	Sample length
m^*	Effective mass
m_c	Cantilever mass
n_e	Normalized effective mass
ϕ	Phase
Q	Quality factor
ρ	Mass density
R	Mass responsivity
σ	Strength
t	Time
T	Temperature
ε	Strain
V	Volume
w	Cantilever width
W_0	Stored vibrational energy
Z_n	Vibration shape of the nth mode

Abbreviations

AFM	Atomic force microscope
CNT	Carbon nanotube
CVD	Chemical vapor deposition
FEB	Focused electron beam
FIB	Focused ion beam
FWHM	Full width at half maximum
GIS	Gas injection system
NEMS	Nanoelectromechanical system
QCB	Quartz crystal microbalance
SEM	Scanning electron microscope
SIM	Scanning ion microscope
SW-CNT	Single-wall carbon nanotube
TEM	Transmission electron microscope

7.1 Introduction

In 1986 the Nobel Prize was given to Ruska for scanning electron microscopy and to Binning and Rohrer for scanning probe microscopy. Referring to the increasing number of publications in this field, the potential of combined or hybrid techniques is about to be explored. Atomic force microscopes (AFMs) with their nanomanipulation capabilities and their cantilever-based sensor derivatives are increasingly combined with scanning electron microscopes (SEMs) and scanning ion microscopes (SIMs). SEMs and SIMs evolved over the last two decades literally into "workshops" which can fabricate tailored 3D nanostructures using gas injection. With the aid of the focused electron beam (FEB) or the focused ion beam (FIB), deposition, etching, and milling can be performed at the nanoscale. Combining these powerful scanning probe observation techniques and their derivatives provides access to the measurement of individual nanostructures, in particular nanowires, nanotubes, and 3D structures in nanoelectromechanical systems (NEMS). Furthermore, FEB and FIB processing of nanostructures can be controlled quantitatively in terms of etching, milling, and deposition rates and can thus be optimized. Figure 7.1 shows the capabilities of SEMs, SIMs, and AFMs and their application in hybrid techniques.

The combination of techniques into hybrid systems is mainly driven by the investigation of *individual* nanostructures and of mechanisms during nanoscale growth and etching. This demands the manipulation of individual nanostructures to probe their physical and chemical properties. In other words, attachment, placement, and release actions must be performed under visual control at nanometer scale to move or to fabricate the nanostructure at the desired place.

We summarize our discussion of AFMs, SEMs, and SIMs in Table 7.1 showing a comparison of several specifications for their use in hybrid systems.

The techniques in Table 7.1 are well known and abundantly discussed in the literature: For information on SEMs the reader is referred to standard textbooks [64, 125]. SIMs are assembled much the same way as their electron counterparts but use other interaction mechanisms [116]. Atomic force microscopy is a very powerful and well-described technique [15,59,63] with derivative techniques—many of them described and discussed in this book series [13].

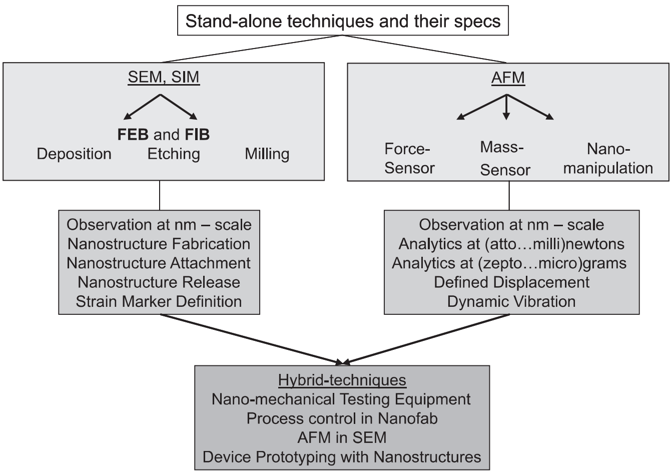

Fig. 7.1. Classic standalone scanning probe techniques and their standalone derivative techniques and tools. Combining their capabilities in observation, nanostructuring, and analytics into hybrid systems opens the world to nanoscale measurements and process control. Acronyms: *AFM* atomic force microscope, *SEM* scanning electron microscope, *SIM* scanning ion microscope, *FEB* focused electron beam, *FIB* focused ion beam

Table 7.1. Comparison of the scanning electron microscope (*SEM*), the scanning ion microscope (*SIM*), and the atomic force microscope (*AFM*)

	SEM	SIM	AFM
Probe	Focused electron beam	Focused ion beam (mostly Ga ions)	Probe tips (functionalized)
Lateral resolution	∼1–10 nm	∼10 nm	∼0.1 nm
Depth resolution	Low (large depth of focus)	Low (large depth of focus)	High (∼ subnanometer)
Environment	Vacuum: $\sim 10^{-6}$ mbar Environmental, ∼1 mbar	Vacuum: $\sim 10^{-6}$ mbar Environmental, ∼1 mbar	Ambient, liquids, vacuum
Analytics	Elemental analysis, crystal orientation, electric field contrast	Elemental analysis, grain orientation	Magnetic force microscopy, friction force microscopy, scanning capacitance microscopy, etc.
Limitations	Projected view	Projected view damage during imaging	Small lateral range
Structuring abilities	Lithography	Lithography, milling, implantation	Assembly of nano-objects via nanomanipulation
Derivative techniques	FEB-induced deposition and etching	FIB-induced deposition and etching	Haptic manipulation, force spectroscopy, mass sensing

FEB focused electron beam, *FIB* focused ion beam

SEMs and SIMs are mainly used for surface imaging at high resolution and for submicron chemical and structural analysis. AFMs are mainly employed for highest-resolution topography imaging and nanomanipulation. This as well as the use of their cantilever-based sensors for force and mass detection makes them particularly attractive for integration into SEMs, SIMs, or dual-beam systems.

Section 7.2 briefly introduces the derivative standalone techniques and their use in hybrid systems. Section 7.3 summarizes the fundamentals of cantilever-based sensors necessary to understand the three hybrid techniques presented in detail in Sect. 7.4.

7.2 Description of Standalone Techniques

7.2.1 FEB/FIB Nanofabrication

FEB/FIB maskless nanofabrication is a standalone technique with many applications in tailored device prototyping covering fields of nanoelectronics [9,37], nanophotonics [91,111,120], and functionalization of scanning probe sensors [10,51,152]. In

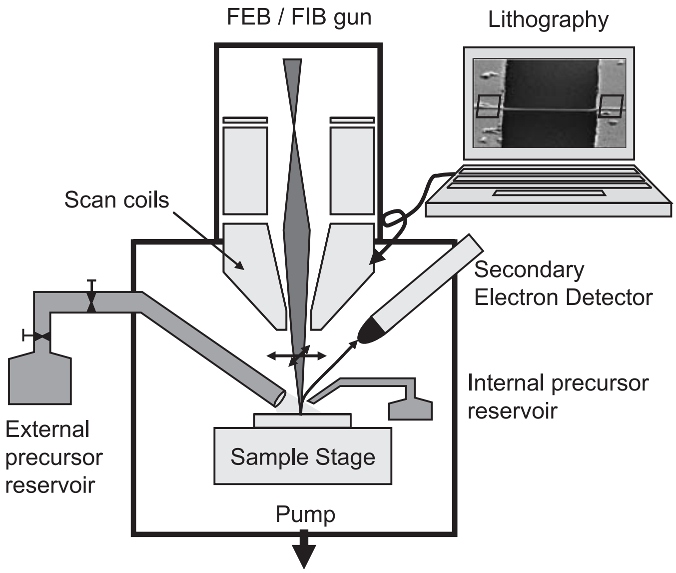

Fig. 7.2. Schematics of FIB and FEB nanofabrication systems. Gas injection systems with external and internal precursor reservoirs are shown. The laptop monitor shows (schematically) FEB- or FIB-written clamps to a nanowire

combination with nanomanipulation, FEB- and FIB-induced deposition is predominantly used as a technique for attachment to the sensor despite the fact that often the mechanical properties of the deposit are unknown. FIB milling by 30-keV Ga ions is predominantly employed as a specimen preparation technique which permits "cutting" nanostructures with dimensions down to sub-100-nm into the bulk. For electrical contacts to carbon nanotubes (CNTs) or nanowires, FEB-induced deposition is frequently used as a soldering technique [21, 54, 55]. The principle of both techniques is presented in Fig. 7.2

7.2.1.1
FEB-Induced Deposition and Etching

The principle of nanofabrication via FEB is shown in Fig. 7.3. Comprehensive overviews in this field are found in [18, 20, 31, 124]. Basically, during deposition and etching surface-adsorbed gas molecules are decomposed by electron irradiation and form either a stable deposit and gaseous by-products or volatile reaction products with the substrates (Fig. 7.3). The molecules are supplied by a gas injection system (GIS). By switching between a deposition gas and an etch gas, one can use this technique as an attach and release tool to/from a nanomanipulator or sensor. Often hydrocarbon molecule sources are present in SEMs coming from oil vapors of the vacuum system and from the contamination on the sample itself. Under electron irradiation they lead to carbonaceous contamination deposits. Historically regarded as an unwanted side effect, they have proved very useful in attaching nanostructures

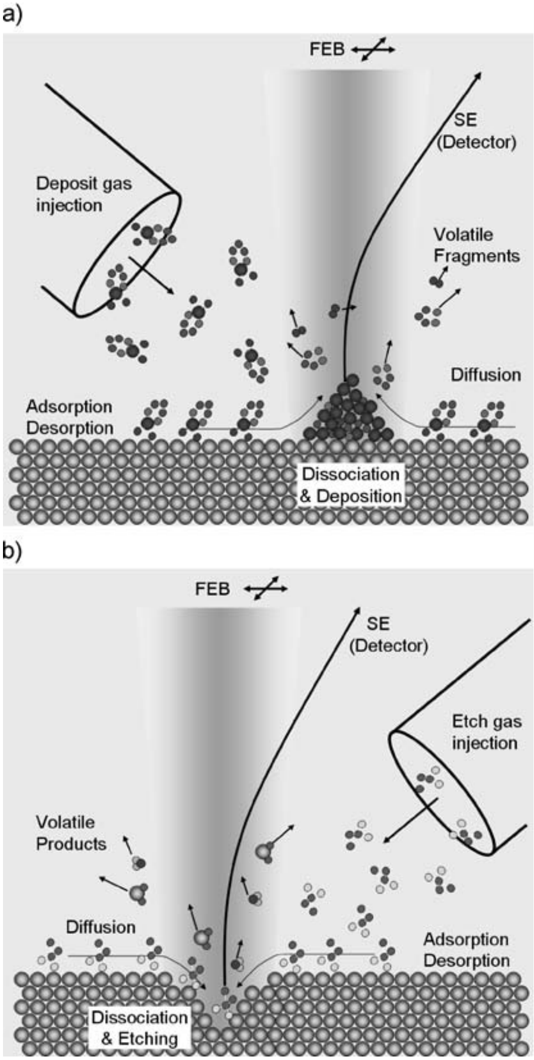

Fig. 7.3. **a** Principle of FEB-induced deposition: Molecules adsorb at the surface and are dissociated under electron impact. Volatile fragments are pumped away and a deposit grows coaxially to the beam. Here molecules are injected by a microtube. In the case of contamination deposits, hydrocarbon molecules originate from the microscope backpressure and the substrate surface. **b** Principle of FEB-induced etching: The surface adsorbed molecules dissociate under electron impact into reactive species and form volatile compounds with the substrate material

to cantilevers and as a marker technique for surface strain quantification detection (Fig. 7.4b,c).

Obviously, reproducibility of "contamination" attachments will strongly depend on the contamination level of the sample and the microscope. Introducing organic precursors into the SEM chamber allows control of the deposit composition and deposition rate [19]. One advantage of FEB-induced deposition is that tuning between mechanical stability and functionality of deposits can be performed using adapted metal–organic precursor molecules (Fig. 7.4a,d). The resulting deposit is a nanocomposite of metal nanocrystals embedded in a stabilizing carbonaceous matrix. In such deposits the metal content varies according to the precursor chemistry and deposition conditions. Higher metal contents are deposited when beam heating effects oc-

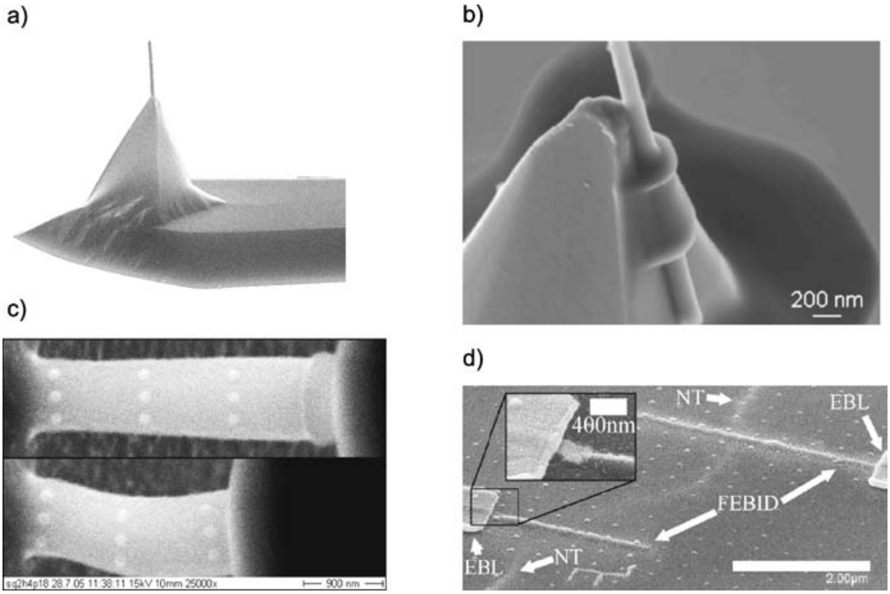

Fig. 7.4. Examples of FEB deposits. **a** AFM sensor functionalized with a magnetic tip deposit. **b** FEB clamp deposit to fix a nanowire to a cantilever tip from a paraffin precursor. **c** FEB marker deposits for strain detection during a compression experiment of micropillars from contamination. **d** FEB contacts to a carbon nanotube using a gold-containing precursor. (**a** From [152], **b** Reprinted with permission from [38]. Copyright (2005), American Institute of Physics, **c** from [105], **d** Reprinted with permission from [21].Copyright (2005), American Institute of Physics)

cur [147, 153]. Examples include FEB deposits from $(CH_3)_2$–Au–$C_5H_4F_3O_2$ [151], $Co_2(CO)_8$ [16], $W(CO)_6$ [88] which result in electrical resistivities about 100–1000 times higher than the corresponding metallic bulk value. Low-resistivity Au contacts comparable to standard Au liftoff techniques were achieved with an inorganic gold precursor $AuClPF_3$ [21] and a mixture of $(CH_3)_2$–Au–$C_5H_7O_2$ and H_2O gas [97]. Etching gases comprise H_2O, O_2 for carbon, and XeF_2 for Si and SiO_2 [124]. Water vapor also attacks the CNT under electron irradiation and can even be used for cutting CNTs [169].

Mechanical properties (strength, density, elastic modulus) of FEB (and FIB) deposits have been poorly investigated owing their small deposit volumes (less than 1 μm^3) and small masses (less than 10 pg) and require special force and mass sensors, as discussed later.

7.2.1.2
FIB-Induced Deposition, Etching, and Milling

Processing with FIB is mainly based on liquid gallium sources being available since the late 1980s. Besides imaging capabilities they provide the ability to remove or add locally material (metals or insulators) at sub-100-nm dimensions [101, 110, 127].

When a focused beam of energetic ions hits a surface, the energy of the incident particles is transferred to the substrate, resulting in ejection of neutral and ionized substrate atoms (sputtering), displacement of atoms (damage), and emission of electrons (imaging, charging) and phonons (heating); see Fig. 7.5a. A substantial number of the impinging ions is implanted into the substrate, leading to contamination problems, or can be used as material doping. The physical sputter process, also named “milling”, is predominantly exploited in FIB nanostructuring. Injection of precursor gases leads to deposition or etching as already described for FEB. Major applica-

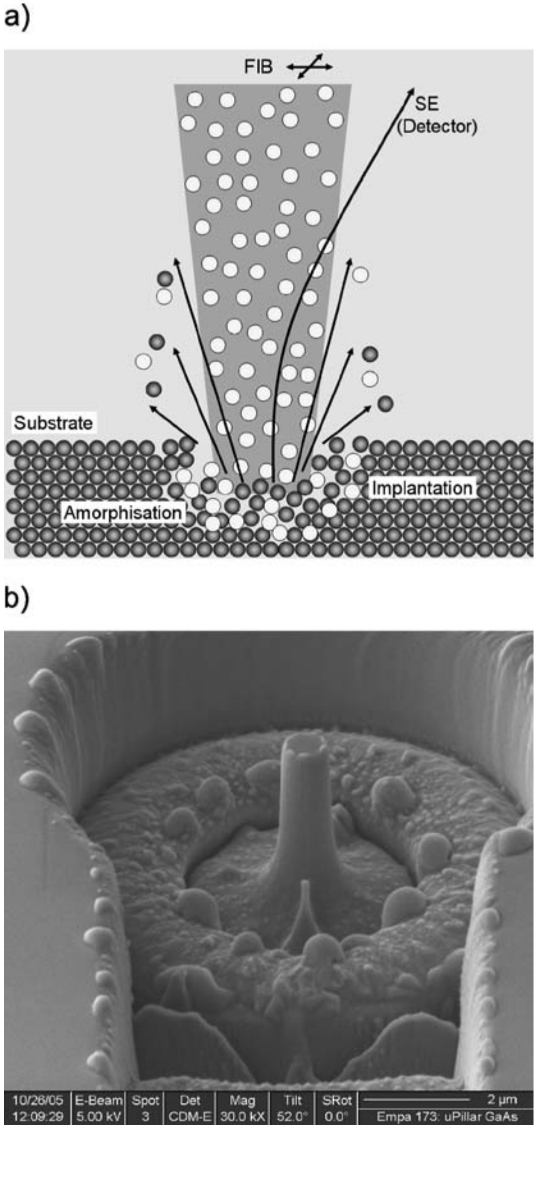

Fig. 7.5. a Principle of FIB milling (or sputtering). **b** GaAs micropillar for compression experiments machined with FIB

tions are device and circuit editing, lamellae preparation for transmission electron microscopy, and structure machining for mechanical tests (Fig. 7.5b). Owing to the ion damage, however, FIB is not well suited for nanostructure imaging and for direct contact writing to nanostructures.

7.2.2 Cantilever as a Static Force Sensor

In conventional AFM force spectroscopy the deflection of a cantilever during an approach–withdrawal cycle with a substrate is monitored and converted into force via the spring constant. Adhesion forces and molecule binding forces have been extensively studied using this technique [13, 26, 115]. Mechanical characterization of nanostructures like nanowires or nanotubes requires physical interaction with the object. With the AFM tip the object can be stretched or bent with nanometer positioning accuracy, while performing measurements with nanonewton to millinewton force resolution (Fig. 7.6). Brittle fracture of semiconductors, the yield point of metallic nanowires, and their Young's modulus can be measured by lying them down on a trench and bending them [14, 69, 119, 131]. The use of the cantilevers in a SEM together with nanomotion control is not only helpful for accurate positioning control, but also to gather additional visual information on the failure mechanism. Section 7.4.1 describes the use of a cantilever in a SEM to perform tensile and bending experiments.

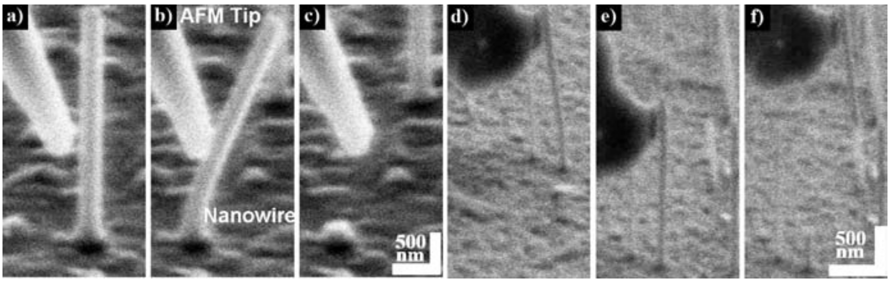

Fig. 7.6. SEM images of a bending and a tensile experiment. **a–c** Bending experiment: an AFM tip is used to manipulate the nanowire. **d–f** Tensile experiment: the freestanding nanowire is attached via a FEB-written bond to the AFM tip. The cantilever of the AFM tip is retracted and used as a force sensor

7.2.3 Cantilever as a Resonating Mass Sensor

Since the mid-1990s there has been significant development in the field of cantilever-based mass sensors for chemical-sensing and biosensing applications. These sensors are based on the fact that the resonance frequency of a vibrating structure depends on external stimuli, such as mass loading.

Using standard AFM cantilevers and equipment, the group of Thundat started to observe mass changes induced by adsorption of molecules on the cantilever

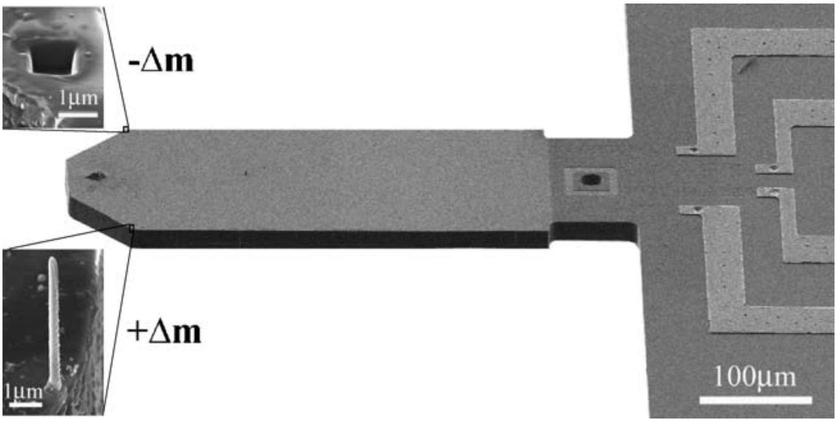

Fig. 7.7. SEM image of a silicon cantilever with integrated piezoresistive Wheatstone bridge (Nascatec, Germany) used as a resonating mass-sensing device in a SEM. The *insets* show FIB milling and FEB deposition on the cantilever surface

surfaces [29, 142, 156]. Further development has led to sensor arrays for the discrimination of volatile organic compounds [66, 89]. Cells of *Escherichia coli* bacteria have been selectively detected with antibody surface coated cantilever beams [76]. Another wide application field of cantilever-based mass sensors is process control, e. g., weight change due to chemical and biological reactions occurring on the cantilever surface. Berger et al. demonstrated in situ measurements of surface stress changes and kinetics during the formation of self-assembled monolayers [11] and thermally induced mass changes [12]. Applications of mass-sensitive resonating cantilever sensors for the detection of gas-phase analytes, liquid-phase analytes, and biological species are reviewed in [90]. Process control of additive or subtractive surface processing techniques based on cantilever sensors is a relatively new field. Sunden et al. [141] showed a weight change due to growth of CNTs on the cantilever surface. In Sect. 7.4.2 we discuss the exploitation of the analytical capabilities of cantilever sensors for studies of ion/electron beam induced processes (Fig. 7.7).

7.2.4 Nanomanipulation

Several kinds of AFM-based nanomanipulation systems have been developed [81, 126, 135] as well as virtual-reality interfaces to facilitate feedback during nanomanipulation [67, 92]. AFMs have been combined with different haptic devices, e. g., NanoMan [155], NanoManipulator^{TM} [1], NanoFeel 300 manipulator [108], and the Omega haptic device [52, 109]. Haptic devices provide the operator with real-time force-feedback. The main drawback of the abovementioned devices is the lack of visual feedback of the manipulation process in real time. Integration of such devices into the SEM would compensate for the lack of real-time visual feedback. Presently nanomanipulation in the SEM is mainly performed with

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