A MEMS nanoindenter with an integrated AFM cantilever gripper for nanomechanical characterization of compliant materials

Z Li¹, S Gao¹, U Brand¹, K Hiller² and H Wolff¹

¹ Physikalisch-Technische Bundesanstalt, 38116, Braunschweig, Germany
² Fakultät für Elektrotechnik und Informationstechnik, Zentrum für Mikrotechnologien, Technische Universität Chemnitz, 09126, Chemnitz, Germany

E-mail: zhi.li@ptb.de

Received 21 October 2019, revised 19 March 2020
Accepted for publication 14 April 2020
Published 11 May 2020

Abstract

This work presents the development of a MEMS nanoindenter that uses exchangeable AFM probes for quasi-static nanomechanical characterization of compliant and ultra-compliant materials. While the electrostatic micro-force transducer of the MEMS nanoindenter provides a maximum indentation depth up to 9.5 µm with a maximum output force of 600 µN, experimental investigations reveal that it can achieve a depth and force resolution better than 4 pm Hz⁻¹/² and 0.3 nN Hz⁻¹/², in air for f ≥ 1 Hz. A passive AFM probe gripper is integrated into the MEMS nanoindenter, allowing the nanoindenter to utilize various AFM probes as an indenter for material testing. A proof-of-principle experimental setup has been built to investigate the performance of the MEMS nanoindenter prototype. In proof-of-principle experiments, the prototype with a clamped diamond AFM probe successfully identified an atomic step (~0.31 nm) within a Si < 111 > ultraflat sample using the scanning probe microscopy mode. The nanomechanical measurement capability of the MEMS nanoindenter prototype has been verified by means of measurements of reference polymer samples using a silicon AFM probe and by means of measurements of the elastic properties of a PDMS sample using a spherical diamond-coated AFM probe. Owing to its compact and low-cost but high-resolution capacitive readout system, this MEMS nanoindenter head can be further applied for in-situ quantitative nanomechanical measurements in AFMs and SEMs.

Keywords: electrostatic MEMS, nanomechanical characterization, nanoindentation, compliant materials

(Some figures may appear in colour only in the online journal)

1. Introduction

Soft materials such as thermoplastic polymers and photoresists, biomacromolecules, gels and colloids are widely used in various scientific and industrial fields including micro- and nano-electronics [1, 2], engineered artificial skin [3, 4], micro- and nano-lithography [5, 6] and soft sensors and actuators [7, 8]. Quantitative nanomechanical characterization of soft materials [9–13] frequently plays an essential role in the design, development and further application of these materials. In comparison to typical metallic and ceramic materials, these soft materials usually have much weaker mechanical properties, and the corresponding elastic moduli can be as large as several GPa or as small as several MPa.

For nanomechanical characterization of such materials, especially in small volumes, the nanoindentation technique...
is frequently utilized, mainly due to its quite limited requirements for sample preparation, its relatively simple measurement procedure, and its well-established data evaluation and interpretation methods [15].

Presently, one common way of performing nanoindentation measurements is to utilize commercially available or self-developed nanoindentation instruments [16, 17]. Because such instruments usually have a maximum indentation force higher than 10 mN with a force resolution down to submicro-Newton, they have proven to be effective in the measurement of common hard and even relatively soft materials with elastic moduli of several GPa [18]. However, the force resolution of these conventional instruments becomes generally inadequate, especially for nanomechanical characterization of (1) ultra-thin (< 200 nm) layered polymers [19], for which the maximum indentation depth \( h_{\text{max}} \) usually is extremely limited (i.e. \( h_{\text{max}} < 20 \text{ nm} \) for obtaining substrate-independent results), and (2) ultra-compliant materials, whose elastic moduli tend to be generally in the MPa range or even lower.

Atomic force microscopes (AFM) generally have high force resolution and have therefore long been used to characterize the mechanical properties of soft materials [20, 21]. However, drawbacks of cantilever-based AFM nanomechanical measurements are also known, including:

- unavoidable tilting and lateral scratching of AFM tip in case of large cantilever deflection for deep indentation;
- quantitative indentation force can only be achieved after careful calibration of the cantilever stiffness and deflection, which becomes increasingly challenging when an AFM probe is equipped with a sharp and fragile tip [22];
- usability only for nearly flat surfaces.

In many cases, these disadvantages can prevent AFMs from reliable nanomechanical measurements in material testing.

A novel nanomechanical measurement system is therefore needed to bridge the gap between the measurement capabilities of traditional nanoindentation instruments and those of AFM-based nanomechanical systems for soft materials.

Microelectromechanical systems (MEMS), especially electrostatic MEMS sensors and actuators, feature high resolution in force and displacement sensing [23], a small size, low costs due to batch fabrication, and medium force and displacement range [24]. Several attempts have already been made to apply MEMS sensors/actuators for nanomaterial testing [25, 26].

However, equipping the MEMS with an indenter is one of the key challenges in MEMS-based nanodimensional and nanomechanical measurement systems. The most common approaches to this problem are:

a) incorporating an in-plane/out-plane tip into the MEMS transducers [27, 28];
b) gluing an indenter (e.g. a diamond tip or a sapphire microsphere) to the MEMS transducer [26, 29];
c) fabricating an indenter tip directly at the end of the MEMS shaft [25].

From an application point of view, the practicability of these approaches is limited because the entire MEMS nanoindenter becomes unusable once the indenter tip is worn out. Furthermore, the third solution needs high-end focused ion beam (FIB) facility, is therefore not cost-effective.

In this manuscript, we present a MEMS-based nanomechanical measurement head (or ‘MEMS nanoindenter’ for short) that is able to use exchangeable AFM probes as an indenter for material testing. The structural design of the MEMS nanoindenter including its passive cantilever gripper is detailed in section 2, prototyping and experimental characterization of the MEMS are illustrated in section 3 and initial measurement results using the MEMS nanoindenter prototype are reported in Section 4.

2. A MEMS nanoindenter: structural design

The goal of this work is to develop a compact and low-cost MEMS nanoindenter consisting of a micro-transducer for nano-force sensing and a passive AFM probe gripper for the utilization of various AFM probes as indenters. This MEMS nanoindenter is designed to perform quasi-static nanomechanical characterizations of compliant and ultra-compliant materials. It is therefore necessary to achieve high force sensitivity over a relatively large indentation depth range.

As in our previous studies [25, 29], the force transducer of the MEMS nanoindenter has been developed on the basis of a lateral electrostatic comb drive actuation and sensing mechanism [30], as this mechanism is not only capable of providing moderate displacement and output force, but is also compatible with various capacitive displacement measurement systems.

2.1. MEMS micro-transducer for nano-force sensing

As illustrated in figure 1, the MEMS force transducer utilizes a set of comb-drives with a differential configuration (\( U^+ \) and
U− in figure 1) to detect the in-plane displacement $\Delta z$ of its main shaft, which is suspended by means of folded springs. The actual force sensitivity of the MEMS transducer is therefore determined by

$$\delta F = \delta z \cdot k_{\text{MEMS}}. \quad (1)$$

where $k_{\text{MEMS}}$ is the axial stiffness of the suspending springs along the $z$-axis.

One way of improving the force sensitivity of a MEMS transducer is to utilize springs with low stiffness ($k_{\text{MEMS}} \to 0$); however, this results in a smaller testing force, higher noise and excessive pull-in and pull-off deflection due to the adhesion surface force \cite{31}. As proposed in \cite{20}, in this manuscript, the MEMS transducer is designed to have a higher stiffness (i.e. $k_{\text{MEMS}} = 25 \sim 70 \text{ N m}^{-1}$) in order to provide an indentation force of up to several 100 $\mu$N.

For constant and prescribed $k_{\text{MEMS}}$, the force sensitivity of the MEMS transducer is mainly limited by the sensitivity of its capacitive displacement readout system.

As shown in figure 2(a), the capacitive variation of the MEMS transducer with respect to the in-plane ($x-z$) displacement of the main shaft can be estimated as

$$\delta C = 4N \cdot \varepsilon_r \varepsilon_0 \cdot \frac{h}{g} \delta z = S_{\text{MEMS}} \cdot \delta z, \quad (2)$$

where $\varepsilon_r$ is the relative static permittivity, $\varepsilon_0$ the electric constant, $h$ the vertical height of the fingers, $g$ the gap between movable and fixed fingers, $N$ the number of finger pairs of $U^+$ or $U^-$ comb drives, and $S_{\text{MEMS}}$ the displacement-to-capacitance coefficient of the MEMS transducer.

To offer a cost-effective MEMS nanoindenter for soft material testing, commercially available capacitive readout ICs, including capacitance-to-digital converters (CDCs), are intended to be used for MEMS signal processing and measurement. It is therefore expected that the capacitive sensitivity of the MEMS transducer $\delta C$ should be at least higher than the resolution of these CDC ICs.

Taking the prerequisites mentioned above and the capability of currently available MEMS fabrication technology into consideration, the out-of-plane (vertical) thickness chosen for the MEMS fingers is set to $h = 75 \mu$m, the in-plane gaps of comb fingers $g = 3 \mu$m, and the overlapped length...
The stiffness of the MEMS is evaluated from the measurement of the piezo stage is measured by the balance. The force-displacement data update rate of this CDC can be up to 90 Hz, it is necessary that the indenter tip be perpendicular to the sample surface for nanoindentation purposes.

Finally, the fundamental parameters of the electrostatic MEMS nano-force transducer are listed in table 1. In addition, the maximum passive output (indentation) force of the MEMS transducer amounts to $F_{\text{max}} = 680 \, \mu \text{N}$ for $\Delta z = 9.5 \, \mu \text{m}$.

### 2.2. Passive gripper for fixing AFM probes as an indenter

In this manuscript, typical Si-based AFM probes are used as indenters for material testing. Because such Si probes usually have a symmetrically etched tip at their cantilever end, it is necessary that the indenter tip be perpendicular to the sample surface for nanoindentation purposes.

Bearing in mind the fact that most nanomechanical probes have a rectangular beam (cantilever) with trapezoidal cross section [32, 33], an AFM cantilever gripper with one pair of L-shaped springs was designed, as shown in figure 4, that clamps a cantilever at its sloped sidewalls. In the current version of the AFM probe holder, the cantilever width should be not smaller than 20 $\mu$m. The in-plane spring constant of the gripper is defined to be $5 \, \text{N m}^{-1}$, yielding a holding force of about 25 $\mu \text{N}$ for a cantilever with $W = 30 \, \mu \text{m}$.

Because the tip height of an ordinary AFM probe is about 15 $\mu \text{m} \pm 5 \, \mu \text{m}$, the gripper is designed in such a way that the tip height of a clamped AFM probe emerging from the gripper amounts to about $H = 9.5 \, \mu \text{m} \pm 5 \, \mu \text{m}$, as demonstrated in figure 4(b).

While this gripper is designed for relatively thick (4 ~ 7 $\mu$m) non-contact AFM probes, rectangular AFM probes with a width $W \geq 25 \, \mu \text{m}$ and a thickness $t_{\text{cantilever}} \geq 1.0 \, \mu \text{m}$ are also applicable to this gripper. Of course, in this case, the thin probes will be clamped on their bottom surface.

### 3. Prototyping and characterization of the MEMS nanoindenter

#### 3.1. Prototyping

The MEMS force transducer numerically designed in section 2 was fabricated by means of deep reactive ion etching combined with a silicon-silicon bonding step (B-DRIE) [34]. By applying this B-DRIE technology, silicon micro-structures with an aspect ratio greater than 25 can be fabricated.

In this way, silicon structures with an out-of-plane thickness up to 75 $\mu \text{m}$ have been successfully obtained. In addition, this fabrication technique was found to be stable over time. Figure 5 shows a prototype of the MEMS nanoindenter and its typical packaging.

#### 3.2. Mechanical and electrical characterization

As indicated in equations (1) and (2), quantitative nanomechanical measurements require that the stiffness $k_{\text{MEMS}}$ and the displacement-to-capacitance sensitivity $\delta C/\delta z$ of the MEMS prototype be quantitatively calibrated.

An independently developed micro-force measurement system [35] has therefore been developed in which a closed-loop nanopositioning system (PIFOC) with sub-nanometer resolution is used to position and move the MEMS transducer, and in which a precision compensation balance (SE2, Sartorius Corporation) is utilized to measure the reaction force of the micro-transducer after engagement. Since the main shaft of the MEMS includes a passive holder, a conical stylus with a spherical tip of 5 $\mu \text{m}$ radius is mounted on the balance to probe the MEMS system, as shown in figure 6(a).

The capacitance variation of the MEMS transducer is read out directly by a commercial CDC IC (AD 7745/46/47, Analog Devices), which has a capacitance measurement range of up to 8 pF with a nominal resolution as low as 4 aF. Since the data update rate of this CDC can be up to 90 Hz, it is adequate for the quasi-static nanodimensional and nanomechanical measurements detailed in section 4.

After engaging with the stylus on the precision balance, the reaction force of a MEMS prototypes with respect to the movement of the piezo stage is measured by the balance. The force-displacement measurement results are presented in figure 6. The stiffness of the MEMS is evaluated from the measurement data to be $k_{\text{MEMS}} = (69.6 \pm 3.2) \, \text{N m}^{-1}$. The compliance of the stiffness measurement setup due to its stiffness of 26.1 $\text{kN m}^{-1}$ was corrected for, during evaluation. In addition, it can be seen from figure 6(b) that the MEMS transducer shows a nonlinearity of less than 0.08% for force sensing.
During the above contact-based MEMS stiffness calibration, the capacitance variation of the MEMS with respect to the piezo movements is also measured by means of the AD7745/46-based capacitive readout system; this variation is illustrated in figure 6(c), from which the real sensitivity of MEMS is found to be $S_{\text{MEMS}} = (645 \pm 15) \text{ aF nm}^{-1}$, coinciding well with the theoretical value in table 1. Furthermore, figure 6(c) shows that the nonlinearity of the MEMS
readout over the whole movement range amounts to less than 0.03 %, which also corresponds well to the specifications of
the CDC IC in use [36].

3.3. Mounting of AFM probes into a MEMS nanoindenter

To demonstrate the feasibility of the gripper design in sub-
section 2.2 for AFM probe holding, a simple assembly is set
up: a prototype of the MEMS nanoindenter facing upwards
is vertically fixed on a platform, and an AFM probe with the
tip pointing also upwards is horizontally mounted to a five-
axis micro-positioning stage, with which the cantilever beam
is adjusted to be perpendicular to the MEMS main shaft. By
means of a vision microscope, the AFM cantilever is inserted
into the passive gripper, and then broken by vertically moving
the substrate of the AFM probe. It should be pointed out that
the mounting of an AFM cantilever into the MEMS shaft is
not an easy operation without adequate exercises and skills. It
becomes especially difficult and damageable, when the stiff-
ness of the AFM probes to be mounted is much higher than
that of the MEMS.

As an example, figure 7(a) shows the top view of a silicon
AFM cantilever held in the MEMS gripper, and figure 7(b)
shows the sideview of the AFM probe.

4. Nanodimensional and nanomechanical
measurements using a MEMS nanoindenter with
different AFM probes

To experimentally investigate the capability of the MEMS
nanoindenter, which uses exchangeable AFM probes as an
indenter for material testing, a proof-of-principle experimental setup is constructed.

### 4.1 System configuration and characterization

As illustrated in figure 8, a three-axis micro-positioning stage equipped with stepper motors (NanoMAX 383, Thorlabs Inc.) is utilized to coarsely position the specimen under test, offering an x–y–z coarse positioning range up to 4 mm × 4 mm × 4 mm with a sub-micrometer resolution. A PZT-based nanopositioning stage (pico-stage) is used for fine positioning and engagement of the sample to the MEMS nanoindenter. This pico-stage provides in-plane (x–y) closed-loop movement with subnanometer resolution over the whole in-plane scanning range of 15 μm × 15 μm, and a vertical open-loop movement range of ±1 μm with sub-50 pm resolution [37]. To move the z-axis of this pico-stage and to acquire its capacitive displacement (analog) signal, a data acquisition system (NI-6259) is utilized, yielding a nominal z-axis sensitivity of about 10 pm mV⁻¹ for the pico-stage.

Finally, as mentioned in subsection 3.2, the z-displacement of the MEMS is directly measured by an AD 7745/46 CDC chip. The nominal resolution of this CDC chip of 4 aF indicates that the MEMS nanoindenter will have a nominal depth and force resolution of 6.5 pm and 0.5 nN, respectively. However, since the proof-of-principle setup works primarily under open-air conditions, the actual performance of the system has to be experimentally characterized.

For this purpose, the displacement readout of the MEMS nanoindenter is initially acquired without contact to any samples. It can be seen from figure 9(a) that the free-standing MEMS nanoindenter with a diamond-coated AFM probe (NC-LC, Adama Innovations, Ltd.) held in its gripper has a noise floor better than 4 pm sqrt(Hz)⁻¹ when the CDC date update rate of 13 Hz is used.

The MEMS nanoindenter is then engaged onto a sapphire sample mounted on the pico-stage with a displacement setpoint of 2 nm; the noise spectrum measured is also illustrated in figure 9(a) (in black), from which it can be seen that a noise floor of about 4 pm sqrt(Hz)⁻¹ can be achieved for frequencies f ≥ 1 Hz.

To further testify the measurement capability of the MEMS nanoindenter, the sapphire sample is moved stepwise with a nominal amplitude of 200 pm and a frequency of 0.1 Hz. Figure 9(b) demonstrates the MEMS readout, which proves that the MEMS nanoindenter within the proof-of-principle setup can have a quasi-static depth sensing resolution better than 60 pm (f ≥ 0.2 Hz), and a force sensing resolution of 3.7 nN in air.

Figure 7. Assembled AFM probe for nanoindentation testing.

Figure 8. Schematic diagram of the experimental setup for nanodimensional and nanomechanical characterization of nano-objects with the MEMS nanoindenter and its capacitive displacement sensing circuit.
4.2. Nanodimensional measurement capability

The ultra-high sensitivity of the MEMS nanoindenter in quasi-static measurements also allows this setup to be used in nanodimensional measurements of low-dimensional samples.

As an example, an atomic step height (ASH) sample made of Si < 111> that was developed at PTB in Braunschweig [38] is positioned on the pico-stage and scanned by the MEMS nanoindenter in an open loop mode. Figure 10(a) shows a microscopic image of the center part of the ASH sample with the regions of interest (ROI) where MEMS nanoindenter measurements are acquired. Figure 10(b) shows an in-situ image of the sample under the MEMS head obtained with a simple zoom microscope.

The typical surface topography of the ASH sample within the ROI and close to its structure edge measured by the MEMS Nanoindenter is illustrated in figure 10(c), and one of the line profiles is detailed in figure 10(d). It can be seen that an atomic step at \( x = 3.3 \) \( \mu m \) can be clearly revealed. Preliminary evaluation shows that the step height at \( x = 3.3 \) \( \mu m \) amounts to \( 0.30 \pm 0.05 \) nm, which is close to the reference values in air \( (0.313 \pm 0.015 \text{ nm, } k = 2) \) published in [38].

4.3. Nanomechanical measurement of soft and ultra-soft materials

The MEMS nanoindenter used in this study is specially designed to bridge the gap between the capabilities of nanoindentation instruments and those of AFMs.

4.3.1. Nanoindentation measurements of soft materials using a silicon AFM probe and an Oliver-Pharr data evaluation model. To demonstrate the capability of the MEMS nanoindenter to perform nanomechanical measurements of soft polymers, typical reference materials such as polycarbonate (PC), polyamide (PA) and low-density polyethylene (LDPE) [20] (delivered from Goodfellow) are measured by means of the experimental setup (shown in figure 8) using a clamped silicon AFM probe. In the preparation of these samples for the nanomechanical measurements with the MEMS nanoindenter, care has been taken to ensure that the sample surfaces should be normal to the test force direction of the MEMS nanoindenter with a misalignment less than \( \pm 1^\circ \).

To perform nanomechanical measurements, a sample is first engaged onto the silicon AFM probe and then withdrawn from the probe until a predefined distance is reached (e.g., \( h_0 = 50 \) nm). Then, a typical three-segmented depth-controlled nanoindentation procedure is followed: the pico-stage moves the sample at a constant speed within a time period \( t_{\text{unload}} \) towards the AFM probe until a predefined position \( z_{\max} \) is reached, holds the sample at \( z_{\max} \) for a duration \( t_{\text{hold}} \) and then brings the sample back to its original position \( z_0 \) within the duration \( t_{\text{unload}} \).

Figure 11(a) illustrates the typical force-depth curves obtained by the MEMS nanoindenter on a bulk PC sample, where \( t_{\text{unload}} = t_{\text{unload}} = 35 \) s, and \( t_{\text{hold}} = 50 \) s.

According to the Oliver-Pharr model [14, 15], the reduced elastic modulus \( E_r \) and the indentation hardness \( H_{IT} \) of the materials can be evaluated as follows:

\[
E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_p(h_c)}}, \tag{3}
\]

\[
H_{IT} = \frac{F_{\text{max}}}{\sqrt{A_p(h_c)}}, \tag{4}
\]

where \( S \) is the slope of the unloading curve at the peak force and \( A_p \) the projected tip area function of an indenter probe; the contact depth \( h_c \) is calculated as \( h_c = h_{\max} - \varepsilon F_{\text{max}}/S \). For the silicon AFM probes, \( \varepsilon \approx 0.73 \) [20].

The tip area function \( A_p \) of a silicon AFM probe in use is usually unknown. While the \( E_r \) of the reference PC has already been obtained by a commercial nanoindentation instrument, the contact area \( A_p \) of this silicon probe can be deduced as \( A_p = \pi/4 \cdot S^2/E_r^2 \). It is known that a fresh silicon AFM probe with sharp tip usually demonstrates unstable tip area function \( A_p \).
because of rapid tip wear. To suppress the influence of tip wear on nanomechanical measurements, in our practice, a fresh silicon tip held in the MEMS nanoindenter needs to firstly make about 150 indentations on the reference PC sample with a moderate indentation force so as to yield a relatively blunt but stable tip. Thereafter, a series of nanoindentation measurements with different \( h_{\text{max}} \) can be performed to evaluate the area function \( A_p \) of the tip.

Following the measurement data evaluation above, we obtain the contact area \( A_p \) of the silicon tip as shown in figure 11(b). Fitting the typical two-term AFM tip area function \( A_p = C_0 \cdot h_c^2 + C_1 \cdot h_c \) [20] to the measurement data, we obtain the area function of this silicon tip as \( A_p = 1.78 \cdot h_c^2 + 548.04 \cdot h_c \). It should be noted that the term \( C_0 = 1.78 \) indicates the semi-apex angle of the silicon tip \( \theta_{\text{tip}} = 37.0^\circ \).

Using equation (4), the hardness of PC is measured by the MEMS nanoindenter over the indentation depth \( h_c \); this hardness is illustrated in figure 11(c). The \( H_{IT} \) measured is found to be \( H_{IT} = (165 \pm 7) \) MPa for \( h_c > 250 \) nm, which coincides well with the reference value obtained by means of other nanoindentation instruments.

The measurement procedure and the data evaluation approach, which are similar, were used to obtain the mechanical properties of LDPE and PA. The measured elasticity and hardness values of these two materials for \( h_c > 250 \) nm are listed in table 2. In comparison with the reference values obtained from the commercial nanoindentation instrument, it can be seen that the measurement results obtained by the MEMS nanoindenter are reasonable.

4.3.2. Nanoindentation measurements of ultra-soft materials using an AFM sphere probe. To demonstrate the MEMS nanoindenter’s capability for performing nanomechanical measurements of ultra-soft materials, the proof-of-principle setup is employed to measure a PDMS (10:1) sample whose nominal elastic modulus is lower than 10 MPa (depending on the aging effect).

To simplify the data evaluation approach, an electron beam-induced deposition (EBID) carbon AFM probe with a spherical tip (biosphere B2000-FM) [39] is mounted in the MEMS nanoindenter, see figure 12(a). This nanoindentation AFM...
Figure 11. Nanomechanical measurement of polymer samples using the MEMS nanoindenter with a silicon AFM tip.

Table 2. Measurement results of PC, PA and LDPE obtained using the MEMS nanoindenter with a silicon tip (the \( E_r \) of PC is used as a reference for evaluating the tip area function, and the reference values are obtained by means of the Hysitron Triboindenter TI-950, a nanoindentation instrument, with a diamond Berkovich tip).

| Materials | Reference value | Measured value | Reference value | Measured value |
|-----------|-----------------|----------------|-----------------|----------------|
| PC        | 3.7 ± 0.4  \(^a\) | 184 ± 20       | 165 ± 7         |
| PA        | 2.3 ± 0.3      | 121 ± 25       | 85 ± 6          |
| LDPE      | 0.26 ± 0.06    | 25 ± 6         | 25 ± 6          |

\( E_r \) probe has a tip radius of 2 \( \mu \text{m} \) ± 10 nm (delivered by the product supplier). Figure 12(b) illustrates one of the typical indentation curves obtained for the PDMS sample, under the conditions of \( t_{\text{load}} = t_{\text{unload}} = 50 \) s and \( t_{\text{hold}} = 10 \) s.

According to literature [40, 41], the relaxation time for PDMS \( \tau_r \) is about 50 ms to 0.5 s; thus, it is reasonable to assume that it is not necessary to consider viscoelastic effects for the measurement data shown in figure 12(b), since the loading/unloading duration in our experiments \( t_{\text{load}} = t_{\text{unload}} \gg \tau_r \) Therefore, to extract the mechanical properties of PDMS from the measurement data, only standard analytical models for adhesive contact [42] have to be applied, e.g. JKR (Johnson-Kendal-Roberts) [43], DMT (Derjaguin-Muller-Toporov) [44].

Only standard JKR or DMT models are considered in this manuscript. To identify which model should be employed, the nondimensional Tabor parameter \( \mu \) [45] has been estimated, which is defined as

\[
\mu = \left( \frac{16R \cdot W}{9K^2 \cdot \epsilon_0} \right)^{1/3},
\]

where \( R \) is the tip radius, \( W \) the work of adhesion, \( K \) the reduced elastic modulus of the material under test, and \( \epsilon_0 \) the equilibrium separation of the tip and the surface (~0.3–0.5 nm). Empirically, the JKR model should be applied in order to process adhesive force-displacement curves when \( \mu > 5 \).

From the experimental data shown in figure 12(b), we obtain the Tabor parameter \( \mu \approx 300 \). Thus, the standard JKR model is employed to evaluate the unloading curves obtained in our experiments; this model is defined as follows [45–47]:

\[
h = \frac{a^2}{R} = \frac{4}{3} \sqrt{\frac{a \cdot F_{\text{PO}}}{R \cdot K}}
\]

\[
a^3 = \frac{R}{K} \left( \sqrt{F_{\text{PO}}} + \sqrt{F + F_{\text{PO}}} \right)^2,
\]
Figure 12. Nanomechanical measurement of PDMS using the MEMS nanoindenter with a sphere AFM probe.

\[ F_{PO} = \frac{3}{2} \pi W R, \quad (8) \]

where \( h \) is the indentation depth and \( a \) is the contact radius. For the measured indentation curve, the indentation depth \( h \) is defined as

\[ h = z - z_0 \quad (9) \]

where \( z_0 \) is the zero indentation point within the unloading curve [47].

By fitting the unloading data to the above model, equations (5)–(8), the zero indentation point \( z_0 \), the work of adhesion \( W \) and the reduced elastic modulus of the material under test \( E_{JKR} \) can be determined.

For the unloading curve in figure 12(b), the fitting result is also plotted, where \( z_0 = 245.5 \, \text{nm} \), \( K = 6.9 \, \text{MPa} \), and \( W = 77.3 \, \text{mJ m}^{-2} \). The elastic modulus \( E_{JKR} \) of PDMS is calculated as follows:

\[ E_{JKR} = \frac{3}{4(1 - \nu^2)} K, \quad (10) \]

where \( \nu \) is the Poisson’s ratio of the material. For PDMS material under a small strain, \( \nu = 0.5 \) [48].

A series of ultra-shallow nanoindentation tests on the PDMS sample have been performed. Using the data evaluation approach detailed above, the elastic modulus \( E_{JKR} \) with respect to the indentation depth \( h \) is shown in figure 12(c). The nearly identical results for two different loading/unloading rates proves that it is not necessary to consider viscoelastic effects for measurements with low loading/unloading rates.

5. Summary

To quantitatively determine the mechanical properties of compliant and ultra-compliant materials, an electrostatic MEMS-based nanomechanical measurement tester (or ‘MEMS nanoindenter’ for short) has been developed. The MEMS nanoindenter features an integrated passive gripper for holding commercial AFM probes as an indenter. The electrostatic force transducer of the MEMS nanoindenter has a maximum indentation depth of 9.5 \( \mu \text{m} \) with a maximum indentation force of greater than 600 \( \mu \text{N} \). The well-designed micro-force transducer allows commercial low-cost capacitive readout ICs to be directly used to detect the indentation depth with a depth noise floor of better than 4 \( \text{pm} \, \text{sqrt(Hz)}^{-1} \) in air, corresponding to a force noise floor of better than 0.3 \( \text{nN} \, \text{sqrt(Hz)}^{-1} \) in air.

Experimental investigations have verified that this MEMS nanoindenter has the following properties:
clamping of different types of commercially available AFM probes as indenters;

- performing nanodimensional measurements with atomic height resolution and some 10 nm lateral resolution;

- performing nanomechanical measurements of compliant and ultra-compliant materials with elastic moduli down to several MPa and offer reasonable measurement results.

Owing to its compact design and small size, it should be possible to integrate the MEMS nanoindenter presented in this manuscript into various AFMs, SEMs or TEMs for in-situ nanodimensional and nanomechanical measurements of micro- and nano-objects. Furthermore, with the supporting of SEM/TEM, the MEMS nanoindenter, is also able to provide a quantitative nanomechanical measurement platform for identification of critical issues in the field of AFM tip-surface interaction on an atomic and nanometer scale, e.g. silicon AFM tip deformation under low and moderate loads.

Acknowledgments

This research project is supported by the European Union and is funded within the scope of the European Metrology Programme for Innovation and Research (EMPIR) project titled ‘Metrology for length-scale engineering of materials (Strength-ABLE)’. The atomic step height samples in this programme for Innovation and Research (EMPIR) project and is funded within the scope of the European Metrology Organisation for Standardization (PTB) Braunschweig.

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