An *In-Situ*, Micro-Mechanical Setup with Accurate, Tri-Axial, Piezoelectric Force Sensing and Positioning

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Abstract

To enable accurate characterization of the mechanical behavior of materials at the micro-scale, e.g., in micro-systems, experiments are required that are representative of the actual (often multi-axial) loading conditions to which these materials are subjected during fabrication and operation. Equally important is the acquisition of mechanical data in the form of multi-axial force measurements, and the measurement of kinematics by in-situ microscopic techniques. To this end, a micro-mechanical testing rig is here realized using commercially available piezoelectric actuators. It is shown that the setup measures forces with a resolution of ~ 0.3 [mN] in the x - and y -directions, and ~ 50 [mN] in the z -direction, over a range of 5 [N], yielding a high dynamic (force) range. Furthermore, displacements can be imposed with a resolution of ~ 1 [nm] over a range of 200 [μ m], in all three directions (x , y , z). The setup is compact, vacuum compatible, and specimens are loaded on top of the setup so that the field of view is unobstructed, allowing for in-situ testing with optical and scanning electron microscopy, and optical profilometry. A generic method is developed for extracting quasi-static forces from the piezoelectric actuators. Furthermore, challenges raised from the use of commercial actuators, for which the public technical specifications are generally incomplete, are overcome and the solution strategy is described. Proof-of-concept experiments on flexible, organic, light-emitting diodes demonstrate the potential of the setup to provide rich micro-mechanical data in the form of tri-axial force and displacement measurements. The commercial availability of the piezoelectric actuators, combined with the proposed engineering solutions lead to a generally accessible micro-mechanical test setup to investigate small-scale specimens under realistic, multi-axial loading conditions.

Keywords Tri-axial force sensing · Micro-mechanics · *In-situ* testing · Piezoelectric actuators

Mechanical characterization of materials in microelectronics devices, e.g., light emitting diodes, computer chips, and solar cells, is challenging due to: (1) the associated

small-scale deformation, (2) the small, multi-directional forces at play within the material layers and their interfaces, and (3) the complex loading conditions to which these systems are subjected during fabrication and operation. To accurately characterize microelectronic materials, high-resolution measurement techniques in terms of displacements and forces are required. Conventional methods are based on experiments in which over-simplified sample geometries and loading conditions are applied, e.g., micro-beam bending or tensile testing of dedicated, micro-fabricated specimens [1–5]. This typically puts high demands on the fabrication processes of dedicated small-scale specimens, while introducing changes to the material behavior, e.g., due to processing-induced size effects [6, 7]. Moreover, these highly specific and simplified experiments do not represent the realistic conditions to which these material systems are subjected during fabrication and/or operation [8–10]. Therefore, to capture the realistic, multi-axial loading conditions in an experiment, while

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overcoming the above-mentioned challenges, a novel testing rig is here proposed. It is designed to mechanically test samples of actual devices, rather than dedicated specimens that are specifically fabricated for one idealized test-case.

Besides the necessity of imposing realistic loading conditions, a major challenge resides in the measurement of the mechanical quantities of interest, i.e., the forces and the displacements in all three directions. Multi-axial force transducers have been realized for, e.g., gripping and passive sensing applications (robotics) [11–13], in-situ scratch testing [14], and biaxial testing of relatively small, cruciform samples [15]. Although tri-axial load-cells are commercially accessible at the larger scale, multi-axial force measurement is not available for the purpose of in-situ, mechanical testing, where deformation must accurately be applied to a small-scale specimen, while measuring forces and imaging the deforming by high-magnification, microscopic techniques. The mechanical testing rig, presented in this article, is compact and vacuum compatible, and specimen loading is performed on top of the setup to realize an unobstructed field of view for in-situ optical and scanning electron microscopy. It consists of two adjacent, and oppositely positioned piezoelectric actuator stacks, enabling an autonomous actuation and force measurement along three perpendicular motion axes (x , y , z). The apparatus is described in “[Methodology](#)”, with a particular focus on the methodology proposed for the force measurement.

The piezoelectric actuators were commercially acquired, making this an economically attractive setup, which is easily accessible to the research community. However, the use of commercially available actuators also raises problems to solve. Indeed, the publicly available technical specifications of commercial actuators is generally incomplete, requiring an extensive characterization of the technical properties of the piezoelectric actuators, the procedure of which is described in “[Characterization of the Setup](#)”. The potential of the realized mechanical test setup is demonstrated by proof-of-concept experiments in “[Proof-of-Concept Measurements](#)”, and the findings are summarized in “[Conclusion](#)”.

Methodology

This section presents a method for adapting a commercial, piezoelectric actuator system, consisting of two piezoelectric actuator stacks, in order to realize a mechanical testing rig with accurate, quasi-static force measuring capabilities in three perpendicular, independent directions, without requiring additional load cells.

Experiment Setup

Two identical, commercially available, vacuum compatible, piezoelectric actuator stacks (manufacturer: Mad City Labs, model type: “nano-3D200”) are positioned oppositely to each other, see Fig. 1(b), and secured to a bottom plate. Each stack consists of three, independent, piezoelectric actuators in order to realize a displacement in three perpendicular directions (x , y , z), without cross-talk between the axes. The displacement of each stack’s platform is accomplished by applying an electric voltage to the piezoelectric element within each actuator (i.e., each independent axis), which thereby deforms mechanically due to the reversed piezoelectric effect. The resulting motion is guided and amplified by an unknown, internal, elastic flexure hinge system to realize a positional range of 200 [μm], and a positioning resolution of 1 [nm], in each direction. Each actuator can be controlled with or without a closed feedback control loop. Piezoresistive sensors measure the motion of each actuator, which is used in the feedback control loop for accurate positioning [16]. These motion sensors, intrinsic to the device, will be utilized for force measuring purposes, as explained in “[Force Measurement Principle](#)”. All experiments shown in this paper have been conducted on top of an anti-vibration table or on the sample platform of the scanning electron microscope (SEM), which also actively cancels out vibrations. No difference, regarding the noise levels, between the in-situ SEM and the ex-situ tests have been observed.

Fig. 1 (a) CAD drawing of a single, tri-axial, piezoelectric actuator stack, consisting of three independent actuators to realize a displacement in three perpendicular, independent directions. Two identical actuator stacks are oppositely positioned to realize a mechanical testing setup, shown in (b), which is used for in-situ testing with, e.g., (c) scanning electron microscopy (SEM)

The setup enables the deformation of small-scale microelectronic specimens between the two actuator stacks in a variety of ways, accommodating tension, compression, bending, shearing, and any arbitrary combination of these loading conditions. The multi-axial load application enables replication of complex conditions to which microelectronic devices are subjected during their fabrication and operation.

Force Measurement Principle

The electric charge generated by a piezoelectric element is proportional to the mechanical force acting on its free surfaces. The quasi-static force measurement, based on this piezoelectric effect, is known to be challenging due to free charge carriers drifting toward the dipoles under static stress in the piezoelectric crystal, leading to leakage of electric charge and current in the electronic circuit and causing significant drift of the measured force

quantity [17, 18]. Solutions proposed in the literature require detailed knowledge and precise manipulation of the system's electronic circuit, and/or of the environmental conditions (e.g., ambient temperature, humidity) to which the system is subjected during operation [18–21]. For off-the-shelf, commercially available piezoelectric actuators, this information is not readily available. However, this problem can be overcome by calibrating one piezoelectric stage against a second piezoelectric stage. Specifically, to measure quasi-static forces in the setup presented here, the embedded piezoresistive motion sensors are utilized, together with an algorithm to circumvent the drift effects. The method is demonstrated in Fig. 2 for an experiment on a linear elastic coil spring loaded along its axis in between the two piezoelectric actuators (x -axis). For each motion axis, the piezoelectric actuator of one of the two piezo-stacks is used to accurately apply deformation to the spring, in axial direction. A feedback control mechanism

Fig. 2 Illustration of the procedure (b)–(c) used to eliminate the noisy plateau regions of the piezoresistive displacement measurement by the actuator operating without a feedback control mechanism, i.e., the “open-loop” actuator (along the x -axis). Thereby, the displacement jumps (green lines in (c)), induced by the step-wise positioning (a) of the actuator operating with the feedback control mechanism, i.e., the “closed-loop” actuator, are recorded and accumulated (d). The force can be calculated from the accumulated open-loop displacement jumps when the system stiffness is known. Two regimes are indicated in the figures: (1) a slow increase of the displacement, followed by (2) a fast increase of the displacement

(see Fig. 2(a)) is thereby used, with a positioning resolution of approximately 1 [nm] (minor deviations may be caused by fluctuating environmental conditions, e.g., vibrations). This piezoelectric actuator operates in the closed-loop configuration, and is henceforth called the “closed-loop actuator”. The opposite piezoelectric actuator is used without the feedback control mechanism to measure the positional change of the actuator, again in axial direction of the coil spring, caused by the force acting on it (see Fig. 2(b)). This load-sensing piezoelectric actuator is henceforth called the “open-loop actuator”. To make the force measurement insensitive to drift, a step-wise positioning of the closed-loop actuator is required, while measuring the resulting positional change of the open-loop actuator. The closed-loop actuator applies a displacement step, and keeps the applied displacement constant for a short period of time (~ 50 [ms]). Because noise and drift affect the uncontrolled open-loop actuator, the constant displacement applied by the closed-loop actuator does not appear as a constant plateau in the open-loop actuator response. To filter out the step from the noise, the levels just before and after the step must be identified accurately, which is done by regression through the respective ranges of the open-loop actuator’s position data points, before and after the step. If the closed-loop actuator moves to a new position, the piezoresistive sensor of the open-loop actuator measures the open-loop actuator’s position and stores that positional value in an array. If the next closed-loop actuator’s position is the same as the previous position (when the closed-loop actuator is on a plateau), another open-loop actuator’s positional value is stored in the same array, and so forth. The next closed-loop actuator position that differs from the previous closed-loop actuator position indicates that the plateau region has come to an end, and a load-step has occurred. Regression is then conducted on the previously measured open-loop actuator’s position data stored in the array. Hence, there is a short time-offset between the step imposed by the closed-loop actuator and the measurement of the corresponding jump of the open-loop actuator, which equals the amount of time each plateau region lasts. The time-frame is adaptable and in this experiment, as mentioned before, that time-frame was set to 50 [ms]. It was found that a linear regression (first order fit) is a stable fitting method, which was the least susceptible to errors at the ends of the fitting range and, therefore, yields the most accurate extrapolation towards the moment of the step. This approach was found to work well when the load steps are temporally spaced close to each other. When slower loading rates are desired, i.e., loads must be kept constant for longer periods of time (in the order of seconds rather than milliseconds, as done here), it may be unavoidable to use higher order regressions. The linear

regressions are shown by the red fit-lines in Fig. 2(b) and (c)). This procedure effectively eliminates noise and drift effects that occur in the open-loop actuator measurements, in between the driving steps induced by the closed-loop actuator. The remaining displacement jumps (linking up the linear fits) of the open-loop actuator (see the green lines of Fig. 2(c)) are thereby effectively recorded and accumulated. The accumulated displacements jumps of the measuring open-loop actuator (see Fig. 2(d)) correspond linearly to the jumps in the axial force when the stiffness of the elastic hinge system of the actuator is known, the calibration of which will be discussed in “[Axial Machine Compliance and Internal Stiffness](#)”. The smaller loading steps in Fig. 2 demonstrate the capability of detecting small jumps, and recapturing the response of the linear elastic coil spring with similar accuracy as when larger, more easily detectable steps are imposed. The algorithm is applied on-the-fly, meaning that the force can be measured real-time during a mechanical experiment (i.e., the force is not post-processed), and the setup can operate autonomously, employing a digital input file for driving the closed-loop actuators of each loading axis (x , y , z). In this fashion, intermittent displacement control with quasi-static force measurement is possible. Force control, for mechanical creep experiments, and fully static force measurement, for relaxation experiments, are unfeasible, due to the long-term, nonlinear drift effects corrupting such measurements. As will be discussed in further detail in “[Characterization of the Setup](#)”, proper operation requires the calibration of the system’s characteristics, such as the stiffness of the elastic flexure hinges in all three directions, and the machine compliance.

Characterization of the Setup

Since the mechanical test setup consists of commercially acquired piezoelectric actuators, detailed technical specifications are not provided to the end user. Therefore, to measure force and displacement accurately, the technical specifications of the setup must be quantitatively assessed first.

This section discusses the assessment procedures and results: (1) the measurement of the axial, machine compliance, affecting the measurement accuracy of the displacement imposed on a test specimen, (2) the calibration of the internal stiffness of the elastic mechanism underlying each loading axis, affecting the force measurement accuracy of the open-loop piezoelectric actuators, (3) the cross-axial compliance that may corrupt the force measurement in the z -direction. Finally, after the setup has been assessed and calibrated, the actual force resolution for each loading axis is determined.

Axial Machine Compliance and Internal Stiffness

Because the testing rig has a finite stiffness, it will deform during a mechanical test. Consequently, accurate measurement of the relative clamp displacement requires knowledge of the machine compliance in each loading direction (x , y , z). The measured displacement of the closed-loop stage of each axis should be corrected in order to recover the actual relative displacement of the specimen clamps. Various factors may contribute to the machine compliance, e.g., the deformation of the elastic flexure hinge system, clearances in the setup, deformation of the machine frame, etc. It is impossible to quantify the individual contributions of such factors in a commercially acquired piezoelectric actuator. The objective here is therefore to quantify the total machine compliance, instead of identifying and characterizing the contributing factors individually.

To measure the machine compliance along each of the three axes, the two piezoelectric stacks are rigidly connected by a relatively thick steel plate, with a cross-sectional area of $30 \times 10 \text{ [mm}^2\text{]}$, which shows negligible deformation under the maximum load allowed by the system: 5 [N] (the manufacturer specifies a maximum load of 10 [N], but an extra safety factor of two is employed for all experiments in this article). For each loading axis (x , y , z), a calibration experiment is conducted,

where the displacement at the closed-loop actuator is prescribed, while the corresponding open-loop actuator's displacement is monitored. When the connection is indeed rigid, the displacement difference between the open-loop actuator and the closed-loop actuator represents the machine compliance Δ_{mc} , i.e., the displacement that is not transferred from the driven closed-loop actuator to the corresponding open-loop actuator. The results of the compliance calibration experiments for each axis are shown in Fig. 3(e), where U_{CL} and U_{OL} are the displacements of the closed-loop (driving) actuator and the open-loop (force measuring) actuator, respectively. The similarity between the axial compliance in the x - and y -directions, and the noticeably different axial compliance in the z -axis results from the likely equivalence in design of the former two stages, since they both drive in the horizontal plane. The z -stage, however, drives in the vertical, out-of-plane direction, which aligns with the direction of gravity, and therefore exhibits a different design than the x - and y -stages. It requires the internal flexure hinges to be designed with a higher stiffness that is needed to carry the combined weight of the x - and y -actuators, and the platform, compared to the x - and y -stages that move perpendicular to the direction of gravity. The solid lines represent linear regressions through the data, from which it is concluded that the machine compliance Δ_{mc} in each direction is linearly dependent

Fig. 3 (a) Photograph of the experiment with a rigid connection (rc) between the two actuator stacks for identifying the machine compliance for all three axes from the data in (e), providing the machine compliance factors C_{mc} . A coil spring (cs) with a stiffness of $K_{coil} = 1.1 \times 10^4 \text{ [Nm}^{-1}\text{]}$ is loaded along the (b) x -, (c) y -, and (d) z -axis for determining the corresponding internal stiffness K_{int} from the data in (f). To load the spring axially, yet trigger the internal stiffness of the y -axis of the setup (recall Fig. 1), the two actuator stacks are both rotated 90° so that their y -axis corresponds with the spring axis. Solid lines represent linear regressions through the data in figures (e)–(f)

Table 1 The machine compliance factor C_{mc} [-], and the internal stiffness $[Nm^{-1}]$ of each loading axis, based on linear regression on the data in Figs. 3(e) and (f), including the 95% confidence intervals of the identified parameters

Axis	x	y	z
C_{mc} [-]	12.42 ± 0.02	10.46 ± 0.02	33.64 ± 0.03
K_{int} $[Nm^{-1}]$	$(3.476 \pm 0.002) \times 10^5$	$(2.925 \pm 0.016) \times 10^5$	$(3.867 \pm 0.023) \times 10^5$

on the open-loop displacement U_{OL} in that direction, i.e.:

$$\Delta_{mc} = C_{mc} U_{OL}. \quad (1)$$

The constant, compliance factors C_{mc} , determined from the linear regressions of Fig. 3(e), are listed in Table 1. Equation (1) is used to compute the compliance C_{mc} , and thereby the relative clamp displacement U_{clamp} between the open-loop and closed-loop actuators of each axis, during a mechanical experiment:

$$U_{clamp} = U_{CL} - U_{OL} - \Delta_{mc}. \quad (2)$$

To convert the open-loop actuator's deformation response to a measured force, the internal stiffness K_{int} of the elastic hinge system of each piezoelectric actuator must first be identified. This is done by using a linear elastic coil spring with a known stiffness of $K_{coil} = 1.1 \times 10^4$ $[Nm^{-1}]$ as a reference specimen and applying deformation with the closed-loop actuator, while measuring the deformation response of the oppositely positioned open-loop actuator, as explained in “Force Measurement Principle”. The response of the open-loop actuator can now be translated into a force by the known stiffness of the coil spring and the measured relative clamp displacement of [equation (2)], for which the machine compliance factors C_{mc} are required:

$$F = K_{int} U_{OL} = K_{coil} U_{clamp}. \quad (3)$$

The results of the coil spring experiments are shown in Fig. 3(f). As expected, the relationship between the force in

the coil spring and the open-loop response of each actuator is linear (linear regressions are represented by solid lines in Fig. 3(f)). From the relation in equation (3) and the results of Fig. 3(f), the internal stiffness K_{int} along each axis (x , y , z) is identified, the values of which are listed in Table 1.

Cross-Axial Compliance

A different type of compliance may jeopardize force measurements in the z -direction. Because experiments are performed on top of the setup (to enable an unobstructed field of view for in-situ microscopy testing), the alignment with respect to the force measuring actuator in the z -direction is not optimal. When forces are applied in the x - and y -directions, a force will also be registered in the z -direction. The in-plane forces: F_x and F_y , induce a moment to the open-loop stage's platform, which thereby rotates around its x - and/or y -axis (see Fig. 1(a)). The piezoresistive displacement sensor of the z -axis, whose internal position is not disclosed by the manufacturer, measures a displacement u_z in the z -direction due to the platform's rotation, which does not represent a displacement caused by a force acting in the z -direction. The situation is illustrated in Fig. 4(a) and (b). The occurrence of this rotation is verified by optical profilometry measurements of a mirror surface, with a sub-nanometer roughness profile, placed on the stage's platform. The profilometry measurements before and after application of forces of 5 [N] in the x - and y -directions, reveal an angular rotation of 0.17°

Fig. 4 (a)–(b) An illustration of the 0.17° angular rotation caused by in-plane forces F_x and/or F_y , resulting in a measured displacement u_z , and (c) the optical profilometry measurements of the stage's platform, verifying its rotation as a result of the applied forces F_x and F_y with a magnitude of 5 N

of the platform around the x - and y -axes. An illustration of this cross-axial coupling and the optical profilometry measurements are shown Fig. 4.

To quantify the cross-axial compliance that affects the measurement of the force in the z -direction, an additional experiment is conducted with a rigid connection between the platforms of the two stages. The connection is the same as the one used for the machine compliance measurements of “Axial Machine Compliance and Internal Stiffness”, Fig. 3(a). Both x - and y -axes are driven in compression and tension directions, across their full ranges and in a scanning manner, while the forces in all directions are recorded. Because of the rigid connection between the stages’ platforms, any force measured in the z -direction should be considered as artificial. The artificial force $F_{z,a}$ is thus a function of the forces F_x and F_y , and its relation appears to be linear, as shown in Fig. 5, i.e., it can be expressed as:

$$F_{z,a} = C_{z,x}F_x + C_{z,y}F_y, \quad (4)$$

where $C_{z,x}$ and $C_{z,y}$ are called the *cross-compliance* factors. The two cross-compliance factors can be straightforwardly characterized from a linear, planar regression through the measured data points of Fig. 5(a). In order to investigate a

potential dependence on the positive and negative regimes of the applied forces F_x and F_y , four planar regressions are made: one in each quartile Q_{1-4} of positive and negative combinations of F_x and F_y . Moreover, the test has been repeated three times over a period of 1 hour, and with 15 minutes intervals in between each test. While the first and third test are performed under equal conditions, the second test is conducted with an added force of 1 [N] in the z -direction, induced by a displacement step of the closed-loop actuator of the z -axis. This was done to investigate the dependence of the cross-compliance on the applied force in the z -direction. The four quartiles and the three tests yield a total of twelve test-cases for the linear regressions. The identified parameters, i.e., the cross-compliance factors, for each test-case are shown in Fig. 5(b) and (c). The spread on the data in Fig. 5(b) and (c) is mainly governed by the quartile location (distinguished by the colors of the data points), and less by the different tests (distinguished by the marker symbols of the data points). The dependence of the cross-compliance factors on the four quartiles suggests that a linear, planar regression on all data points is inadequate, since it does not properly capture the dependence of $F_{z,a}$ upon F_x and F_y . Accordingly, [Eq. 4] should be extended. To this end, polynomial regressions of different orders are

Fig. 5 (a) The artificial force $F_{z,a}$ in the z -direction is caused by the forces F_x and F_y . Separate linear regressions are made on the data in the quartiles Q1, Q2, Q3, and Q4, and for three separate tests, to determine cross-compliance factors $C_{z,x}$ and $C_{z,y}$ for all twelve test-cases, the values of which are graphically shown in (b) and (c), respectively

Fig. 6 (a) Measurement of the artificial force in the z -direction (blue data points). A second order polynomial regression is made on the data points, and used to determine the cross-axial compliance factors, which is subsequently used to correct for the artificial force $F_{z,a}$, as is illustrated by the red data points. (b) RMSD vs. order of polynomial regression

made on all data points of all three tests combined. The root-mean-square of deviation (RMSD) between regressions and the measured data points is plotted against the polynomial order of the regressions, as shown in Fig. 6(b).

It is clear that the linear regression is significantly less adequate than the higher order regressions. A second order regression polynomial yields an improvement of 34% with respect to the linear one. Higher (than second) order polynomial regression introduces higher order fluctuations, due to non-physical measurement noise, while not reducing the RMSD significantly with respect to the second order polynomial regression. Hence, a second order polynomial regression is adopted and the artificial force $F_{z,a}$ is written as:

$$F_{z,a} = C_{z,x}F_x + C_{z,y}F_y + C_{z,xx}F_x^2 + C_{z,xy}F_xF_y + C_{z,yy}F_y^2, \quad (5)$$

where $C_{z,i}$ are the cross-axial compliance factors as determined from the second order polynomial fit through the data from the calibration experiment, as shown in Fig. 6(a) and listed in Table 2. Equation (5) is used to correct the force extracted from the open-loop displacement $U_{OL,z}$ as measured by the open-loop actuator in the z -axis:

$$F_z = K_{int,z}U_{OL,z} - F_{z,a}, \quad (6)$$

where $K_{int,z}$ is the stiffness of the respective actuator system (see Table 1). For the calibration experiment, the measured

z -force is thereby reduced to nearly zero, as shown by the red data points in Fig. 6(a).

In spite of this correction, the artificial force may still negatively affect the resolution of the force measurement in the z -direction, as will be shown in the next section. Besides the artificial force, a bending moment may be applied to the specimen due to the rotation of the platform. However, it is small (compared to typical bending moments caused by alignment errors in regular macroscopic tensile tests), because the angular rotation was measured to remain below 0.17° at the maximum force of 5 [N], while approximately scaling down linearly with lower applied forces. Since the bending moment is small, it has not been further characterized.

It was also verified whether artificial forces also occur in the x - and y -directions, for which a displacement is imposed along each of the two other axes, while measuring the corresponding forces by the open-loop actuators (not shown). To this end, the in-plane displacements, measured by the piezoresistive motion sensors of the open-loop x - and y -actuators (used for force measurements) were also measured by 2D digital image correlation on images of the moving stage platform. It was found that the x - and y -displacement of the platform correspond to that of the piezoresistive sensors within measurement accuracy. Therefore, it can be concluded that the cross-axial machine compliance can be neglected for the x - and y -force measurements.

Table 2 The cross-axial compliance factors $C_{z,i}$ [-] with the 95% confidence intervals, as determined from a second order polynomial regression through the calibration data of Fig. 6(a)

$C_{z,x}$	$C_{z,y}$	$C_{z,xx}$	$C_{z,xy}$	$C_{z,yy}$
-0.4222 ± 0.0027	-1.7920 ± 0.0040	-0.0136 ± 0.0015	-0.0286 ± 0.0030	-0.0393 ± 0.0031

Fig. 7 Linear regressions on the force-displacement curves from uni-axial experiments of linear elastic coil spring specimens loaded in three different directions. The root-mean-square deviation with respect to the regressions reveals the force measurement resolution for each axis

Force Resolution

To quantify the force resolution of the calibrated system, a coil spring specimen, with a (very low) stiffness of $125 \text{ [Nm}^{-1}\text{]}$, is elongated by $200 \text{ [}\mu\text{m]}$ by the closed-loop actuator, while the force is measured by the open-loop actuator. The measurement is conducted separately for each direction, where the spring is loaded along its axis, equivalent to the test configurations shown in Fig. 3(b)–(d). The resulting force-displacement curves are shown in Fig. 7. The linear regressions verify the ability of the measurement algorithm to capture the linear elastic behavior of the coil spring.

The root-mean-square deviation (RSMD) with respect to the linear regression represents the resolution of the force measurement by the open-loop actuator of each axis. The force resolution values are listed in Table 3, while the maximum allowed force for these piezoelectric actuators is 5 [N] . However, for the z -direction, the RSMD of the second order polynomial regression through the artificial z -force data in Fig. 6 is two orders of magnitude larger than the RMSD of the regression from Fig. 7. Here, the cross-compliance will still corrupt the force resolution in the z -direction (if x - and y -loads are present). Therefore, the RSMD of the artificial z -force regression is taken as a measure for the resolution instead, as seen in Table 3. Since the load bearing capacity of the actuators is here limited to

Table 3 The force measurement resolution in $[\text{N}]$, for open-loop actuator of each axis, which should be compared to the load range of 5 [N] , yielding the dynamic range

Axis	x	y	z
Resolution $[\text{N}]$	1.8×10^{-4}	2.8×10^{-4}	7.0×10^{-2}
Dynamic range $[-]$	2.8×10^4	1.8×10^4	7.1×10^1

5 [N] , the dynamic range, defined as the ratio between the maximum load and the force resolution, is particularly high for the x - and y -directions.

Proof-of-Concept Measurements

First, a three-point-bending experiment is described in “[Three-Point Bending Experiment](#)”, characterizing a well-defined spring steel rod specimen. The potential of the setup is further demonstrated with several other proof-of-concept experiments in “[Organic Light-Emitting Diode](#)”.

Three-Point Bending Experiment

A three-point bending experiment is conducted on a cylindrical, spring steel rod. The results are validated by analytic calculation, making use of the Euler-Bernoulli beam theory. Since special grippers are used for this experiment, the previously measured machine compliance in the x -direction (direction of bending) may be incomplete. Therefore, the total machine compliance in x -direction is measured again for the system, now including the specific grippers: $C_{mc,x} = 18.738 \pm 0.02 \text{ [-]}$.

The cylindrical rod specimen has a length of $L = 4.07 \pm 0.006 \text{ [mm]}$ (measured optically between the outer loading points), and a diameter of $D = 400 \pm 1 \text{ [}\mu\text{m]}$. The force F is measured in the x -direction, along which the bending deformation is applied, by the corresponding open-loop actuator, see Fig. 8.

The relative displacement of the two piezoelectric actuators represents the deflection of the bending rod. Using the Euler-Bernoulli equation for this problem, the maximum deflection w at $x = \frac{1}{2}L$ is:

$$w|_{x=L/2} = \frac{FL^3}{48EI}, \quad (7)$$

where E is Young’s modulus and I is the second moment of area of this cylindrical specimen. Using Young’s modulus as the unknown material parameter, applying equation (7) on the experimental data allows to identify the Young’s modulus as: $211 \pm 5 \text{ [GPa]}$, which is in agreement with the expected Young’s modulus of the spring steel: 210 [GPa] . The error bounds on the computed value of the Young’s modulus are determined from the error margins on the variables of equation (7), as specified throughout this article. This experiment demonstrates that a setup based on two commercially available piezoelectric actuator stacks can indeed be used to perform highly accurate mechanical tests on small-scale specimens.

Fig. 8 Micrographs of the bending experiment: (a) zoomed-out image of the clamped specimen, (b) zoomed-in image of the bending specimen. (c) Force versus relative clamp displacement in the drive direction x , as measured by the piezo testing rig

Organic Light-Emitting Diode

Samples from a flexible sheet of an organic light-emitting diode (OLED) are mechanically deformed to demonstrate the potential of the setup for tri-axial, micro-mechanical testing. The samples consist of a polyethylene naphthalate (PEN) substrate with three moisture barrier layers, each consisting of an acrylate (organic) coating for planarization (OCP) [22, 23], and a 150 ± 1 [nm] thick Si_3N_4 -coating. The thickness of the PEN substrate is $H = 160 \pm 1$ [μm], and the total thickness of all three (equally thick) OCP-layers is $h = 75 \pm 1$ [μm]. The specimens are cut to a size of 7 ± 0.01 [mm] in length and 4 ± 0.01 [mm] in width, by a thin razor blade with a thickness of ~ 10 [μm]. To facilitate viewing and recording of the deformation of the specimen over its cross-section and along its length-direction by optical microscopy, it is ground by using consecutively finer sanding paper, with ~ 5.5 [μm] particles for the finest step. Loctite universal super glue is used to glue the specimen to the side planes of the grippers of the mechanical testing rig. At the side of the substrate, the specimen is adhered over the full length and width of the specimen. At the side of the OCP-layers, the specimen is adhered partially over a length of ~ 2 mm and the full width of the specimen. A schematic illustration of the OLED specimen and the locations at which it is loaded by the mechanical testing rig (the boundary conditions), is shown in Fig. 9.

Four in-situ experiments are conducted in which interface delamination between the OCP-layers is triggered under different loading modes: (a) normal opening by driving the stage 100 [μm] along the x -axis, (b) in-plane shear opening by driving the stage 100 [μm] along the y -axis, (c) out-of-plane tearing by driving the stage 100 [μm] along the z -axis, and (d) a combination of the former loading modes, with 100 [μm] along x , and 50 [μm] along y and z , consecutively imposed in that order. In each of the four

mechanical tests interface crack propagation was observed and the delamination was induced in the OCP-stack between the first and second OCP-layers (counting from the substrate), as illustrated in Fig. 9(b). Before the specimens were loaded, a pre-crack was first produced by loading the specimen in the x -direction, while observing the force evolution. The moment when the force in the x -direction was observed to suddenly drop, the loading was stopped. The result of this loading path is a pre-crack of ~ 2 [mm], spanning the length of the specimen over which the right-hand boundary condition (glue) is applied, see Fig. 9(a). The pre-cracked specimen was first unloaded before being reloaded in one of the four experiments (a)-(d).

The forces in all directions were recorded during the four experiments, see Fig. 10, together with several micrographs of the deformation which occurred during the experiments.

The progressive increase of the x -force in the normal opening test-case of Fig. 10(a) can be explained by the fact that the delaminating layers are stretched as the experiment

Fig. 9 Illustration of the OLED specimen that corresponds to the microscopic point of view, where opening between the layers can be visualized. The specimen is glued to the grippers of the mechanical testing rig, at the schematically indicated locations. (b) After loading in, e.g., horizontal direction, delamination is triggered between the OCP-layers. $H = 160 \pm 1$ [μm], $h = 75 \pm 1$ [μm]

Fig. 10 Force evolution in all directions (x , y , z), as measured by the open-loop actuators of the mechanical setup during in-situ tests under (a) normal opening, (b) in-plane shearing, (c) out-of-plane tearing, and (d) a combination of loading modes, consecutively imposed in the order as mentioned above. The induced delamination is visualized by (e) optical microscopy, (f) scanning electron microscopy (SEM), of which a photograph of the setup inside the vacuum chamber is seen in (g), and optical profilometry (h)

progresses and the interface crack propagates. Interfacial failure is visualized by optical microscopy, as shown in Fig. 10(e), and more detailed visual information of, e.g., the crack-tip is captured by scanning electron microscopy, as seen in Fig. 10(f). The induced delamination is not purely mode-I, thereby preventing the measured force to reach a plateau or decrease, as is typically observed for pure mode-I loading conditions. For the in-plane shearing and the out-of-plane tearing test-cases, no significant decrease in the force slopes is observed at all, yet crack propagation, and thus delamination, is clearly observed in both experiments. The specimen of the tearing test-case shows crack propagation along the length of the interface. By using surface metrology techniques, such as optical profilometry, the out-of-plane delamination of OCP-layers subjected to tearing, can be quantitatively visualized, see Fig. 10(h).

The specimen processing method, prescribed above, does not produce specimens with a consistent, reproducible pre-crack length, and therefore does not allow for comparison of the results between different samples. The experiments only serve to demonstrate the tri-axial capabilities of the device. The setup's in-situ microscopic testing capabilities assist in acquiring high resolution images of the underlying microscopic (failure) mechanisms. Such experimental data facilitate the identification and validation of small-scale mechanical models. The tri-axial force measurements, in combination with high resolution microscopy and computational modeling tools, e.g., the finite element method, provide rich mechanical data and allow for in-depth investigation of realistic, three-dimensional mechanics, which would be impossible with conventional, uni-axial mechanical experiments, such as bending or tensile tests.

Conclusion

Understanding the intricate, mechanical behavior of densely stacked materials, as used in, e.g., microelectronic applications, requires mechanical experiments that mimic the realistic setting in which these material systems are fabricated and used. This article presents a versatile experimental approach to measure quasi-static, tri-axial forces using commercially available piezoelectric actuators. A tri-axial, micro-mechanical testing rig with sub-millinewton force sensing resolution, a high dynamic (force) range, and nanometer positioning resolution has been realized.

The leakage of charge in piezoelectric materials poses a major challenge in measuring quasi-static forces by the piezoelectric effect. The generic solution, proposed in this work, relies on the use of two piezoelectric actuators for each loading axis (x , y , z) of the mechanical testing rig. One actuator is used with closed-loop feedback control to impose mechanical deformation to a specimen in a step-wise manner. The other actuator is used without the feedback control loop to measure the force in the specimen. Because of the step-wise method for driving the closed-loop actuators, the corrupting drift effects, resulting from charge leakage, can be eliminated.

The use of commercial actuators makes the device accessible, but also presents particular challenges, since the design details are not disclosed by the manufacturer. This problem is overcome through a series of dedicated calibration experiments, which characterize the device's technical properties, i.e., the internal stiffness, and the axial and cross-axial machine compliance. To demonstrate that the calibration tests are truly representative of multi-axial

loading conditions, the calibrations must be done over the whole range of loads and cross-loads that the stage can apply in all three directions.

This work resulted in an accurate, in-situ, tri-axial, mechanical testing rig with a relatively simple design, and constructed from commercially available components, which makes it highly accessible for academic research or industrial investigatory applications. At present there is no equivalent instrument offering these three-axes capabilities with these specifications. A shortcoming of the setup is, however, its incapability of performing mechanical creep or relaxation experiments. The setup demonstrates its potential for conducting realistic, micro-mechanical, quasi-static experiments. The combination of tri-axial displacement control and measurement, simultaneous tri-axial force measurement, versatile load path application, and in-situ microscopy testing, enables state-of-the-art and future experimental mechanics, whereby the focus lies more and more on investigating and characterizing the three-dimensional, micro-mechanical behavior of materials.

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Appendix: Motion Verification Under Load

In order to verify the closed-loop mechanism and its performance under load, a total of eight experiments have been conducted. One of the actuators has been commanded to move from 0 to 200 [μm] with 20 [μm] steps, while optical micrographs have been captured of the moving platform, which are then used in a digital image correlation (DIC) procedure to measure the displacement of the platform (independently from the internal piezoresistive position sensor of the actuator). A global DIC scheme [24], employing polynomial basis functions of zeroth order, was used to capture the rigid body motion of the stage's platform. This experiment was first performed without any load acting on the platform, followed by three tests

Fig. 11 Photographs of the setup in which a static load of 4 [N] is applied by (a) hanging a mass by a string over a pulley for the x -, y -directions. (b) For the z -direction, the mass is placed on top of the stage's platform

that include a static load of 4 [N], applied by a mass acting in the x , y , and the z -directions (in separate experiments), as shown in Fig. 11. These four experiments have been performed while the actuator operated in the closed-loop (CL) configuration, and, once again, while the actuator operated in the open-loop (OL) configuration, thus yielding a total of eight experiments. The results are represented in Fig. 12 and clearly show that in the closed-loop configuration the actuator moves to the commanded position, regardless of the load (in any direction) acting upon the platform. The linear regression through the closed-loop data points highlights the actuation accuracy of the

Fig. 12 The positioning performance of the actuator when operated in the closed-loop (CL) and the open-loop (OL) configurations, under loads of 4 [N] in different directions (tests without a load are labeled “free” in the legend). The error bars represent the uncertainty estimate for the DIC measurement which originate from: erroneous pixel size assumptions, optical lens distortions, interpolation errors within the DIC algorithm, image noise. The linear regression through the closed-loop data points highlights the actuation accuracy of the actuator when operated in the closed-loop configuration

actuator when operated in the closed-loop configuration. It is also clear that in the open-loop configuration the load has no significant effect. As expected, the actuation is generally inaccurate in the open-loop configuration, due to the absence of a closed-loop feedback control mechanism. The open-loop configuration is therefore not used for driving an experiment, but is only used for measuring forces. The closed-loop configuration is used for driving the experiment, as the actuator is then most accurate and unaffected by forces acting upon the stage.

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