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Traceable Micro-Force Sensor for Instrumented Indentation Calibration

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ABSTRACT

Instrumented indentation testing (IIT), commonly referred to as nanoindentation when small forces are used, is a popular technique for determining the mechanical properties of small volumes of material. Sample preparation is relatively easy, usually requiring only that a smooth surface of the material to be tested be accessible to a contact probe, and instruments that combine sophisticated automation with straightforward user interfaces are available commercially from several manufacturers. In addition, documentary standards are now becoming available from both the International Standards Organization (ISO 14577) and ASTM International (E28 WK382) that define test methods and standard practices for IIT, and will allow the technique to be used to produce material property data that can be used in product specifications. These standards also define the required level of accuracy of the force data produced by IIT instruments, as well as methods to verify that accuracy. For forces below 10 mN, these requirements can be difficult to meet, particularly for instrument owners who need to verify the performance of their instrument as it is installed at their site.

In this paper, we describe the development, performance and application of an SI-traceable force sensor system for potential use in the field calibration of commercial IIT instruments. The force sensor itself, based on an elastically deforming capacitance gauge, is small enough to mount in a commercial instrument as if it were a test specimen, and is used in conjunction with an ultra-high accuracy capacitance bridge. The sensor system is calibrated with NIST-traceable masses over the range 5.0 µN through 5.0 mN. We will present data on its accuracy and precision, as well its potential application to the verification of force in commercial instrumented indentation instruments.

INTRODUCTION

Small-scale mechanical devices are becoming ever more ubiquitous in our lives, particularly in digital systems, where hard-disk drives continue to shrink and where arrays of more than one million tilting micro-mirrors form the heart of popular digital light projection (DLP) systems. Even in multilayer electronic circuits that have no specific mechanical application, knowledge of the mechanical properties of the various materials and thin films from which they are built is a key to understanding their reliability, as differential stresses can lead to premature failures, particularly at interfaces.

Low-force instrumented indentation testing (IIT), or nanoindentation, has become a popular method of determining key mechanical properties – particularly hardness and elastic modulus - of thin films and other small volumes of material[1]. Commercially produced instruments for this purpose are available from several manufacturers and are in widespread use around the world. However, the mechanical properties data these instruments generate is only as good as the calibration of the force and displacement transducers they incorporate. A recently approved ISO standard[2] now lays out force and displacement calibration requirements for IIT instruments, and a related ASTM document[3] is close to being approved. These requirements can be difficult to meet, particularly for instrument owners who need to verify the calibration of their instruments at their site.

In this paper, we describe recent work in the characterization of a small capacitive force transducer that may be suitable for use as a force calibration device, traceable to the International System of Units (SI), in the force range from 5 μN to 5 mN. The transducer's small size allows it to be easily mounted in most, if not all commercial instrumented indenters, as if it were a specimen to be tested. The indenter system can then apply forces, and those forces can be compared to transducer's output. We characterize the sensor's accuracy and reproducibility, and use it in an indentation system to investigate issues related to its use as a force transfer standard[4], such as its sensitivity to off-axis loading.

DESCRIPTION OF FORCE SENSOR AND ELECTRONICS

The force sensor studied in this work was an elastically deformable capacitance transducer consisting of an electrically conductive capacitor plate constrained by a spring suspension to move linearly between two fixed, parallel conducting plates. Dimensions of the transducer are 12 mm by 12 mm by 4 mm high, with motion in the vertical (4 mm) direction. The spring suspension has a nominal stiffness of 200 N/m, and allows travel of approximately 15 micrometers above and below the center position. Two transducers were purchased from their manufacturer. One was studied extensively, with the results reported here. The second received more limited testing, but its performance appeared to be fully comparable to the first in all tests performed on it.

The moving capacitor plate had a hole at its center, aligned with larger holes in the top and bottom fixed plates; this allowed for the mounting of an electrically insulating Delrin® post that extended up through holes in both the top capacitor plate and a grounded brass enclosure that was constructed to shield the sensor from stray electric fields. The post, 5 mm in diameter at the top, was designed with a recessed square glass loading area, 1.5 mm x 1.5 mm, centered on the axis of the post, as well as a v-notch cut into the outer perimeter of the post to facilitate the application of cylindrical wire deadweight masses during calibration. The sensor and post are shown schematically in Figure 1.

In both the calibration of the sensor and the use of the sensor in measuring force in an instrumented indenter, the data collected consisted of the measured change (increase) in capacitance between the sensor's moving plate and fixed lower plate with applied mass or force. The upper plate was not used, and was always held at ground potential. Nominal sensor capacitance, when loaded only by the weight of the post, was 6.0 pF. During all experiments, the capacitance was measured with an Andeen-Hagerling 2500-A capacitance bridge ("AH bridge"). This bridge makes a three-terminal apacitance measurement with a resolution of 0.5×10^{-6} pF (0.5 attofarads) for capacitance values of 10 pF or less, and contains an internal reference capacitance that is held at constant temperature. The stability of the bridge was checked using an external NIST silica reference capacitor of nominal capacitance 10 pF. The bridge read 10.000270 pF +/- 0.000010 pF for room temperatures spanning the range 22.5 °C to 25.0 °C, well within the stability requirements of the current work. AH bridge readings were logged by computer via a GPIB interface.

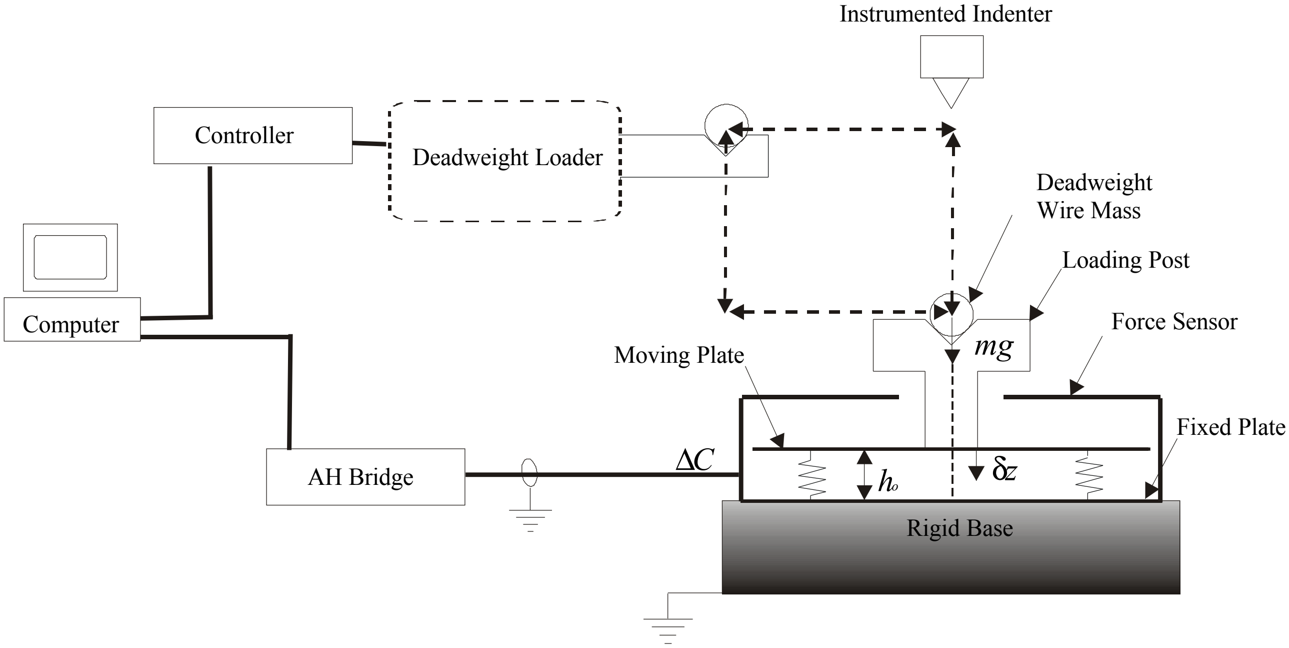

Figure 1. Schematic representation of the force sensor calibration and subsequent transfer to the instrumented indentation machine. The sensor is calibrated using deadweight forces applied by an automated loading system, and is then used to record force applied by an instrumented indentation instrument.

SENSOR CALIBRATION

The sensor was calibrated using a set of stainless steel wire deadweight masses prepared in our laboratory. Masses ranged from 0.5 mg to 500 mg, resulting in nominal loading forces from 5 µN to 5 mN. The deadweights were ultrasonically cleaned, and were weighed on an electronic balance with a calibration that was verified with NIST-traceable mass sets. The masses used, with their absolute uncertainties, are shown in Table 1. Also shown is the resulting force each applies, with its relative uncertainty, obtained using a value of *g*, the acceleration due to gravity, of $g = 9.801033\ \mathrm{m/s^2}$ +/- $0.000004\ \mathrm{m/s^2}$ measured in the room where the calibrations were performed. Deadweights were applied and removed from the v-notch in the sensor loading post using an automated turret system, as show schematically in Figure 1 and described elsewhere[5]. Each mass was applied and removed ten times, and multiple capacitance readings were taken and averaged for each application. The sensor's baseline capacitance values were measured before and after each deadweight application, and each change in capacitance was measured relative to those baseline observations.

Table 1. Masses and uncertainties of stainless steel wire deadweights used in the calibration of the force sensor, as well as the observed change in capacitance resulting from the application of each mass.

Nominal Mass (mg)	Actual Mass (mg)	Mass Uncertainty (mg)	Actual Force (μN)	Force Uncertainty (%)	ΔC (pF)	ΔC Uncertainty (%)
0.5	0.50707	0.00060	4.96981	0.12	0.001653	0.24
1	1.04196	0.00041	10.2123	0.039	0.003291	0.12
2	1.98956	0.00071	19.4997	0.036	0.006473	0.046
3	3.08438	0.00106	30.2301	0.034	0.009853	0.041
4	4.04711	0.00077	39.6659	0.019	0.012963	0.039
5	5.04575	0.00067	49.4536	0.013	0.016414	0.024
10	10.0345	0.00071	98.3485	0.0071	0.033434	0.015
20	20.01974	0.00077	196.214	0.0038	0.067078	0.0060
30	29.96073	0.01069	293.646	0.037	0.100993	0.0050
40	39.96126	0.00054	391.662	0.0014	0.135449	0.0074
50	49.98958	0.00070	489.950	0.0014	0.171906	0.0041
100	99.70782	0.00068	977.240	0.00068	0.353855	0.0028
200	200.01729	0.00180	1960.376	0.00090	0.758260	0.0024
300	299.93127	0.00116	2939.636	0.00039	1.220238	0.0014
400	399.96515	0.00123	3920.072	0.00031	1.755643	0.0014
500	500.74129	0.00055	4907.782	0.00011	2.390454	0.00092

The change in sensor capacitance resulting from the deadweight loadings, with the relative uncertainty, is shown in Table 1 and plotted in Figure 2a. Because the sensor was later used to measure applied forces, the axes have been switched to show force, F, as a function of capacitance change, ΔC. It was found that $F(\Delta C)$ could be well-fit over individual decades by general cubic polynomials of the form $F(\Delta C) = A_3\Delta C^3 + A_2\Delta C^2 + A_1\Delta C + A_0$. The residuals to those fits are shown in Figure 2b.

Sensor calibration was also checked by rotating the sensor +/- 120 degrees in the horizontal plane, relative to the orientation used for the primary calibration, a procedure that parallels that specified in ASTM Standard E 74 for higher-force measuring instruments[6]. Resulting data for one rotated orientation agreed with the primary calibration within 0.0156% or better at all forces tested. Force readings from the third orientation, however, ranged between 0.71 % and 0.80 % higher than the primary calibration values. Rotation of the sensor required rotation of the Delrin® loading post such that the v-notch remained aligned with the wire masses as applied by the turret system. This rotation may have slightly changed the alignment between the post and the capacitor center plate to which it is mounted, and the practice of rotating the post between calibrations was discontinued.

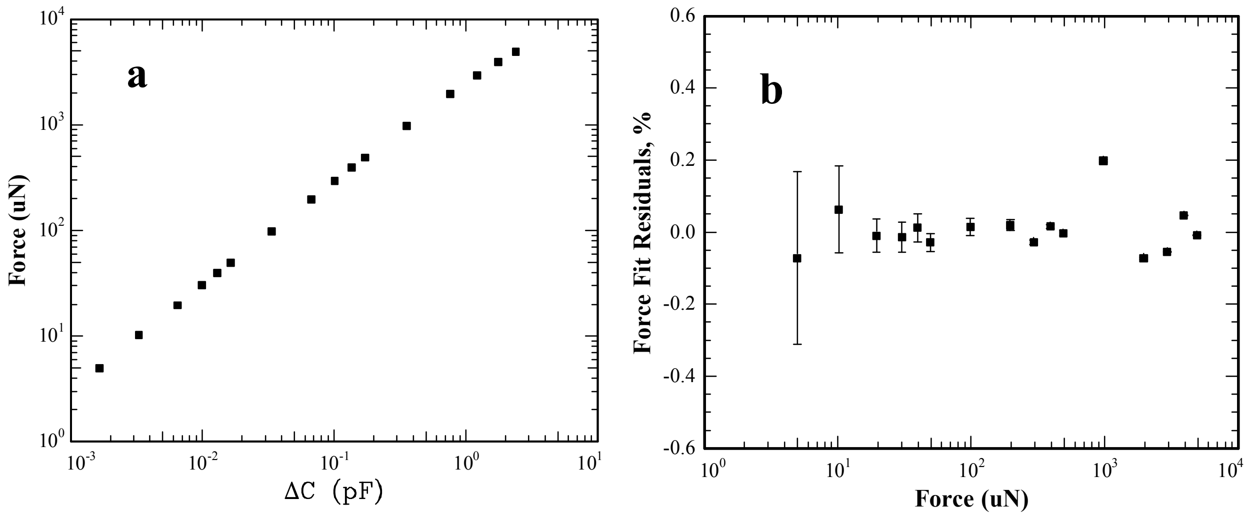

Figure 2. a) Change in sensor capacitance, in picofarads, vs applied force, measured from 5 μN to 5 mN. b) Residuals in force from cubic fits to the data in a), expressed as a percentage of each applied force value. In Figure 2a, error bars based on uncertainty in either force or capacitance data would be smaller than the size of the symbols used, and hence are not shown.

The sensor used in this study has been in our laboratory for several years, and it has been calibrated several times, beginning in September, 2004, to check its long-term stability. A comparison of that calibration to the most recent, performed in September, 2006, showed that values of ΔC were between 1.3 % and 2.2 % less in 2004 than those measured most recently, for the same applied masses. In addition, the baseline (unloaded) capacitance of the sensor varied between 6.06 pF and 6.25 pF over the same period. Very preliminary studies indicate that the temperature dependence of the sensor's baseline capacitance is approximately 0.004 pF/°C for temperatures near 22 °C, where we have calibrated and used the sensor. Studies of the effects of relative humidity have not yet been undertaken.

SENSOR APPLICATION

After calibration, the sensor was mounted in a commercial instrumented indentation system, as if it were an indentation specimen, to assess the use of the sensor as a possible force transfer artifact, and to compare sensor and IIT force readings. The force and displacement of the IIT instrument had been calibrated immediately prior to the sensor installation, following the instrument manufacturer's recommended procedures. These procedures included the hanging of masses from the indenter shaft to calibrate force, and the use of a helium-neon laser interferometer system to calibrate the displacement transducer by moving the indenter shaft over a distance equivalent to 20 interference fringe spacings (approximately 6.3 μm). The masses used were weighed on the same electronic balances used for the sensor calibration masses, and have mass uncertainties similar to those shown in Table 1 for comparable values of mass.

The IIT system was programmed to perform multiple force applications on the sensor loading post over a range of forces. Each maximum force was held for 30 seconds, during which time capacitance and force data were recorded simultaneously. When the sensor was to be used at several values of maximum force, it would first be "exercised" with applications of the highest force in the series, a procedure that again parallels the recommendation of ASTM E 74 for higher-force devices[6].

The relative difference between the forces reported by the indenter system and the forces determined from recorded changes in sensor capacitance, as calculated via the cubic fitting functions described above, are plotted in Figure 3, expressed as a percentage referenced to the indenter force readings. One can see that the sensor's force readings are systematically lower than those reported by the indenter, typically by 3% - 4%. Most of the indenter contacts were made at the center of the glass platen, which itself was centered on the loading post axis. However, additional contacts were made near the edges of the glass platen, at positions 700 μm from the sensor's center axis (open diamond), and even on flat regions of the surrounding Delrin® post, at radial distances from 1.7 mm to 2.2 mm from the axis (open circle), to check the sensor's sensitivity to off-axis loading. These data, also shown in Figure 3, indicate no significant difference in sensor readings between on-axis and off-axis loading, particularly for contact anywhere on the glass platen.

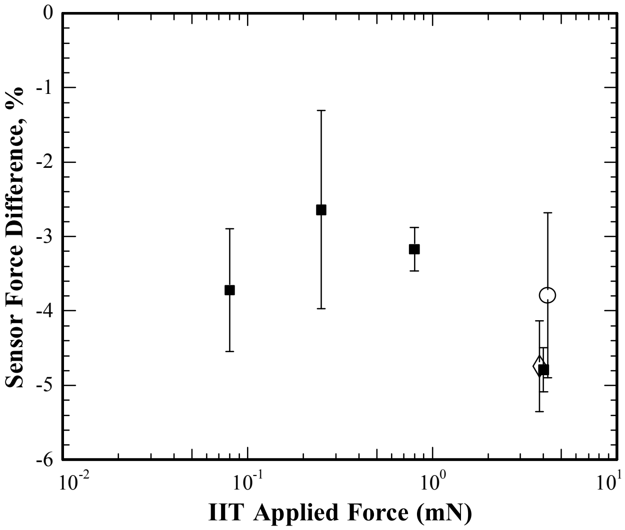

Figure 3. The relative difference between force applied by an instrumented indenter, as recorded by that instrument, and the force determined by the capacitance sensor, referenced to the indentation force reading. Solid squares: on-axis loading; open diamond (behind square): 0.7 mm off-axis; open circle: Delrin® contact, 1.7 mm to 2.2 mm off-axis.

CONCLUSIONS

We have characterized the performance of an elastically deformable capacitance force sensor of suitable design and size for potential application as a force calibration device for micro- and nano-mechanical test instruments such instrumented indenters, and have calibrated it in a manner that is traceable to the SI. We then probed the sensor with a commercial instrumented indenter that had also been calibrated with techniques traceable to the SI. The force readings obtained from the two devices differed by more than expected based on the uncertainties in the force and length references used, implying a systematic error in the experimental methodology.

Although the alignment of the axes of motion to gravity were not known precisely for either the sensor or the indenter, the force sensor's alignment was not changed significantly between its calibration and use in the indenter, and the indenter's alignment, as determined through an installation consistent with the manufacturer's recommendations, is not expected to be off by more than 2°. As most alignment errors are expected to enter as the cosine of the misalignment angle, it is unlikely that this could explain the discrepancy. We plan further investigations into the temperature and humidity dependence of the force sensor.

Disclaimer: Certain commercial materials and equipment are identified to specify the experimental procedure. Such identification does not imply endorsement by NIST, or that the material or equipment identified is necessarily the best available for the purpose. This work was performed under the auspices of the U.S. Department of Energy by the University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48

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