IN-SITU TRACEABLE CHARACTERIZATION OF THE DEPTH SENSING SYSTEM OF A NANOINDENTATION INSTRUMENT

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Abstract

Nanoindentation testing has proven to be effective for the determination of the mechanical properties of small structures. Accurate material measurements demand that nanoindentation instruments, especially their depth sensing system, be carefully calibrated.

Here a homodyne laser interferometer is realized for in-situ investigation of the displacement measuring performance of a commercial nanoindentation instrument. By means of real-time inspection of the axial movement of a spherical indenter mounted on the transducer of the nanoindentation instrument, the developed laser interferometer is capable of traceable calibration of the instrument's depth sensing system with nanometric resolution, whilst needing no additional measurement mirror.

Preliminary experimental results reveal that the nanoindentation instrument under calibration demonstrates adequate linearity within a limited measurement range. The interferometric calibration efforts will help to improve the measurement accuracy not only of conventional mechanical properties like Young's modulus and hardness, but also for the time constant and the complex modulus of viscous materials.

Keywords: Indentation instrument, depth sensing indentation, traceable calibration, homodyne laser interferometer

1. INTRODUCTION

Nanoindentation testing, also referred to as depth sensing instrumented indentation testing, is one of the most important methods for determining the mechanical properties of small structures , including ultra-thin films/coatings, nanoparticles, nano-wires/tubes, etc.. In the past decades, effective analysis and interpretation methods [1-3] for instrumented indendation technique (IIT), especially in the field of elasto-plastic materials testing, have been well developed and standardized [4-5].

According to the classification of ISO 14577 [4] nanoindentation testing is defined as having maximum indentation depths less than 200 nm. Within this region, the measurement accuracy of typical nanoindentation testing depends [5], in general, on

(1) the uncertainty of the zero point of contact,
(2) systematic measurement errors of the force generation system and
(3) of the depth sensing system, and
(4) drift of the instrument.

The last two factors play a very important role when the specimen under test has time-dependent mechanical behavior, what for example various engineering polymers show.

Calibration of the depth sensing system has gained therefore increasing interest over the last few years. To date, one of the typical approaches to traceable investigation of the performance of a nanoindentation instrument's depth sensing system is to fix an additional measurement mirror to the instrument so that a traditional laser interferometer [6] can be directly utilized [7]. In many cases, however, potential measurement errors would be introduced, when the mass of the additional mirror has a large deviation from that of the indenters in use.

In this paper, a practical laser interferometer, aiming to in-situ calibration of a commercial nanoindentation instrument without the need of an auxiliary measurement mirror, is proposed. The detailed principle of this interferometer is illustrated in section 2, and preliminary experimental results are reported in section 3, which proves the feasibility of the proposed interferometer.

2. PRINCIPLE

In the field of indentation testing of materials, there exist already indenters with various tip shapes for different testing purposes. Ball-shaped (spherical) indenters feature, compared especially with pyramidal-like indenters, much more evenly distributed stress distributions in the indentation region and allow a relatively simple data interpretation for elasto-plastic materials [8]. And, compared with materials testing using flat-punch-type indenters, spherical indentation, in which spherical indenters are utilized, is generally not very sensitive to the error sources like misalignment between the indenter and the surface of specimen under test. As a result, spherical indenters have long become one of the standard indenters associated with commercial nanoindentation instruments.

Here a practical laser interferometer for the calibration of a nanoindentation instrument is proposed, whose fundamental principle [9] is illustrated in Fig. 1.

The laser light coming from a frequency-stabilized laser (not shown in the schematic diagram) is firstly coupled into a polarization-maintaining (PM) single-mode (SM) fiber, and then delivered into the interferometer block. An aspheric lens is used to collimate the laser light from the PM-SM fiber. The collimated beam is then split by a polarization beamsplitter (PBS) into two beams, i.e. measuring beam and reference one. For the sake of adjustment of the interference contrast, an additional $\lambda/2$ waveplate is positioned before the PBS to rotate the polarization direction of the collimated input beam. The reference beam is reflected by the reference mirror (MR) back to the PBS, and its polarization direction has already been rotated by 90^o due to the 45^o-placed $\lambda/4$ waveplate.

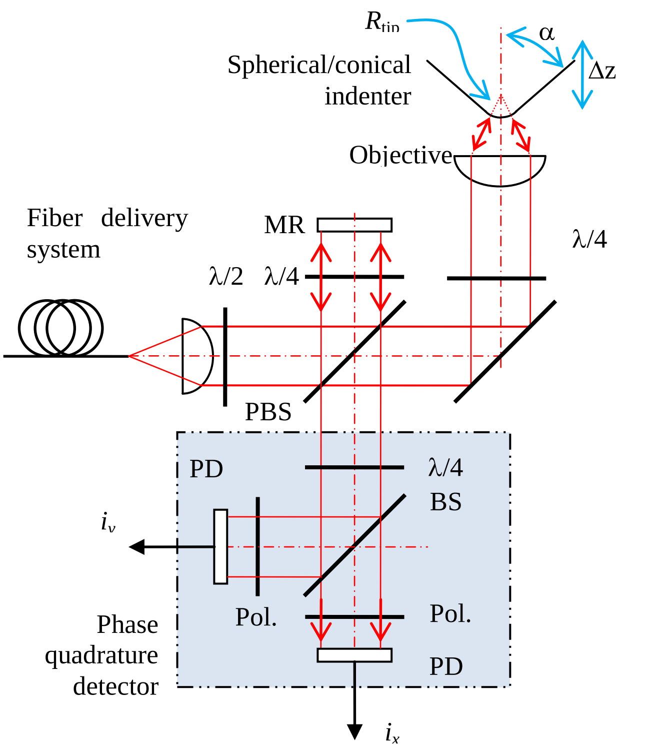


Fig. 1: Fundamental principle of a laser interferometer for measurement of the displacement of an indentation instrument.

The measuring beam is reflected up into the direction of the indenter by a 90° folding mirror. After passing through an objective, the measuring beam can finally sense the axial displacement of the spherical indenter.

The reflected measuring beam and the reference beam are combined together again by the PBS, and sent to a phase-quadrature detector, where the linearly polarized measuring and reference beams are firstly converted to circularly polarized beams by a 45°-placed λ/4 waveplate, and then split into two parts by a non-polarising beam splitter BS. Before each part of the circularly polarized measuring and reference beam reach the photo detector PD, a polarizer is inserted into the optical path, in order to yield an interference signal at each PD. Since the polarization directions between the two polarizers are orthogonal, two channels of interference signals (i_x, i_y) obtained from the two PDs will have 90° phase difference, which helps to measure both displacement and the displacement direction (i.e. forward or backwards.).

Before the aforementioned principle of the laser interferometer could be really applied for instrument calibration, there are still some practical issues to be considered.

1. Target surface

In principle, either the top surface or the central plane (perpendicular to the moving axis of the instrument) of the spherical indenter can be used as the target of the measuring beam. Taken into account that the surface of a real indenter, especially a used indenter might be, in general, not ideal for optical purposes, here the horizontal central plane of the indenter is suggested as the target plane of this interferometer.

2. Microscopic objective

A practical spherical/conical indenter usually would have limited apex angle α. It can be imagined that high loss of the measuring beam would happen if the microscope objective used in Fig. 1 would have too high numerical aperture A_N. In the mean time, an objective with too low A_N usually means poor capability of positioning the indenter under test. Therefore, in the ideal case, the microscope object used in the real calibration interferometer should have a numerical aperture A_N comparable to the apex angle α of the indenter in use, whilst a lateral resolution which is much smaller than the radius of the indenter in use R_{tip}, i.e.

$$\lambda/R_{tip} \ll A_N \le \sin(\alpha)\ . \qquad (1)$$

3. Measurement range

The measurement range Δz of the interferometrical calibration setup depends, to a large extent, on the depth of focus of the microscope objective. Taken into consideration that the proposed system is essentially a homodyne laser interferometer, here a more strict criterion is introduced to define the measurement range of the calibration setup, i.e.

$$\Delta z < 0.9\,\lambda/A_N^2, \qquad (2)$$

the latter is actually the axial resolution of a confocal microscope using the objective shown in Fig. 1.

3. CALIBRATION SETUP

The nanoindentation instrument (Hysitron Triboscope TI-950, Hysitron Inc.) involved in this research has a transverse capacitive transducer for force generation and indentation depth sensing and features a depth sensing resolution down to subnanometer [10]-[11].

However, similar to other transducers of capacitive type, the linearity of the depth sensing system is usually questionable, and therefore such systems have to be carefully calibrated. In addition, instabilities of environmental conditions including long-term pressure, humidity and temperature variation could have a strong influence on the stability of the capactive transducer, including systematic drift.

The calibration setup based on the proposed laser interferometer is illustrated in Fig. 2: The capacitive transducer to be calibrated is mounted on a 3D-piezo-scanner (not shown in the schematic) for indentation testing and surface/post indent imaging. Coarse positioning of the transducer together with the piezo-scanner is realized by a z-axis translation stage.

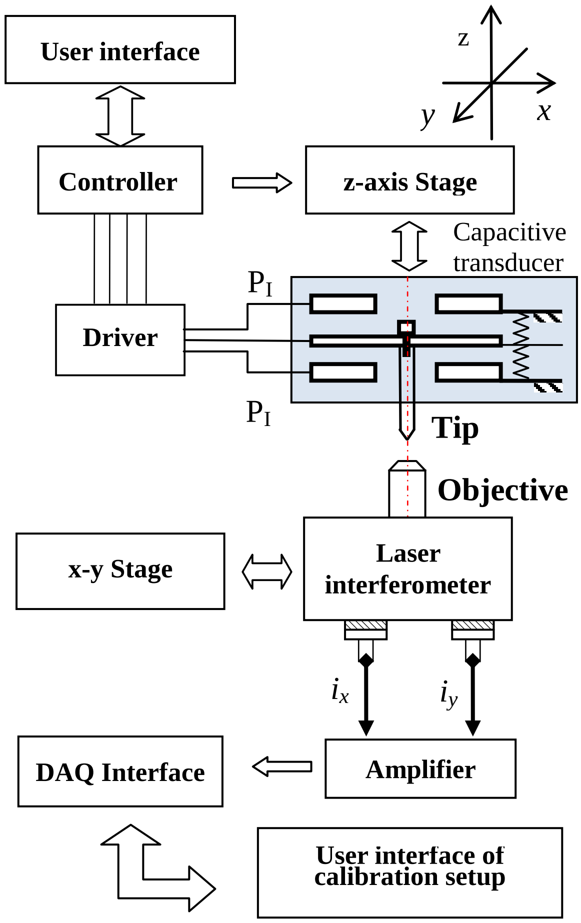
To calibrate the depth sensing performance of the transducer, a spherical indenter with a tip radius R_{tip} =

100 µm (nominal value) is used in the calibration setup.

The home-developed laser interferometer employs a frequency-stabilized He-Ne Laser (λ = 632.8 nm) with a fiber delivery system. In accordance with the indenter used (Eq. (1)), a microscope objective with A_N = 0.3 is chosen for the interferometer. Finally the whole interferometer system is mounted on the motorized x-y sample table, with which fine lateral positioning between the microscope objective and the spherical indenter is realized.

The phase quadrature signals from the interferometer are firstly amplified and then acquired by a DAQ system, finally sent to the measurement computer for further analysis, including nonlinear correction and low-pass filtering.

Fig. 2: An interferometric calibration setup for investigation of the depth sensing performance of a nanoindentation instrument.



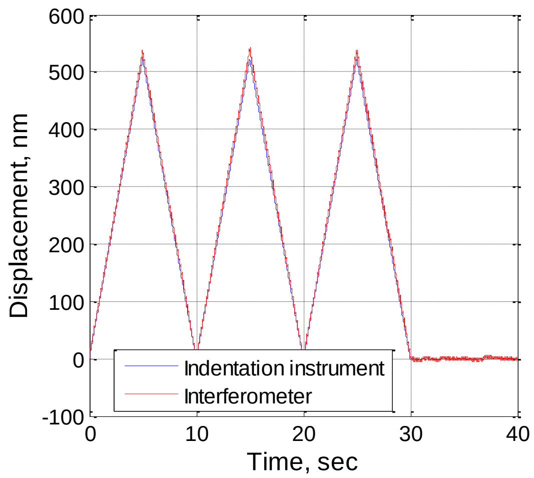
It has to be mentioned that the electronic signals from the transducer to be calibrated is not accessible, therefore, direct acquisition of the displacement of the transducer is not possible. A typical calibration procedure consists of the following steps:

(1) An indentation curve (open loop) is defined in the user interface of the indentation instrument,
(2) An air-indent according to the predefined curve is made,
(3) In the meantime, the vertical displacement of the indenter tip is measured by the interferometer,
(4) Finally, the displacement value given by the user interface of the nanoindentation instrument and that obtained by the laser interferometer are compared by means of a simple correlation analysis.

4. PRELIMINARY EXPERIMENTAL INVESTIGATION

The preliminary experimental results are shown in Fig. 3. The transducer shown in Fig. 2 was driven to make three cycles of indent in air within 30 s, and then kept unmoved for 10 s in order to investigate the stability of the calibration setup.

Fig. 3: Typical calibration procedure used in the



experiments.

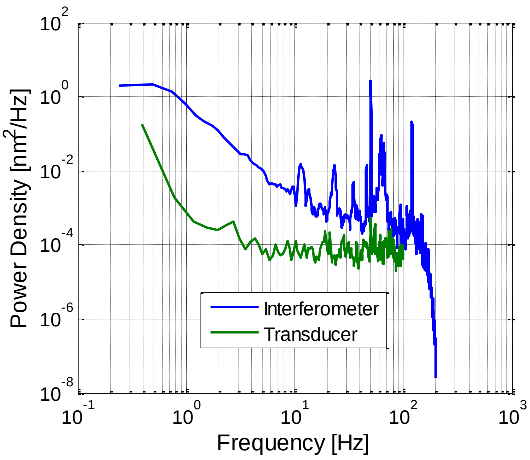
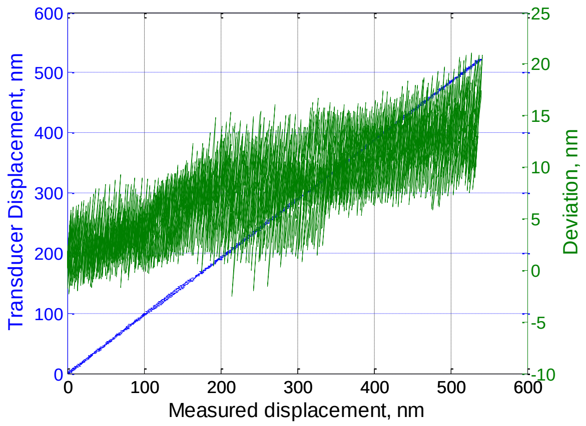
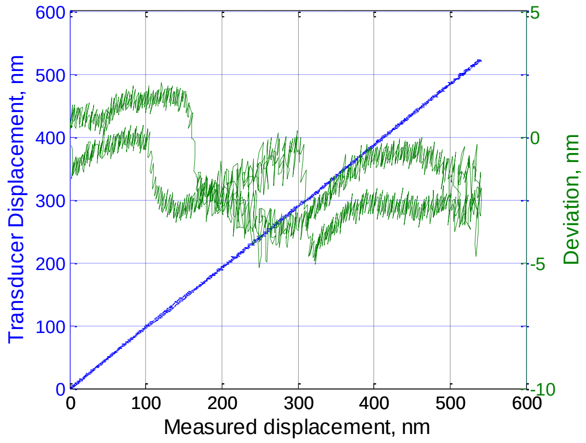


Fig. 4: Comparison between the noise spectrum of the interferometer and that of the transducer.

From the measurement data during the stable test period at the end of the experiment, the noise spectrum of both, the transducer and the interferometer, can be obtained (see Fig. 4). It's worthwhile to mention that the interferometrical calibration setup has measured actually the overall stability of the nanoindentation instrument, i.e. not only the noise within the transducer, but also that from the coarse x-y-z positioning system. Of course, the typical 50 Hz electric noise can also be clearly revealed from the interferometer signal.



(a) Systematic deviation of the capactive transducer



(b) Hysteresis within one test cycle (the scale error has already been removed)

Fig. 5: Preliminary calibration results of the transducer's depth sensing system.

Comparison between the indenter displacement measured by the interferometer and that by the transducer is shown in Fig. 5. It can be seen Fig 5(a) and Fig 5(b) that

(1) the transducer has a scale deviation of 2.6% ,
(2) there exists a hysteresis during the loading and unloading procedure of the transducer when it works in open-loop.,
(3) the nonlinearity within the measurement range is negligible.

4. SUMMARY

For in-situ investigation of the displacement measuring performance of a commercial nanoindentation instrument, here a homodyne laser interferometer is developed By means of real-time inspection of the axial movement of a spherical indenter mounted on the nanoindentation instrument, the home developed laser interferometer is capable of traceable calibration of the instrument's depth sensing system with nanometric resolution, whilst needing no additional measurement mirror mounted instead of the indenter.

Preliminary experimental investigation of an indentation instrument indicates that within a small displacement range the transducer of the instrument demonstrates a clear scale deviation of 2.6 %. One of the potential reasons for this scale deviation is that the scale of the transducer is usually determined within a large displacement range (e.g. several microns, as is the case of self-calibration with a force up to 600 µN).

Hysteresis phenomenon has also been detected even when the transducer works in open-loop and relatively low loading and unloading rates (e.g. ±40 µN/s), indicating the limited bandwidth of transducer.

Further experimental investigation of this instrument, including a larger displacement range and the long term stability, will be carried out in the near future.

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