In-situ characterisation of the metrological properties of nanoindentation instruments

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Abstract

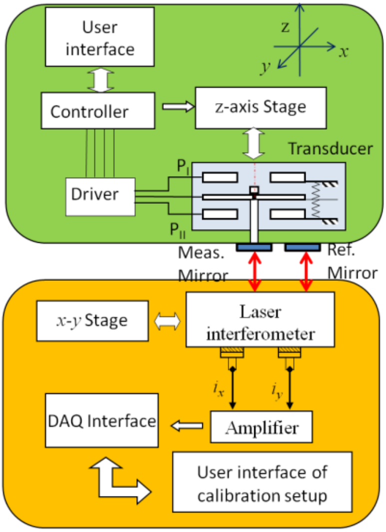
In this paper our efforts to characterise the force generation actuator and the displacement/depth sensing system of a nanoindentation instrument are detailed. An interferometric depth calibration setup has been developed to in-situ determine the performance of the instrument's depth sensing system. The quasi-static indentation force generated by the instrument down to the µN range is in-situ calibrated using a modified compensation balance. Moreover the qausi- and dynamic performance of the instrument in the sub-µN range is characterised with a MEMS-based electrostatic nanoforce transfer standard.

1. Introduction

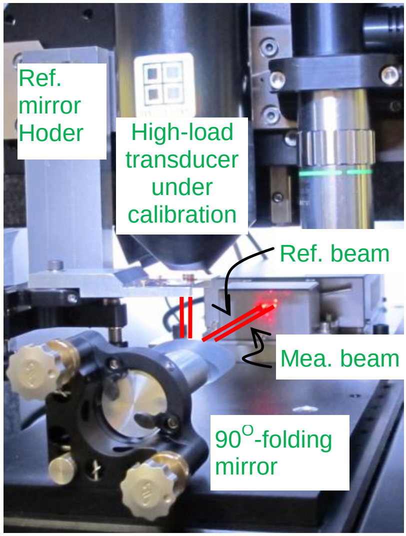
Nanoindentation testing offers fundamentally an excellent methodology for determining the mechanical properties of small structures, including ultra-thin films, nanoparticles, nano-wires/tubes and many more. For the purpose of material testing with high accuracy, the performance of nanoindentation instruments in use, especially their depth sensing system and force sensing units, should be carefully characterised [1].

2. In-situ calibration of the depth sensing system of a nanoindentation instrument

A differential laser interferometer [2] is employed to investigate the performance of the depth sensing system of a nanoindentation instrument [3]. As shown in Fig. 1, the reference and measurement beams coming from the laser interferometer are reflected by a 90º-folding mirror to the reference and measurement mirrors, respectively. The common-path configuration of this calibration ensures that the calibration results are immune to environmental instabilities.

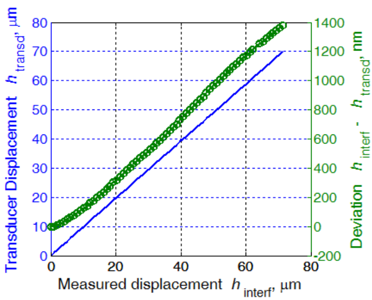


(a) Schematic of the interferometric depth calibration system

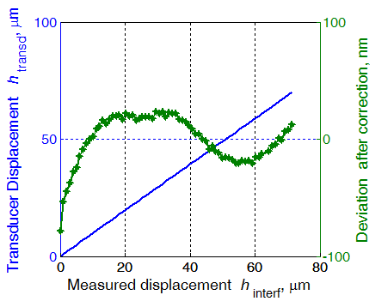


(b) Photography of the in-situ indentation depth calibration setup

Figure 1: In-situ calibration setup for the depth sensing system of a nanoindentation instrument.



(a) Comparison between the transducer readout and interferometer output.



(b) Residual error of the transducer after linear correction

Figure 2: investigation of the depth sensing system of a nanoindentation instrument

One of the typical depth calibration results is shown in Fig. 2(a), and a linear deviation between the transducer readout and the laser interferometer output has been revealed, i.e.

$$h_{\text{Interf}} = 0.9794*{}_{\text{Transducer}} + 78.4 \text{ nm},$$

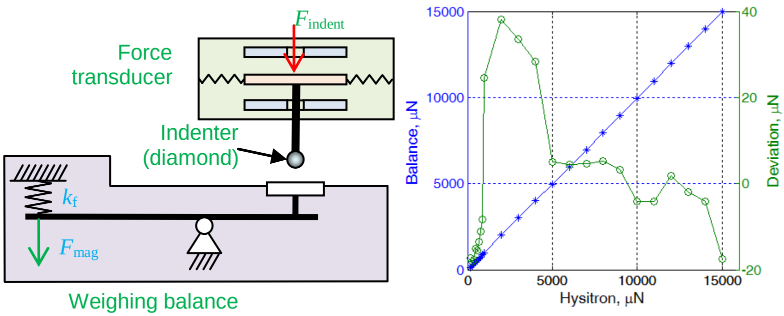
which indicates that the depth scale factor of the transducer under calibration has to be corrected. After correction, a residual displacement error of 20 nm (1σ) over the whole indentation depth range (70 μm) has been achieved, as shown in Fig. 2(b).

3. Traceable calibration of the indentation force generated by a nanoindentation instrument

The nanoindentation instrument under calibration is equipped with a low-force and a high load transducer. The former is able to generate indentation forces down to sub-micronewton, and the later creates test force up to 2 N.

3.1 Using a compensation balance

To traceable characterise the indentation force generated by the nanoindentation instrument from μN to tens of mN, a precision compensation balance [4] has been employed, which features small height, and a high force sensitivity (down to 10 nN). As illustrated in Fig. 3(b), it was found that over the whole calibration range (15 mN) the high load (HL) transducer shown in Fig.1(b) demonstrates a force deviation less than 17 μN (1 σ).



(a) Schematic of the force calibration system for nanoindentation instruments (b) Force calibration result of the HL-transducer shown in Fig.1(b)

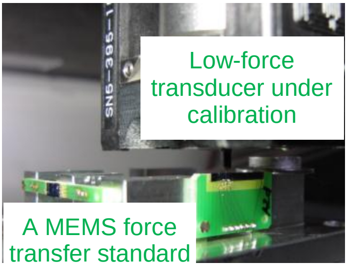
Figure 3: Traceable measurement of the indentation force using a precision compensation balance

Calibration of the low force transducer using this precision balance and the related results are detailed in Ref. [5].

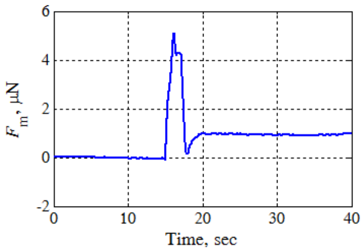
3.2 Application of a MEMS-based force transfer standard

Although the precision balance mentioned in subsection 3.1 possesses indeed high resolution, its relatively slow response capability (on the order of 10 s) prevents it from dynamic applications. To investigate the dynamic performance of the low-force transducer, here a MEMS-based nano-force transfer standard [6] is used.

Fig. 4(b) demonstrates the indentation force variation during a typical engagement process with a pre-load of 1 µN, in which an overshoot of about 4 µN is detected.



(a) Nano-force calibration using a MEMS force standard



(b) Dynamic force response of the low-force transducer during the engagement process

Figure 4: Measurement of the indentation force in the sub-µN range using an electrostatic nano-force standard.

4. Summary

A comprehensive calibration approach for quantitative characterisation of the test force and the indentation depth of a nanoindentation instrument has been presented. The calibration methods presented in this paper show that it is possible to achieve an uncertainty level of 1 - 0.5 % for the indentation depth and indentation force of a commercially available nanoindentation instrument.

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References

[1] ISO 14577-2 (2002): Metallic Materials-Instrumented indentation test for hardness and materials parameters-2:Verification and calibration of testing machines
[2] SP-DS Miniature Interferometer, SIOS GmbH, Ilmenau, Germany, www.sios.de/ENGLISCH/PRODUKTE/SP-DS_E_2012.HTM
[3] Hysitron Triboscope TI950 of the Hysitron Inc., Minneapolis, USA
[4] Compensation balance WZ2P-CW from Sartorius GmbH, Göttingen, Germany
[5] Z. Li, U. Brand: Towards Quantitative characterisation of the small force transducer used in nanoindentation instruments. Mod. Instrument., 2013, 2, 61-67
[6] S. Gao, Z. K. Zhang, Y. Wu, K. Herrman. Proc. 9th Euspen International Conference, 2-5 Juni, 2009, San Sebastian, Spain, Vol. 1, 267-270.