

TRIB2004-64363

CONTACT MECHANICAL IMPEDANCE METHOD IN NANOINDENTATION

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ABSTRACT

Performance of quantitative nanoscale characterization instruments in quasistatic and force modulation modes is evaluated by the newly introduced analytical method. The proposed analytical method is based on the mechanical impedances superposition and allows optimization of dynamically contacting nanoindenter and elastic indentation contact for ideal materials where dynamic response equations are merged with the Hertz's contact mechanics. Nanoscale contact impedance values are calculated as a function of elastic contact area and oscillation frequency. Theoretical dynamic response curves are calculated for commercially available quantitative nanoscale characterization devices.

INTRODUCTION

Dynamic indentation or so-called force modulation technique has been introduced into quantitative nanometer scale measurements a while ago [1, 2] and was commercialized by the leading nanoindenter manufacturers such as MTS, Hysitron by the names of "Continuous Stiffness Measurement" and "nanoDMA™", respectively. Here, loss and storage moduli are derived from the dynamic response of oscillating nanoindenter in contact with the sample. Due to the low bandwidth of nanoindentation instruments, quantitative nanoscale measurements are targeting time-dependant polymeric materials such as elastomers, plastics and rubbers. Inconsistency of the data between vendors often leads to the questioning of instrumentation and the proposed techniques.

The mechanical impedance matching method [3] was introduced to evaluate quasistatic and dynamic contacting metrology tools [4]. Theoretical guidelines for materials dynamic testing at nanoscale are provided for variety of commercially available quantitative nanomechanical test instruments. The proposed systematic analytical approach allows finding the best instrument for the desirable range and materials to be tested.

MECHANICAL IMPEDANCE APPROACH

By definition, mechanical impedance Z is a ratio of driving force F and resultant velocity v derived at the driving point or at the point of dynamical interaction between oscillating nanoindenter and sample [5]:

$$Z = \frac{F}{v} \quad (1)$$

The basic idea behind mechanical impedance approach for the dynamically contacting instrument and the sample is expressed by matching corresponding mechanical impedances [3, 6], i.e.:

$$Z_c \approx Z_v \quad (2)$$

here, Z_c and Z_v are mechanical impedances of the semi-infinite elastic contact and oscillating nanoindenter, respectively.

For frictionless dynamic contact, the contact mechanical impedance Z_c of a semi-infinite sample excited normally by means of rigid spherical indenter tip consists of dissipative term r_c , inertial term m_c , contact compliance term q_c and can be expressed as follows:

$$Z_c = r_c + jm_c - j \frac{1}{\omega q_c} \quad (3)$$

An elastic Hertzian contact compliance of the radius R_c and corresponding cross section area A_c is defined by:

$$q_c = \frac{1}{2R_c} \left(\frac{1-\nu_s^2}{E_s} + \frac{1-\nu_t^2}{E_t} \right) = \frac{\sqrt{\pi}}{2\sqrt{A_c}} \left(\frac{1-\nu_s^2}{E_s} + \frac{1-\nu_t^2}{E_t} \right) \quad (4)$$

It has been shown [4] that for dynamic nanoindentation testing range (0 – 300Hz) dissipative r_c and inertia m_c terms are

negligibly small to compare with the compliance term q_c . So, the contact impedance is inversely proportional to the contact compliance q_c multiplied by the excitation cyclic frequency ω .

The second important parameter which needs to be evaluated is mechanical impedance of the dynamic oscillator. For nanoindentation assisted dynamic measurements, mechanical impedance of the oscillator can be derived from the single degree of freedom forced oscillator model in the form:

$$Z_v = \sqrt{(k_v - m_v \omega^2)^2 + \omega^2 c_v^2} \quad (5)$$

here, k_v , m_v and c_v are instrument's stiffness, mass and damping, respectively. The contact stiffness and machine compliance terms are not included. It can be noted that for the single DOF mass driven system all terms, i.e., mass, stiffness and damping of the instrument need to be considered. In order to increase sensitivity of the measurement, oscillating nanoindentation instrument is operated in vicinity of its rigid body resonance mode.

MODELING RESULTS AND DISCUSSION

Mechanical impedances calculated according to the Eq. 5 for six commercially available nanoindenters are plotted versus frequency and shown in Fig. 1 together with the contact mechanical impedance of ideal polymers and metals for nanometer scale elastic contacts. Parameters used in mechanical impedance calculations of nanoindenters are presented in the Table 1. Here, numbers in parenthesis are related to the modeling result curves plotted in Fig. 1. Parameters were obtained experimentally or from the data sheets of the instrument vendors.

Table 1. Mechanical properties used to model nanoindentation assisted dynamic oscillators for commercially available nanoindenters and instrument under development.

Instrument\parameter	Stiffness k_v , N/m	Mass m_v , kg	Damping c_v , kg/s
TriboScope Hysitron (1)	150	$0.26e^{-3}$	0.05
XP-DSM, MTS (2)	100	$0.1e^{-3}$	0.015
NanoIndenter II, MTS (6)	100	$6e^{-3}$	5
Multirange NanoProbe (1N), Hysitron (3)	15000	$6e^{-3}$	0.1
Multirange NanoProbe (2N), Hysitron (4)	27000	$6e^{-3}$	0.1
Nanohead-1 CETR (5)	400	$5e^{-3}$	0.05

The contact impedance Z_c was calculated and plotted for nanometer scale contacts versus excitation frequency for ideal very soft (0.02GPa) and hard polymers (4GPa). Most

commonly used nanoindentation tests elastic contacts of 1 – 10 μm radii for polymers and frequency ranges of 0 – 15kHz were investigated.

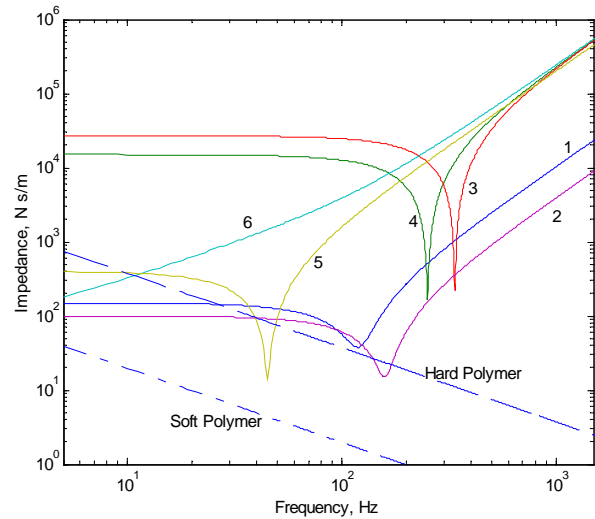


Figure 1. Mechanical impedance of nanoindenters and contact mechanical impedance of ideal polymers for nanometer scale contacts versus frequency.

Curves 1, 2 and 5 represent moderately damped instruments, while curves 3, 4 correspond to underdamped and 6 – overdamped systems. This result makes it obvious why MTS's DCM module (2) and CETR's Nanohead-1 (5) has a better dynamic performance for entire range of ideal polymeric materials nanoscale contacts. Mechanical impedance of these instruments in the vicinity of resonance matches perfectly with the contact mechanical impedance of polymeric materials. Hysitron's TriboScope (1) dynamic response corresponds to the critically damped oscillator case due to the design and manufacturing constrains and misses impedance of polymeric materials to be matched. Here, it should be pointed out that some impressive experimental results on harder polymeric materials such as PMMA were reported by the numerous researchers using this instrument [7]. Also, it can be observed that resonance peak related to the TriboScope's (1) dynamic response resonance peak reaches the hard polymers contact impedance curve indicating consistency with experimental findings.

On the same note, Hysitron's Multirange Nanoprobe instrument (3, 4) is not well suited for dynamic measurements either. High stiffness value of the instrument results in sharp resonance peaks and does not match mechanical impedance of the polymeric materials at all. Only lowering stiffness by a factor of 10 and keeping the same mechanical Q-value could potentially place these two instruments on impedance matching zone.

MTS's NanoIndenter II has a critically overdamped instrument dynamic response curve (6) which passes through the polymers contact impedance function. Theoretically, this instrument cannot benefit in sensitivity of resonance peak because of rolling out dynamic response but should perform way better than (1, 3, 4). This hypothesis is supported by the multiple successful experimental studies reported in the peer review papers [8]. Stiffness of each instrument can be observed at the "0" frequency in Fig. 1.

CONCLUSIONS

The mechanical impedance matching approach is introduced to evaluate nanoscale quasistatic and dynamic contact metrology tools. Here, mechanical impedance of the semi-infinite contact and oscillating instrument are matched in order to find optimal performance criteria. An analytical mechanical impedance expression of nanoindentation instrument is derived from a single degree of freedom oscillator frequency response function. Expression for mechanical impedance of semi-infinite elastic contact is based on the Hertz elastic contact formula.

Theoretical mechanical impedance curves representing best performing instruments in the field were compared with the optimum dynamic testing range for ideal polymeric materials. The following are modeling results:

- Values of instrument internal damping, stiffness and overall bandwidth are the most important in tuning with in order to match mechanical impedance of polymeric materials. Stiffness range of 100 – 500N/m, damping of 0.015 – 0.05 N/ms and first resonance between 20 – 150Hz.

- Excessive internal damping and high bandwidth can shift mechanical impedance toward mismatch. Also, lower bandwidth of the instrument and higher stiffness could place mechanical impedance into a perfect match position.

Nevertheless, all commercially available quantitative nanoscale instruments are driven by the adequate electronics and control software what can enhance performance by various analog and digital signal processing options such as filtering, data points averaging etc.

REFERENCES

- [1] Oliver, W.C., Pharr, G.M., 1997, *J. Mater. Res.*, **6**, pp. 1564 – 1572.
- [2] Asif, S. A. S., Wahl, K.J., Colton, R.J., 1999, *Rev. Sc. Instr.*, **70**, pp. 2408-2415.
- [3] Daugela, A., 1997 “Mechanical Impedance Approach for Contact Impedance Sensors,” PhD Theses, Gifu University, Japan.
- [4] Daugela, A., Misaki, A., Fujii, H., 1996, “Nondestructive Mechanical Contact Impedance and Compliance Testing of Rubberlike Materials,” *JSME Intern. J.*, **A, 39**, 4, pp. 640-648.
- [5] Harris, Crede, *Handbook on Shock and Vibrations*, McGraw-Hill, 7th ed. 2001.
- [6] Lucas, B.N., Oliver, W.C. Swindeman, J. E., 1998, *Mat. Res. Soc. Symp. Proc.* **522**, pp. 3 - 10.
- [7] Asif, S.A.S., Wahl, K.J., Colton, R.J., Warren, O.L., 2001, *J. Appl. Phys.*, **90**, 1192 – 1198.
- [8] Oliver, W.C., Pharr, G.M., 2004, *J. Mater. Res.*, **19**, pp. 3-22.