Force modulation with a scanning force microscope: an analysis

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A magnetic force modulation microscope (FMM) has been employed to measure the dynamic behavior of a contact between a scanning force microscope (SFM) tip and a surface. Our experimental results show the inefficiency of the classical models (two Kelvin–Voigt elements in parallel). A new model which takes into account the normal and tangential stiffness of the contact, and also the geometrical and mechanical properties of the cantilever which hold the tip, is proposed. This model shows that the natural frequency is sensitive to the normal stiffness, only if the ratio of the normal contact stiffness to the cantilever stiffness is between 0.2 and 200. Above this domain, the natural frequency is sensitive to sliding (Mindlin theory).

Keywords: scanning force microscope, force modulation, cantilever natural frequency, contact stiffness, Young modulus, model

1. Introduction

Since its invention in 1986, the scanning force microscope (SFM) [1] has become a routine instrument for surface investigations [2,3]. In addition to topographic features, SFM was developed to produce image maps of mechanical properties of materials at the nanometer scale. Quantitative friction force [7–11] and adhesive force imaging [12–16] has been realized. Furthermore, force curves have been employed to measure the Young modulus of surfaces with coherent results [17–21] and force modulation microscopes (FMM) have supposedly enabled the elastic imaging of surfaces [4,22–25].

The principle of FMM is to create a harmonic modulation force on the contact and to measure the response of the cantilever. Two types of FMM, direct and indirect mode, can be used [4,5]. In the case of direct FMM, the force acts directly on the cantilever. Alternatively, for indirect FMM, the modulation is done via a vertical modulation of the sample (or cantilever) position. Some limitations are related to FMM. In both types the tilt of the cantilever causes a horizontal displacement of the contact point. The consequence is that the response of the cantilever is influenced by both indentation and friction. Due to the lack of a suitable model no relation can be given correlating the amplitude of vibration of the cantilever and the material properties of the sample.

Furthermore some drawbacks are relevant to indirect FMM in particular:

(i) The cantilever stiffness must be at least of the same order of magnitude as that of the normal contact stiffness. This leads to a major limitation at high applied forces.

(ii) There is always a horizontal displacement when the piezo is modulated in a vertical direction.

(iii) For a constant driving voltage, the vertical displacement amplitude of the piezo is dependent on the frequency. The natural frequency peak of the cantilever is not clearly visible on a frequency response curve because it is mixed with the natural frequency peaks of the piezo (fig. 1). Furthermore, the response of the piezo depends on the amplitude of the driving voltage and of the mass of the sample.

In order to overcome these drawbacks, we have chosen to build a FMM where the modulation is applied directly to the cantilever [4]. Direct FMM presents great advantages:

(i) The natural frequencies of the cantilever without contact are known.

(ii) The amplitude of vibration, and thus the sensitivity, is one or two orders of magnitude higher near the natural frequency compared to low frequencies.

(iii) The natural frequency is not sensitive to variation of the “A–B” signal (variation of the laser beam intensity, electronic noises, piezo vibrations at low frequencies...).

Fig. 1. Response frequency curve of the cantilever with a driving voltage by a piezo electric ceramic. The natural frequency peak of the cantilever is not clearly visible.
(iv) The band width of the natural frequency peak gives information about the damping of the material.

The challenge of FFM is to realize quantitative elastic imaging of a surface. We believe that it is possible by acquiring the natural frequency of the cantilever. The aim is to study the differences between the elastic properties of small volumes (nanometer scale) and bulk properties, the elastic properties of thin layers (from organic films to oxide layers) or local heterogeneity of the Young modulus.

2. Materials and methods

2.1. Magnetic force modulation microscope

The principle of our instrument could be compared with systems developed recently [4,26–28]. The instrument is a commercial SFM (Autoprobe CP, Park Scientific Instruments, USA) with a lateral force microscope (LFM) head and a “signal access module”. The cantilever is made magnetic by a coating of cobalt. A magnetic field is generated by a coil (Sigma SC30 1 mH, resistance \( R \approx 60 \), inductivity \( L \approx 0.8 \) mH), which creates a force acting on the cantilever. The frequency is generated and analyzed with an impedance/gain-phase analyzer (Solartron 1260, Schlumberger, Germany) piloted by home-made software via an IEEE interface (KPC-488.2, Keithley, USA) (fig. 2). This enables us to work in a frequency range of 1–300 kHz. At lower frequencies, the response of the cantilever is noisy due to piezo vibrations. At higher frequencies the amplitude of vibration of the “A–B” signal becomes too low due to the impedance of the coil, the band width and noise of the SFM pre-amplifier. The modulation force is about 20 nN at low frequencies but could be easily increased by one order of magnitude.

The response frequency curves are generated by sweeping the driving voltage frequency.

2.2. Magnetic cantilever

The classical technique to give magnetic properties to a cantilever is to glue a magnet onto the cantilever [4,26–28]. This technique has some drawbacks:

(i) The glued magnet changes the mechanical properties of the cantilever. There is a lack of information about the stiffness and the mass of the cantilever.

(ii) These cantilevers are not easy to produce in a routine way.

(iii) The cantilevers made by this technique are not reproducible because the glued magnet is made by crushing a bigger one [4]. The geometry of the magnet is unknown, which leads to a lack of information about its magnetic behavior and the localization/orientation of the modulation force (normal or/and lateral excitation).

For these reasons we chose to coat the cantilevers with a thin layer of cobalt. Our cantilevers are Si$_3$N$_4$ commercial cantilevers (Young modulus 130–150 GPa, density 2.8–3.1 [29,30]) (Microlever, Park Scientific Instrument, USA) with a pyramidal tip \( H \approx 3 \), \( R \approx 50 \) nm). We used both rectangular and triangular shape cantilevers with different stiffnesses (table 1). A cobalt layer is deposited on the backside of the cantilever under low argon pressure using cathodic sputtering. This layer is about 30–40 nm thick.

3. Theory of force modulation

3.1. Hertz theory

The elastic contact between two bodies has been modeled by Hertz [6]. In the case of a sphere of radius \( R \) and a plane pressed together with a normal force \( F_n \), the radius of contact \( a \), the indentation depth \( \delta \), and the normal contact stiffness \( k_n \) are equal to:

\[
a = \left( \frac{3F_nR^2}{4E^*} \right)^{1/3},
\]

\[
\delta = \left( \frac{9F_n^2}{16RE^{-*2}} \right)^{1/3},
\]

\[
k_{n,c} = 2aE^* = \left( 6F_nRE^{-*2} \right)^{1/3},
\]

with

\[
E^* = \left( 1 - \nu_1^2 \right) \left( 1 - \nu_2^2 \right) E_2 \left( E_1 \right)^{-1}
\]

and \( E_1, E_2 \) and \( \nu_1, \nu_2 \) are the Young modulus and the Poisson ratio of the two surfaces respectively.

3.2. Model

It is said that the equivalent model of the tip/surface contact, for direct force modulation, is a parallel arrangement of two Kelvin–Voigt elements [4,5] (fig. 3). The normal contact stiffness \( k_{n,c} \) adds to the cantilever linear stiffness \( k_{1,t} \) resulting to an increasing of the natural frequency \( f_c \). For a cantilever of constant effective
mass $M$ and no damping, the relative resonant frequency $f_r$, ratio of $f_c$ to the natural frequency in vacuum $f_v$, should be equal to:

$$f_r = \frac{f_c}{f_v} = \left(\frac{k_{1,1} + k_{n,c}}{k_{1,1}}\right)^{1/2} \left(\frac{M}{k_{1,1}}\right)^{1/2} = (k_{1,1} + 1)^{1/2},$$

(5)

with

$$k_{1,1} = \frac{k_{n,c}}{k_{1,1}}.$$

(6)

4. Experimental results

The first stage of our approach was to measure the natural frequency of the cantilever without contact $f_a$ and when the tip is in contact with the surface $f_c$. The aim is to measure the normal stiffness $k_{n,c}$ of the contact between the tip and the surface in order to see the ability of our system to probe the Young modulus of the surface.

4.1. Cantilever without contact

The natural frequencies of a cantilever without contact, $f_a$, have been measured. The first natural frequency was found to be less than the theoretical value. The cobalt coating causes a decreasing of the natural frequencies of the cantilever. Theoretically, the natural frequencies of a homogeneous beam of length $L$, Young modulus $E$, moment of inertia $I$ and of mass $M$, are equal to [31]:

$$f_v = C_n \left(\frac{EI}{ML^3}\right)^{1/2},$$

(7)

with

$$M = \rho WTL$$

(8)

and $C_n$ a coefficient available for each mode (which depends on the boundary conditions) given in table 2 [31]. In the case of a rectangular beam:

$$f_v = C_n \frac{T}{\sqrt{12L^2}} \left(\frac{E}{\rho}\right)^{1/2}.$$

(9)

In the case of a cantilever made with two layers of thickness $T_a$ and $T_b$ of two different materials of Young modulus $E_a$ and $E_b$, $EI$ is equal to [32]:

$$EI = \frac{WT_a^3T_aE_aE_b}{12(T_aE_a + T_bE_b)}C,$$

(10)

with

$$C = 4 + 6 \frac{T_a}{T_b} + 4 \left(\frac{T_a}{T_b}\right)^2 \frac{E_b}{E_a} \left(\frac{T_a}{T_b}\right)^3 \frac{E_bT_b}{E_aT_a}.$$

(11)

The natural frequencies found are close to the theoretical values. Fig. 4a shows the response of a rectangular cantilever (type B) as a function of the frequency. The 1st, 2nd, 3rd and 5th peaks are respectively corresponding to the four first modes of flexion vibration, the 4th peak is probably a torsion mode of vibration, as shown in fig. 4b.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Geometrical and mechanical characteristics of the cantilevers used (taken from the manufacture sheet)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cantilever</td>
<td>Shape</td>
</tr>
<tr>
<td>A</td>
<td>triangular</td>
</tr>
<tr>
<td>B</td>
<td>rectangular</td>
</tr>
<tr>
<td>C</td>
<td>triangular</td>
</tr>
<tr>
<td>D</td>
<td>triangular</td>
</tr>
<tr>
<td>E</td>
<td>triangular</td>
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<tr>
<td>F</td>
<td>triangular</td>
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</table>

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Coefficients $C_n$ for the natural frequencies of a beam</th>
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</thead>
<tbody>
<tr>
<td>Mode</td>
<td>1</td>
</tr>
<tr>
<td>&quot;clamped-free&quot; beam</td>
<td>$C_n$</td>
</tr>
<tr>
<td>&quot;clamped-hinged&quot; beam</td>
<td>$C_n$</td>
</tr>
<tr>
<td>&quot;clamped-clamped&quot; beam</td>
<td>$C_n$</td>
</tr>
</tbody>
</table>

Fig. 3. Schematic of the "two Kelvin–Voigt" mechanical equivalent model. The two Kelvin–Voigt elements correspond to the cantilever and the contact stiffness.
4.2. Tip in contact

4.2.1. Influence of the cantilever

The frequency response curve, when the tip is in contact with the surface, shows several peaks indicating a system with \( n \) degrees of freedom. The first natural frequencies for different cantilevers, for the same surface of glass and a constant force has been measured (fig. 5). The ratio of the first natural frequency in contact to the first natural frequency without contact \( f_r \) is about 4.1 for a triangular shape cantilever, whatever the cantilever stiffness \( k_l \); it may be, and about 4.7 for a rectangular shape cantilever (table 3). This indicates that \( f_r \) is more influenced by the geometrical shape than its stiffness.

Furthermore in air for a cantilever of type C, the amplitude of vibration is about 2 \( \mu \)m for a driving voltage of about 20 nN. When the tip is in contact with a glass surface with a force of about 50 nN, the contact stiffness should be about 300 N/m (Hertz theory). With the same driving voltage, the amplitude of the “A–B” signal is two orders of magnitude lower at low frequencies (fig. 5). If we consider that the amplitude of the “A–B” signal is only due to indentation, the indentation modulation depth must be about 20 nm, requiring a modulation force of about 6 \( \mu \)N, which is two orders of magnitude higher than the real modulation force. Because the “A–B” signal is sensitive to the changes in position angle of the edge of the cantilever, two movements or a combination of them, can be measured: the vertical or the horizontal displacement (in the axis of the cantilever).
beam) of the tip. In this case, this means that the modulation amplitude of the “A – B” signal is mostly influenced by horizontal motion of the tip and then friction.

4.2.2. Influence of the surface

The first natural frequency of the rectangular cantilever (type B), when the tip is in contact with a surface, \( f_c \) has been measured for different materials. We chose a rectangular cantilever because it is more easy to describe mechanically. The surfaces have been chosen for their flatness and to be a representative sampling of surfaces (Young modulus, hardness, density, nature of the material) [33]. The values of the \( f_c \) are indicated in fig. 6, for different forces and surfaces. It is notable that the first natural frequencies are close together, whatever the surface or the applied force may be, and could not be directly linked to the mechanical properties of the sample (table 4).

5. Discussion

5.1. Inefficiency of the ‘‘two Kelvin–Voigt’’ model

The inefficiency of the ‘‘two Kelvin–Voigt’’ is due to the fact that the modulation force is not perfectly applied in the same axis as the applied force. This model could not explain our results for six reasons:

(i) \( f_c \) is between 60 and 63 kHz whereas it should be between 300–2300 kHz (table 5).

(ii) For \( F_n \) greater than 5 nN, \( f_c \) decreases with the applied force whereas it should increase.

(iii) For constant force, \( f_c \) could not be directly linked to the Young modulus of the surface whereas it should increase with the Young modulus of the surface.

(iv) For constant \( F_n \) and the same surface, \( f_r \) remains stable with an increasing of \( k_{11} \) whereas it should decrease.

(v) There are several resonance peaks whereas there should be a single peak.

\[
\left( \frac{E_{\text{sample}}}{\rho_{\text{sample}}} \right)^{1/2} \gg 2\pi f_A, 
\]

with \( A_v \), amplitude of vibration and \( f \), frequency. Furthermore to influence the effective mass, this volume must be of the same order of magnitude as the cantilever dimension (~2400 \( \mu \text{m}^3 \)) which represents a half sphere of about 10 \( \mu \text{m} \) in radius?

5.2. New model

From these considerations, a new 2D model which takes into account the geometrical and mechanical properties of the cantilever and the normal and tangential contact stiffness has been built (fig. 7). This model does not take into account the damping of the system, the torsion of the cantilever, its static deformation, or the angle between the cantilever and the sample.

In our model the cantilever is decomposed into \( n \) elements, of equal length, defined by geometrical nodes. Each node has two degrees of freedom (flexion and rotation). The edge of the cantilever is linked with a coil spring (for the normal contact stiffness) and a spiral spring (for the lateral contact stiffness). We have calculated the first natural frequencies of the cantilever using finite element analysis.

Table 4

<table>
<thead>
<tr>
<th>Material</th>
<th>sapphire</th>
<th>steel</th>
<th>gold</th>
<th>glass</th>
<th>PET</th>
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</thead>
<tbody>
<tr>
<td>Young modulus (GPa)</td>
<td>400</td>
<td>200</td>
<td>80</td>
<td>70</td>
<td>3</td>
</tr>
<tr>
<td>Hardness (MPa)</td>
<td>5000</td>
<td>~1000</td>
<td>40</td>
<td>7200</td>
<td>~20</td>
</tr>
<tr>
<td>Density (kg/m³)</td>
<td>3900</td>
<td>7800</td>
<td>19300</td>
<td>2500</td>
<td>~1200</td>
</tr>
</tbody>
</table>

Table 5

Predicted first natural frequencies (kHz) by the “two Kelvin–Voigt” model taking \( k_{11} = 0.02 \text{ N/m, } R = 50 \text{ nm, } E_{\text{tip}} = 130 \text{ GPa and } f_0 = 13.17 \text{kHz} \)

<table>
<thead>
<tr>
<th>Force (nN)</th>
<th>Sapphire</th>
<th>Steel</th>
<th>Gold</th>
<th>Glass</th>
<th>PET</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>1482</td>
<td>1384</td>
<td>1186</td>
<td>1153</td>
<td>462</td>
</tr>
<tr>
<td>20</td>
<td>1876</td>
<td>1744</td>
<td>1494</td>
<td>1452</td>
<td>582</td>
</tr>
<tr>
<td>40</td>
<td>2106</td>
<td>1917</td>
<td>1677</td>
<td>1630</td>
<td>654</td>
</tr>
<tr>
<td>60</td>
<td>2253</td>
<td>2094</td>
<td>1794</td>
<td>1744</td>
<td>699</td>
</tr>
</tbody>
</table>

Fig. 6. First natural frequency of a cantilever when the tip is in contact with a surface as a function of the applied force for several materials \( (f_0 = 13.17 \text{kHz}) \).
Fig. 7. Schematics of our mechanical equivalent model. The edge of the cantilever is linked to a spiral spring and a coil spring. The arrows represent the possible movement of each node.

5.2.1. Case of a beam linked with two springs

The first natural frequency of the cantilever when the edge of the cantilever is linked to a linear spring $k_1$ and a rotational spring $k_\theta$ has been estimated by computer calculation. This model shows that $f_1$ depend only on $k_{1,c}$ and $k_{\theta,c}$. Simulation results are presented in fig. 8. $f_1$ does not exceed 6.25, the value corresponding to the ratio of the first natural frequency of a “clamped-clamped” beam to the first natural frequency of a “clamped-free” beam [31].

5.2.2. Case of a cantilever in contact with an elastic surface

The tangential stiffness of an elastic contact between a sphere and a plane has been studied by Mindlin [6]. If there is no sliding the tangential stiffness $k_{1,c}$ is equal to:

$$k_{1,c} = \frac{F_1}{dx} = 8aG^s = \frac{4k_{n,c}G^s}{E^s}, \quad (13)$$

with

$$G^s = \left( \frac{2 - \nu_1}{G_1} + \frac{2 - \nu_2}{G_2} \right)^{-1}$$

$$= \left( \frac{2(1 + \nu_1)(2 - \nu_1)}{E_1} + \frac{2(1 + \nu_2)(2 - \nu_2)}{E_2} \right)^{-1}. \quad (14)$$

If $E_1 \ll E_2$ the relation between $k_{1,c}$ and $k_{n,c}$ becomes:

$$k_{1,c} \approx \frac{2(1 - \nu_1)}{(2 - \nu_1)} k_{n,c}. \quad (15)$$

The Poisson ratio $\nu_1$ varies from 0.1 (for diamond) to 0.5 (for rubber). So the ratio of $k_{1,c}$ to $k_{n,c}$ varies from 2/3 to 18/19 with an average value of 0.85. The first natural frequency of a cantilever in contact with a surface for an elastic contact could be calculated (fig. 9) with the equivalent rotation stiffness $k_{\theta}$ equal to (if $\theta \approx 0$):

$$k_{\theta} = \frac{\sin \theta}{\theta} Hk_{1,c} \approx Hk_{1,c}. \quad (16)$$

It is interesting to see that the first natural frequency is sensitive to normal stiffness or tangential stiffness only for some values of $k_{1,c}$, $f_c$ is no longer sensitive to $k_{1,c}$ if the natural frequency exceed a limit value $f_l$ equal to:

$$f_l = 4.375 f_c, \quad (17)$$

with $f_c = 1.02 f_n [30]$ and 4.375 the value corresponding to the ratio of the first natural frequency of a “clamped-hinged” beam to the first natural frequency of a “clamped-free” beam.

In our experiments, $f_n \approx 13.17$ kHz, the calculation leads to $f_l$ equal to 58.8 kHz. $f_c$ was at least equal to 60.2 kHz, a value only sensitive to the tangential stiffness. In this domain, if there is a partial slip the relative displacement $dx$ is equal to:

$$dx = \frac{3\mu F_n}{16aG^s} \left[ 1 - \left( 1 - \frac{F_1}{\mu F_n} \right)^{2/3} \right]. \quad (18)$$

For a modulation of force which leads to an amplitude of vibration of 1 $\mu$m without contact, the sliding distance would be about 5 nm. If we consider that the apparent tangential stiffness is equal to the tangential force divided by the total displacement, the apparent tangential stiffness is considerably lower than the value given by the formula (13). For example for a sliding distance of 10 nm, the apparent tangential stiffness is equal to 1/10 of the tangential stiffness without slipping (fig. 10). Furthermore, if there is sliding, a part of the energy is dissipated, introducing a damping effect.

These two phenomena could explain why the first natural frequency decreases with the applied load. An increase of the sliding distance or an increase of the damping leads to a decrease of the natural frequency.

6. Conclusions

Experimental results of direct FMM show:

(i) The inefficiency of the “two Kelvin–Voigt” model.

(ii) That the amplitude of vibration of the “A–B” signal is not, as usually said, representative of the vertical movement of the tip. It is mostly influenced by the horizontal movement (in the axis of the beam) of the tip. The
contrast of direct FMM image is essentially due to friction and could not be used for elastic imaging.

A new model which is able to explain our experiment is proposed. This model shows that direct FMM should be able to probe the Young modulus of a surface by measuring the first natural frequency of the cantilever. The important information is that the first natural frequency is sensitive to the normal stiffness of the contact only if it is of the same order of magnitude as the cantilever stiffness. For SFM classical experiments (Si$_3$N$_4$ cantilever, Young modulus of the tip $\approx 130$ GPa, tip curvature radius $\approx 20–50$ nm, applied force $\approx 1–200$ nN, Young modulus of the sample $\approx 0.1–1000$ GPa) the normal contact stiffness estimated by the Hertz theory is equal to $1–1000$ N/m. The stiffness of the cantilever (in the case of a rectangular beam) must be equal to 5 N/m to be able to give the normal stiffness of the contact. If the stiffness of the cantilever is too small, the natural frequency is sensitive to the friction and could not be exploited to measure elastic properties of materials.

In the case of indirect force modulation, the classical model (two Kelvin–Voigt elements in series) is probably not correct because it does not take into account the geometrical and the mechanical properties of the cantilever and the tangential contact stiffness. We believe that the response of the cantilever is also mostly influenced by friction, especially for low cantilever stiffness. Some models of indirect force modulation have been realized, and will be presented in a following paper.

Acknowledgement

The authors are grateful to: Joël Perret-Liaudet for his availability and patience during discussions about mechanical vibrations and for reviewing this manuscript; Emmanuel Varenne and Etienne Georges for technical assistance during cobalt laying.

Appendix: nomenclature

$\alpha$ radius of contact circle
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_v$</td>
<td>vibration amplitude</td>
</tr>
<tr>
<td>$C$</td>
<td>coefficient</td>
</tr>
<tr>
<td>$E$</td>
<td>Young modulus</td>
</tr>
<tr>
<td>$E'$</td>
<td>reduced Young modulus</td>
</tr>
<tr>
<td>$f$</td>
<td>frequency</td>
</tr>
<tr>
<td>$f_a$</td>
<td>first natural frequency of cantilever without contact in air</td>
</tr>
<tr>
<td>$f_c$</td>
<td>first natural frequency of cantilever when the tip is in contact with a surface</td>
</tr>
<tr>
<td>$f_r$</td>
<td>ratio of $f_c$ to $f_v$</td>
</tr>
<tr>
<td>$f_v$</td>
<td>first natural frequency of cantilever without contact in vacuum</td>
</tr>
<tr>
<td>$F_n$</td>
<td>normal force</td>
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<tr>
<td>$F_t$</td>
<td>tangential force</td>
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<td>$G$</td>
<td>shear modulus</td>
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<td>$G'$</td>
<td>reduced shear modulus</td>
</tr>
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<td>$H$</td>
<td>tip height</td>
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<tr>
<td>$I$</td>
<td>moment of inertia</td>
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<td>$k_{nc}$</td>
<td>contact normal stiffness</td>
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<td>$k_{1c}$</td>
<td>contact tangential stiffness</td>
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<td>$k_{11r}$</td>
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<td>$k_{01r}$</td>
<td>ratio of $k_{0c}$ (or $k_\theta$) to $k_{01}$</td>
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