 CMC, vol.37, no.2, pp.123-134, 2013

The Analytical and Numerical Study on the Nanoindentation of Nonlinear Elastic Materials

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Abstract: In nanoindentation testing of materials, the analytical/numerical models to connect the indentation load, indentation depth and material properties are crucial for the extraction of mechanical properties. This paper studied the methods of extracting the mechanical properties of nonlinear elastic materials and built general relationships of the indentation load and depth of hyperelastic materials combined with the dimensional analysis and finite element method (FEM). Compared with the elastic contact models and other nonlinear elastic contact models, the proposed models can extract the mechanical properties of nonlinear elastic materials under large deformation simply and effectively.

Keywords: Nanoindentation, dimensional analysis, hyperelasticity, contact mechanics.

1 Introduction

Nanoindentation technique is an important experimental method of measuring the mechanical properties of materials at micro-nano scales. In recent years, nanoindentation is becoming an increasingly popular materials characterization tool in various soft materials such as soft polymers, gels, biological materials and soft tissues [Briscoey, Fiori and Pelillo (1998); Ebenstein and Pruitt (2006); Lin and Horkay (2008); Cakmak, Schöberl and Major (2012)] because of its obvious advantages such as simple sample preparation, high load and displacement resolution. The traditional characterization methods which is developed based on the theory of elastic contact [Johnson (1987)], are mainly applied to the elastic or elastic-plastic properties for metals and ceramics [Oliver (2004); Fischer-Cripps (2006)]. However, most soft materials have complex composition and microstructure characteristics [Kurapati, Lu and Yang (2010); Pulla and Lu (2012); Oyen (2013)], low elastic modulus and high sensitivity to external stimuli [Oyen (2008); Hu, Zhao, Vlassak

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and Suo (2010); Chan, Deeyaa, Johnson and Stafford (2012)], their indentation behaviors usually exhibit large deformation or time dependence[Duan, An, Zhang and Jiang (2012); Oyen (2013)]. Therefore it is vital to develop appropriate analysis models for the characterization of their nonlinear or time-dependent mechanical properties.

For soft materials, the elastic modulus is usually very small on the order of 1 Mpa or even smaller, large deformation can be easily achieved. In the range of large deformation, most soft materials have typical nonlinear stress-strain characteristics that the stiffness changes as the strain increases [Basar and Weichert (2000)].The analysis methods based on the elastic contact models can be reliably applied to the characterization of metal and ceramic materials. It is common to apply these methods to analyze the indentation load-depth curve of soft materials at the small-strain range, for example, controlling the ratio of contact radius and depth less than 0.1 to meet the Hertz contact model in spherical indentation problems. Consequently, errors are frequently incurred and the transition from linear to nonlinear stress-strain behaviors may be ambiguous [Lin and Horkay (2008)]. It is very necessary to study the characterization techniques which can capture the large deformation and material nonlinearity characteristics currently.

It is difficult to obtain accurate analytical solutions of the nonlinear contact problem which involves geometry, material and boundary nonlinearity, so the related research is relatively less. Choi [Choi and Shield (1981)] and Sabin [Sabin and Kaloni (1983)] derived the general solutions(S-K model) of isotropic incompressible hyperelastic contact problems by second-order nonlinear analysis in a relatively small deformation ($a/R << 1$). Applying the second-order hyperelastic theory and finite element simulation, Giannakopoulos [Giannakopoulos and Triantafyllou (2007)] studied the spherical indentation problems of incompressible rubber-like materials and established a different analytical model (G-T model) with S-K model, finite element calculations and experimental results showed that the model was reliable under the conditions$h/R \leq 0.1$.Lin [Lin, Dimitriadis and Horkay (2007)] derived a series of approximate expressions of the relationships between indentation load and depth based on the hyperelastic strain energy functions by applying the concepts of equivalent indentation strain and stress. Based on the Hertz contact model and second-order approximation of spherical indenter shape function, Liu [Liu, Zhang and Sun (2010)] derived a nonlinear elastic contact model, finite element calculations and experimental results show that the model can be used to predict the large indentation behavior of materials. Fig.1 and Fig.2 compare the indentation responses given by Hertz model, S-K model, G-T model and Liu model. As can be seen from the figures, when the deformation is small, the normalized load-displacement curves are in good agreement. With the deformation increasing, the normalized load-displacement curves of S-K model and G-T model show significantly deviation, as their applications are limited by the second-order nonlinear analysis and only valid for$h/R \leq 0.1$. Liu model shows more softer load than the Hertz model, which is mainly due to the indenter shape function using the second-order approximation of spherical functions, when the indentation depth is same, the contact areas is smaller.

To effectively use nanoindentation to characterize the mechanical properties, it is necessary to build the proper analytical or numerical models which relate the indentation load, depth and the mechanical parameters. To date, it is still very difficult to measure the nonlinear mechanical properties as their nonlinear mechanical characteristics and complex stress state in indentation process. In this paper, the nonlinear contact problems were investigated combined with theoretical analysis and finite element simulation. Dimensional analysis was carried out to characterize the relationships between the indentation responses and the hyperelastic properties of indented materials. FEM calculations were performed to obtain the explicit expressions of indentation load and displacement. The proposed models were verified and compared with the other contact models to illustrate the validation and application.

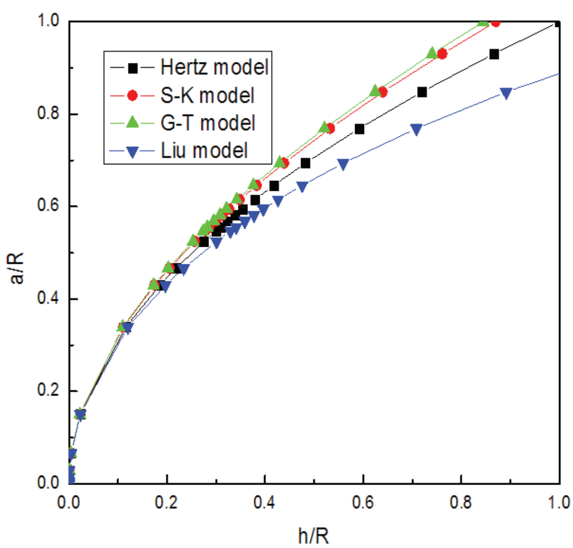

Figure 1: Relation between contact radius and depth by different contact models

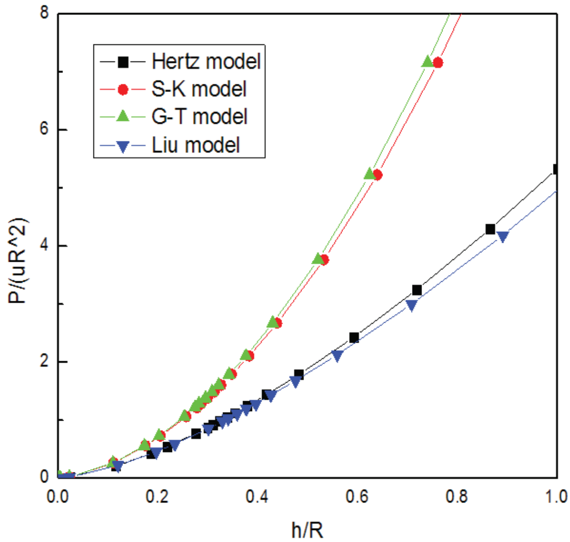

Figure 2: Normalized load-displacement curves by different contact models

2 Dimensional analysis

Dimensional analysis presented in early twentieth century is a method of establishing mathematical models in the field of physics which can build the relationships between different physical quantities by identifying their dimensions. Dimensional analysis has been a powerful tool for the application of physical theory to solve practical problems, which can not only simplify the processing of the physical rules, but also can be used to check the correctness and completeness of physical equations and empirical formula. Dimensional analysis provides an important methodto study the problems of indentation [Cheng and Cheng (1998 and 2004); Cao and Lu (2004); Cao, Ji and Feng (2010)] and is helpfull to promote the understanding and application of indentation technique.

For linear isotropic elastic materials, the constitutive model can be described by Hooke's law $\sigma = E\varepsilon$, where E is the elastic modulus, σ and ε are stress and strain, respectively. Consider the indentation of a rigid indenter with arbitrary profile pressed into a homogeneous, isotropic and linear elastic material,assuming no friction between the contact surfaces. The profile of the indenter can be described by a finite number of geometric parameters

$$S_{\text{indenter}}=\phi(c_0, c_1, c_2, \ldots, c_m) \quad (1)$$

where subscript m is an integer, c_m are geometric parameters; φ is a real function.

During the indentation experiments, the indentation load P can be expressed as a function of the related independent variables:

$$P = f(h, E, \upsilon, c_0, c_1, c_2, \ldots, c_m) \tag{2}$$

where f is a real function, h is the indentation depth. For the spherical indentation of linear isotropic elastic materials, the radius of the spherical indenter R is the only geometric parameter, Poisson's ratio is assumed to be a constant. The indentation depth and elastic modulus has the independent dimensions. According to Pi-theorem in dimensional analysis, the general expression of the load-depth relationship for the spherical indentation of a linear elastic solid is given as

$$\frac{P}{Eh\sqrt{Rh}} = \Pi(\frac{h}{R}) \tag{3}$$

where Π is a dimensionless function.

Hyperelastic constitutive models are usually used to describe the nonlinear mechanical behaviors of materials such as rubber-like material, soft tissues, cells and DNA, the stress-strain relationships can be derived through a strain energy density function. In order to describe the various deformation responses of materials, many strain energy density functions are proposed where Neo-Hookean models, Mooney-Rivlin models and Arruda-Boyce models are commonly used [Mooney (1940); Arruda and Boyce (1993)]. Neo-Hookean model for hyperelastic material can describe the mechanical responses of materials under small and finite strain, the strain energy density function can be expressed by

$$\psi = c_{10}(I_{C1} - 3) + \frac{1}{D_1}(J-1)^2 \tag{4}$$

where ψ is strain energy density function, I_{C1} is the first deviatoric strain invariants, J is the volume ratio of deformed state and undeformed state,D_1determines whether the material is compressible, if D_1 is 0,the materials is completely incompressible. c_{10} is the material parameter, the initial shear modulus is $\mu_0 = 2c_{10}$, the initial bulk modulus is $k_0 = \frac{2}{D_1}$. Similar to linear elastic indentation, taking the indentation depth h and the initial shear modulus μ_0 as the independent variables in dimensional analysis, the general expression of the load-depth relationship for the spherical indentation of a hyperelastic solid can be given as

$$\frac{P}{\mu_0 h\sqrt{Rh}} = \Pi(\frac{h}{R}) \tag{5}$$

Due to the limitations of dimensional analysis, the load-depth relationships remain semi-quantitative, the dimensionless functions or constants also need to be further determined by other methods. In the indentation, the specific forms of the dimensionless function generally can be determined through the finite element methods and then the explicit expressions of indentation load-depth relationships can be obtained.

3 Finite element analysis and verification

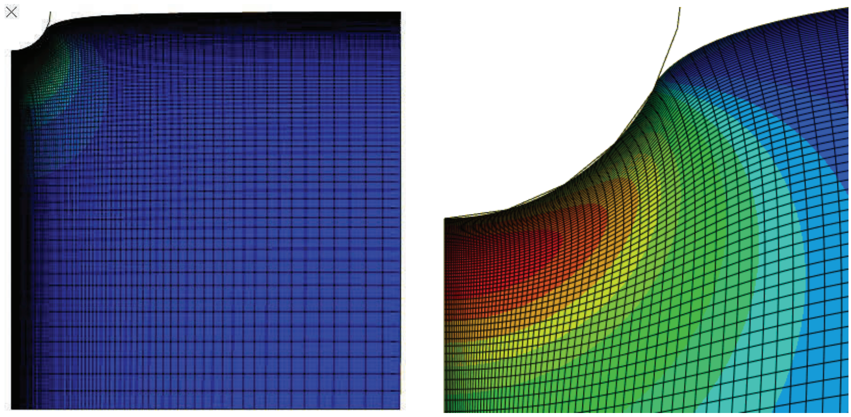

Figure 3: Spherical indenter and FEM mesh

The indentation method based on the linear elastic contact theory such as compliance method can successfully be used to extract the linear elastic properties of soft materials under small deformation. But for the general soft materials, their mechanical responses generally involve large deformation and the stress-strain relationships exhibit strong non-linear characteristics. Because of the complexity of the contact problems, finite element method (FEM) has become a powerful tool in indentation problems. Considering geometric and material nonlinear characteristics of materials, the finite element software ABAQUS 6.11[ABAQUS (2009)] was used to simulate the nanoindentation process of the nonlinear elastic materials.

In the FEM calculations, a 2D axisymmetric model with a spherical indenter was used to simulate the indentation tests, the spherical indenter and the finite element mesh were shown in Fig.3. Spherical indenter had a radius of 50um and was modeled as an analytically rigid. The substrate is an axisymmetric deformable solid and discretized by applying the bilinear axisymmetric reduced integration elements, a refined mesh was employed in the contact zone beneath the indenter tip to ensure the accuracy of the numerical results. The lower surface nodes of the sample are fixed, the forces is exerted at the reference point of the indenter. The contact between the spherical indenter and the sample was defined as frictionless. The choice of material parameters had no effect on the determining of the dimensionless functions, the initial shear modulus 2MPa and Poisson's ratio 0.5 are selected, the specific parameters of hyperelastic materials [Chen and Diebels (2012)] are given in Tab.1.

Table 1: Material parameters

Material models	Neo-Hookean	Mooney-Rivlin		Yeoh model		
Material parameters	c_{10}/MPa	c_{10}/MPa	c_{01}/MPa	c_{10}/MPa	c_{20}/MPa	c_{30}/MPa
	1	0.256	0.744	1.0	2.0	5.0

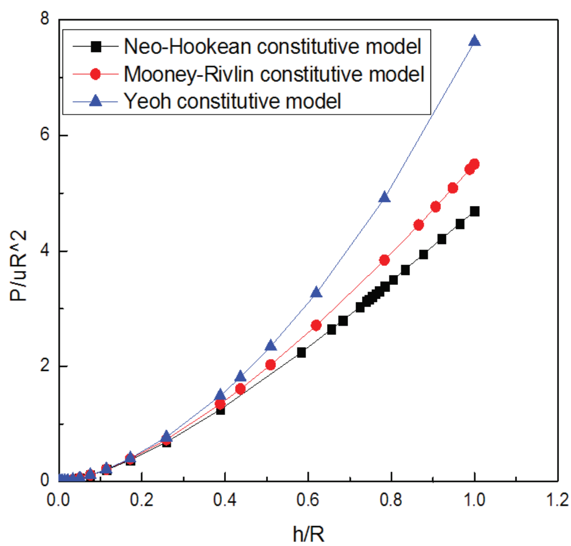

Figure 4: The results of FEM calculations

Based on the results of dimensional analysis, the explicit expressions of load-displacement relationships for the indentation of hyperelastic materials still need to be further determined through the finite element calculations. The process of indentation experiments for the Neo-Hookean, Mooney Rivlin and Yeoh models were simulated, the calculated indentation load and displacement data were used to determine the dimensionless functions. Fig.4 shows the dimensionless indentation load and displacement curves of three hyperelastic materials. As can be seen that three different hyperelastic materials show different indentation behaviors especially when the deformation is large enough though the three curves agree very well under small deformation, as the indentation depth increases, the deviation is greater. Therefore, different hyperelastic constitutive models have different non-dimensional functions. Combined with dimensional analysis and finite element calculations, the explicit expressions of load-displacement relationships for the indentation behaviors of Neo-Hookean, Mooney Rivlin and Yeoh hyperelastic models can be obtained, the general relationship of indentation load and displacement for Neo-Hookean materials is

$$P = \mu_0 h\sqrt{Rh}\left[5.34931\text{-}0.40638\times(h/R)\text{-}0.25329\times(h/R)^2\right] \tag{6}$$

Correspondingly, Hertz contact model can be written in dimensionless form:

$$\frac{P}{\mu_0 h\sqrt{Rh}} = \frac{16}{3} \tag{7}$$

The difference between Eq. (6) and Eq. (7) is a function of h/R and totally shows gradually increasing trend with the increase of h/R. Application of Hertz model to determine the initial shear modulus will bring some errors and basically the errors increase monotonely with the increase of h/R. The initial shear modulus extracted through Hertz model will significantly deviate from the true values under large deformation.Fig.5 compares the dimensionless indentation load and depth curves of Hertz contact model, the Liu model and Eq.(6). As can be seen that when the deformation is small (less than 0.1), the dimensionless load-displacement relationships are in good agreement; with deformation increase, the dimensionless load-displacement relations are significantly different. Compared with the Hertz model, Liu model and Eq. (6) both show softer loads.Liu model applies the second order approximation of spherical functions as the indenter shape function, the contact area is smaller when indentation depth is the same. Eq.(6) in which the geometric and material nonlinear are both considered, is mainly due to that Neo-Hookean material exhibits softening in larger deformation.

The indentation experiments of several different Neo-Hookean materials are simulated with the FEM model to verify the proposed models. The simulation results of Neo-Hookean materials with the initial shear modulus 1 Mpa and the fitted results by the Hertz model are shown in Fig.6.It can be shown that the proposed model presents an excellent prediction of the indentation behavior under large deformation. Hertz model can well fit the FE results under small deformation, but shows obvious deviation as the deformation increases. The initial shear modulus obtained by Hertz model is 0.91Mpa with as error of 9%, the proposed model can describe accurately the indentation behavior of soft materials under large deformation.

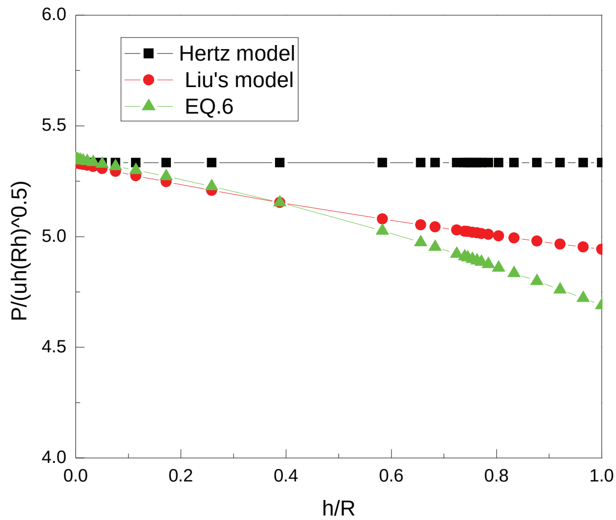

Figure 5: Comparisons among Hertz model, Liu model and the present model

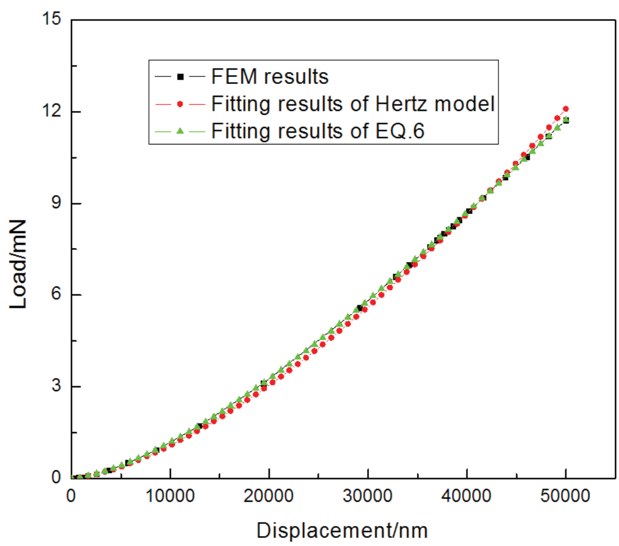

Figure 6: FEM verification

4 Conclusions

In this paper, the indentation methods of extracting the mechanical properties of nonlinear elastic materials were investigated and a general relationship between indentation load and depth of hyperelastic materials was built through dimensional analysis and FE simulation. The numerical examples show that the model is available for the large indentation responses of nonlinear elastic materials. Although the present analysis method is only applicable to the cases of the given constitutive response of materials, relative to the Hertz model and other nonlinear elastic contact model, it is a simple and effective method to extract nonlinear elastic properties under large deformation. In addition, this paper considers only the initial shear modulus of hyperelastic materials. The characterization method for other parameters of hyperelastic materials still needs to be further studied.

Acknowledgement: Financial support from the Natural Science Foundation of China under grant #11172012 and National Key Technology R & D Program under grant # 2012BAK29B00 is gratefully acknowledged.

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