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Dimensionless Analysis to Determine Elastoplastic Properties of Thin Films by Indentation

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Abstract: By assuming the elastoplastic properties of thin-film materials, a reverse analysis method is proposed by deriving a dimensionless function for the indentation process. The substrate effect is taken into account by assuming a perfect interface between thin-film and substrate materials. In order to obtain the applied load–penetration depth (P – h) curves, the indentation process is numerically modeled as an axisymmetric problem with a rigid-body Berkovich indenter on the semi-infinite substrate when performing finite element (FE) simulations. As a typical soft film/hard substrate problem, the elastic substrate is assumed and the power-law model is used to describe the constitutive properties of thin-film materials. Varying elastic modulus (10–50 GPa), yield strength (60–300 MPa), and hardening exponent (0.1–0.5) characterize different elastoplastic mechanical properties of thin-film materials with film thickness of 10–30 μm . Owing to the good trending P – h curves with the maximum indentation depth up to the 2/3 film thickness for different elastoplastic thin-film materials, a dimensionless function is derived and validated based on the predictions by reliable FE simulations. The proposed dimensionless function elegantly elucidates the essential relationship between the elastoplastic mechanical properties of the thin-film material and indentation responses (e.g., loading and unloading variables). The elastoplastic constitutive curves predicted by the proposed reverse method are confirmed to be in good agreement with the stress-strain curves of materials by FE simulations with the randomly selected elastoplastic mechanical properties and film thicknesses. This study provides a theoretical guidance to understand the explicit relationship between elastoplastic mechanical properties of the thin-film material and indentation responses.

Keywords: indentation; thin-film; reverse analysis; dimensionless function; finite element simulation



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1. Introduction

Thin film/substrate systems are of significance in numerous critical engineering applications such as micro-electronics, optoelectronics, display panels, and many other devices, as illustrated in Figure 1. Processing techniques, for instance, sputtering, vapor deposition, ion implantation, and laser glazing are employed to fabricate thin film/substrate systems [1–3]. The reliability of microelectronic devices relies primarily on how reliably the thin films adhere, or “stick” to each other and substrates [4], thus the adhesion measurement of thin film has been of great interest in recent years. Carbon nanotubes and graphene are successfully applied to energy conversion systems, including solar cells and fuel cells, due to their excellent structures and properties in many vital areas [5]. A new domain of technical physics, integrated optics, is stimulated by the technical problems related to the widespread application of laser technology, both for actual aims (communication, information processing, ranging, etc.) and for physical research [6]. In addition, there are various applications, from computer hardware and sensors to thin films and coatings, where important parts are manufactured in small sizes and low thicknesses [7,8]. Depth-sensing nanoindentation measurement techniques are frequently utilized to determine the mechanical properties of surface layers and ultrathin coatings of bulk materials [9]. In fact,

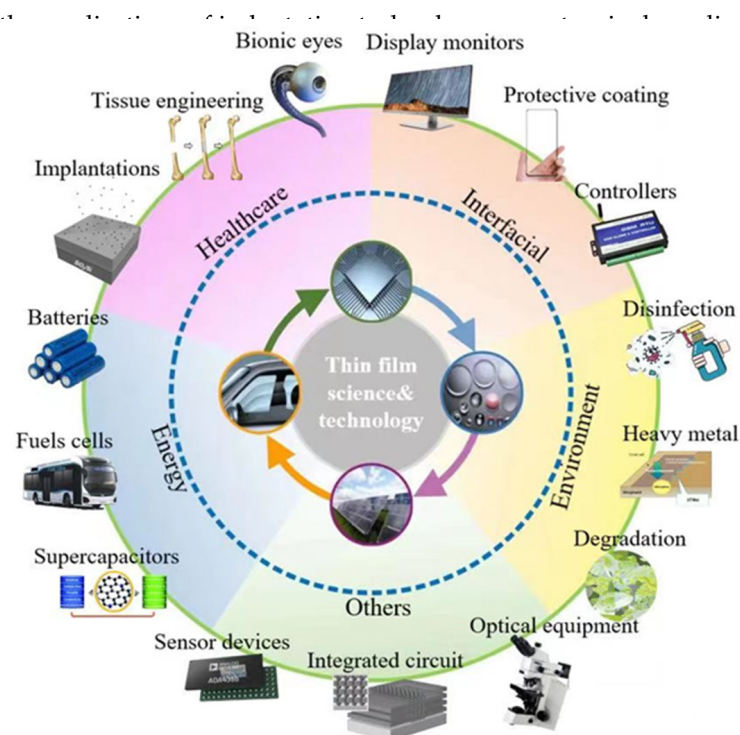


Figure 1. Schematic diagram of thin-film applications in various areas.

The method proposed by Oliver and Pharr in 1992 to measure hardness and elastic modulus by indentation techniques, has extensively been applied and used for characterization of small-scale mechanical behavior [10]. In recent decades, because of its good performance in characterizing the mechanical properties of each material at low scales, the nanoindentation theory technique has been applied to measure the mechanical properties of micro-electronics and nano-scale component materials as well for local mechanical properties of materials [11]. For anisotropic materials, the question is what modulus is measured by the indentation technique due to load and displacement [12]. On the microscopic aspect, Bouzakis et al. [13] investigated the effect of surface roughness of the substrate material due to different size shapes in nanoindentation experiments based on FE simulation. Rauchs [14] performed nanoindentation experiments to determine the material parameters of an elastic-viscoplastic material model with nonlinear isotropy and kinematic hardening by minimizing the least-squares difference between experimental data from indentation tests and FE predictions. Shelef and Bar-On [15] related the indentation modulus magnitude and loss parameters to the modulus of the original film to derive the film-substrate energy storage and energy dissipation capabilities, and outlines a method to back-calculate the elements by simple linear scaling. Kampouris et al. [16] presented a new approach to interpreting Vickers microindentation data, using a gradient elastic framework fitting formulation, and showed experimentally the potential of the formulation to model such microdeformation issues. With regard to improving the accuracy of material parameters measured by nanoindentation, Chollacoop et al. [17] proposed an empirical method to improve the accuracy of material parameter measurement by enriching the indentation method with utilizing double sharp indenters with different tip-top angle. Treier et al. [18] combined the genetic algorithm with the requested parameters and applied to the identification of material parameters. Kim et al. [19] proposed a size effect model by investigating the effect of roughness in nanoindentation experiments. Based on the first-order asymptotic solution, a simple analytical approximation is suggested for the indentation scaling factor that takes into account the elastic layer's finite thickness as well as the effect of the elastic substrate [20]. By conducting indentation experiments, Long et al. [21–24] investigated the residual stresses in Sn-3.0Ag-0.5Cu lead-free solder

after annealing treatment, and discussed the effects of different annealing temperatures and durations on the applied load–penetration depth (P - h) curves. Long et al. [25] established a quantitative relationship between indentation strain rate and uniaxial tensile strain rate by introducing the concept of rate factor, which is based on a set of polynomial functions connecting the instantaneous and residual plastic zone radii and helps to break the uniqueness of determining the unique elastic-plastic properties of a material by indentation. Researchers focus on the effect of surface stress by pre-stressing the materials to be indented by a Berkovich indenter [26]. In order to improve the accuracy of the reverse calculation, a great deal of work has been done using different shapes of material properties of the indenter [27–29], proposing advanced algorithms for FE simulations [30,31]. By using dimensionless analysis, FE results are first combined with dimensionless functions, and then a series of nonlinear fitting equations are proposed to determine the dimensionless equations for the mechanical properties of elastic-plastic solids with work hardening [32]. It is noticed that these studies extended the application of indentation to characterize mechanical properties such as surface stresses, residual stresses, and plastic properties of elastoplastic substrates. However, existing studies were limited to the cases with only the substrate model. This means that the methods and conclusions from existing studies cannot shed light on the cases for film/substrate model. This underlines the urgent necessity to incorporate thin-film materials in the FE model and consider the influence of the presence of thin films on the whole process of indentation.

The purpose of this paper is to develop a method involving a combination of indentation techniques and FE simulations to describe the mechanical behavior of thin films. The reliability of the modeling is investigated mainly through simulation to verify the experimental phenomena. The film is further assigned on the basis of a pure substrate model in order to find out the influence of film properties on the indentation process. Finally, a reverse analysis based on P - h curves as indentation response is presented in this paper to acquire the mechanical properties of the substrate material. However, the theory of the indentation technique is not well established especially for thin film materials. Hence, a dimensionless algorithm based on the P - h curve as the indentation response is proposed to obtain the mechanical properties of the thin film material.

2. Indentation Simulation by FE Method

2.1. Validation with No-Film Model

As thin metal films are widely used in microelectronic devices, optics, and other fields, the mechanical properties of the films are also required in practice. Hence, it is especially important to experimentally and numerically study the mechanical properties of thin films. The indentation method can better address these problems and obtain the measurement results with sufficient accuracy. However, the indentation method inevitably brings some errors in the process of testing, such as local wear of the indenter, microscopic defects on the surface of the specimen, and small changes in the experimental environment. These minor error sources can be neglected in macro-scale studies. But these error sources can greatly reduce the accuracy of measurement results in the nano-scale. With the help of FE analysis, the application of indentation characterization of various mechanical theoretical models can be significantly improved, by improving the mechanical properties of materials in terms of measurement accuracy. Therefore, the information provided by FE simulations combined with indentation tests can be more comprehensive. By performing FE simulations, the predictions based on the three-dimensional Berkovich model are in general accordance with those by using the two-dimensional equivalent model as found by Zhuk et al. [32]. Essentially, the standard Berkovich indenter enables the consistency in terms of projected area by equivalent sharp indenters (e.g., conical, triangular pyramidal and square-based pyramidal indenters) for the indentations [33,34].

Figure 2 shows the schematic diagram of the FE model for indentation simulation with a film/substrate structure by using a Berkovich indenter. The film is attached on top of the substrate by assuming a perfect interfacial adhesion. This means that no delamination

failure is taken into account in the present FE model. Regarding the loading condition, displacement control is applied on the indenter by means of a reference point. For the Berkovich indenter, the FE model can be simplified as an axisymmetric model with the indentation half-angle of 70.3° . The substrate is regarded as a semi-infinite space body with a height of $3000\ \mu\text{m}$, and the film thicknesses are selected as $10\ \mu\text{m}$, $20\ \mu\text{m}$, and $30\ \mu\text{m}$ for the parameter identification of subsequently proposed dimensionless functions, and the maximum penetration depth is selected as $2/3$ of the film thickness. The mesh discretization of the FE model is confirmed to be small enough to simulate such a semi-infinite space problem after performing mesh sensitivity study.

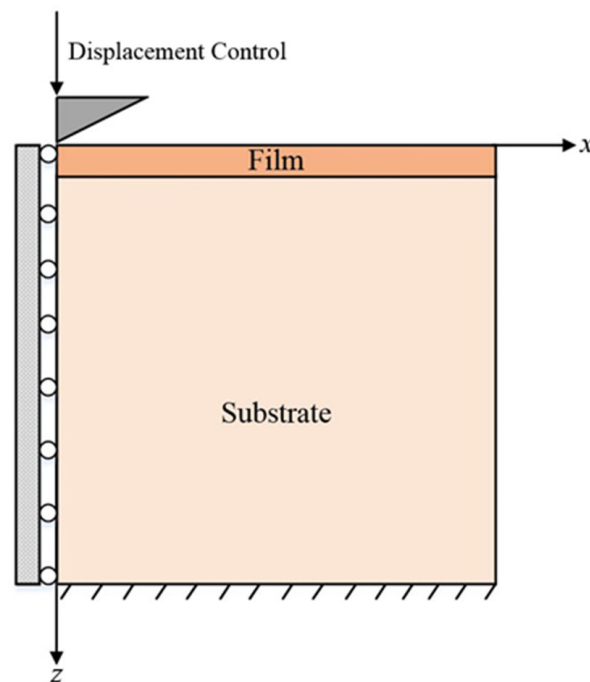


Figure 2. Schematic diagram of the indentation model with a film/substrate structure under a Berkovich indenter.

For the axisymmetric boundary condition, appropriate constraints are applied to the left and lower sides in the semi-infinite substrate to simulate the real experiments. As the stress distribution in the substrate induced by the indenter penetration is localized, the left side of the semi-infinite substrate is free. The vertical displacement is applied at the reference point of the indenter to penetrate the indenter into the film/substrate structure. The applied load P on the indenter and the penetration depth h can be recorded throughout the indentation process, so that the P - h curves under different working conditions are utilized to characterize the mechanical response of the film/substrate structure. In the elastoplastic deformation process, the substrate is considered to be elastic as the induced stress is not significant, while the film material is identified by a piecewise elastoplastic model with the plastic stage described by a power-law model which is usually expressed as

$$\sigma = \begin{cases} E\varepsilon & \varepsilon \leq \varepsilon_y \\ R\varepsilon^n & \varepsilon > \varepsilon_y \end{cases}, \quad (1)$$

where E is the Young's modulus, R is the strength coefficient, n is the hardening exponent, and the yield strain ε_y is the corresponding to the yield strength which can be expressed as

$$\sigma_y = E\varepsilon_y = R\varepsilon_y^n, \quad (2)$$

The total strain can be represented by the sum of the elastic strain ε_e and the plastic strain ε_p as

$$\varepsilon = \varepsilon_e + \varepsilon_p, \quad (3)$$

Combining the above equations, the stress σ can be written as

$$\sigma = \frac{\sigma_y}{\varepsilon_y^n} (\varepsilon_e + \varepsilon_p). \quad (4)$$

For validating the established FE models for simulating indentation response, a series of comparisons is made for the FE predictions with the reported experimental results in the literature. First, considering a simpler indentation condition without the composition of film and substrate in the FE model, the predicted P - h curves are compared with the experimental results [34]. Table 1 lists the mechanical properties for both elastic Berkovich indenter and elastoplastic substrate. A wide range of film materials is elastoplastic materials with the film thickness of 10–30 μm and the mechanical properties for Young's modulus of 10–50 GPa, yield strength of 60–300 MPa, hardening exponent of 0.1–0.5, and Poisson's ratio of 0.07. As shown in Figure 3, the solid line indicates the FE simulations and the spheres represent the experimental measurements [34], both of which are in good agreement, confirming the accuracy of the FE models when predicting the indentation response.

Table 1. Mechanical properties for indenter and substrate.

-	E (GPa)	ν	n	σ_{fy} (Mpa)
Berkovich indenter	1060	0.07	-	-
Substrate	215	0.28	0.13	330

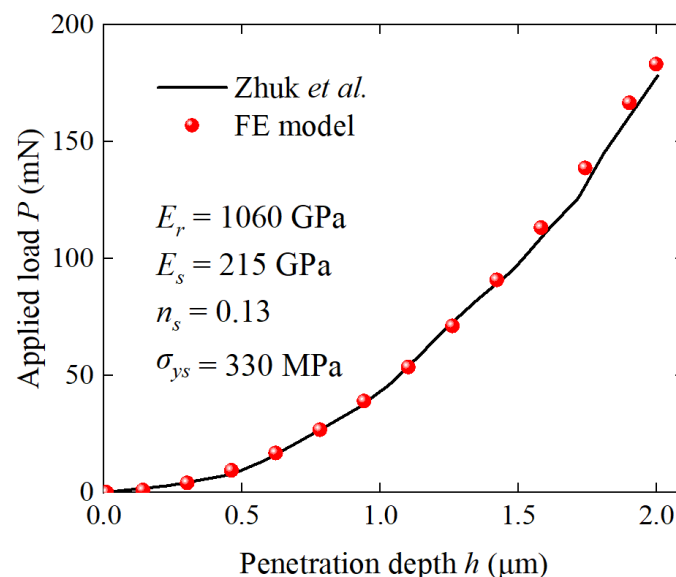


Figure 3. Response comparison of the established FE model and published result [34].

In addition to the validation of loading stage, the full process for both loading and unloading is adopted to validate the indentation prediction accuracy of the established FE model with the published results [35], as shown in Figure 4. In this validation, the maximum value of penetration depth is 200 nm. For substrate, the Young's modulus is 65 GPa and the yield strength is 300 MPa. For indenter, the Young's modulus is 1141 GPa and the Poisson's ratio is 0.07. As shown in Figure 4, compared with the validation case with 2 μm , the penetration depth of 200 nm is much smaller. This means the established FE models are nano and micro-scales capable of predicting the indentation behavior for both nano and micro-scales.

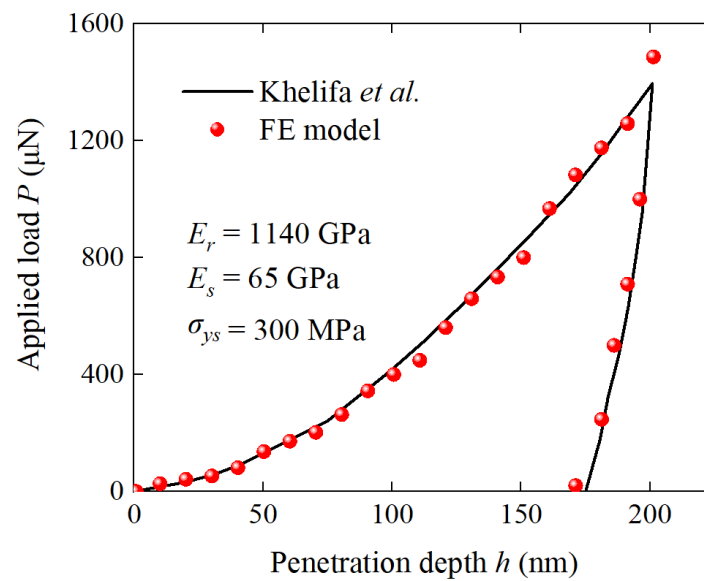


Figure 4. Response comparison of the established FE model and published result [35].

2.2. Validation with Film/Substrate Model

After validating the FE model for the indentation of homogenous substrate materials, the experimental result of composite materials with both film and substrate [36] are also taken into account for validating the prediction accuracy of the established thin film/substrate model. The indenter material is diamond with Young's modulus of 1141 GPa, and Poisson's ratio of 0.07. The film material is TiN with the thickness of 2 μm , Young's modulus of 427 GPa, Poisson's ratio of 0.25, and yield strength of 13,500 MPa. For the substrate, the Young's modulus is 218 GPa, Poisson's ratio is 0.3, yield strength is 1800 MPa. With the good agreement of P - h curves as shown in Figure 5, the established FE model is confirmed to be satisfactory to predict the indentation behavior of thin film/substrate structure. Moreover, after further examining the stress distribution in film/substrate as shown in the insert of Figure 6, severe plastic deformation is induced in the thin film but elastic deformation is dominant for the substrate materials. So, it is reasonable to assume elastic substrate material and focus on the elastoplastic behavior of thin-film materials in the subsequent FE simulations with film/substrate of the present study.

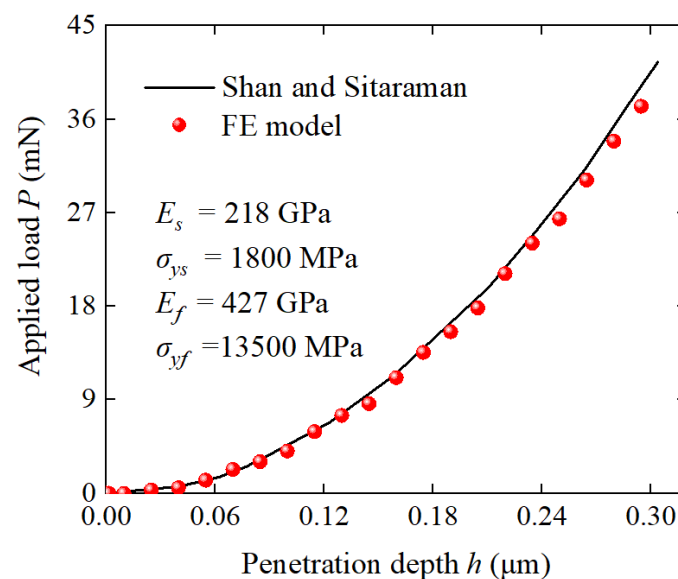


Figure 5. Response comparison of the established FE model and the published result [36].

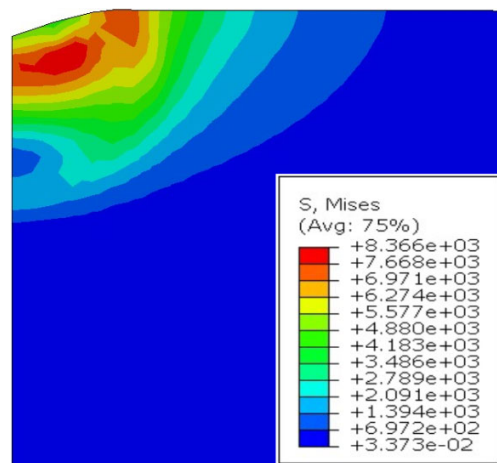


Figure 6. Stress deformation diagram for indentation depth of 0.3 μm .

Subsequently, the indentation on hard film/soft substrate and soft film/hard substrate material systems was comprehensively investigated. The soft and hard materials are defined as isotropic elastic-perfectly plastic materials. Young's modulus and yield strength of the soft material are taken as 100 GPa and 1000 MPa, respectively. The corresponding values for the hard material are 200 GPa and 20,000 MPa. The depth of substrate is 200 μm , while the thickness of thin films is 2 μm . The distributions of equivalent plastic strain in both cases are compared in Figures 7 and 8. It was found that the deformation and the corresponding value PEEQ under different working conditions are very close to each other.

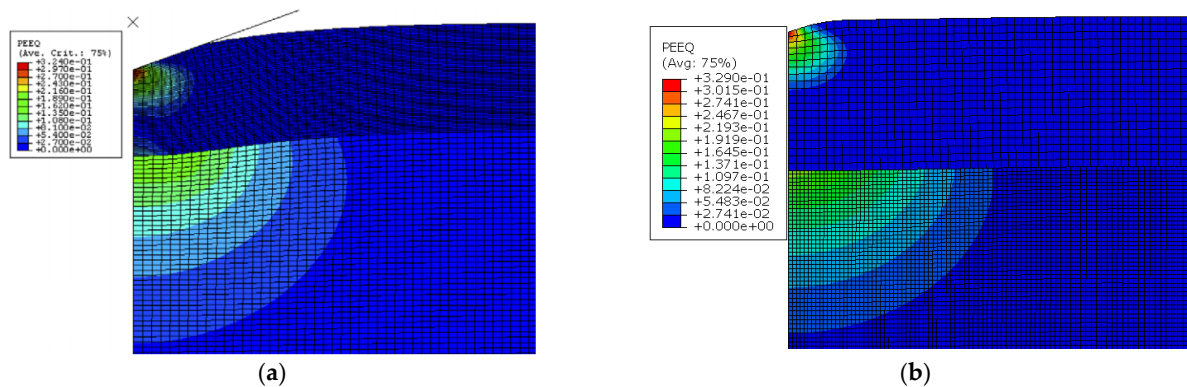


Figure 7. PEEQ comparison in the hard film/soft substrate model at $h_{\text{max}} = 900 \text{ nm}$: (a) reported result by Pelegri et al. [37] (maximum value is 0.3240); (b) FE result (maximum value is 0.3290).

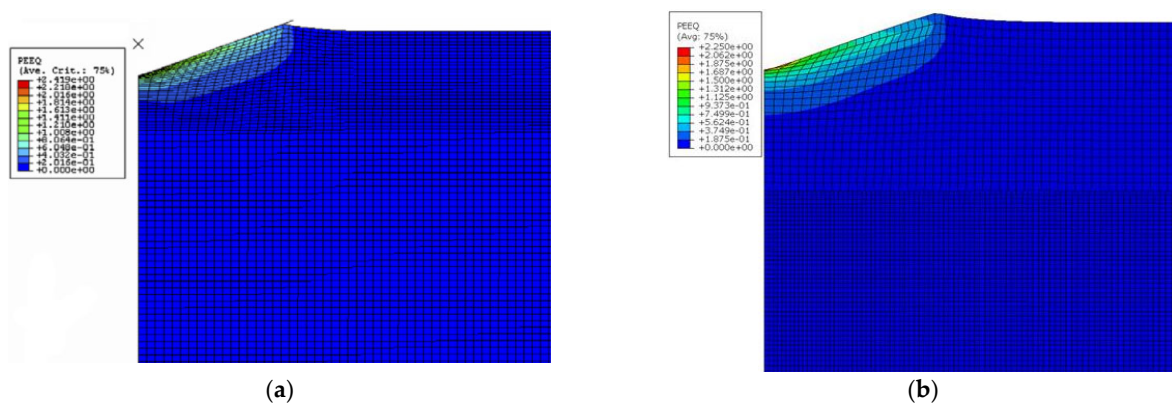


Figure 8. PEEQ comparison in soft film/hard substrate model at $h_{\text{max}} = 900 \text{ nm}$: (a) reported result by Pelegri et al. [37] (maximum value is 2.419); (b) FE result (maximum value is 2.250).

3. Dimensionless Analysis

Indentation tests are versatile in terms of controlling the applied displacement, load, and time sequence, which can be illustrated in Figure 9. An indentation process includes usually at least a sequence of loading and unloading. In the local region underneath the indenter, elastic and plastic deformations occur when the indenter is penetrated into the substrate material until the maximum penetration depth h_{max} . The indentation develops with a shape corresponding to the indenter shape. Since the plastic part does not fully recover after the complete unloading of the load on the indenter, only the part of the elastic deformation can be recovered until the residual penetration depth h_r . Elastic properties can be extracted from the elastic part of the unloading curve. This will facilitate the use of the contact stiffness S as the initial slope of the unloading curve to quantify the elastic properties such as the Young's modulus. Considering the composition of film and substrate of interest in the present study, the mechanical properties of the films can be further elucidated by proposing the dimensional analysis based on extensive FE simulations.

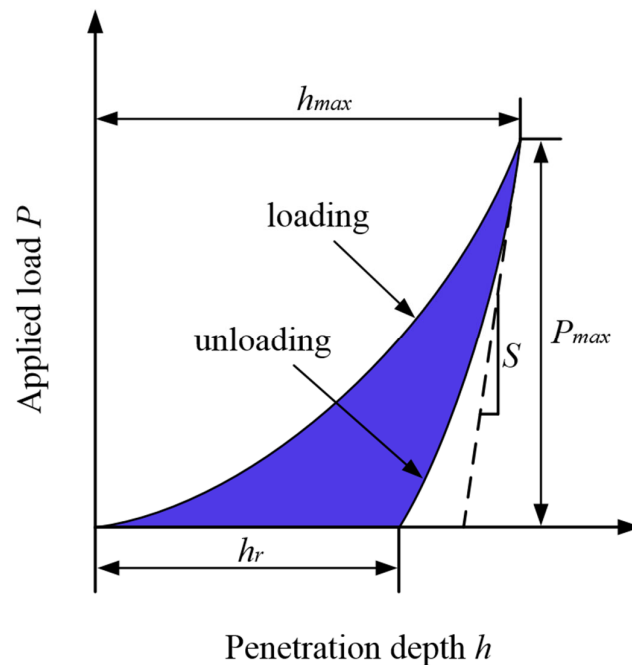


Figure 9. Typical P - h curve under the thin film/substrate model.

The continuous measurement of the applied load during the loading stage can be utilized to measure the hardness. As the average contact pressure of the material under the indenter [37], hardness exhibits the material ability to resist local deformation, especially plastic deformation, indentation, or scratching. Conventionally, hardness is defined by

$$H = \frac{P}{A_c}. \quad (5)$$

where P is the applied load that is obtained directly from the P - h curve, and A_c is the projected contact area of the indenter tip in the substrate material which can be determined for a Berkovich indenter as

$$A_c = 24.5h^2. \quad (6)$$

For a conical indenter in this axisymmetric problem, the contact stiffness S can be used to calculate the Young's modulus of the material and denoted as

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A_c}. \quad (7)$$

where dP/dh is the initial slope of the unloading curve, and E_r is the reduced Young's modulus of the material which can be defined as

$$\frac{1}{E_r} = \frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_i^2}{E_i}. \quad (8)$$

The reduced Young's modulus E_r is evaluated from the indentation measurements according to the following equation

$$E_r = \frac{\sqrt{\pi}}{2} \frac{(dP/dh)_{\text{unload}}}{\beta \sqrt{A_c}}. \quad (9)$$

where $(dP/dh)_{\text{unload}}$ is the slope of the unloading curve evaluated at the position of maximum load. β is a dimensionless parameter related to the geometry of the indenter. For the case of tapered indenter $\beta = 1$ [38].

However, it should be noted that the above equations cannot be applied directly for the mechanical characterizations of thin films which are supported by their substrates, as the substrate effect cannot be neglected. Usually, in order to avoid the influence of the substrate for measuring reliable mechanical properties, the penetration depth should be less than 10% of the film thickness. On the other hand, if the penetration depth is too small by satisfying this limitation, the size effect should be concerned for such shallow indentations. Therefore, the above equations can still be used but with a compromised penetration depth for thin-film materials. This work is motivated to propose a method to determine the mechanical properties of thin-film materials by considering the substrate effect.

The applied load P generated by the entire indentation process is extremely dependent on the mechanical parameters between the substrate and the film. As revealed by the FE predictions, similar trend is expected to be captured by the proposed dimensionless function in a similar way of the works done by Long et al. [39,40]. During the elastic-plastic indentation deformation, the applied load P can be given as

$$P = P(E_s, E_f, n_f, \sigma_{fy}, h, t), \quad (10)$$

where E_s is the Young's modulus of the base material, E_f and n_f are the Young's modulus and hardening exponent of the Berkowitz indenter, σ_{fy} is the yield strength of the film, h is the penetration depth of the indenter, and t is the thickness of the film. Since the indenter parameters are constant, the material and geometric parameter related with the indenter is not considered.

As identified in Equation (10), seven variables with three independent dimensions (that is, length, mass, and time) are utilized. By proposing four dimensional terms, the dimensionless functions are rewritten as

$$\frac{P}{E_f \cdot h^2} = \Pi \left(\frac{n_f \cdot E_f}{E_s}, \frac{\sigma_{fy}}{E_f}, \frac{h}{t} \right). \quad (11)$$

By identifying the values of the dimensionless parameters, the dimensionless formulae are derived in an elegant way to better reproduce the FE predictions. In order to increase the reliability of the parameter fitting for the proposed dimensional analysis, random work conditions with statistical distributions are considered when preparing the database of FE simulations. For the selection of working conditions in this study, in addition to varying the values with certain increments to cover the whole range of mechanical parameters, some more working cases are also determined randomly so as to enrich the generalization of the indentation database. For instance, the histogram of the determined thickness t is shown in Figure 10. The other parameters such as Young's modulus, yield strength, and hardening exponent are also randomly determined in their individual ranges. The indentation database with both incremental and random conditions can effectively enhance the parameter fitting of the proposed dimensionless function.

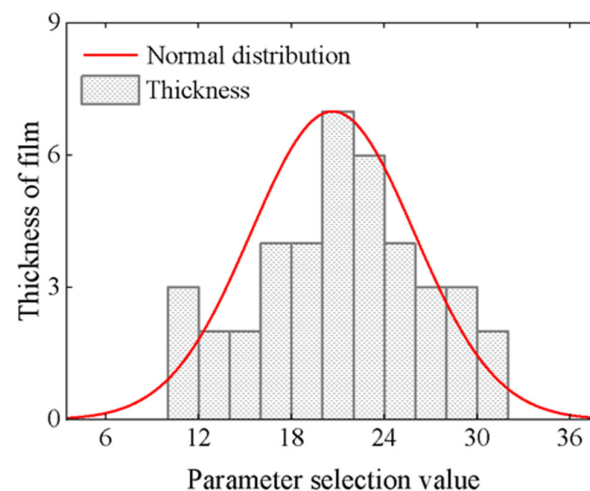


Figure 10. Distribution histogram of the thickness randomly determined in the range of interest.

4. Results and Discussions

Young's modulus of the substrate is much larger than that of the film, which is the standard case for the soft film/hard substrate of interest in this study. For the parameter selection of FE simulations, the reasonable ranges of mechanical properties for the thin-film materials are $E_f = 10\text{--}50$ GPa, $\sigma_{yf} = 60\text{--}300$ MPa, $n = 0.1\text{--}0.5$, and $t = 10\text{--}30$ μm , while the substrate material remains elastic. The variation of these mechanical parameters covers most of the thin-film materials in the industries. The film constitutive behavior is described according to Equation (1). After performing extensive FE simulations, the effects of mechanical properties and film thickness on the P - h curves are further investigated.

As illustrated in Figure 11, it can be seen that the effects of these four parameters on the curves are consistent. The applied load exponentially increases with the increase of mechanical properties. As the penetration depth increases, greater difference is found for the applied load due to the increase of these parameters gradually. Moreover, the applied load decreases as the film thickness increases.

Apparently, the influences of these factors of material parameters and film thickness can be well described by exponential fitting functions, which significantly guides the explicit form of the proposed dimensional function.

In this work, the commercial analysis software Origin 2018 64 Bit is used to fit the dimensionless formula. By focusing on the effect of material parameters and film thickness on the corresponding P - h curve during the indentation, the indentation problem is symmetrically investigated by performing FE analysis. Based on the indentation method proposed by Oliver and Pharr [10] and the elastic-fully plastic material model, mechanical parameters have been assumed. To perform the reverse analysis of constitutive parameters of thin-film materials based on the indentation responses, a parameterized dimensionless function is achieved based on the obtained FE results as

$$\frac{P}{E_f \cdot h^2} = a \cdot \frac{\sigma_{fy}}{E_f} \cdot \left(1 + b \cdot \frac{h}{t}\right) \cdot \exp\left(c \cdot \frac{n_f \cdot E_f}{E_s}\right) + d \cdot \frac{h}{t}. \quad (12)$$

where $a = 0.04797$, $b = 1.19646$, $c = 2.82563$, and $d = 3.87933 \times 10^{-4}$. Furthermore, the fitting accuracy is measured by the coefficient of determination R^2 of 0.90 for the FE predictions in the database of various working conditions. This proves that the dimensionless function agrees well with the FE simulations. It should be noted that the proposed dimensionless function can be utilized to guide the parameters tuning when performing indentation tests or simulations on film/substrate structures.

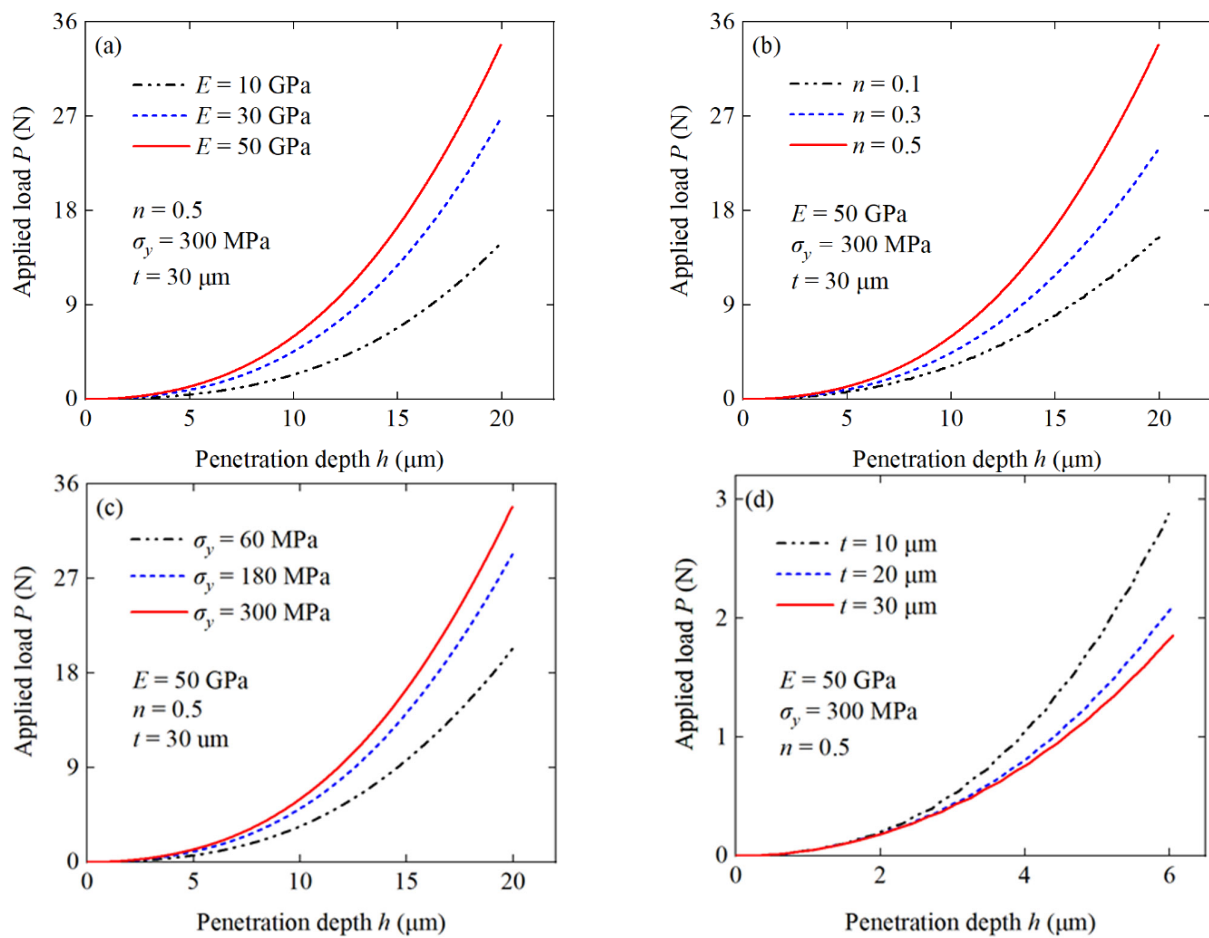


Figure 11. Effect of material parameters and film thickness on the P - h responses: (a) Young's modulus; (b) hardening exponent; (c) yield strength; (d) film thickness.

In order to further examine the reliability of the dimensionless formulation, three working conditions are randomly determined as listed in Table 2 (Case1: $E = 41,905.8$ MPa, $n = 0.48$, $\sigma_{fy} = 252.5$ MPa, $t = 24$ μm ; Case2: $E = 20,251.6$ MPa, $n = 0.40$, $\sigma_{fy} = 225.7$ MPa, $t = 20$ μm ; Case3: $E = 31,448.7$ MPa, $n = 0.19$, $\sigma_{fy} = 112.4$ MPa, $t = 13$ μm). It should be noted that all cases are out of the database for the parameter identification. Thus, the comparisons between the predictions by the proposed dimensional method and the established FE model can be utilized to confirm the prediction accuracy of the proposed dimensional method.

Table 2. Three new randomly determined working conditions.

-	E (MPa)	n	σ_{fy} (Mpa)	t (μm)
Case1	41,905.8	0.48	252.5	24
Case2	20,251.6	0.40	225.7	20
Case3	31,448.7	0.19	112.4	13

The solid line denotes the FE simulation results, and the scattered spheres corresponds to the predicted results at different penetration depths. As shown in Figure 12, the final data show that the dimensionless formulation is highly predictive for FE simulations, testifying to the credibility of Equation (12).

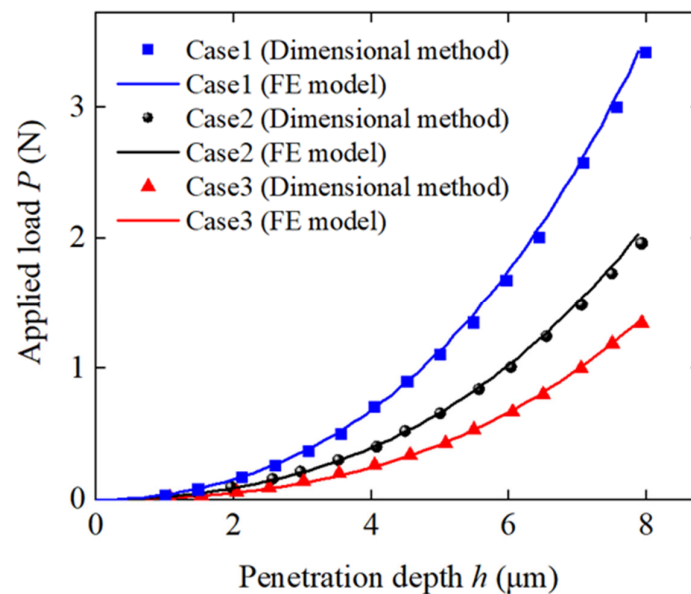


Figure 12. Prediction comparisons by the proposed dimensional method and the established FE model for three new randomly determined conditions.

5. Conclusions

A dimensionless analysis is proposed to estimate the elastoplastic properties of thin-film materials by the indentation method. After the simulation validation of the published results, an FE method is used to parametrically investigate the extensive mechanical parameters of the thin-film material. The prediction of the FE results shows a consistent loading trend for various mechanical properties and film thicknesses, which makes it possible to explore the relationship between the individual variables. With the parameters to describe the indentation problem for dimensional analysis, a dimensionless function related to the applied load is proposed. Using the proposed dimensionless function, the penetration depth within 6 μm is fitted with FE predictions up to 0.90. This means that unlike simulations, the dimensionless function provides a reliable method of analysis. With the film material and its mechanical properties established, the thickness of the film required to withstand external loads may be a practical application technique that can be developed. The applied loads were estimated for the films with different material properties and the obtained results fit well with the results. FE simulations can verify the accuracy of experiments, shorten the experimental cycle, and reduce material losses, and the dimensionless method has good application prospects and provides theoretical guidance for establishing parameter relationships by numerical methods.

Author Contributions: X.L.: conceptualization, methodology, funding acquisition, supervision, writing—review and editing; J.L.: investigation, methodology, writing—original draft, visualization, software; Z.S.: methodology, writing—original draft, visualization; Y.S.: methodology, software, writing—review and editing. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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