Numerical Analysis for High-Throughput Elastic Modulus Measurement of Substrate-Supported Thin Films

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Abstract: Micro/nano-thin films are widely used in the fields of micro/nano-electromechanical system (MEMS/NEMS) and flexible electronics, and their mechanical properties have an important impact on the stability and reliability of components. However, accurate characterization of the mechanical properties of thin films still faces challenges due to the complexity of film-substrate structure, and the characterization efficiency of traditional techniques is insufficient. In this paper, a high-throughput determination method of the elastic modulus of thin films is proposed based on the strain variance method, the feasibility of which is analyzed by the finite element method (FEM), and the specific tensile configuration with array-distributed thin films is designed and optimized. Based on the strain difference between the film-substrate region and the uncoated region, the elastic modulus of multiple films is obtained simultaneously, and the influences of film width, spacing, thickness, and distribution on the measurement of elastic modulus are elucidated. The results show that the change in film width has a more obvious effect on the elastic modulus determination than film spacing and thickness, i.e., the larger the film width is, the closer the calculation results are to the theoretical value, and the change in calculation results tends to be stabilized when the film width increases to a certain length. Specifically, the simultaneous measurement of the elastic modulus of eight metal films on a polyimide (PI) substrate with a length of 110 mm and a width of 30 mm can be realized, and the testing throughput can be further increased with the extension of the substrate length. This study provides an efficient and low-cost method for measuring the elastic modulus of thin films, which is expected to accelerate the development of new thin film materials.

Key words: high-throughput; thin films; elastic modulus; finite element method; strain variance method

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0 Introduction

Thin films are widely used in fields such as aero-engines, flexible electronics, and biomedical engineering due to their superior mechanical, and electromagnetic properties[1-8]. Thin-film structures often face complex loads such as tensile and compressive deformation and cyclic bending during service, and behaviors such as fatigue damage and failure caused by complex loads greatly affect the functional properties of thin-film structures. Therefore, the study of the mechanical properties of thin-film structures is of great significance. It has been shown[9] that the thin film morphology of metals is one of the key factors affecting the differences in mechanical properties of their macroscopic morphology, and the accurate characterization and efficiency of their mechanical properties are still a challenge. Elastic modulus is a physical quantity that describes the deformation resistance of a material and is one of the most important indicators for the performance design and reliability assessment of metallic thin

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films, therefore, this paper investigates the high throughput measurement method of elastic modulus of thin-films based on the finite element method.

The main methods currently used to characterize the mechanical properties of nanoscale metal films are the bending method[10], the bulge method[11], the nanoindentation method[12], and the tensile testing method[13], among others[14]. The bending method is a commonly used method to measure the elastic modulus by measuring the bending strain of metal beams. In recent years, a lossless bending measurement method has been proposed[15]. Based on the nonlinear Euler-Bernoulli beam theory, an experimental system for measuring the elastic modulus of thin films with a precision laser sensor has been constructed. This method can not only satisfy the measurement accuracy but also ensure that no damage is caused to the specimen. The bending method generally requires specialized experimental equipment and metal beams, and it suffers from the problems of low accuracy and limited applicability. To address the challenges associated with complex equipment and the low accuracy of the bending method, Liu et al.[10] introduced a strain measurement technique based on the finite element theory about the static elastic modulus. This method necessitates neither specialized equipment nor specific specimens, offers enhanced precision in testing, and has been extensively applied in engineering practice. The bulge method involves adhering a film to one side of a substrate that has holes, gradually increasing a uniform pressure on the opposite side, and measuring the pressure-induced change in the film's central height. By integrating this data with an appropriate mechanical theory model, one can determine the stress-strain curve, elastic modulus, and residual stress of the film[16]. Compared to general measurement techniques, the bulge method is characterized by low cost, simple equipment, and no contact. The nanoindentation method entails the use of a nanoindentation instrument to repeatedly indent a thin film specimen mounted on a substrate. By examining the correlation between the indentation depth and the number of impacts, one can ascertain the fatigue damage and deformation characteristics of the film[17]. It is noteworthy that the utilization of a substrate is a critical factor influencing the precision of the measurement outcomes. Microtensile testing is another method for measuring the mechanical properties of freestanding thin films. This method involves conducting a tensile test on a dog-bone specimen with half of it coated with the film. After the test, the strain of the film and the substrate is extracted, and then the elastic modulus of the film is calculated using specific formulas. In our previous work[18], we proposed a technique for measuring the elastic modulus of nano-films based on the "strain variance method" by combining microtensile testing with a dual telecentric digital image correlation (DIC). This technique has simple experimental equipment, a wide range of applications, accurate measurement results, and can also achieve the purpose of non-destructive.

Although the above techniques can accurately characterize the mechanical properties of nano-films, only one mechanical parameter of a film can be measured in one experiment, and the measurement techniques are not efficient. To improve the efficiency of characterizing the mechanical properties of nano-films, high-throughput characterization of the mechanical properties of nano-films has received extensive attention. For example, Zhang et al.[19] proposed a bulge method for fast and efficient high-throughput array characterization of thin film mechanical properties of circular thin film specimens. They obtained the full-field displacements of the arrayed films by 3D-DIC analysis and then calculated the elastic modulus and Poisson's ratio of multiple films using the established relationship between the elastic constants and film displacements. Burger et al.[20] arrayed a series of silicon cantilevers and then plated a 1 μm Cu film on the silicon cantilevers by a combination of radio frequency magnetron sputtering systems to investigate a series of stress amplitude by the vibration of the silicon cantilevers with different frequencies to study the fatigue life and damage formation of metal films at a range of stress amplitudes. In the study of thermal spraying coating and interfacial fracture resistance, the traditional bending method of cantilever beam may cause some problems such as crack deflection or crack propagation asymmetry. To overcome these shortcomings, Mishra et al. [21] proposed an improved cantilever bending method for measuring the interfacial fracture toughness of ceramic/metal interfaces. Finite element simulations were used to determine the energy release rate and phase angle of an interfacial crack in a bi-layered cantilever, considering varying elastic modulus ratio, thickness ratio of coating to substrate, and beam length to thickness ratio. The characteristic of this method is the ability to extract a large number of interfacial fracture energy measurements from the same sample. Johnson et al.[22] proposed a novel high-throughput spherical micro indentation measurement and analysis scheme for the cyclic response of metal specimens, which can be used for the rapid screening of metal alloys. Li et al.[23] applied spherical nanoindentation to an additive manufacturing specimen and measured its stress-strain curve on a microscopic scale. The nanoindentation method has the advantage of high throughput, but the requirements for specimen preparation and test conditions are relatively high. Ojeda et al.[24] conducted tensile experiments on high-density unidirectional silver nanowires bonded to the substrate and analyzed their fracture strain in combination with DIC. In comparison to in-situ mechanical testing, this method significantly enhances efficiency. However, it is constrained by the alignment and resolution capabilities of the optical microscope, potentially leading to the loss of some data. Up to now, there has been a relative scarcity of research on high-throughput characterization methods for the mechanical properties of metal thin films based on the tensile method. Therefore, this paper proposes a new method that is simple, efficient, highly accurate, and applicable to both crystalline and amorphous material thin films. Compared with other high-throughput characterization methods, this method is easy to operate and non-destructive to the specimens.

1 Methodology Principle

Some researchers have proposed a method for determining the elastic modulus of thin films based on strain variance[18]. As shown in Fig.1, where F represents the force applied to both sides of the specimen, the yellow part is the coated area and the blue part is the uncoated area, a dog bone specimen was subjected to a microtensile experiment, and the strains in the coated and uncoated areas were measured by a DIC system. The elastic modulus of the film is then calculated using the strain variance method.

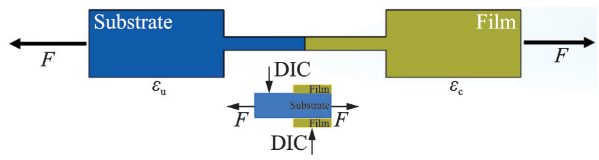

Fig.1 Schematic diagram of a dog bone specimen

The strain variance method equation is

$$\frac{E_f t_f}{E_s t_s}=\frac{(\varepsilon_u)_{11}^{s}}{(\varepsilon_c)_{11}^{f}}+\frac{\nu_f-\nu_s}{1-(\nu_s)^2}\frac{(\varepsilon_c)_{22}^{f}}{(\varepsilon_c)_{11}^{f}}-\frac{1-\nu_f\nu_s}{1-(\nu_s)^2} \quad (1)$$

If $\nu_f=\nu_s$, Eq.(1) can be simplified as

$$\frac{E_f t_f}{E_s t_s}=\frac{(\varepsilon_u)_{11}^{s}}{(\varepsilon_c)_{11}^{f}}-1 \quad (2)$$

where E is the modulus of elasticity, t the thickness, ε the strain, and ν the Poisson's ratio. The subscripts c and u are the coated and uncoated regions (pure substrate), respectively; subscripts 11 and 22 the longitudinal and transverse directions, respectively; and superscripts f and s the film and the substrate, respectively.

In general, the difference between Poisson's ratio of the substrate and the film has a very small effect on the result of the calculation, and the elastic modulus can be calculated with Eq.(2). The thickness and Poisson's ratio of the substrate and the film, as well as the elastic modulus of the substrate, can be determined in advance, and the elastic modulus of the film can be calculated by simply obtaining the strains of the substrate and the film after tensile.

By arraying multiple films on a flexible substrate, the elastic modulus of multiple films can be calculated in a single experiment based on Eq.(2). This high-throughput approach improves efficiency and saves costs. In this paper, the full-field strains of the films and the substrate are obtained by ABAQUS simulation, and the elastic modulus of multiple films is successfully determined, which verifies the feasibility of this method.

2 Finite Element Simulation

To demonstrate the feasibility of high throughput measurement of elastic modulus of thin films, numerical simulations were carried out using ABAQUS. Due to the symmetrical nature of the structure, a quarter of the specimen (*XZ* and *XY* symmetry planes) was taken to model the structure for computational efficiency, where the *X*-axis is the direction of loading, the *Y*-axis is the transverse direction, and the *Z*-axis is the out-of-plane direction.

2.1 Geometric modeling

If the approximate elastic modulus of the film is determined, it is important to choose a suitable film thickness. According to the error analysis[25], to obtain an acceptable measurement accuracy, e.g., the uncertainty of the elastic modulus is less than 10%, the dimensionless ratio is required to be greater than 0.13 according to the relationship between the uncertainty of the elastic modulus and the dimensionless ratio *R*. Then the thickness of the film is not less than $\frac{0.13E_s t_s}{E_f}$.

The uncertainty of the elastic modulus is

$$\frac{\delta E_f}{E_f}=\sqrt{\left(\frac{\delta R}{R}\right)^2+\left(\frac{\delta E_s}{E_s}\right)^2+\left(\frac{\delta t_s}{t_s}\right)^2+\left(\frac{\delta t_f}{t_f}\right)^2} \tag{3}$$

The dimensionless ratio *R* is

$$R=\frac{E_f t_f}{E_s t_s} \tag{4}$$

The area and arrangement of the film also need to be considered. If the area of the film is too large or the arrangement is too widely spaced, the number of films will be reduced accordingly, and the high throughput effect will not be achieved. If the film area is too small or the spacing is too small, the calculated results will have a large error due to the edge effect.

According to GB/T 41477—2022 rectangular cross-section tensile specimen preparation requirements, an initial film size was selected, as shown in Fig.2, the polyimide (PI) film was selected as the substrate, 66 mm in length, 30 mm in width, with a thickness of 125 μm; the tungsten metal film (W film) was 30 mm in length, 10 mm in width, and the thicknesses of all of them were 400 nm. The spacing between the films was 2 mm.

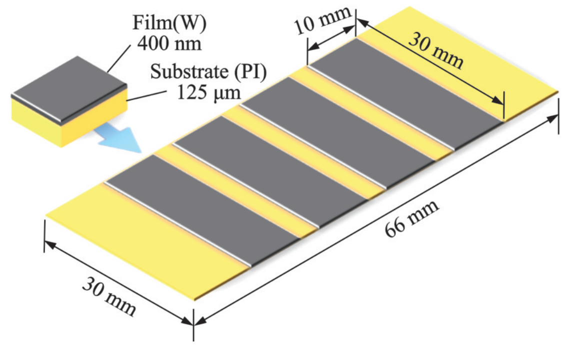

Fig.2 3D model of array distributed film-substrate structure

2.2 Meshing

According to the characteristics of the structure, the 3D solid part of the C3D8 cell grid is used to mesh the substrate, and the 2D shell layer part of the S4 cell grid is used to mesh the W film. The global seed size was set to 0.03. The meshing is shown in Fig.3.

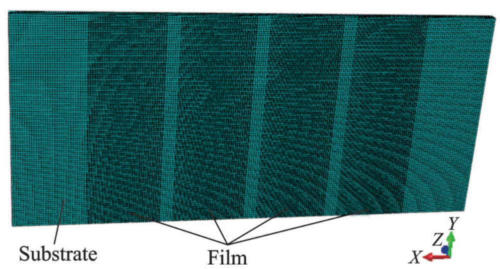

Fig.3 Schematic diagram of meshing

2.3 Defining material properties

The substrate used was a PI film with elastic modulus set at 4 GPa and Poisson's ratio of 0.34. The film was a metallic W film with elastic modulus set at 375 GPa and Poisson's ratio of 0.28.

2.4 Creating loads and boundary conditions

Tie constraints were used between the film and the substrate to simulate the strain transfer at the interface. As shown in Fig.4, a uniform load is directly applied at the left end of the substrate, which is gradually increased by 20, 33.3, 46.7, 60, 73.3, and 86.7 N, respectively, and the constraints are set up at the right end in the *X*-direction, and the symmetric boundary conditions (*XZ* and *XY* symmetric planes) are applied to the center plane of the substrate to avoid rigid-body motions. Under geometric nonlinear effects, the geometry of the deformed structure affects the internal stress distribution, leading to stress concentration or stress redistribution. Therefore both the film and the substrate are assumed to be isotropic and linearly elastic.

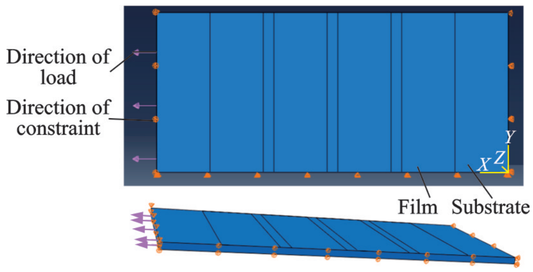

Fig.4　Schematic of loads and constraints

2.5　Analysis results

The full-field axial strain maps of the coated and uncoated regions are obtained after analysis, as shown in Fig.5. Due to edge effects, the strain distribution at the edges of the thin film is non-uniform. Consequently, the average strain within a square area, centered on the film and one-third the size of the film's dimensions, is taken to represent the strain of that section of the film.

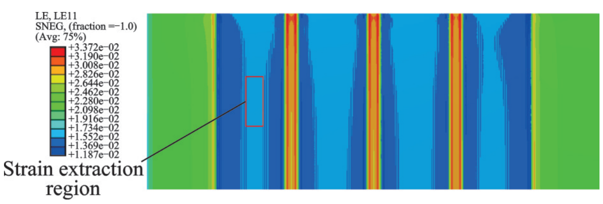

Fig.5　Full-field axial strain diagram

The calculated results of the extracted strain and elastic modulus of the substrate and film are shown in Table 1, where f1—f4 are different films. It can be seen that the calculated elastic modulus of the film has a large error compared to the true value (In this paper, the error between the calculated value and the theoretical value is less than 10% as an acceptable range), so the size and distribution of the film need to be optimized to reduce the error.

Table 1　Strain and elastic modulus calculation results and errors for substrate and film

Parameter	Strain	E/GPa	Error/%
Substrate	0.021 67	—	—
f1	0.015 71	474.2	26.5
f2	0.015 73	472.0	25.9
f3	0.015 80	464.4	23.7
f4	0.015 63	483.0	28.8

3　Film Size and Distribution Optimization

3.1　Effect of film width

To study the effect of film area on the simulation results, the length of the film is fixed at 30 mm and the simulations are carried out with film widths of 10, 12, 14, 16, 18, 20, 22, and 24 mm, respectively. The film spacing and thickness are 2 and 400 nm, respectively, and the same method described above is used to analyze and calculate the films with different widths. To reduce the likelihood of random errors, four films with identical parameters were allocated to a single group for the determination of the elastic modulus. The mean value obtained from these measurements was then considered as the true value of the elastic modulus for that specific set of parameters. The analyzed results are shown in Fig.6. From the figure, it can be seen that the calculated value is getting closer to the theoretical value as the film width increases. When the width of the film is increased to 22 mm, the effect of increasing the film width on the simulation results is already small, in which case it may be the influence of other factors.

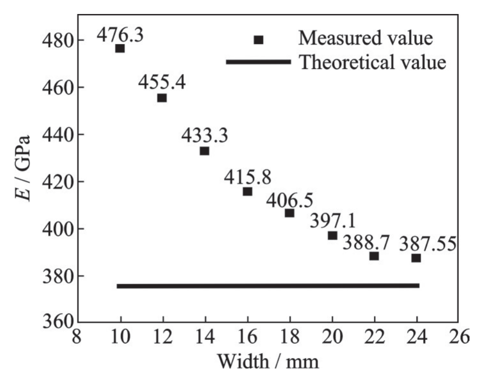

Fig.6　Simulation results for different film widths

3.2　Effect of film spacing

The results in Subsection 3.1 indicate that the error is relatively small when the film width is

22 mm. Therefore, three sets of simulation experiments with different parameters were set up with a film width of 22 mm, film thickness of 400 nm, and film spacing of 1, 2, and 3 mm, respectively. The simulation results are shown in Table 2. It can be seen that the film spacing has little effect on the results of the simulation.

Table 2 Simulation results for different film spacings

Film spacing / mm	E/GPa	Average
1	393.0	387.6
	385.3	
	383.6	
	388.3	
2	392.5	388.7
	386.4	
	386.1	
	389.9	
3	392.4	389.8
	389.3	
	388.1	
	389.4	

3. 3 Effect of film thickness

The film edge length of 22 mm, film pitch of 2 mm, and film thickness of 200, 400, 600, and 800 nm are selected for simulation. The simulation results are shown in Fig.7. The green solid line is the theoretical value, and the red and blue dashed line are the upper limit of error and the lower limit of error, respectively (the results are allowed to have a 5% error from the theoretical value). It can be observed from the figures that the measurement results for films of varying thicknesses are all within

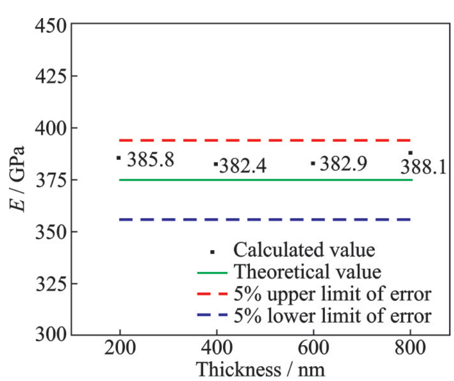

Fig.7 Simulation results for different film thicknesses

the margin of error and relatively close to each other. Consequently, it can be concluded that the thickness of the film has a minimal impact on the calculation of the elastic modulus.

3. 4 Effect of film distribution

Through the above work, a set of film dimensions with superior measurement results were determined: Film width of 22 mm, film spacing of 2 mm, and film thickness of 400 nm, and the simulation results are shown in Fig.8. Under this arrangement, the calculated error in the elastic modulus of the thin film is within 5%. Therefore, it is feasible to consider reducing the size of the thin film to achieve the goal of increasing throughput. According to the table of rectangular cross-section tensile scale specimen dimensions in GB/T 41477—2022, the width of the substrate can only be taken up to a maximum of 30 mm, while the length can grow by designing different thicknesses. Consequently, the number of thin films can only be increased by reducing their dimensions in the Y-direction, thereby allowing for additional rows to be arrayed in that direction.

Fig.8 Simulation results of thin film arrays in the X direction

To increase the number of films, the films were arrayed in the X-Y direction with a film length of 22 mm, a width of 14 mm, and a spacing of 2 mm, and their arrangement is shown in Fig.9. The computational results of this arrangement are shown in Fig.10, where f1—f8 denote different films. As can be seen from the figure, the calculated results of the eight films simulated are all within the error range. This indicates that the size and arrangement of such films are feasible. The Y-direction array of three rows of films was also simulated subsequently, but due to the small area of the film, the error of the calculation results was too large by the edge effect, and it was not considered.

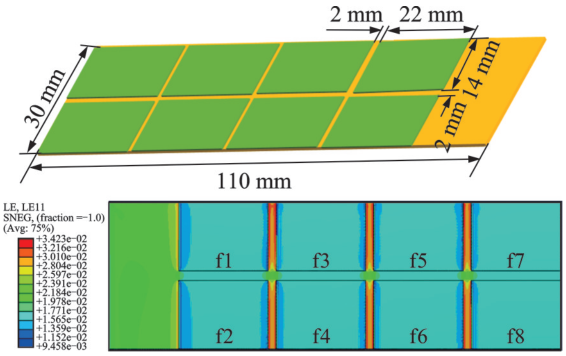

Fig.9 Simulation results of thin film arrays in the X-Y direction

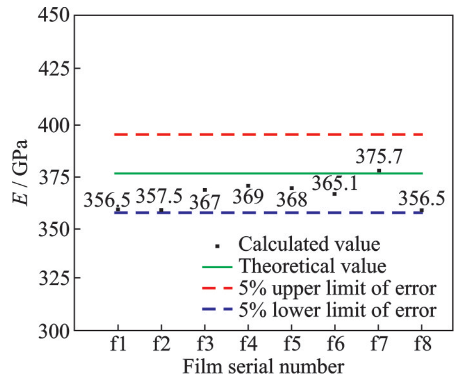

Fig.10 Calculated results for thin film arrays in the Y direction

4 Conclusions

Based on the strain variance method, a high-throughput determination method of the elastic modulus of thin films is proposed, by which the elastic modulus of many thin films can be obtained simultaneously in one measurement. Numerical experiments with array-distributed thin films were designed, and the effects of film width, film spacing, film thickness, and film arrangement were analyzed. From the results and analyses, the following conclusions can be made:

(1) When the film width is varied between 10 and 24 mm, the measured values of elastic modulus are closer to the theoretical values as the film width increases, and stabilizes when the film width reaches 22 mm. Changes in film spacing and thickness have less effect on the measured values.

(2) An optimized film size and arrangement are obtained to ensure that the measured values of the elastic modulus of thin films are within the error range. With this method, the elastic modulus of eight thin metal films of 22 mm in length and 14 mm in width can be measured on a substrate of 110 mm in length and 30 mm in width in a single experiment, which lays the foundation for the realization of high-throughput experimental measurements of thin films.

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Author contributions Dr. **HAN Meidong** provided suggestions and improvement plans for the finite element analysis study and the revision of the paper. Mr. **ZOU Jinluo** built the model, performed the analysis, interpreted the results and wrote the paper. Dr. **HE Wei** designed the study and provided the research proposal and ideas. Mr. **LI Yu** provided valuable suggestions for the revision of the manuscript. All authors commented on the manuscript draft and approved the submission.

Competing interests The authors declare no competing interests.

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膜基结构中薄膜弹性模量高通量测量的数值分析

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摘要:微/纳米薄膜广泛应用于微/纳机电系统(Micro/nano-electromechanical system, MEMS/NEMS)和柔性电子领域,其力学性能对元器件的稳定性和可靠性有着重要影响。然而,由于薄膜-基底结构的复杂性,薄膜力学性能的准确表征仍然面临挑战,此外传统技术的表征效率也不足。本文基于应变方差法提出了一种薄膜弹性模量高通量测量方法,通过有限元方法(Finite element method,FEM)验证了其可行性,并设计优化了具有阵列分布薄膜的特定拉伸构型。基于薄膜-基底区域与未涂覆区域之间的应变差异,同时获得了多个薄膜的弹性模量,并阐明了薄膜宽度、间距、厚度和分布对弹性模量测量的影响。结果表明,薄膜宽度的变化对弹性模量测定的影响比薄膜间距和厚度更为明显,即薄膜宽度越大,计算结果越接近理论值,当薄膜宽度增加到一定长度时,计算结果的变化趋于稳定。具体来说,可以在聚酰亚胺(Polyimide, PI)基底上同时测量8个金属薄膜的弹性模量,随基底长度增加测试通量可进一步提升。本研究提供了一种高效且低成本的薄膜弹性模量测量方法,有望加速新型薄膜材料的开发。

关键词:高通量;薄膜;弹性模量;有限元方法;应变差异法