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Uniqueness test for thin film fitting in spectroscopic ellipsometry

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Abstract: The thickness and dielectric constants of thin films usually have certain correlation in the fitting procedure of spectroscopic ellipsometry (SE). The choice of different dispersion models may also influence the results and cause errors. As the fitting is influenced by the dispersion models adopted in the analysis, the uniqueness test has been introduced into SE fitting. The results of uniqueness test have been compared with different dispersion models, different film thicknesses, different wavelength ranges and different incident angles using titanium dioxide samples as an example. It is indicated that uniqueness test is efficient in evaluating the fitting for SE measurement. Uniqueness test can also provide quantitative comparison among different dispersion models and contribute to fitting precision.

Key words: spectroscopic ellipsometry, uniqueness test, thin film, titanium dioxide
PACS: 78.20.Bh, 78.20.Ci, 07.05.Kf, 81.70.Fy

唯一性检测在椭圆偏振光谱薄膜拟合中的应用

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摘要：利用椭圆偏振光谱进行薄膜样品的测量数据分析拟合时，薄膜厚度与介电常数通常具有一定的关联性. 不同色散模型的选取也会对拟合结果产生明显的影响，引起较大误差. 介绍了唯一性检测在椭偏拟合中的实现方法. 并以二氧化钛样品为例，利用唯一性检测对比了不同色散模型、不同厚度、不同测量波段、不同入射角度时的唯一性检测结果. 结果表明，唯一性检测能够有效标定出椭偏测量和拟合过程中所产生的误差，同时能够对不同色散模型进行量化对比，提升拟合精度.

关 键 词：椭圆偏振光谱；唯一性检测；薄膜；二氧化钛

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Introduction

Spectroscopic ellipsometry (SE) is routinely used in investigating the thickness and dielectric constants of thin films. Ellipsometry is an indirect technique. The measured ellipsometric data at certain wavelength range are fitted to the optical model for thin film, which introduces uncertainty into the final results. Unique results are usually difficult to evaluate due to the correlation between dielectric constants and film thickness, especially for absorbing materials[1-2].

Although SE measurement may not be the best way for the characterization of thin absorbing films, its non-contact and non-destructive characteristics are ideal for many situations when film thickness or dielectric constants are needed[1, 3-5]. Typically, the sample needs no special preparation before ellipsometry measurement. Moreover, rich information including film thickness, dielectric constants, optical band gap and surface roughness of material could be revealed at once[6]. The compatibility of SE makes it valuable for in-situ, in-line or many other situations.

Varies methods were used to enhance the uniqueness in SE measurement, including interference enhancement[7-8], multiple samples analysis[9-10], multiple angles analysis[11], systemic error elimination[12-13], simultaneous analysis of SE[14-15], and optical constant parameterization[1, 6]. However, the lattice mismatch between different layers or thickness dependent of dielectric constants will also induce uncertainty in ellipsometry[6, 16], which makes it hard to compare the sensitivity and reliability of these methods. All the methods above need a route to determine the uniqueness of the SE fitting results. Hence, uniqueness test has been developed and used as an important method in comparing SE fitting results for this purpose[1,7,17].

In this work, uniqueness test is described in detail. As an example, titanium dioxide (TiO_2) thin films are fitted with varies ellipsometric parameters for comparison. The concept of thickness deviation is introduced to examine the consistency of uniqueness test. The results demonstrate that uniqueness test is efficient in determining fitting quality and can provide the error range of SE as well.

1 Methods

1.1 Spectroscopic ellipsometry

The measurement in SE is recorded as two values which are related to the parallel p- and perpendicular s-polarized light. The polarization change is described by the amplitude ratio of reflected p- to s- polarized light (ψ) and the phase shift difference between the two (Δ)[1].

$$\rho = \tan(\psi)e^{i\Delta} = \frac{R_p}{R_s} \quad , \quad (1)$$

where R_p and R_s represent the p- and s- Fresnel reflection coefficients, respectively. The values of Ψ and Δ are collected in SE measurement and fitted to the optical model based on film structure[1]. In the simplest case, a single isotropic transparent thin film on known absorbing substrate, the real part of dielectric constants (ε_1) and film thickness (d) can be determined. With these two measured parameters (ψ, Δ), the unique result is easily obtained.

Compared with transparent film, the imaginary part of dielectric constants (ε_2) are nonzero for metal and semiconductor films, which leads to three unknown parameters with only two measured SE values. Although the dielectric constants must conform to the Kramers-Kronig (K-K) consistent[1], the dispersion models used in the SE fitting are still variable.

A key parameter in qualifying the differences between fitting results and experimental data is mean squared error (MSE), which is defined as[2]:

$$\mathrm{MSE}^2 = \frac{1}{2N-M}\sum_{i=1}^{N}\left[\left(\frac{\Psi_i^{\mathrm{mod}}-\Psi_i^{\mathrm{exp}}}{\sigma_{\psi,i}^{\mathrm{exp}}}\right)^2+\left(\frac{\Delta_i^{\mathrm{mod}}-\Delta_i^{\mathrm{exp}}}{\sigma_{\Delta,i}^{\mathrm{exp}}}\right)^2\right] \quad , \quad (2)$$

where σ is the measurement error bars. N and M are number of Ψ(or Δ) measured and number of fitting parameters, respectively. A small MSE is necessary in SE fitting, but it may also indicate that too many fitting parameters in dispersion model or film structure were used. Therefore, the uniqueness test is very necessary to distinguish good fitting results and redundant parameters.

1.2 Uniqueness test

A uniqueness test is a series of simulation procedure with one selected fit parameter being tested. The selected parameter is fixed at certain values near the best fitting result, and other fitting parameters are coordinated to approach the smallest MSE. Then a series MSE is obtained and the normalized MSE can be plotted versus the selected fit parameter. Thickness is the most selected parameter in uniqueness test as it is independent on wavelength and easily compared with other characterization methods. Figure 1(a) is an example of uniqueness test of film thickness in a thin film sample. The range which normalized MSE lower than 1.1 is referred to as uniqueness range[2].

In many cases, the uniqueness test needs to evaluate samples with different thickness. However, the uniqueness range will vary in these samples although all fabricating and measuring methods are identical, which makes it inconvenient for comparison. Hence a concept of thickness deviation is introduced, which is defined as:

$$\frac{d-d_0}{d_0} \times 100\% \quad , \quad (3)$$

where d is a variable value and d_0 is the best fit of thickness. The normalized uniqueness range makes it possible to apply uniqueness test among the same batch of samples. Figure 1(b) shows the transferred uniqueness test of TiO_2 thin films.

2 Results and discussions

The TiO_2 samples were deposited on n-type Si (100) using electron beam evaporation (EBE) methods, then treated by thermal annealing process[18-20]. The characteristic analysis of surface morphology was performed by atomic force microscopy (AFM, Veeco. Atomic Force Microscope System VT-1000) in tapping

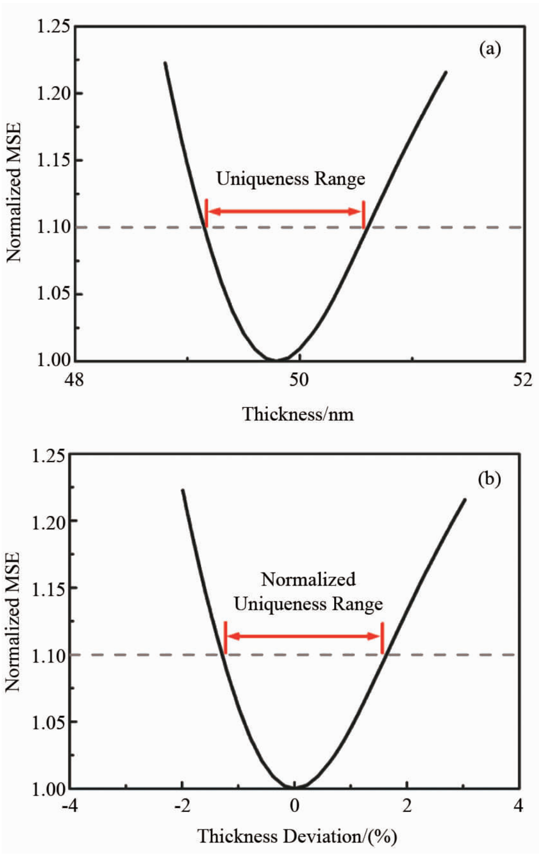

Fig. 1　Uniqueness test of TiO_2 thin film. (a) The Normalized MSE versus the varied thickness. (b) The Normalized MSE versus the thickness deviation

图 1　TiO_2 薄膜的唯一性检测 (a)归一化均方误差关于厚度的变化,(b)归一化均方误差关于厚度偏移的变化

mode. The SE measurements were done by rotating-polarizer-analyzer ellipsometer (RPAE) at various incident angles[21-22]. Ellipsometric spectra were measured over the wavelength range of 300-800 nm at three different incident angles: 65°, 70°and 75°, respectively.

Four TiO_2 thin films with different thickness were fabricated. As shown in the AFM image in Fig. 2, the surfaces of TiO_2 are very smooth. The thickness and root mean square roughness (RMS roughness) of the samples are about 1 nm. As the thicknesses of TiO_2 samples are much thicker than the RMS roughness, the structure configuration in the fitting is substrate/TiO_2/ambient air without a roughness layer[1].

Figure 3 shows the uniqueness test results of TiO_2 films with different thickness. The normalized uniqueness ranges listed in Table 1 shows only a little variation, which proves that the results of uniqueness test on samples with different thickness are consistent. The variation among these four samples is introduced in manufacturing and measuring procedures. The influence of roughness layer is important in SE fitting. Pervious works have demonstrated that the thickness of roughness layer in SE fitting is about 1.5 times of measured RMS roughness[23]. The RMS roughness of TiO_2 samples is within the uniqueness ranges, which means that introducing the roughness layer will not improve the fitting accuracy.

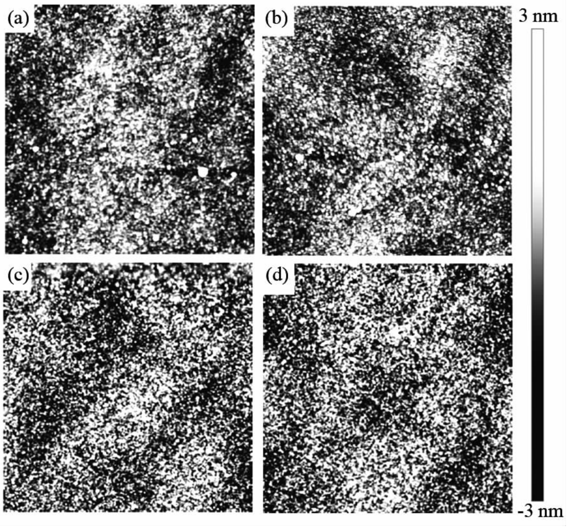

Fig. 2　AFM image of TiO_2 thin films (2 μm × 2 μm)

图 2　TiO_2 薄膜的原子力显微镜扫描图 (2 μm × 2 μm)

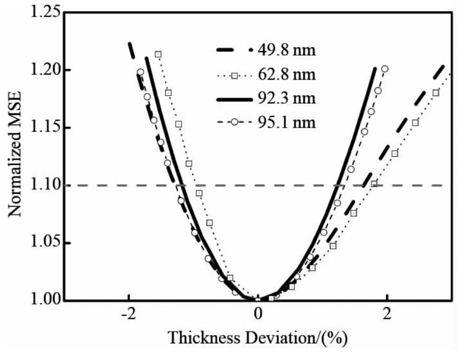

Fig. 3　Uniqueness test results of TiO_2 thin films with different thicknesses

图 3　不同厚度 TiO_2 薄膜的唯一性检测结果

Table 1　Physical Parameters and Fitting Results of the TiO_2 thin films

表 1　TiO_2 薄膜的物理参量和拟合结果

Sample	Thickness /nm	RMS roughness /nm	Uniqueness Range/nm	Normalized Uniqueness Range/(%)
A	49.8	0.85	1.4	2.9
B	62.8	0.88	1.7	2.7
C	92.3	1.07	2.3	2.5
D	95.1	1.08	2.5	2.6

Sample A was selected as an example to perform further investigation. A valid dispersion model is important in SE fitting. For semiconductors, the most commonly used models are Tauc-Lorentz Model[24], F-B Model[25], Lorentz Oscillator Model[6] and Cauchy Potential Model[1]. Each model has its physical significance and has been proved to be efficiency in many situations. However, for a particular material, it is hard to compare the "goodness" of dispersion models based on the physical significance or MSE. Table 2 shows the fitting results for different dispersion models. The thickness differences revealed by these models are within the RMS roughness and the MSE are all acceptable, which makes it hard to determine the best dispersion model. Figure 4 (a) compares the uniqueness test results when TiO_2 film was fitted using different dispersion models. From the normalized uniqueness range it is easily concluded that F-B Model is the best dispersion model for the TiO_2 sample. As a method of pure mathematics, uniqueness test can provide a valuable criterion in selecting dispersion models.

Table 2 Fitting results of different dispersion models for TiO_2 film (49.8 nm)

表 2 采用不同色散模型对 TiO_2 薄膜(49.8 nm)的拟合结果

Dispersion Model	Best Fitted Thickness/nm	MSE
Tauc-Lorentz	49.5	2.70
Lorentz	51.4	1.97
F-B	49.8	0.98
Cauchy Potential	49.9	1.53

It has been widely acknowledged that one of the best way to increase the uniqueness of SE is by acquiring more information, such as multiple angles analysis and measuring wavelength range extension[1,11]. With additional information obtained from measurement, the correlation between thickness and dielectric constant is increased.

Uniqueness tests were applied on different wavelength ranges in Fig. 4(b). It can be noted that the normalized uniqueness range is smaller with wider wavelength range. Extending the wavelength range is efficient in promoting the uniqueness of SE measurement. Furthermore, from the test the minimum wavelength range can be estimated to ensure the uniqueness of the thickness. In this case, 450-800 nm is acceptable in determining the thickness of TiO_2 film, but the normalized uniqueness range of 600-800 nm is larger than 10%, which indicates that the wavelength range needs to be extended.

Figure 4(c) gives the uniqueness test of multiple incident angles. The normalized uniqueness range of three incident angles (65°, 70°and 75°) is smaller than that from one incident angle (75°), but the promotion is not as significant as that from extend wavelength range. Figure 4(d) shows the measured Ψ and Δ at three incident angles. The angle variation is not large enough and the Ψ data are only slightly varied. So the information

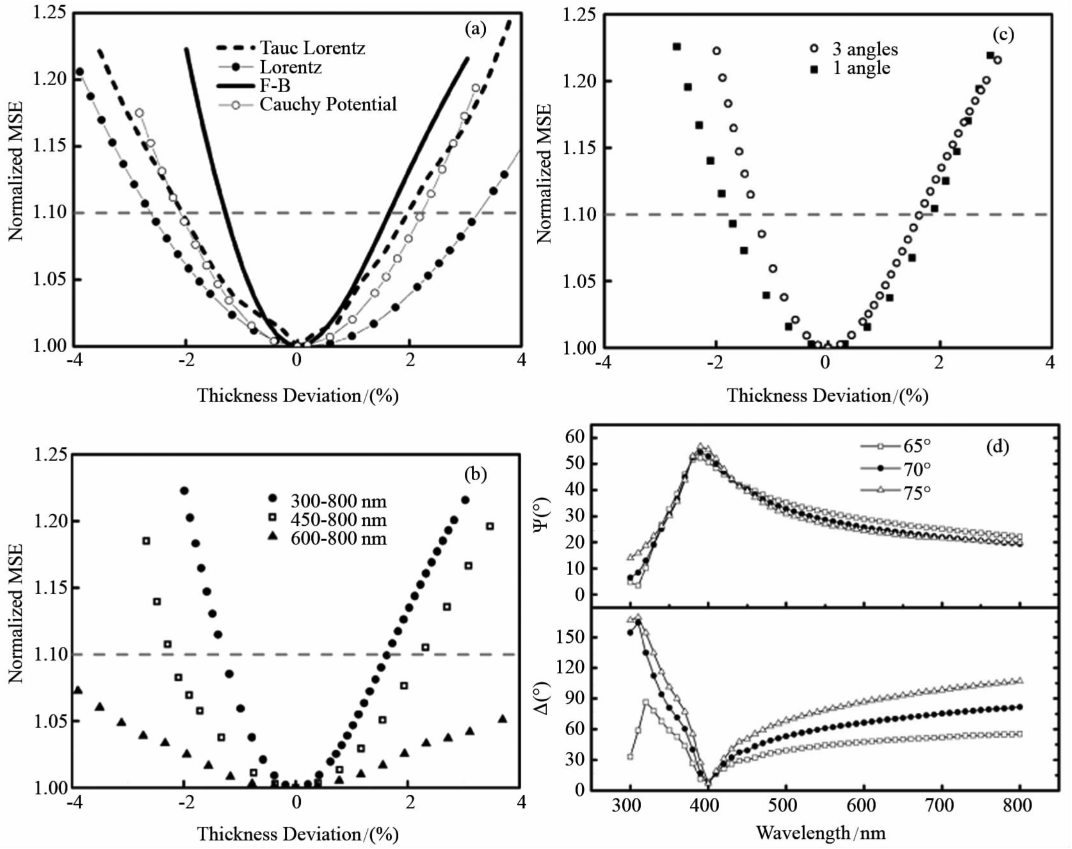

Fig. 4 The uniqueness test results of TiO_2 thin film with (a) different dispersion models, (b) different wavelength ranges, (c) different numbers of incident angles. (d) Measured ellipsometric parameters of TiO_2 thin films under different incident angles

图 4 对 TiO_2 薄膜(a)不同色散模型,(b)不同波长范围,(c)不同入射角度数量的唯一性检测结果,(d)测量得到的 TiO_2 薄膜在不同入射角下的椭偏参量

obtained from multiple incident angles is less useful than that from extended wavelength range, although it is still effective in increasing the uniqueness of SE measurement.

3 Conclusions

In summary, the uniqueness test has been introduced in SE fitting and the normalized uniqueness range is used as an evaluation criterion. As an example, TiO_2 samples have been fabricated using EBE and analyzed by AFM and SE. The consistency of uniqueness test is proved by comparison of multiple samples. Further analyses have demonstrated that the uniqueness range of SE fitting can be revealed by uniqueness test quantitatively. The uniqueness test is beneficial for the selection of dispersion model, determination of wavelength range, testing of multiple incident angles and many other situations. The results demonstrate that the uniqueness test will shed new light on future SE related research.

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