

Study on multi-wavelength thin film thickness determination method

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Abstract: This work introduces a novel method for measuring thin film thickness, employing a multi-wavelength method that significantly reduces the need for broad-spectrum data. Unlike traditional techniques that require several hundred spectral data points, the multi-wavelength method achieves precise thickness measurements with data from only 10 wavelengths. This innovation not only simplifies the process of spectral measurement analysis but also enables accurate real-time thickness measurement on industrial coating production lines. The method effectively reconstructs and fits the visible spectrum (400-800 nm) using a minimal amount of data, while maintaining measurement error within 7.1%. This advancement lays the foundation for more practical and efficient thin film thickness determination techniques in various industrial applications.

Key words: film thickness determination, transmittance and reflectance spectra, fitting, spectral reconstruction

多波长薄膜厚度检测方法研究

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摘要: 本工作提出了一种新的薄膜厚度检测方法, 该多波长方法显著地降低了膜厚检测对于宽光谱数据的需求。不同于需要几百个光谱数据点的传统技术, 多波长方法在仅采用 10 个波长数据点的情况下即可实现精确的膜厚测量。这一创新不仅简化了光谱测量分析的过程, 同时也使得工业化镀膜产线上实时的膜厚准确检测成为可能。该方法能够在少量数据的情况下有效地恢复和拟合可见波段光谱 (400~800 nm), 同时能够很好地将膜厚检测误差保持在 7.1% 以内。这一进展为许多工业应用中更加实用而高效的薄膜厚度检测技术奠定了基础。

关键词: 膜厚检测; 透反射谱; 拟合; 光谱还原

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Introduction

Thin film materials play a pivotal role in energy-saving construction, automotive glass, optoelectronics, and other fields. In energy-saving buildings, thin films are mainly used for window glass coatings, effectively controlling indoor temperatures and reducing energy consumption^[1-2]. These films reflect or absorb solar thermal energy while allowing visible light to pass through, improving the energy efficiency and comfort of buildings. In automotive glass, thin films enhance safety and comfort by blocking ultraviolet rays, reducing interior temperature increases, and improving glass shatter resistance^[3-4]. Thin film materials also play a significant role in artificial structural colors, applicable in anti-counterfeiting and information encryption^[5]. Thus, thin film materials are crucial for enhancing energy efficiency and optimizing user experience.

During the deposition of optical thin films, thickness is a key factor affecting film performance. Rapid online thickness determination technology is critical for achieving specific thicknesses. Commonly used methods in the research field include profilometry method^[6], interferometric fringe method^[7], ellipsometry method^[8-10], and micro area composition measurement techniques^[11-12]. Profilometry method, although stable and high-resolution, has limitations such as requirement for step features on the film, potential scratching, slow scanning speed, and limited measurement area. Ellipsometry method, a non-contact optical method, offers high sensitivity and accuracy but is challenging for online detection and requires expensive equipment. These methods are difficult to apply for real-time measurement in coating equipment. Xingxing Liu also proposed a method using an interference-assisted film layer to accurately determine the film thickness^[13], but this method requires the use of a substrate coated with an assisted film layer as a monitoring film.

Real-time thickness monitoring is increasingly important in industrial coating equipment, especially for manufacturing high-performance coated products. Maobin Xie proposed a method capable of detecting the optical constants of thin films *in situ*^[14]. Zhuangzhuang Cui proposed a method to accurately measure the optical constants of low-dimensional materials using optical microcavities^[15], but it is not applicable to real-time measurements on industrial production lines. The thickness and uniformity of the optical film layer may also have an impact on the scattering of the optical system^[16]. Existing film thickness detection methods in industrial coating systems are dominated by single-wavelength detection^[17], which is very simple but highly affected by the dispersion of the material. There are also traditional methods for detecting film thickness using visible spectral data, but full spectral data information needs to be collected. With the increasing demand for film performance, it is becoming critical to measure film thickness accurately and in real time. This is not only essential for improving coating quality, but also extremely important for data-driven process control. Accurate film thickness measurement can

achieve more optimized production processes, improving product consistency and performance, while also reducing production costs. Therefore, developing and optimizing thickness determination technologies is key to efficient, high-quality coating production.

Unlike single-wavelength methods affected by material dispersion and limited to detecting films with optical thicknesses at quarter-wavelength multiples, and also different from traditional full spectrum methods that require wide spectral data, the multi-wavelength film thickness determination method proposed in this paper is able to accurately determine the thickness information of the film layer by spectral reduction and film thickness fitting for films of different materials and thicknesses.

The multi-wavelength method investigated in this paper is a new technique for film thickness determination. It is based on spectral reconstruction of multi-wavelength spectral data, using spectral data at different wavelengths for the entire visible wavelength band (400-800 nm) and film thickness fitting. This method requires the use of data at only 10 wavelengths and enables rapid acquisition of spectral data, effectively simplifying the traditional full-spectrum method. Through experimental validation, the errors of this method on the film thickness determination of different material film layers are less than 7.1%, indicating its accuracy and practicality, which is especially suitable for real-time inspection of film thickness on production lines.

1 Methods

1.1 Multi-wavelength thickness determination method overview

The multi-wavelength thickness determination method uses spectral data from 10 different wavelengths (405, 450, 488, 505, 532, 635, 650, 670, 685, 780 nm) to reconstruct the visible spectrum (400-800 nm) and determine film thickness. This approach avoids the limitations of single-wavelength methods and the requirement of broadband spectral data, significantly improves measurement accuracy, simplifying the thickness measurement system. It is highly suitable for industrialized coating production lines, laying a theoretical foundation for developing multi-wavelength thickness online monitoring equipment.

The basic principle of this method is that different models can be used to fit the propagation of light in different types of film layers. For materials such as Si₃N₄ and AZO (aluminium doped zinc oxide) that are transparent in the visible band, the Cauchy model can be chosen as the material model, with a fitting formula of

$$n(\lambda) = A + \frac{10^4 \cdot B}{\lambda^2} + \frac{10^9 \cdot C}{\lambda^4} \quad , \quad (1)$$

where A is a dimensionless parameter of refractive index, and B and C affect the curvature of refractive index and the whole amplitude.

For metal materials such as Ag and NiCr that are opaque in the visible band, the Drude model can be used as the material model, and the fitting formula is

$$\varepsilon = \varepsilon_{\infty} - \frac{\omega_p^2}{2\pi \cdot f (i\nu_c + 2\pi \cdot f)} \quad , \quad (2)$$

where ε_{∞} is the static dielectric constant, ω_p is the plasma resonance frequency, and ν_c is the plasma collision frequency.

According to the transfer matrix method:

$$\begin{bmatrix} B \\ C \end{bmatrix} = \left\{ \prod_{j=1}^K \begin{bmatrix} \cos\delta_j & \frac{i}{n_j} \sin\delta_j \\ in_j \sin\delta_j & \cos\delta_j \end{bmatrix} \right\} \begin{bmatrix} 1 \\ n_{k+1} \end{bmatrix} \quad , \quad (3)$$

where n_{k+1} represents the outgoing admittance of the ($k+1$ st) layer, and n_j represents the dielectric admittance of the j^{th} layer, $\delta_j = \frac{2\pi}{\lambda} n_j d_j$ represents the optical phase.

The reflectance R and transmittance T of thin films are defined as:

$$R = \left(\frac{n_0 B - C}{n_0 B + C} \right) \left(\frac{n_0 B - C}{n_0 B + C} \right)^* \quad , \quad (4)$$

$$T = \frac{4n_0 n_{k+1}}{(n_0 B + C)(n_0 B + C)^*} \quad , \quad (5)$$

where n_0 is the admittance of the incident medium. From this, the transmittance and reflectance spectra of the thin film can be calculated. Finally, the film thickness is used as a fitting parameter through the least squares method. By changing the thickness of the film layer, the corresponding film system spectrum is fitted, and the film thickness is continuously changed to obtain the transmittance/reflectance spectrum that is closest to the actual situation. This completes the determination of the film layer thickness.

The main factor to consider when selecting wavelength points in the range of 400-800 nm is to ensure that the selected wavelength points cover the entire wavelength band as evenly as possible, so as to maximize the spectral information carried by these ten wavelength points, which is beneficial for later spectral reconstruction and film thickness fitting. On the other hand, in order to develop equipment that can use multi-wavelength method for film thickness determination of coating products, we consider using existing commercial lasers as detection light sources and conducted research and summary on the center wavelength of the existing laser output light. Based on the above two principles, we have identified 10 wavelength points in the 400-800 nm wavelength range.

1.2 Experimental design and setup

The core flow of the whole experiment is shown in Fig. 1, which can be divided into three major components: spectral acquisition, spectral reconstruction and film thickness acquisition.

Sputtering coating technology is a physical vapour deposition technology^[18-23], which can make the substrate at a lower temperature to complete the coating, and the film layer obtained by good adhesion, densification, uniformity, is a commonly used in the industrial production of coated glass coating means. Physical vapor deposition technology can also be used for the manufacturing of optical metasurfaces^[24]. The coated samples involved in this paper are prepared by Bühler coating machine using the

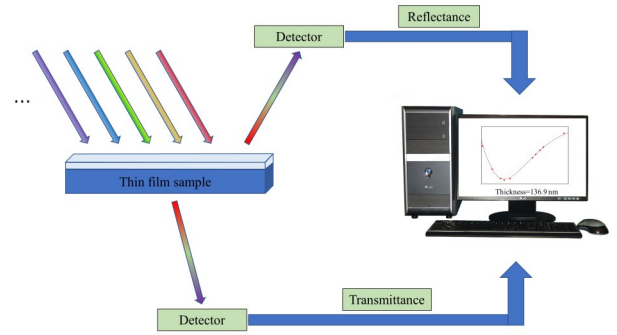


Fig. 1 Schematic diagram of multi wavelength spectral reconstruction and film thickness fitting experiment

图1 多波长光谱还原和膜厚拟合实验流程示意图

sputtering coating principle. The substrates of the coated samples are selected from the raw glass commonly used in the automotive glass manufacturing industry, and silicon wafers. The materials of the coated layers are selected from the Si_3N_4 , AZO, Ag, NiCr, which are commonly used in the manufacturing of the automotive glass industry. A Zeiss-branded scanning electron microscope (SEM) was used to photograph the dissected sections of the wafer substrate samples to obtain accurate information on the thickness of the film layers for comparison.

Spectral reconstruction and thickness fitting were performed using CODE 3.80 software from W. Theiss Hard and Software. During the fitting process, the corresponding material model and glass model are first used to establish the film system. Then, the film thickness to be solved and the parameters A , B , and C in the Cauchy model, as well as the parameters ω_p and ν_c in the Drude model, will be set as variables. Finally, input spectral data for fitting (e. g. full spectral data or transmittance and reflectance spectral data at 10 different wavelength points). Transmittance and reflectance spectral data can be used simultaneously, or separately, depending on the working environment. Using spectral data of both transmittance and reflectance are generally considered to be the most informative and accurate in terms of results, and the fitting can generally be started by setting a reference weight of 1:1 for the input transmittance and reflectance spectra. During the fitting process, the setting of initial values has a significant impact on the fitting results. Therefore, it is best to determine the initial values based on the approximate thickness range of the coated layer, in order to achieve a more accurate fitting effect. The software uses the least squares principle to adjust thickness and spectral reconstruction, stopping when the error is minimized to provide thickness results.

1.3 Data acquisition and processing

After the preparation of the coated samples, the transmittance and reflectance spectra of the thin film samples of various different materials were collected using an Agilent spectrophotometer, where the reflectance spectrum was collected at an angle of 6° and the transmittance spectrum was collected at an angle of normal incidence.

Due to the influence of the measurement system and

environmental factors on the acquisition of the transmittance and reflectance spectra, corrections are required to reduce the influence of noise on the subsequent fitting process. For this purpose, a self-written MATLAB program is used to correct the transmittance and reflectance spectra in order to eliminate the effects of system noise and random noise.

2 Results

SEM measurements were first used to obtain accurate thickness values for high-absorption, low-absorption, and metallic thin film samples. The multi-wavelength method was then applied to determine the thickness of these samples.

The fitted values of film thickness for different thicknesses and types of samples using the full spectrum fitting method (1 data point per nm of the transmittance & reflectance spectra in the 400-800 nm band) and the multi-wavelength method (using transmittance & reflectance spectral data at 10 wavelengths in the visible band) and their magnitude of error with respect to determining the thickness values by SEM and the multi-wavelength method, are shown in Table 1. According to the results in Table 1, it can be seen that for the two types of transparent dielectric single-layer film samples, when the film layer is thin, the base of the film thickness is small, resulting in a larger proportion of the error in the case of the absolute value of the error is not too large. In the case of the film thickness becomes thicker, due to the complexity of the spectral feature information, the amount of information carried by the spectrum is also relatively large, so the accuracy of the estimation of the thickness of the film layer based on the spectra can be improved, and the error is also reduced to a lower level. For metal film layers with interlayer protection against oxidation, the thickness of the protective film layer needs to be fixed during fitting to prevent interference with the fitting results. Under this premise, the thickness of the metal film layer can also be accurately fitted to the error level of about 1%. The errors for all samples remained within 7.1%.

Table 1 Comparison table between the fitting thickness of the full spectrum method/multi wavelength method (Ten points) and the SEM thickness

表1 全光谱法/多波长法拟合厚度(10点)与扫描电镜测试厚度结果比较表

film type	SEM thickness (nm)	full spectrum fitting thickness (nm)	ten points fitting thickness (nm)	ten points method deviation
Si ₃ N ₄ -1	38.0	38.6	35.3	-7.1%
Si ₃ N ₄ -2	78.0	74.5	79.5	+1.9%
Si ₃ N ₄ -3	142.5	143.9	136.9	-3.9%
Si ₃ N ₄ -4	283.2	283.8	281.3	-0.7%
AZO_1	10.4	9.8	9.7	-6.7%
AZO_2	42.6	42.4	42.5	-0.2%
AZO_3	137.7	135.6	133.8	-2.8%
AZO_4	239.4	235.7	234.0	-2.3%
AZO/Ag/AZO	54.6/14.7/43.7	54.6/14.7/43.7	54.6/14.9/43.7	+1.4%
Si ₃ N ₄ /NiCr/Si ₃ N ₄	51.2/8.9/39	51.2/8.9/39	51.2/8.8/39	-1.1%

During the acquisition of the spectral signals, both systematic and random noise may affect the intensity of the signals, making them deviate from the theoretical spectra and affecting the effectiveness of spectral reduction and film thickness fitting. For this reason, a self-written MATLAB program was used to correct the acquisition results. The correction was carried out iteratively by comparing the measured spectrum of the thin films system with the theoretical spectrum, using the scale factor and the additive and subtractive coefficients to adjust the intensity of the overall spectral signals in order to reduce the gap between the measured spectral signals and the theoretical spectral signals. The spectral of the thin films system, which was fitted out by CODE software, was used as the theoretical spectra (i. e., the target of correction), and the measured spectral intensity multiplied by a proportionality coefficient a and plus or minus a coefficient

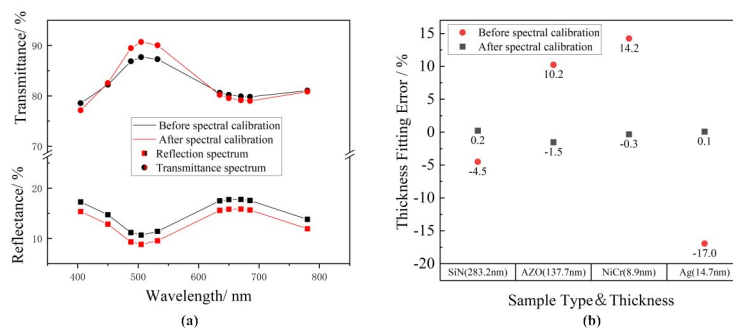


Fig. 2 (a) Comparison chart of measured reflectance and transmittance spectra before and after program calibration processing of Si₃N₄ (283.2 nm) sample; (b) thickness fitting error between fitting thickness and SEM thickness of different samples before and after spectral calibration (using full spectrum data points)

图2 (a)程序修正处理前后 Si₃N₄ (283.2 nm)样品的透反射光谱对比图;(b)光谱修正前后不同样品的拟合厚度与SEM厚度之间的厚度拟合误差(使用全光谱数据点)

cient b (assuming that the measured light intensity signal value is x under one wavelength, then the corrected spectral signal value under this wavelength is $a \cdot x + b$). After that a corrected spectrum is obtained. Subtracting all the corrected spectral intensity values at each wavelength from the theoretical spectral intensity values gives a difference, and all the differences in the visible wavelength band are summed to give a total difference, and the coefficient a and coefficient b that minimise this difference can be found using the MATLAB program. The measured spectrum intensity will be multiplied and added with the two coefficients a and b , that is to say, the correction is completed once, and according to the result of the correction, it can be corrected for a number of iterations. The corrected spectrum can effectively reduce the influence of systematic noise and random noise in the spectral signal, which is conducive to improving the accuracy of spectral reconstruction and film thickness fitting.

Figure 2(a) shows the comparison of the measured transmittance and reflectance spectra after the correction process. It can be seen that the corrected spectra have

been effectively adjusted compared with the original experimental spectra, and the characteristic information of the original spectra is retained, which proves that the correction process reduces the systematic noise and random noise of the transmittance and reflectance spectra and retains the key information related to the film thickness, which lays a foundation for the subsequent spectral restoration and the good effect of the film thickness fitting.

The measured spectral data before and after the correction process were used to fit the film thicknesses, and the errors between the fitted film thicknesses and the physical film thicknesses determined by SEM were compared, and the results are shown in Fig. 2(b). It can be seen from the results in Fig. 2(b) that, before spectral correction, the inclusion of system noise and environmental noise in the measured transmittance and reflectance spectra leads to significant fitting errors when the spectra are used for thickness fitting. After correction, the error in thickness fitting using the full spectrum data can be reduced to below 15% of the error before processing.

Figures 3(a) and 3(b) show the effective reconstruction of the visible wavelength band's reflectance and

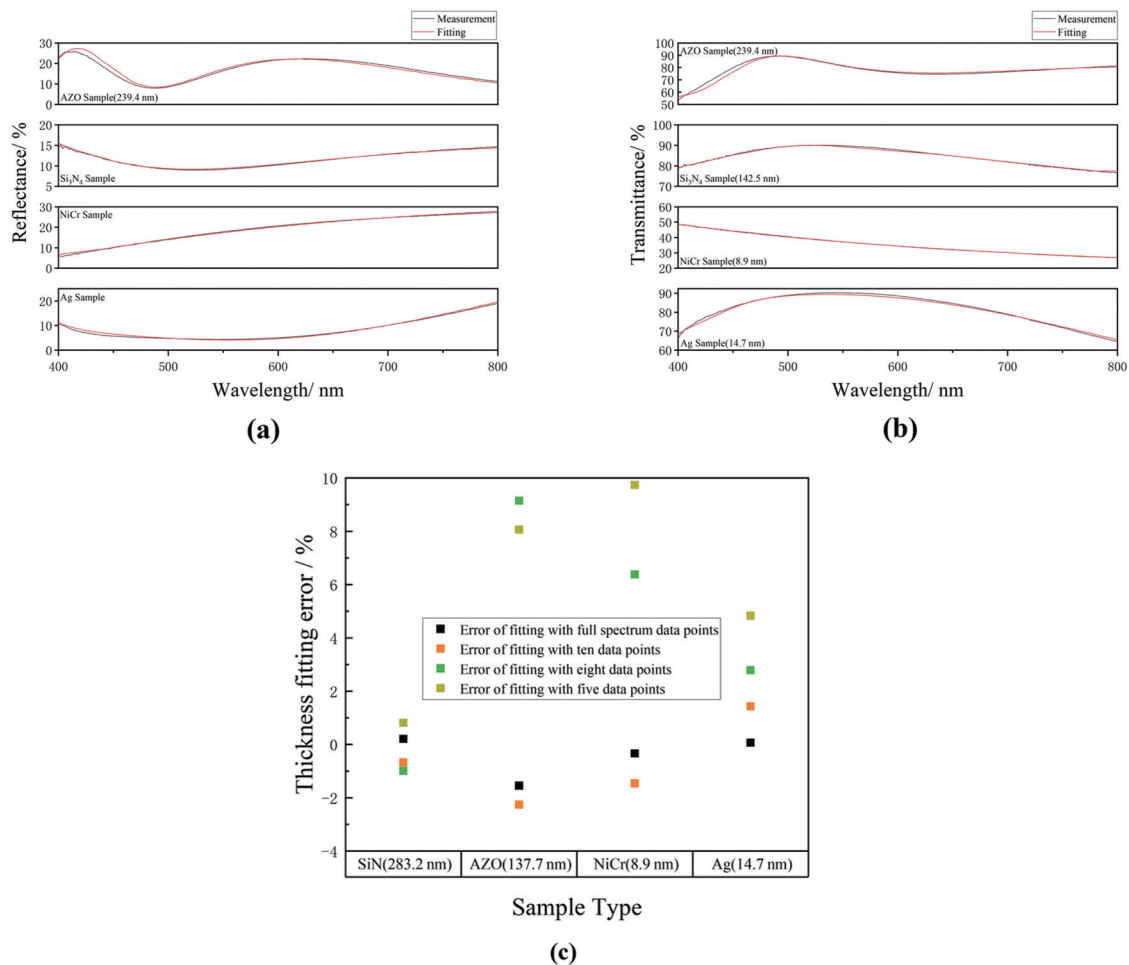


Fig. 3 Spectral fitting images using 10 different wavelength information of coating samples with different material compositions: (a) reflectance spectrum; (b) transmittance spectrum; (c) thickness fitting error between fitting thickness and SEM thickness of different samples when using different numbers of data points for fitting

图3 不同材料成分镀膜样品使用10个不同波长信息获得的光谱拟合图像:(a)反射光谱;(b)透射光谱;(c)使用不同数量波长数据点进行拟合时,不同样品的拟合厚度与SEM厚度结果相比的误差

transmittance spectra for thin film samples of different material compositions using the multi-wavelength method with ten data points.

To investigate the impact of the number of data points on the accuracy of film thickness fitting due to their different information content, thin film samples of four different materials were fitted using the spectral data of the entire visible range (400-800 nm), spectral data at ten wavelengths, and corresponding spectral data at eight and five wavelengths. The fitting errors were compared with the thickness results determined by SEM, as shown in Fig. 3(c). The figure illustrates that the accuracy of thickness fitting using the full spectrum data is the highest due to its maximum information content and complete spectral characteristic information. While using data from ten wavelengths for thickness fitting, despite a reduction in information content compared to full spectrum fitting, still retains most spectral information, resulting in high fitting accuracy with errors below 7.1%. As the number of data points used for fitting decreases to eight or five, the lack of sufficient information leads to larger fluctuations in fitting results and increasing errors, with errors reaching around 10% in extreme cases with five data points, indicating significant errors in thickness fitting.

Overall, the number of data points used in the multi-wavelength spectral method for thickness fitting represents the amount of spectral information related to film thickness to some extent. More data points mean more spectral characteristic information is included, leading to better reconstruction effects of the sample's transmittance and reflectance spectra during the fitting process, and more accurate film thickness fitting results. In the fitting process for thin film samples of four common coating materials, using spectral data from ten wavelengths shows little difference from using full spectrum data, with errors controlled within 7.1%, allowing for relatively precise fitting and detection of sample layer thickness. This significantly reduces the data volume required for thickness detection and simplifies the equipment needed for such measurements.

3 Discussion

The multi-wavelength method for thickness determination reduces the need for hundreds of data points, as required by traditional full-spectrum methods, to just ten data points. This greatly reduces the amount of data and processing time needed, making it highly suitable for the rapid, real-time determination of film thickness in industrial coating production lines. Traditional full-spectrum thin film thickness determination methods require spectrometers to collect spectra across the entire visible range or even longer wavelengths, and software to fit the collected spectra to determine the thickness of the coated film. This process is challenging due to the large volume of data and high costs, making it difficult to meet the rapid detection requirements of assembly lines. When using the multi-wavelength method for film thickness determination, there is no need to detect the entire spectrum of

the sample, only parts of the spectral intensity data at certain wavelengths, allowing for flexible fitting of both transmittance and reflectance spectra according to specific scenarios, thus reducing costs and simplifying application. This approach enables the use of inexpensive light sources and detectors for testing, avoiding the need for expensive precision spectrometer equipment. The reduction in spectral data points also shortens the time for data collection, transmission, and processing, and lowers the demands on computer system processing capabilities, enabling fast and accurate low-cost detection. The introduction of the multi-wavelength method significantly simplifies the process of measuring film thickness through spectral analysis and greatly reduces the economic costs involved.

Compared to traditional full-spectrum methods, the multi-wavelength method is cost-effective, has a simpler optical path, and is easy to integrate into coating chambers on production lines. Thus, adopting the ten-point data multi-wavelength method provides the coating industry with a low-cost, rapid, real-time, and accurate means of thickness determination.

According to the experimental results of multi-wavelength thickness determination on thin film samples of different compositions and thicknesses, the multi-wavelength method, despite sacrificing a small portion of spectral information, achieves effective reconstruction of the transmittance and reflectance spectra in the visible range (400-800 nm) through the application of spectral reconstruction technology, combined with the model information of the coated materials and substrates. This allows for accurate thickness determination of different composition samples with errors below 7.1%.

For conventional thin films, their visible spectra become more complex with increasing film thickness, i. e., the number of spectral peaks and valleys increases, along with the information content. Within a certain thickness range, the wavelengths selected by the multi-wavelength method can cover the peak and valley information within the band, leading to effective spectral reconstruction, simple equipment structure, low cost, and fast measurement speed. However, as film thickness increases to a certain level, its spectral characteristic information becomes complex, and using ten data points may not cover all spectral characteristics. At this point, sufficient information can be obtained by increasing the number of wavelengths in the collected spectrum. For thin films with a thickness of 10-300 nm, the ten-wavelength method can rapidly determine their thickness effectively; for thicker films, increasing the number of wavelengths detected can ensure the accuracy of thickness determination.

When implementing the multi-wavelength thickness determination method in the coating industry, we can choose common commercial lasers as light sources, combined with filters and detectors for different wavelengths, integrating them into a unified online thickness detection device. Alternatively, common LED light sources and halogen lamps with spectroscopic devices can be used for

multi-wavelength spectral thickness determination. Depending on the actual conditions and cost considerations of the coating production line, different schemes can be flexibly chosen to achieve the purpose of thickness determination using the multi-wavelength method.

4 Conclusions

In summary, this paper introduces a new, simple, and suitable multi-wavelength spectral data-based thickness determination method for online use in coating lines. This method requires only spectral data at ten wavelengths to reconstruct the entire visible spectrum (400–800 nm) of thin film samples and accurately obtain thickness information. Our experimental tests on samples of different material compositions demonstrate that the method's error in film thickness determination is below 7.1%, verifying its accuracy and practicality.

This approach significantly reduces the amount of data required for film thickness determination, marking a notable decrease in the complexity of spectral analysis and data requirements in film thickness detection. In situations where traditional spectral methods require hundreds of data points for accurate measurements, our multi-wavelength method effectively reduces this to just ten points without compromising accuracy. This innovation simplifies the detection equipment structure, greatly reducing costs, and is particularly suited for real-time online thickness determination in industrial coating applications.

Overall, our research establishes a new paradigm for industrial application of film thickness determination, highlighting the potential of multi-wavelength spectral thickness determination method. This method not only significantly improves efficiency and cost-effectiveness but also ensures high accuracy, making it a powerful tool in the fields of materials science and industrial manufacturing.

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