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Temperature-Dependent Residual Stresses and Thermal Expansion Coefficient of VO_2 Thin Films

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Abstract: This study aims to investigate the thermomechanical properties of vanadium dioxide (VO_2) thin films. A VO_2 thin film was simultaneously deposited on B270 and H-K9L glass substrates by electron-beam evaporation with ion-assisted deposition. Based on optical interferometric methods, the thermal–mechanical behavior of and thermal stresses in VO_2 films can be determined. An improved Twyman–Green interferometer was used to measure the temperature-dependent residual stress variations of VO_2 thin films at different temperatures. This study found that the substrate has a great impact on thermal stress, which is mainly caused by the mismatch in the coefficient of thermal expansion (CTE) of the film and the substrate. By using the dual-substrate method, thermal stresses in VO_2 thin films from room temperature to 120 °C can be evaluated. The thermal expansion coefficient is 3.21×10^{-5} °C^{-1}, and the biaxial modulus is 517 GPa.

Keywords: thin film; vanadium dioxide; residual stress; thermal stress; thermal expansion coefficient; biaxial modulus

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1. Introduction

Optical thin films have been applied in various optical components; in particular, the stability of optical thin film filters in the environment is particularly important [1]. When optical thin films are used in solar panels, optical communications, displays, lasers, optical detectors, and sensors in the visible and infrared spectra, the operating temperature range of these components will vary with changes in environmental temperature. As the temperature of the optical thin film rises, it will be affected by thermal stress, causing changes in the optical thickness or refractive index of the optical thin films and resulting in a drift in the center wavelength with temperature changes [2]. Vanadium dioxide (VO_2) has garnered widespread attention due to its unique insulator-to-metal phase transition characteristics. With temperature variations, VO_2 undergoes a reversible transition between insulating and metallic states, leading to significant changes in its electrical and optical properties [3]. The metal-to-insulator phase transition (MIT) behavior in VO_2 was first reported by Morin in 1959 [4]. In some study cases, vanadium dioxide (VO_2) exhibits a thermally induced structural phase transition from a monoclinic (M1) to rutile tetragonal (R) structure at 68 °C under no strain. In fact, thermal stress in thin films is complexly affected by multiple parameters, including the coefficient of thermal expansion (CTE), substrate properties, film thickness, and thermal conductivity. These factors collectively influence the overall thermal stress experienced by the thin film system [5,6]. Residual stress is a major challenge that can induce functional defects, often resulting in significant changes in shape and structural integrity. These effects may manifest as lamination, buckling, or even the formation of cracks. In 2012, Tsai and Chin et al. [7] discovered a relationship between the residual stress of VO_2 film deposition and the phase transition temperature

(Tt). Residual stress was quantitatively measured by X-ray diffraction. It has been verified that the reduction in the phase transition of polycrystalline VO_2 films with (011) orientation is due to shrinkage caused by residual tensile stress. The degree of crystallization of the VO_2 film will change the residual stress in the deposited state. In 2014, Sakai et al. [8] reported that substrates with larger thermal expansion coefficients cause larger out-of-plane lattice spacings in both Pt and VO_2 and lower transition temperatures for VO_2 films.

In order to address the issue of thermal stress in thin films, Chen et al. [9] reported the correction of deformation, thermal stress, and temperature relationships along the sample thickness direction and established models through the analysis of some experimental methods to determine the thermal expansion coefficient and biaxial modulus of thin film materials. When small temperature gradients are induced along the sample thickness direction, it has a significant impact on the accurate measurement of thin film materials and the biaxial modulus of thermal expansion coefficient and therefore should be considered in the analysis. If the influence of this temperature gradient is carelessly ignored in physical modeling, it may lead to errors in the obtained thermal expansion coefficient and biaxial modulus. Additionally, in order to evaluate the anisotropic stress of thin films, deformation must be measured in different directions. Tien et al. [10] used the fast Fourier transform (FFT) method to measure the anisotropic thermal stress in thin films and combined it with dual-substrate technology to explore the anisotropic thermal expansion coefficient and biaxial modulus of optical thin films. The functionality and reliability of multi-layer systems are strongly influenced by thermal elastic stresses. Zhang [11] decomposed the total strain into uniform strain components and bending strain components and developed a closed-form solution to overcome the complexity of traditional analysis. Subsequently, an alternative analytical model was developed based on the curvature radius of the neutral axis, used for zero normal strains and normal strains at the interface between the substrate and the thin film. In 2022, Wang et al. [12] investigated the simulation of thermal stress in VO_2 thin films based on a finite element method; their objective was to identify control parameters via simulation to fine-tune the design of a premium VO_2 thin film/substrate system, aiming for optimized performance and quality.

This study presents a thermal stress evaluation method for vanadium dioxide thin films. The values of thermal stress, the coefficient of thermal expansion (CTE), and the biaxial modulus of VO_2 thin films were determined by the proposed method. The thermal stress in VO_2 thin films may exhibit greater complexity due to stress redistribution caused by their phase transitions. There are not many relevant studies in the literature on the thermal stress of VO_2 films. This study intends to explore the thermal stress changes of VO_2 films and measure the biaxial modulus and thermal expansion coefficient (CTE) of VO_2 thin films deposited on different substrates by ion-assisted electron beam evaporation. The results can be used as a reference for the process design of VOx-based multi-layer film structure applications.

2. Materials and Methods

2.1. Thin Film Preparation

In this study, B270 and H-K9L glass substrates were characterized by their known Young's modulus and coefficient of thermal expansion. For the residual stress evaluation, we used two different glass substrates, with one side of each substrate surface being a rough surface and the other side polished to a flatness of one wavelength. The size of each glass substrate was 1.5 mm in thickness and 25.4 mm in diameter. These glass substrates utilized in the experiment were carefully cleaned with a cleaning agent, and then acetone was used in the ultrasonic cleaner for the final cleaning step. During the coating process, vanadium dioxide thin films were deposited on B270 and H-K9L glass substrates by using a SHOWA electron beam evaporation system with an ion-assisted deposition technique. Before initiating the film deposition process, the vacuum chamber underwent evacuation to achieve a base pressure of less than 9.0×10^{-4} Pa. The substrate heating temperature was set at 250 °C during the process. The coating material was bombarded by the electron beam generated by the electron gun and then deposited onto the substrate in an upward direction. The process vacuum was set at higher than 1.0×10^{-3} Pa. The high-vacuum evaporation system was outfitted with an optical monitor and a quartz crystal to ensure precise control over film thickness and deposition rate. The physical thickness and deposition rate of the films during the process were monitored using quartz monitoring, and the deposition rate in this study was set at 0.1 nm/s. The thin film deposition process involved operating the electron gun at a maximum power output of 10 kW, with voltage and current settings of 10 kV and 1 A, respectively. For ion-assisted deposition, the anode current ranged from 0.5 to 10 A, while the anode voltage varied from 80 to 300 V, with ion energy levels between 50 and 200 eV. The film thickness of the VO_2 layer was set to 60 nm, and the argon gas flow rate was maintained at 16 sccm.

2.2. Residual Stress Measurement

The intrinsic stresses within a film cause bending of the film/substrate system, thereby promoting partial relaxation. Tensile stresses occur when the substrate bends concavely upward, while compressive stresses occur when the substrate bends in the opposite direction. Figure 1 shows an illustration of the tensile stress and compressive stress behaviors. If the thin film generates compressive stress and tends to expand relative to the substrate, then the substrate is bent outward into a convex shape. On the contrary, a thin film containing internal tensile stress causes the substrate to bend upwards into a concave shape.

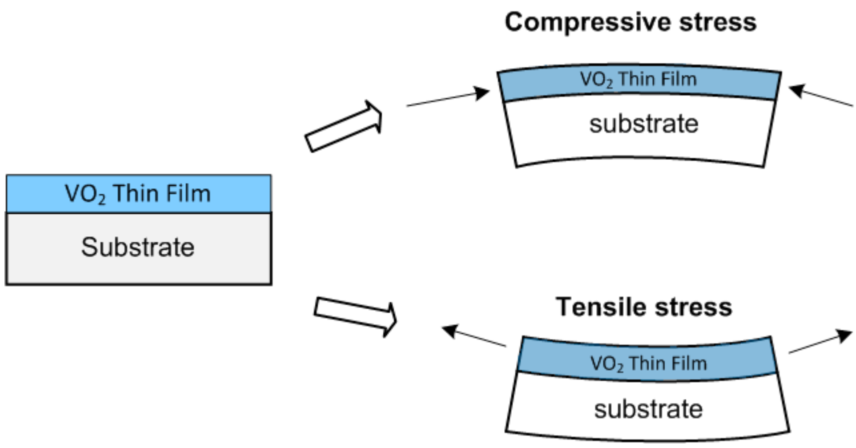

Figure 1. Schematic diagrams of the thin film/substrate system under tensile or compressive stress.

This study used a homemade, improved Twyman–Green interferometer [13] to measure residual stress in thin films. The interferometer is equipped with a heater and an NIR thermal imager, as shown in Figure 2. The measurement system uses a helium–neon laser with a wavelength of 632.8 nm as the light source, forming a point source of laser light through a spatial filter. The light then passes through a collimating lens to generate a uniform parallel beam, splitting into two beams through a beamsplitter. These two beams are reflected by a reference mirror and the surface of the test object, then recombined into a single beam by the beamsplitter, forming an image on a rotating screen and creating interference fringes. Finally, the interference pattern is captured using a CCD camera, and the thin film surface profile is reconstructed by fast Fourier transform and phase retrieval methods to detect the curvature radius value of the test object. Subsequently, the residual stress in the thin films can be calculated using the Stoney formula, as follows [14].

$$\sigma = \sigma_i + \sigma_{th} = \frac{1}{6}\frac{E_s}{(1-v_s)}\frac{{t_s}^2}{t_f}\frac{1}{R}, \frac{1}{R} = \frac{1}{R_2} - \frac{1}{R_1}, \tag{1}$$

where σ is the residual stress; σ_i represents the internal stress; σ_{th} represents the thermal stress, E_s represents the Young's modulus of the base material; ν_s is the Poisson's ratio of the substrate material; t_s is the thickness of the substrate; t_f is the thickness of the film;

R_1 and R_2 are the curvature radius values of the substrate before and after the coating process; and R represents the change in the curvature radius of the substrate before and after coating.

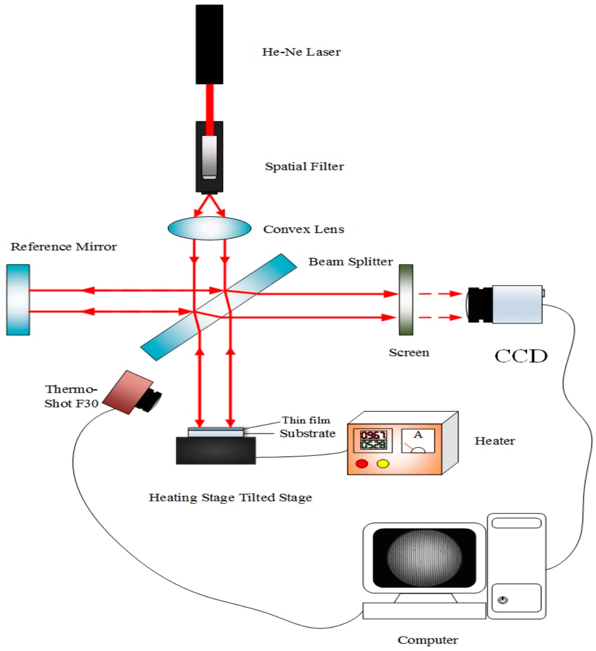

Figure 2. Schematic diagram of the measurement system for measuring residual stress and thermal stress.

The thermal stress (σ_{th}) arises from the mismatch in thermal expansion between the thin film and the substrate. The thermal stress can be written as follows:

$$\sigma_{th} = \left(\alpha_s - \alpha_f\right)\frac{E_f}{1-\nu_f}(T_2 - T_1), \quad (2)$$

where α_f and α_s represent the thermal expansion coefficients of the substrate and film, respectively, and E_f and ν_f are the Young's modulus and Poisson's ratio of the thin film. $\frac{E_f}{1-v_f}$ is the biaxial modulus of the film. T_1 and T_2 are the thin film temperature differences before and after substrate heating. From Equation (2), it can be proven that the slope of the experimental stress–temperature curve is equal to the following:

$$\frac{d\sigma}{dT} = (\alpha_s - \alpha_f)\frac{E_f}{1-\nu_f}. \quad (3)$$

Here, it is assumed that the values of α_s, α_f, E_f, and ν_f are independent of temperature. Given the lack of information on either α_f or $\frac{E_f}{1-v_f}$, both values can be obtained by simply determining $d\sigma/dT$ on each of two substrates with known values of α_s and solving two equations in the form of Equation (3) for α_f and $\frac{E_f}{1-v_f}$ simultaneously. This approach is called the double substrate method [15]. The principle is to deposit the same thin film on substrates made of different materials in order to measure the stress–temperature curve relationship of the thin film on different glass substrates. The thermal expansion coefficient and biaxial modulus of the thin films are determined by simultaneously solving Equations (4) and (5) [16,17].

$$\alpha_f = \frac{\alpha_{s1}\frac{\sigma_2}{T} - \alpha_{s2}\frac{\sigma_1}{T}}{\frac{\sigma_2}{T} - \frac{\sigma_1}{T}} \quad (4)$$

$$\frac{E_f}{1-\nu_f} = \frac{\frac{\sigma_2}{T} - \frac{\sigma_1}{T}}{\alpha_2 - \alpha_1} \tag{5}$$

where α_{s1} and α_{s2} are the thermal expansion coefficients of different glass substrates; α_f is the thermal expansion coefficient of the thin film; Ef and ν_f are the biaxial modulus and Poisson's ratio of the thin film material, respectively; and $\frac{\sigma_1}{T}$ and $\frac{\sigma_2}{T}$ are the two slope values of the stress–temperature curves of the thin film deposited on two different substrates. Table 1 indicates the physical parameters of the two different glass substrates.

Table 1. Physical parameters of different glass substrates.

Glass Substrate	B270	H-K9L
CTE (°C^{-1})	8.2×10^{-6}	7.6×10^{-6}
Young's modulus (GPa)	71.5	79
Poisson ratio	0.219	0.214
Thickness (mm)	1.5	1.5

3. Results

As the temperature rises from ambient temperature to 120 °C, the average residual stress significantly increases. The increase in temperature causes the residual stress of the VO_2 thin film deposited on the B270 and H-9KL glass substrates to shift towards a more tensile state. The coefficient of thermal expansion (CTE) of the VO_2 thin films can be determined by analyzing the stress–temperature relationship using the double substrate method, which takes into account factors such as the substrates' properties, film thickness, and temperature variations. The residual stress variation of the VO_2 film coated on the B270 and H-9KL glass substrates during heating is approximately linear.

3.1. VO_2 Coated on B270 Substrate

Firstly, we observe the thermal stress changes of the VO_2 thin film deposited on the surface of the B270 glass. Figure 3 shows the NIR thermal images, interference fringes, and 3D surface profile of the VO_2 film coated on the B270 glass substrate at different heating temperatures. The changes in the 3D contour map also indicate that as the heating temperature increases, the contour of the film surface changes from slightly concave to more concave, indicating that the radius of curvature changes from large to small. Figure 4 shows that as the heating temperature increases from room temperature to 120 °C, the compressive stress decreases and transforms into tensile stress. As the heating temperature increases, the stress value varies from −0.238 to 0.819 GPa. The residual stress changes from compressive stress to tensile stress in the heating process. We found that there is no significant difference in the thermal stress changes of VO_2 thin films deposited on B270 glass substrates during heating and cooling processes. The residual stresses in the VO_2 thin films on the B270 glass change almost proportionally to temperature changes during heating and cooling cycles, as shown in Figure 4. The residual stress is related to the morphology, texture, and grain size of the VO_2 thin films, typically including tensile stress formed during island structure coalescence and compressive stress generated by defects, vacancies, and impurities. It was found that the transmittance loop of the VO_2 films when thermally cycled differs between films with different manufacturing conditions, resulting in a different transition temperature (Tt) than that of a strain-free VO_2 single crystal [18]. However, when the temperature changes between 60 and 70 °C, the stress changes rapidly, which is speculated to be related to the phase transition temperature of the VO_2 film. It is known that VO_2 undergoes a structural phase transition from monoclinic to tetragonal, and each structure is stable at temperatures below and above the transition temperature. When the temperature rises above the phase transition temperature, a transition from compressive stress to tensile stress occurs, which is consistent with the expected volume expansion (~0.32%) associated with the transformation from monoclinic to tetragonal crystals [19]. The local volume change is related to the change in the relative position of V and O atoms.

The contraction and expansion of V-O bonds cause octahedral deformation, providing a basis for reversible stress changes during the phase transition process.

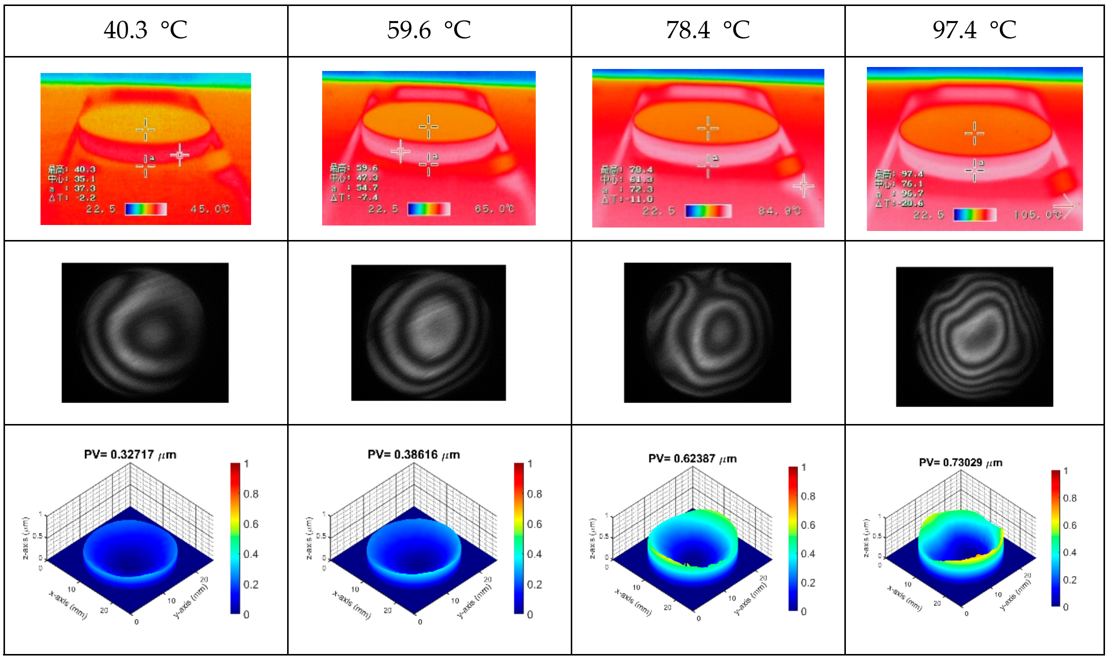

Figure 3. Thermal images, interference fringes, and 3D surface profile of the VO_2 film coated on the B270 glass substrate at different heating temperatures.

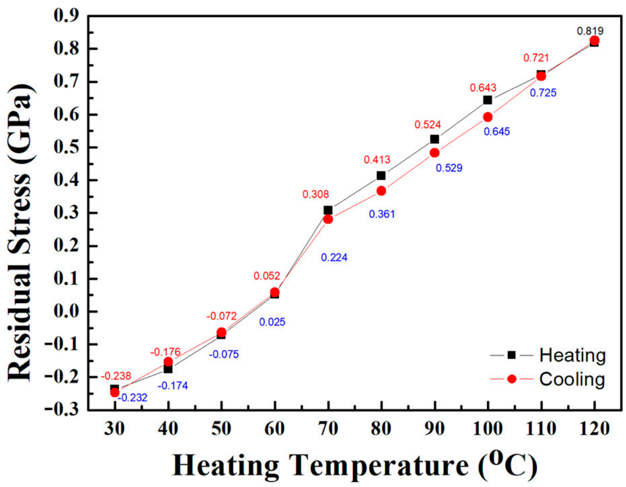

Figure 4. Residual stress vs. heating temperature for VO_2 film coated on B270 glass.

By conducting curve fitting in Figure 4, we determined that the slope of the stress–temperature plot for VO_2/B270 glass is 5.6×10^{-3} GPa/°C. This indicates a transition in the stress state of the VO_2 film deposited on the B270 glass. The observed behavior suggests a gradual shift in residual stress from compressive to tensile as the heating temperature rises. This transition underscores the influence of temperature fluctuations and the mismatch in the coefficient of thermal expansion (CTE) on thermal stress.

3.2. *VO_2 Coated on H-K9L Substrate*

Figure 5 shows the NIR thermal images, the interference fringes, and the 3D surface profile of the VO_2 thin film deposited on the H-K9L glass substrate, which were examined at various heating temperatures. The measurement results show that the number of interference fringes gradually increases with increasing temperature. The changes in the 3D profile also show that as the heating temperature increases, the film's surface profile changes from slightly concave to more concave, which means the radius of curvature changes from a large value to a smaller value. Figure 6 illustrates the residual stress in the VO_2 thin film deposited on H-K9L glass substrates plotted against temperature. The plot shows the measured average stress (open circles) data versus heating temperature. When examining the impact of temperature on the average residual stress, we assess changes in substrate curvature before and after film deposition in all radial directions. As the temperature increases, the film stress values fluctuate within the range of −0.247 to 0.825 GPa. The residual stress changes from compressive stress to tensile stress in the heating process. We found a linear correlation between residual stress and heating temperature. As the temperature rises from room temperature to 120 °C, the residual stress in the VO_2/H-K9L sample transitions from compressive to tensile stress. Both samples display a rise in residual stress with increasing heating temperatures, depicting the stress function of VO_2 films on various substrates in response to temperature changes. Through curve fitting in Figure 6, we determined that the slope of the stress–temperature plot for the VO_2/H-K9L sample is 3.2×10^{-3} GPa/°C. It should be noted that a significant stress alteration in the VO_2 thin film coated on the H-K9L glass occurs when the temperature exceeds 60 °C. As the temperature rises, a transition from compressive to tensile stress in the VO_2 film can be observed.

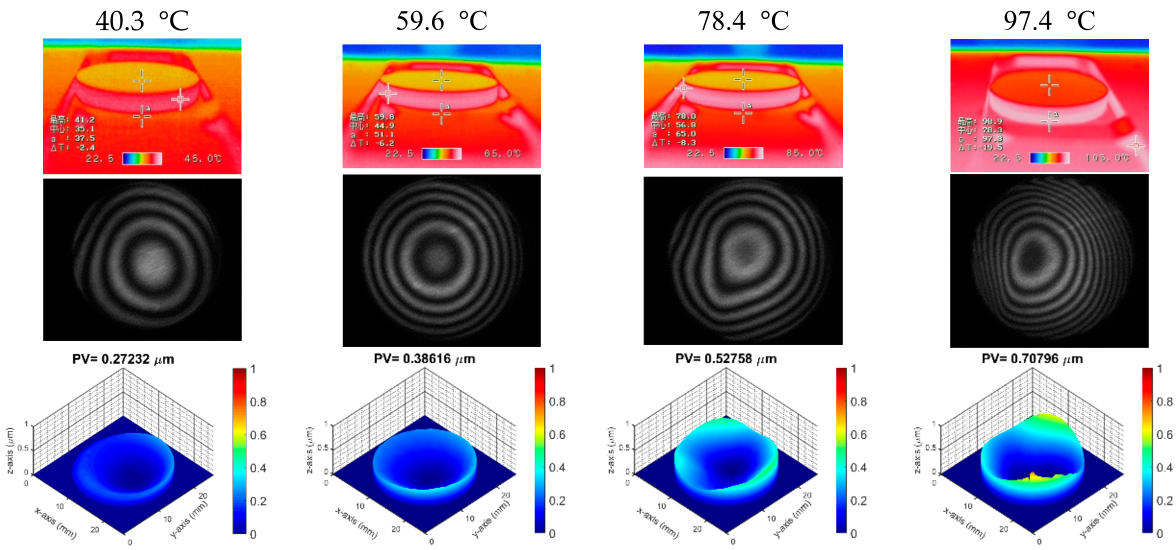

Figure 5. Thermal images, interference fringes, and 3D surface profile of the VO_2 film coated on the H-K9L glass substrate at different heating temperatures.

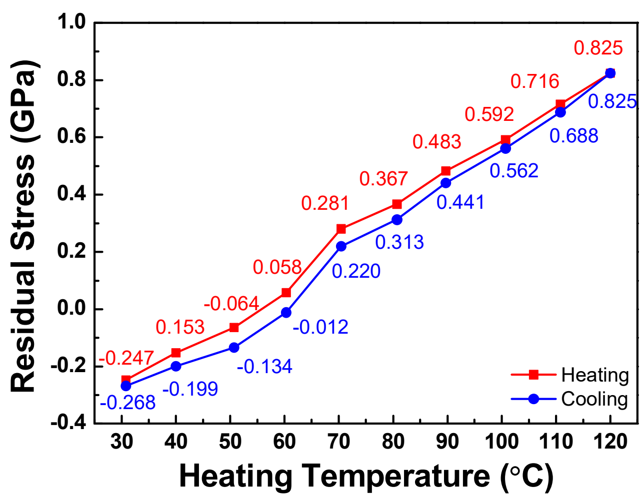

Figure 6. Residual stress vs. heating temperature for VO_2 film coated on H-K9L glass.

3.3. Evaluation of the CTE and Biaxial Modulus for VO_2 Thin Films

The results of the residual stress versus heating temperature show that VO_2 thin films coated on the HK9L and B270 glass substrates exhibit compressive stress after coating. As the temperature increases from 60 to 100 °C, the compressive stress gradually moves toward the tensile stress state. Figure 7 shows the linear fitting diagram of the residual stress vs. heating temperature for the VO_2 films coated on the B270 and H-K9L glass substrates.

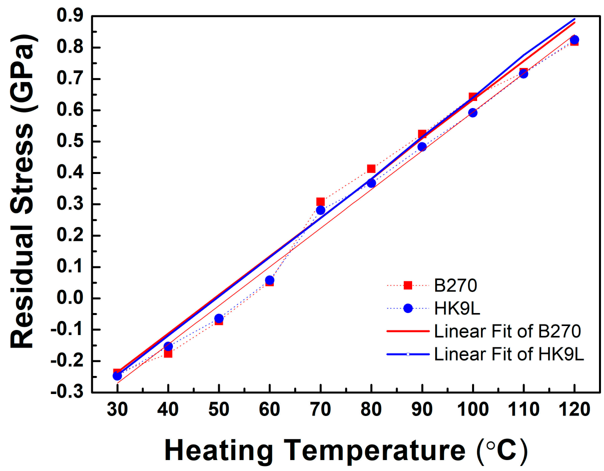

Figure 7. Linear fitting diagram of the residual stress vs. heating temperature for VO_2 film coated on B270 and H-K9L glass substrates.

The CTE and biaxial modulus are determined using linear regression analysis over the temperature range from room temperature to 120 °C. Intuitively, the expected average CTE of the VO_2 film over the analyzed temperature range is estimated to be 3.21×10^{-5} °C^{-1}, assuming the physical properties of the film and substrate remain constant over the elastic range. The biaxial modulus of the VO_2 film was determined to be 517 GPa. These findings are consistent with CTE data reported in the literature [20,21].

The observation of the relationship between residual stress and temperature can be divided into two parts. When a 60 nm thick VO_2 thin film is deposited on B270 and HK9L, the residual stress is negative due to the compressive stress relationship at lower temperatures of 30–50 °C. However, as the surface temperature of the thin film increases, a significant decrease in compressive stress is observed. When the heating temperature exceeds 60 °C, the residual stress value is positive due to the tensile stress, and the residual stress value increases with temperature. At a temperature of 70 °C, the residual stress value slightly increases, which may be related to the thermochromic nature of VO_2 films, causing it to deviate from a linear relationship.

It can be observed from the linear fitting plots of the residual stress vs. heating temperatures of the B270 and HK9L substrates that there are some slight fluctuations between 60 and 70 °C. Currently, it is inferred that this is due to changes in the internal structure, and it cannot be determined whether it is affected by thermal stress. The distribution of thermal stress growth shows linear growth. Through calculations based on simultaneous equations, it is found that the thermal expansion coefficient of vanadium dioxide composite films is 3.21×10^{-5} °C^{-1}, and the biaxial modulus is 517 GPa. Based on the above data, it can be inferred that the thermal stress of the B270 and HK9L substrates shows linear growth from 30 °C to 120 °C at different temperatures, with significant fluctuations at 60–70 °C. As thermal stress results from discrepancies in the thermal expansion coefficients between the substrate and the thin film, resulting in size variations, the thermal stress of the film does not increase significantly before and after 60 °C and exhibits linear growth. From this, it can be inferred that the intrinsic stress may be affected by changes in the internal structure of the film, and further research is needed to determine the actual changes in the internal structure of the film.

4. Conclusions

This experiment was conducted by measuring vanadium dioxide films that were simultaneously coated on B270 and H-K9L glass substrates, and we measured the residual stress from room temperature to 120 °C. Although the VO_2 phase transition mechanism remains controversial, considerable progress has been made in the modification of phase transitions in recent years. In this work, the experimental investigation focused on exploring the temperature-dependent coefficient of thermal expansion (CTE), the residual stress, and the biaxial modulus of VO_2 thin films. When thermal gradients cause thermal stress, it may lead to the failure of the thermochromic thin film structure. Therefore, for the measurement of the thermal–mechanical characteristics of optical thin films, a thorough understanding of thermal stress in the thin film structure is necessary. By using the dual-substrate method, thermal stresses in VO_2 thin films from room temperature to 120 °C can be evaluated. The thermal expansion coefficient is 3.21×10^{-5} °C^{-1}, and the biaxial modulus is 517 GPa. The experimental findings provide valuable insights for optimizing the excellent VO_2 film/substrate system settings, enhancing its potential for various applications in smart windows, optoelectronic switches, and intelligent heat dissipation devices.

Author Contributions: Conceptualization, C.-L.T. and C.-Y.C.; methodology, C.-L.T.; writing—review and editing, C.-L.T. and C.-Y.C.; validation, C.-L.T. and C.-Y.C.; formal analysis, C.-L.T. and C.-C.W., data curation, C.-C.W. and S.-C.L. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data presented in this study are not available due to privacy.

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

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