Research Article

Thickness Measurement of V_2O_5 Nanometric Thin Films Using a Portable XRF

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Nanometric thin films have always been chiefly used for decoration; however they are now being widely used as the basis of high technology. Among the various physical qualities that characterize them, the thickness strongly influences their properties. Thus, a new procedure is hereby proposed and developed for determining the thickness of V_2O_5 nanometric thin films deposited on the glass surface using Portable X-Ray Fluorescence (PXRF) equipment and the attenuation of the radiation intensity $K\alpha$ of calcium present in the glass. It is shown through the present paper that the radiation intensity of calcium $K\alpha$ rays is proportional to film thickness in nanometric films of vanadium deposited on the glass surface.

1. Introduction

Thin films have always been intended to be utilized primarily for decorative purposes; however they are currently being used for multiple purposes. It has contributed to the technological revolution of integrated circuits and telecommunications [1–4].

Moreover, when physical and chemical techniques are combined, thin films can be produced from any material including organic or inorganic, metals, metal oxides, and polymers [5, 6].

Thin films are layers whose thickness is naturally decisive for their electromagnetic and quantum characteristics [7–9]. Nowadays, several techniques are used to measure thin films thickness, even in an indirect way. In this paper, the techniques of Rutherford which are back scattering [10, 11], Scanning Electron Microscopy, profilometry [12, 13], interferometry, and the low explored X-ray fluorescence [14–19] are highlighted.

The thickness measurement of materials, especially thin films, is currently being made by sophisticated and very expensive devices which often need a proper sample treatment to perform the desired measurement.

Among those techniques, the Portable X-Ray Fluorescence (PXRF) presents a low cost that dispenses sample preparations. Furthermore, it can be installed for *in situ* measures, which has a notably good experimental error when compared with the other existing methodologies or measurement techniques [20–24]. An application of the PXRF technique to measure the thickness of the vanadium pentoxide (V_2O_5) has not been found in the literature, which is widely used in the production of electrooptical devices. The objective was to investigate the analytical potential of a Portable X-Ray Fluorescence (PXRF) system for vanadium oxide thin films (or layers) thickness measurement. Along these lines we propose a fast and precise empirical method based on the attenuation law of radiation in order to determine the thin films thickness vanadium pentoxide (V_2O_5) deposited on the glass surface.

FIGURE 1: V_2O_5 thin films were used in the calibration techniques with 160, 110, and 48 nm of thickness, respectively, from left to right, measured by SEM.

2. Methods and Materials

2.1. Preparing the Samples. Vanadium oxide thin films were deposited by resistive thermal evaporation in high vacuum. Glass plates as substrates were used for both microscope and X-rays fluorescence measurements. The substrates were cleaned in ultrasonic acetone baths solutions followed by isopropanol, which were subsequently dried in hot plate to complete the evaporation of solvents and water. Vanadium oxide films of different thicknesses were deposited with an amount variation of V_2O_5 powder mass, Figure 1.

The evaporation bath containing the oxide to be evaporated in tablet form is made of metallic tungsten. The material in tablet form was obtained from uniaxial quantities of 130 mg, 100 mg, 55 mg, 45 mg, and 35 mg from V_2O_5 powder (Sigma-Aldrich, 99.9%) weighted on an analytical scale. The film depositions were done in a brand system called HHV (Auto 306). The substrates were held in a rotated substrate holder to ensure a better uniformity in the film thickness during the deposition.

The substrate holder and the substrates were kept in room temperature. A glow-discharge process was performed to ensure the cleanliness of the substrates before the deposition. The back pressure was 7.89×10^{-6} mbar and 1.15×10^{-5} mbar during the deposition.

2.2. Scanning Electron Microscopy. In the Scanning Electron Microscopy (SEM) an electron beam is accelerated with a potential difference of thousands of volts; when it reaches the sample, it is reflected and directed to the detector. In case of nonconductive samples as the studied films, a thin film conductor ought to be deposited on its surface so that an electric charge of the surface does not occur; this could lead to an electrostatic deviation of the incident electrons.

A SEM was employed to determine the thin films thickness from 100 mg, 70 mg, and 35 mg of V_2O_5 powder; this thickness is subsequently confronted with the Portable X-Ray Fluorescence technique average thickness. A FEI Quanta 200 microscope (Oregon, USA) which belongs to the University of Londrina Electron Microscopy Laboratory and Microanalysis was used. The images were performed employing an acceleration voltage of 30 kV.

2.3. Instrumentation. A home-made Portable X-Ray Fluorescence (PXRF) spectrometer was employed for the analysis with the following items.

2.3.1. X-Ray Tube. A MAGNUM MUHV Mini X-ray tube manufactured by Moxtec (Moxtec Inc.) with both silver target (Ag) electric current and electric voltage is controlled by a high-voltage source. This tube can be operated in up to 40 kV and 100 μ A with a maximum power of 4 W, although it was employed at 28 kV maximum voltage and maximum current 15 μ A in our analysis.

For a better system performance Ag filter with 100 μ m thick was used in the outlet of it. This filter was selected because it allows a strong filtering of the Ag K line excitation increasing the sensitivity and decreasing the background that is highly produced from the major constituent of substrate (glass) SiO_2 .

Therefore, the use of the Ag filter provides a better value for the peak/background ratio, allowing an accurate analysis of the elements in the energy range of 3–15 keV [25] which is especially suitable for the energies $K\alpha$ 4.95 and $K\beta$ 5.43 keV of vanadium and calcium ($K\alpha$ = 3.69 and $K\beta$ = 4.01 keV) [26].

2.3.2. Detector. An XR-100CR Si-PIN detector with a preamplifier, a thermoelectric cooling system, a conjugated high-tension source module, and an amplifier was also employed. This detector has 6 mm² crystal by 500 μ m thick with 12.5 μ m Beryllium window thick and 145 eV energy resolution for Mn- $K\alpha$ of 5.9 keV. It was connected to a multichannel analyzer model MCA8000A (Amptek Inc.).

2.3.3. Geometry. X-rays from the samples were collimated using a 1 cm long by 0.5 mm diameter Ag cylinder. This collimator was used to minimize the dead time in the measurement and the Compton scattering. Both the detector and the tube were positioned at 45° on the samples surface.

The measurements were performed with the samples 2 cm away from the tube and 2.5 cm from the detector; as a result the analyzed area was approximately 0.8 cm². The excitation and detection time for each measurement was 300 seconds. Each sample was analyzed in triplicate.

The spectral analyses were performed using WinQXAS software. The whole system is shown in Figure 2.

2.3.4. Theoretical Background. When the X-ray photons go through the matter, the emerging beam intensity (I) is smaller than the incident beam intensity (I_0) due to the interaction (absorption and scattering) with the constituent atoms of the material. It is experimentally verified that the decreased number of photons of the incident beam is directly proportional to the absorbing material thickness, which is related to it through the law of radiation attenuation in the matter given by

$$I = I_0 e^{-\mu' x}, \quad (1)$$

where μ' is the Linear Attenuation Coefficient (cm⁻¹) of the material and its value depends on the energy of the incident

FIGURE 2: Portable X-Ray Fluorescence system: (1) sample support, (2) X-ray minitube, (3) X-ray detector, (4) standard electronic (5) laptop computer.

FIGURE 3: PXRF spectra obtained for the substrate. Measurement time 300 s.

radiation, the attenuating material density, and its chemical composition; x represents the material thickness in centimeters; I_0 is the incident beam intensity in the sample; I is the emerging beam intensity in the sample.

If we consider a simple case of a thin film of vanadium deposited on a glass flat surface (substrate), the film thickness could be determined by the measurement of a constituent element attenuation intensity of the substrate [27–29].

Critical analyses of five substrates were previously performed to identify the main constituent elements of the substrate (glass) and then determine which element had the best peak/background (P/B) ratio and also the best resolution (FWHM). An illustrative spectrum is presented in Figure 3.

3. Results and Discussions

As it can be seen in Figure 2, three elements have been found in the glass container: K, Ca, and Fe. Among all of elements found only two of them (Ca and Fe) were selected to be

TABLE 1: Average values for the ratio peak/background and FWHM for the glass substrate number 5.

Element	Energy (keV)	Peak/background	FWHM	
			(Channel)	(keV)
Fe	6.40	1.15	4.99	0.14
Ca	3.69	1.07	4.12	0.11

FIGURE 4: Theoretical attenuation according to (1) for the Ca-K α (3.69 keV) through the vanadium film.

analyzed, P/B and FWHM, due to the fact that both presented a high intensity; there is a considerable distinction between the others and in almost all of them a noninterference with the peaks of vanadium K α = 4.95 and vanadium K β = 5.43 keV. The results are shown in Table 1.

Based on the results shown in Table 1, it has been determined that the calcium element was the one that presented the best resolution and the best peak/background ratio among the twelve existing elements in the glass substrate. Thereby, we used the attenuation of the Ca-K α line at 3.69 keV to estimate the thickness of the vanadium films, according to (1).

Thus, assuming that a thin film with a nanometric vanadium thickness is deposited on a substrate of another element (Ca), the measured intensity for the X-rays in which characteristic of the Ca-K α is exponentially attenuated as an example is simulated in Figures 4 and 5 of the theoretical attenuation according to (1) for Ca-K α (3.69 keV) through the vanadium film.

Despite the fact that the attenuation of the radiation by the matter is exponential, for sub-micrometers thicknesses (μm) and nanometers (nm) as can be seen in the theoretical simulated graphs of Figures 4 and 5, it is possible to use a linear approximation to determine the thickness of the film [30–33]. Moreover, an appropriate calibration curve for PXRF system must be carried out, especially to decrease the uncertainties arising from the geometry used and minimize the background and scattered radiation.

TABLE 2: Thicknesses of deposited films in terms of material mass that has been used for each evaporation.

Mass (mg)	Thickness measured by SEM (nm)	Surface density ($\mu\text{g}/\text{cm}^2$)
100 ± 2.5	160 ± 5.41	53.7 ± 3.8
70 ± 2.5	110 ± 3.45	36.9 ± 3.0
35 ± 2.5	48 ± 7.4	16.1 ± 3.3

FIGURE 5: Theoretical attenuation according to (1) for the Ca-K α (3.69 keV) through the vanadium film.

3.1. Calibration Curve. To determine the thickness of the vanadium film in unknown samples using PXRF with the proposed theoretical model a calibration method for the portable spectrometer is required. The calibration method proposed was made with a series of standard samples with known thicknesses where a net area curve (counts) under the peak calcium (K α -3.69 keV) was plotted against thicknesses of vanadium film, V_2O_5 .

Three samples of vanadium film, V_2O_5 , with known masses were deposited on a glass substrate as described in Section 2.1 and their respective thicknesses were determined by Scanning Electron Microscopy (SEM) to determine the calibration curve. For all the samples, a calibration curve was made by calculating the mean of three measurements of the net area of Ca-K α . The films thicknesses utilized to determine the calibration curve are shown in function of their mass deposition in Table 2.

Figure 6 demonstrates a micrograph of a vanadium oxide + glass film. The measured thickness of V_2O_5 film is 160 nm, for the evaporated mass of 100 mg.

Figure 7 presents a graph of calibration curve determine where the net area of the Ca-K α line is Y-axis and vanadium thickness is X-axis.

As can be seen in Figure 7, the reasonable fit of the data points is shown. It is an indication of the absence of significant matrix effects in our samples. An excellent agreement with the theoretical model used is also evidenced in the calibration curve, especially for thickness of nanometer order, where

FIGURE 6: Scanning Electron Microscopy of the deposited film with 100 mg evaporated over the glass substrate.

FIGURE 7: Calibration curve for net area of K α line of calcium (Ca) versus thickness of vanadium determined by SEM.

the absorption of X-rays of K α line of calcium by vanadium is small but not negligible.

Analysis of variance or ANOVA has been carried out to verify the quality of the adjustment of the model used in the calibration curve. The result is presented in Table 3, where the equation is determined ($Y = 10611.42 - 13.34X$), where Y is the net area (counts) of K α line of calcium and X is thickness of vanadium.

Although R^2 found for the model is 0.98 the sum of the quadratic regression corresponds to 98.85% of the total variance, indicating that the linearity of the model is very good. This fact is also evidences with very low waste values and indeed for thicknesses analyzed in this scientific paper, there is a good linear relationship between the net area of K α line of calcium (counts) and the thickness of vanadium.

3.2. Unknown Samples Analyses. Three samples with different mass were produced with the same methodology used in the preparation of the calibration samples. Each sample had the size of 7.5 cm by 2.5 cm and the analyses were done

TABLE 3: Analysis of adjustment variance of the model presented in Figure 7.

Model	Source of variation	Quadratic sum	Degrees of liberty	Quadratic average	R^2
$Y = 10611.42 - 13.34X$	Regression	0.70939	1	0.70939	0.98
	Residues	0.00102	6	0.00204	
	Total	0.71041	7		

TABLE 4: Thickness of unknown samples of the vanadium films calculated.

Mass (mg)	Net area Ca-K α	Thickness (nm)	Superficial density ($\mu\text{g}/\text{cm}^2$)
45 ± 2.5	9783 ± 108	62 ± 3.3	20.8 ± 0.2
55 ± 2.5	9512 ± 167	75.6 ± 4.1	25.4 ± 0.4
130 ± 2.5	8132 ± 93	185.7 ± 6.0	62.4 ± 0.9

FIGURE 8: Vanadium thickness on the glass substrate calculated using a calibration equation.

in three different points. The net area of Ca-K α line for the spectra obtained by PXRF was determined, and the correspondent thickness was calculated using the equation obtained with the calibration curve. The results found for the thickness and superficial densities of vanadium with their respective deviations for three samples are presented in Table 4. Results show that the three measures have relatively small uncertainties, all of them below 5%.

Figure 8 shows a plot of the Ca-K α line net area experimentally obtained for the three unknown samples as a function of the vanadium thickness determined by the calibration equation. The correlation coefficient found for three samples was 0.997.

A typical spectrum obtained with the Portable X-Rays Fluorescence system for the sample of thickness 185.7 ± 1.2 nm is shown in Figure 9.

In this spectrum we can clearly see the presence of other elements as Si, Ar, Ag, W, and Au. The presence of W may be attributed to a contamination that occurred in the sample preparation process at issue [34]. The other elements are from the equipment used and of course there is air between

FIGURE 9: PXRF spectrum of vanadium layer on a glass substrate for the thickness of 185.7 nm. Measurement time 300 s.

FIGURE 10: Thickness determined by PXRF for all the samples of V_2O_5 versus that determined by the deposited mass.

the films of vanadium and the detector, since the samples were not analyzed in a vacuum system. In Figure 10 there is a graph that shows the calculated thickness of the model versus the vanadium oxide superficial density deposited on the glass substrate determined by the Portable X-Ray Fluorescence to all the samples, three of them with their thickness previously determined by the SEM and another three determined by the proposed methodology.

A variance analysis or ANOVA has been used to verify the adjustment quality for all the samples. The result is presented in Table 5.

TABLE 5: Adjustment of variance analysis of the points presented in Figure 10.

Source of variation	Quadratic sum	Degrees of liberty	Quadratic average	R^2
Regression	7202.264	4	7202.264	1
Residues	0.01599	6	0.004	
Total	7202.27999	11		

Figure 10 demonstrates a linear relationship vanadium thickness (nm) determined by PXRF and the mass ($\mu\text{g}/\text{cm}^2$) for all the samples. The variance analysis presented in Table 5 also shows the linearity of the model, which is strongly evidenced by very low residues amounts and R^2 equals 1.

Even though there is a relationship between the intensity attenuated by the film thickness and a theoretical exponential relationship, one can perform a linear approximation for the region to be examined as it has an extremely thin thickness range.

4. Conclusions

In this scientific work, thickness measurement of nanometric thin films made by a Portable X-Ray Fluorescence technique based on the attenuation of the radiation field is proposed. Although the methodology is simple, the use of calibration curves for unknown samples led to consistent results within the expected range for the thickness. In fact, the main issue which can eventually make interpretation of data difficult is the correct determination of the calibration curve using reliable standards. Therefore, this empirical method is fast and accurate for measuring thicknesses of nanometric films. In addition, it provides a simple and convenient method for thickness monitoring.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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