The Effect of Annealing Temperature on Statistical Properties of WO₃ Surface

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We have studied the effect of annealing temperature on the statistical properties of WO₃ surface using atomic force microscopy techniques (AFM). We have applied both level crossing and structure function methods. Level crossing analysis indicates an optimum annealing temperature of around 400°C at which the effective area of the WO₃ thin film is maximum, whereas composition of the surface remains stoichiometric. The complexity of the height fluctuation of surfaces was characterized by roughness, roughness exponent and lateral size of surface features. We have found that there is a phase transition at around 400°C from one set to two sets of roughness parameters. This happens due to microstructural changes from amorphous to crystalline structure in the samples that has been already found experimentally.

In practice, one of the effective ways to modify the surface morphology is annealing process at various temperatures. So far, most of morphological analysis related to the WO₃ surface were accessible through the experimental methods. Usually these analysis are rigorous and time consuming. Moreover, lack of the suitable analysis for AFM data to find the nano and microstructural properties of surfaces was feeling perfectly.

In this article, we introduce the methods: roughness analysis and level crossing as suitable candidates and show that we can get easily the structure and morphological properties of a surface in a fast manner, only using the AFM observation as an initial data.

The roughness of a surface has been studied as a simple growth model using analytical and numerical methods [18, 19, 20, 21, 22, 23, 24, 25, 26]. These studies quite generally proposed that the height fluctuations have a self-similar character and their average correlations exhibit a dynamic scaling form. Also some authors recently use the average frequency of positive slope level crossing to provide further complete analysis on roughness of a surface [27]. This stochastic approach has turned out to be a promising tool also for other systems with scale dependent complexity, such as in surface growth where one would like to measure the roughness [28]. Some authors have applied this method to study the fluctuations of velocity fields in Burgers turbulence [29] and the Kardar-Parisi-Zhang equation in (d+1)-dimensions [30] and analyzing the stock market [31].

In this work, we have used the scaling analysis to determine the roughness, roughness exponent and the lateral size of surface features. Moreover, level crossing analysis has been utilized to estimate the effective area of a surface.

This paper is organized as follows: In section II, we have discussed about the film preparation and experi-
mental results obtained from AFM, XPS and UV-visible spectrophotometer for the annealed samples at the various temperatures. In section III, we have introduced the analytical methods briefly. Data description and data analysis based on the statistical parameters of WO$_3$ surface as a function of annealing temperatures are given in section IV. Finally, section V concludes presented results.

II. EXPERIMENTALS

Thin films of WO$_3$ were deposited on microscope slide glass using thermal evaporation method. The deposition system was evacuated to a base pressure of $\sim 4 \times 10^{-3}$ Pa. Thickness of the deposited films was considered about 200 nm measured by the stylus and optical techniques. More details about the other deposition parameters of the films are recently reported elsewhere [32].

To study the effect of annealing temperature on surface structure and optical properties of the samples, they were annealed at 200, 300, 350, 400, 450, and 500°C in air for a period of 60 min. Optical transmission and reflection measurements of the deposited films were performed in a range of 300-1100 nm wavelength using a Jasco V530 ultraviolet (UV)-visible spectrophotometer with resolution of 1 nm.

X-ray photoelectron spectroscopy (XPS) using a Specs EA 10 Plus concentric hemispherical analyzer (CHA) with Al $K\alpha$ anode at energy of 1486.6 eV was employed to study the atomic composition and chemical state of the tungsten oxide thin films. The pressure in the ultra high vacuum surface analysis chamber was less than 1.0 $\times$ 10$^{-7}$ Pa. All binding energy values were determined by calibration and fixing the C(1s) line to 285.0 eV. The XPS data analysis and deconvolution were performed by SDP (version 4.0) software. The nanoscale Surface topography of the deposited films was investigated by Thermo Microscope Autoprobe CP-Research atomic force microscopy (AFM) in air with a silicon tip of 10 nm radius in contact method. The AFM images were recorded with resolution of about 20 nm in a scale of $5 \times 5 \mu m$.

A. XPS Characterization

The elemental and chemical characterizations of the films were performed by XPS. Figure 1a shows the W(4f) core level spectra recorded on the "as deposited" WO$_3$ sample, and the results of its fitting analysis. To reproduce the experimental data, one doublet function was used for the W(4f) component. This contains W(4f$_{5/2}$) at 35.6 eV and W(4f$_{7/2}$) at 37.8 eV with a full-width at half-maximum (FWHM) of 1.75 $\pm$ 0.04 eV. The area ratio of these two peaks is 0.75 which is supported by the spin-orbit splitting theory of 4f levels. Moreover, the structure was shifted by 5 eV toward higher energy relative to the metal state. It is thus clear that the main peaks in our XPS spectrum attributed to the W$^{6+}$ state on the surface [1, 2, 33]. In stoichiometric WO$_3$, the six valence electrons of the tungsten atom are transferred into the oxygen p-like bands, which are thus completely filled. In this case, the tungsten 5d valence electrons have no part of their wave function near the tungsten atom and the remaining electrons in the tungsten atom experience a stronger Coulomb interaction with the nucleus than in the case of tungsten atom in a metal, in which the screening of the nucleus has a component due to the 5d valence electrons. Therefore, the binding energy of the W(4f) level is larger in WO$_3$ than in metallic tungsten. If an oxygen vacancy exists, the electronic density near its adjacent W atom increases, the screening of its nucleus is higher and, thus, the 4f level energy is expected to be at lower binding energy [1].

By increasing the annealing temperature it was observed that the position of W(4f) peak did not obviously change. But for WO$_3$ thin film annealed at 500°C (Fig. 1b), the W(4f) peak moved to a lower binding energy so that W(4f$_{7/2}$) position was observed at 35.0 eV. This can be related to oxygen vacancy at this high annealing temperature and formation of W$^{5+}$. 

![FIG. 1: W(4f) core level spectra of WO$_3$ thin films: a) "as deposited" and b) annealed at 500°C.](image-url)
B. Optical Characterization

The transmittance and reflectance spectra in the visible and infrared range recorded for the WO$_3$ thin films before and after annealing at different temperatures (Fig. 2a). It is seen that, the transmittance of the "as deposited" films in the visible range varies from about 80 up to nearly 100% (without considering the substrate contribution). Correspondingly, maximum value of the reflectance for both the film and the substrate is about 20% (the reflectance from the bare glass substrate was measured about 10%). The sharp reduction in the transmittance spectrum at the wavelength of $\sim 350\,\text{nm}$ is due to the fundamental absorption edge that was also reported previously [1, 2, 3].

The oscillations in the transmission and reflection spectra are caused by optical interference. The optical transmittance of WO$_3$ films strongly depends on the oxygen content of the films. In fact, non-stoichiometric films with composition of WO$_{3-x}$ show a blue tinge for $x > 0.03$ [34].

The "as deposited" pure tungsten oxide films were highly transparent with no observable blue coloration, under our experimental conditions. As can be seen from Fig. 2a after annealing process at 200 to 400°C, the transmittance and reflectance of the WO$_3$ films have not changed significantly. Only, the position of the oscillations altered due to thickness reduction and film condensation after the heat-treatment process [1]. At 500°C transmitance and reflectance of the annealed WO$_3$ film is reduced about 10%, therefore at this temperature, the film turn into non-stoichiometric composition, so that it could be seen from changing color of the film.

The optical gap ($E_g$) was evaluated from the absorption coefficient ($\alpha$) using the standard relation: $(\alpha h\nu)^{1/\eta} = A(h\nu - E_g)$, in which $\eta$ depends on the kind of optical transition in semiconductors, and $\alpha$ was determined near the absorption edge using the simple relation: $\alpha = ln[(1 - R)^2/T]/d$, where $d$ is thickness of the film. More useful explanation about the optical band gap calculation reported in [36]. The relationship between the optical band gap energy and annealing temperature for WO$_3$ thin films has been shown in Fig. 2b. As can be seen from it, the optical band gap for the "as deposited" WO$_3$ evaluated 3.4 eV. Amorphous structure of the "as deposited" WO$_3$ causes to $E_g$ is bigger than 2.7 eV. After annealing samples at 200 and 300°C, the optical band gap decreased slightly about 0.1 eV which can be related to condensation of the films. But the optical band gap of the WO$_3$ annealed at 400°C reduced to 3.1 eV due to crystallization of the film. This reduction continues to 2.5 eV for the sample annealed at 500°C. Reason of the $E_g$ becomes smaller than 2.7 eV is oxygen vacancy at this temperature as was seen in Fig 1b. It is to note that for evaporated WO$_3$ films one has found $2.7 < E_g < 3.5$ eV [1].

![Band Gap Energy vs Temperature](image)

**FIG. 2:** a) Optical transmittance (T) and reflectance (R) and b) Optical band gap energy of the WO$_3$ thin films annealed at different temperatures.

C. AFM Analysis

To study the effect of the annealing process on the surface morphology of the films, we have shown AFM images of the WO$_3$ surfaces annealed at the different temperatures: 200, 300, 350, 400, 450, 500°C in Figure 1. As can be seen from Fig. 1, for the annealed film at 200°C, it seems that the surface morphology of the film is relatively the same with a smooth surface, amorphous structure and nanometric grain size, as also reported by other investigators for WO$_3$ films [33, 36]. We have also ob-
served similar image for the "as deposited" WO₃ which is not shown here. For WO₃ thin films, increasing annealing temperature to 350°C did not significantly effect on surface parameters because it is low temperature for crystallization of WO₃ [1]. But at higher annealing temperatures 400, 450 and 500°C, surface grain size and

FIG. 3: AFM images of WO₃ thin films annealed at various temperatures a) 200, b) 300, c) 350, d) 400, e) 450 and f) 500°C, respectively.
roughness begin to increase. The more precise analysis of these surfaces are given in the next section.

III. STATISTICAL QUANTITIES

A. Roughness Analysis

It is also known that to derive the quantitative information of the surface morphology one may consider a sample of size $L$ and define the mean height of growing film $h$ and its roughness $\sigma$ by:

$$\sigma(L, t) = \langle (h - \bar{h})^2 \rangle^{1/2}$$  (1)

where $t$ is growing time and $\langle \cdots \rangle$ denotes an averaging over different samples, respectively. Moreover, growing time is a factor which can be applied to control the surface roughness of thin films.

Let us now calculate the roughness exponent of the growing surface. Starting from a flat interface (one of the possible initial conditions), it is conjectured that a scaling of length by factor $b$ and of time by factor $b^z$ ($z$ is the dynamical scaling exponent), rescales the roughness $\sigma$ by factor $b^\alpha$ as follows [18]:

$$\sigma(bL, b^z t) = b^\alpha \sigma(L, t)$$  (2)

which implies that

$$\sigma(L, t) = L^\alpha f(t/L^z).$$  (3)

For large $t$ and fixed $L$ (i.e., $x = t/L^z \to \infty$) $\sigma$ saturate. However, for fixed and large $L$ and $t \ll L^z$, one expects that correlations of the height fluctuations are set up only within a distance $t^{1/z}$ and thus must be independent of $L$. This implies that for $x \ll 1$, $f(x) \sim x^\beta g'(\lambda)$ with $\beta = \alpha/z$. Thus, dynamic scaling postulates that

$$\sigma(L, t) = \left\{ \begin{array}{ll}
        t^\beta, & t \ll L^z; \\
        L^\alpha, & t \gg L^z.
        \end{array} \right.$$  (4)

The roughness exponent $\alpha$ and the dynamic exponent $\beta$ characterize the self-affine geometry of the surface and its dynamics, respectively. In the present work, we see the surfaces at the limit $t \to \infty$ and so we will only obtain the $\alpha$ exponent.

The common procedure to measure the roughness exponent of a rough surface is use of the surface structure function depending on the length scale $l$ which is defined by:

$$S^2(l) = \langle (h(x + l) - h(x))^2 \rangle.$$  (5)

It is equivalent to the statistics of height-height correlation function $C(l)$ for stationary surfaces, i.e. $S^2(l) = 2\Delta_0^2(1 - C(l))$. The second order structure function $S^2(l)$, scales with $l$ as $l^{2\alpha}$.

B. Level Crossing Analysis

Let $\nu_\alpha^+$ denotes the number of positive slope crossing of $h(x) - \bar{h} = \alpha$ for interval $L$.

Since the process is homogeneous, if we take a second time interval of $L$ immediately following the first we shall obtain the same result, and for two intervals together we shall therefore obtain [28]:

$$N_\alpha^+(2L) = 2N_\alpha^+(L),$$  (6)

from which it follows that, for a homogeneous process, the average number of crossing is proportional to the interval $L$. Hence

$$N_\alpha^+(L) \propto L,$$  (7)

or

$$N_\alpha^+(L) = \nu_\alpha^+ L,$$  (8)

where $\nu_\alpha^+$ is the average frequency of positive slope crossing of the level $h(x) - \bar{h} = \alpha$. We now consider how the frequency parameter $\nu_\alpha^+$ can be deduced from the underlying probability distributions for $h(x) - \bar{h}$.

Consider a small length scale $\delta x$ of a typical sample function. Since we are assuming that the process $h(x) - \bar{h}$ is a smooth function of $x$, with no sudden ups and downs, if $\delta x$ is small enough, the sample can only cross $h(x) - \bar{h} = \alpha$ with positive slope if $h(x) - \bar{h} < \alpha$ at the beginning of the interval $L$. Furthermore, there is a minimum slope at $x$ if the level $h(x) - \bar{h} = \alpha$ is to be crossed in interval $\Delta x$ depending on the value of $h(x) - \bar{h}$ at position $x$. So there will be a positive crossing of $h(x) - \bar{h} = \alpha$ in the next interval $\Delta x$ if, at $x$,

$$h(x) - \bar{h} < \alpha \quad \text{and} \quad \frac{\Delta [h(x) - \bar{h}]}{\Delta x} > \frac{\alpha - [h(x) - \bar{h}]}{\Delta x}. $$  (9)

As shown in [28], the frequency $\nu_\alpha^+$ can be written in terms of joint PDF (probability distribution function ) of $p(\alpha, y')$ as follows:

$$\nu_\alpha^+ = \int_0^\infty p(\alpha, y') y' \, dy'.$$  (10)

and then the quantity $N_{\text{tot}}^+$ which is defined as:

$$N_{\text{tot}}^+ = \int_{-\infty}^{+\infty} \nu_\alpha^+ |\alpha - \bar{\alpha}| \, d\alpha.$$  (11)

will measure the total number of crossing the surface with positive slope. So, the $N_{\text{tot}}^+$ and square area of growing surface are in the same order. Concerning this, it can be utilized as another quantity to study further the roughness of a surface [27].
IV. RESULTS AND DISCUSSION

Thin films of WO$_3$ were deposited by using thermal evaporation method and then surface micrographs of WO$_3$ samples were obtained by AFM technique after annealed at different temperatures (Fig.3). These micrographs were then analyzed using methods from stochastic data analysis have introduced in the last section. Figure 3 shows AFM images of WO$_3$ thin films annealed at 200, 300, 350, 400, 450, and 500°C. The "as deposited" and annealed sample at 200°C (Fig. 3a) have columnar structure, indicating that up to 200°C no significant changes in the microstructure occurs. However, at higher temperatures (figs. 3b-3f) we have observed increased grain size and rougher surface. Specifically at 500°C (Fig. 3f) we observe stark changes in the micrograph which is accompanied by composition changes in the surface. This can be related to the phase transition to Magneli phase e.g. WO$_{3-x}$ in the annealing process [36]. This is also confirmed by our XPS and UV-visible spectrophotometry analysis (Sec. II). These are shown the significant formation of W$^{5+}$ state in the surface at 500°C.

Also our analysis shows that below 400°C the surfaces are in amorphous phase with the same behavior for all scales, but as soon as the crystalline phase appears the system behaves differently which diagnostics at small and large scale for temperatures above 400°C. By using parameters of the analytical method given here, these transitions can be quantified.

Now, we will use the statistical parameters introduced in the last section and will obtain some quantitative information about the effect of annealing temperature on the surface topography of the WO$_3$ samples.

The structure function $S^2(l)$ as defined in Eq.(5) can be used to quantify the topology of a rough surface. The structure function $S^2(l)$ is plotted against the length scale of the sample in Fig.4. The saturated $S^2(l)$ is an indication of the surface roughness, as $2\sigma^2$. The most obvious observation indicates that roughness is raised with increasing annealing temperature. Roughness has a minimum of 0.91nm at 27 and 200°C and a maximum of 48nm at 500°C. This is because higher temperatures create higher peaks (i.e. peaks with more deviations from the average). All exponents which is derivable from $S^2(l)$ have been summarized and given in Table I.

As depicted in Fig.4, the structure function $S^2(l)$ has a different behavior in the various temperatures. So that, in the annealing temperature range 27-350°C it has a typical behavior in all scales, but in the higher temperature range 400-500°C its behavior is different in the small and large scales. In the other words, the phase transition is occurred at 400°C, because for higher temperatures, there are two sets of roughness parameters needed to simulate the surface morphology. It can be related to the phase transition in the structure of the surface from amorphous to crystalline phase has been yielded from the band gap energy (see the section II.B).

The slope of each $S^2(l)$ curves at the small and large scales yields the roughness exponents $\alpha$ and $\alpha'$ of
FIG. 6: The normalized $N_{\text{tot}}^+ (27^\circ C)$ behavior as a function of annealing temperature. The solid line is plotted according to Eq.(12) around $400^\circ C$.

The corresponding surface. Hence, it is seen that the mono roughness exponent increases with the addition of annealing temperature up to $400^\circ C$. In the higher temperatures, we have obtained two roughness exponents ($\alpha-\alpha'$) equal to the 0.40-0.14, 0.71-0.20, and 0.69-0.24 for temperatures 400, 450 and 500$^\circ C$, respectively. Difference in the $\alpha$ values, in these temperatures, are in agreement with changes of correlation length. Where the correlation length, is the distance at which the structure function behaves differently.

The range of the scaling upon correlation length listed in the forth column in table I. The value of $C_0^+$ denotes the correlation length at small scales and $C_\infty^+$ for large scales. The higher $C_\infty^+$ value represents a smoother surface (as we expected from Fig.3). The correlation length obtained from the structure function is also a measure of minimum lateral size of surface features at each annealing temperature.

The another important $WO_3$ film parameter is the effective area of the sample which has an important role in the gas sensitivity of $WO_3$ surfaces. To obtain a measure for this, we utilize the level crossing analysis. As shown in Fig.5, the average frequency $\nu_{\alpha}^+$ as a function of height $h$, is plotted for the various annealing temperatures. The broad curves indicate the higher magnitude of height fluctuations around the average, and sharp curves show that the most of fluctuations are around the height average. This conclusion is in the correspondence with the results obtained from Fig.3.

According to the Eq.(11) $N_{\text{tot}}^+$ i.e. The total number of the crossing surface with positive slope is proportional to the square of area of the growing surface. To obtain the optimum value of the effective area, we have calculated the ratio of effective areas with respect to the area of the "as deposited" surface ($27^\circ C$). The values are given in the last column in Table I. It means, although the roughness increases by the annealing temperature but the effective square area of the rough surface has a maximum value of $N_{\text{tot}}^+ [27]$.

For more clarity, we have calculated the temperature dependence of normalized $N_{\text{tot}}^+$ numerically (Fig.6) around $400^\circ C$, and we have obtained the three following functions for this quantity:

$$N_{\text{tot}}^+(T) = (5.0718 - 0.0223 \times T + 2.72 \times 10^{-5} \times T^2)^{-4}$$

$$\ln(N_{\text{tot}}^+(T)) = -6.3632 + 0.0344 \times T - 4.20 \times 10^{-5} \times T^3$$

$$N_{\text{tot}}^+(T) = -8.7057 + 0.0520 \times T - 6.37 \times 10^{-5} \times T^3$$

According to this figure, the maximum value of the effective area is at $400^\circ C$ (with respect to its value at $27^\circ C$) with the relative value equal to 2.00. Thus, applying this analysis easily shows that if one follows the condition which the effective area as an important parameter in the gas sensitivity of $WO_3$ surfaces is optimum and furthermore, the film composition has not been changed (e.g. The Magneli phase transition has not been occurred), should choose the annealed surface at $400^\circ C$ for better performance.

V. CONCLUSIONS

We have investigated the role of annealing temperature, as an external parameter, to control the statistical properties of a rough $WO_3$ surface. The AFM microstructure of the surfaces is just needed to apply in our analysis. We have computed the statistical quantities such as roughness exponent, roughness and lateral size of surface features of the "as deposited" and annealed...
surfaces at 200, 300, 350, 400, 450, and 500°C, using the structure function. We have seen a phase transition at 400°C, because for higher temperatures there are two sets of roughness parameters, due to structural changes from amorphous to the crystalline phase. Moreover, using the level crossing analysis we have obtained an optimum annealing temperature, 400°C in which the surface of the WO₃ has maximum value about twice relative to the “as deposited” film without any changes in the film composition that may increase surface reaction of the WO₃ film as the gas sensor or photo-catalyst.

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