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Height and Feature Parameters Study of thermally evaporated ZnS thin films By AFM

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ABSTRACT

AFM has been used to get microscopic information of the surface structure and to plot topographies representing the surface relief. AFM technique can be used to visualize the surface relief, specify the growth of thin films, and determine Height parameters. In this work we have studied height and feature parameters for Zinc Sulphide thin films such as the root mean square height RMS, Surface Skewness, Surface Kurtosis, Arithmetical mean height, Density of peaks and Arithmetic mean peak curvature. It can be seen on optical micrographs the fine grains with different sizes which are distributed over a smooth homogeneous background that may correspond to the amorphous, or polycrystalline phase of ZnS films. Some of the grains are seen to be united/fused- forming agglomerates. The surface roughness parameters were determined by using the software of ISO 25178 standard provided with the microscope.

Keywords: Roughness (R), Waviness (W), Root Mean Square height (RMS), Surface Skewness (S_{sk}), Surface Kurtosis (S_{ku}), Arithmetical mean height (S_a), Density of peaks and Arithmetic mean peak curvature**,**

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دراسة برامترات الخشونة لأغشية ZnS الموضَّعة حرارياً على ركائز من الزجاج بمجهر القوة الذرية

سهام الطرابيشي(1)

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الملخص

يسمح مجهر القوة الذرية بالحصول على معلومات مجهرية عن بنية سطوح الأغشية وبرسم صور لطبوغرافيتها تمثل تضاريس السطح، ومراقبة نمو الأغشية الرقيقة وتحديد برامترات الخشونة. قمنا في هذا العمل بدراسة برامترات الخشونة للسطح ومعالمه. من البرامترات المدروسة جذر متوسط مربعات الارتفاعات **RMS** (S_q) والعزم الإحصائي ذو المرتبة الثالثة **Skewness** (S_{sk}) الذي يصف انحراف توزع ارتفاعات السطح عن التناظر، والعزم الإحصائي ذو المرتبة الرابعة **Kurtosis** (S_{ku}) الذي يصف تفلطح توزع ارتفاعات السطح، والمتوسط الحسابي للارتفاعات (S_a)، وكثافة القمم (S_{pd}) في السطح والمتوسط الحسابي لانحناءات القمم (S_{pc}). وقد أمكن في الصور الضوئية لهذه الأغشية رصد حبيبات دقيقة مختلفة الأبعاد موزعة على خلفية متجانسة يرجح أن تكون لابلورية أو متعددة التبلور. وقد لوحظت في الصور تجمعات لبعض الحبيبات. وأمَّا برامترات الخشونة فقد حُدّدت باستخدام البرمجيات المرافقة لمجهر القوة الذرية.

الكلمات المفتاحية: الخشونة، التموج، جذر متوسط مربع الارتفاعات، العزم الإحصائي الثالث (الانحراف عن التناظر)، العزم الإحصائي الرابع (وصف التفلطح)، المتوسط الحسابي للارتفاعات، كثافة القمم، المتوسط الحسابي لانحناءات القمم.

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I. Introduction

Zinc sulphide (ZnS) is an important (II-VI) semiconducting material with a wide direct band gap of 3.65eV in the bulk[1], a high refractive index (2.35) and high dielectric constant[2]. ZnS thin films with a wide direct band gap and n- type conductivity are promising candidates for optoelectronic device applications. These properties of zinc sulphide thin films are promising for short wavelength optoelectronic device applications. It has potential applications in optoelectronic devices such as blue light emitting diodes[3]. ZnS can also be used for light emitting diodes in the blue ultraviolet region thanks to its wide band gap. ZnS is also widely used as the base materials for cathode-ray tube luminescent materials, catalysts, electroluminescent devices, and UV semiconductor lasers for optical lithography [4,5]. ZnS crystals reveal high photoluminescence and thermo –luminescence properties above room temperature. The nanostructures made up of ZnS materials find attractive applications in electronic and optoelectronic nano-devices [5],electroluminescent devices and photovoltaic cells[6]. In thin film solar cells based on CuGaIn (S.Se)2 absorbers, a CdS buffer layer is generally required in order to obtain high conversion efficiency. However, there are toxic hazards with respect to the production and use of the CdS layer. Therefore research in developing Cd-free buffer layers has been encouraged. This has lead to the investigation of ZnS as a buffer layer in ZnO/ZnS/CuInS2 devices[7]. ZnS has a wider energy band gap than CdS, which results in the transmission of more high energy photons to the junction and to the enhancement of the blue response to the photovoltaic cells.

Several techniques such as thermal evaporation [8], molecular beam epitaxy [9], metal-organic vapor phase epitaxy [10], chemical vapor deposition[11], spray pyrolysis[12], and chemical bath deposition CBD[13] have been used to produce ZnS thin films for photovoltaic applications because of its efficient, cost effective and large scale capability[15].

II. Definition of Roughness and Feature parameters [16, 17, 18]

The Roughness of a surface is defined as the main component of the relief containing the smallest wavelengths measured on the sample. The roughness gives indication to the nature of the material and the machining type used. The wavelengths under the cut-off are kept in the roughness, the others, higher, are kept in the waviness.

The height properties, according to surf manual are described by six parameters, which give information about the statistical average properties, the shape of the height distribution histogram and about extreme properties. All the parameters are based on two-dimensional standards that are extended to three dimensions.

A. Height (Roughness) Parameters (ISO 25178) (Surface)

Parameters related to roughness are general and valid for any M × N rectangular image.

Some of the parameters depend on the definition of a local minimum and a local maximum. Here, a local minimum is defined as a pixel where all eight neighboring pixels are higher and a local maximum as a pixel where all eight neighboring pixels are lower.

Height parameters are a class of surface finish parameters that quantify the Z-axis perpendicular to the surface. The reference plane for the calculation of these parameters is the mean plane of the measured surface.

1. Root mean square height, S_q

Standard deviation of the height distribution, or RMS surface roughness. It is defined as:

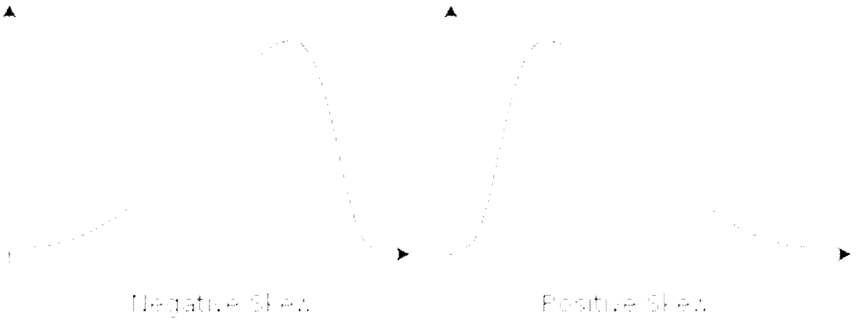
$$S_q = \sqrt{\frac{1}{MN}\sum_{k=0}^{M-1}\sum_{l=0}^{N-1}[z(x_k, y_l)]^2}$$

It computes the standard deviation for the amplitudes of the surface (RMS).

2.The Surface Skewness or skewness of the height distribution, S_{sk}, is defined as:

$$S_{sk} = \frac{1}{MN\ S_q^3}\sum_{k=0}^{M-1}\sum_{l=0}^{N-1}[z(x_k, y_l)]^3$$

which is the third statistical moment, qualifying the asymmetry of the height distribution histogram. A negative S_{sk} (Fig. 1a) indicates that the surface is composed of principally one plateau and deep and fine valleys. In this case, the distribution is sloping to the top. A positive S_{sk} (Fig. 1b) indicates a surface with lots of peaks on a plane. The distribution is sloping to the bottom. Due to the big exponent used, this parameter is very sensitive to the sampling and to the noise of the measurement.



a **b**

Fig. 1

If $S_{sk} = 0$, a symmetric height distribution is indicated, for example, by a Gaussian like curve. If $S_{sk} < 0$, it can be a bearing surface with holes and if $S_{sk} > 0$ it can be a flat surface with peaks. Values numerically greater than 1.0 may indicate extreme holes or peaks on the surface.

3.The Surface Kurtosis or Kurtosis of the height distribution, S_{ku}, is defined as:

$$S_{ku} = \frac{1}{MN\, S_q^4} \sum_{k=0}^{M-1} \sum_{l=0}^{N-1} [z(x_k, y_l)]^4$$

which is the fourth statistical moment, qualifying the flatness of the height distribution. Due to the big exponent used, this parameter is very sensitive to the sampling and to the noise of the measurement.

For Gaussian height distributions S_{ku} approaches 3.0 when increasing the number of pixels. Smaller values indicate broader height distributions and *vice versa* for values greater than 3.0.

A high kurtosis distribution has a sharper *peak* and longer, fatter *tails*, while a low kurtosis distribution has a more rounded peak and shorter thinner tails.

4. Maximum peak height, S_p, Height between the highest peak and the mean plane.

5. Maximum pit height, S_v, Depth between the mean plane and the deepest valley.

6. Maximum height, S_z, Height between the highest peak and the deepest valley.

7. Arithmetical mean height, S_a or Mean surface roughness is defined as

$$S_a = \frac{1}{MN}\sum_{k=0}^{M-1}\sum_{l=0}^{N-1}|\, z(x_k, y_l)\,|$$

B. Definition of Feature Parameters [16, 17, 18]

Feature parameters are derived from the segmentation of a surface into motifs (hills and dales). Segmentation is carried out in accordance with the watersheds algorithm. For the moment, all feature parameters are calculated after a discrimination by segmentation using a Wolf pruning of 5% of the value of the Sz parameter (Maximum height). The most important of these are:

1- Density of peaks, S_{pd}, Number of peaks per unit area.

$$S_{pd} = \frac{Number\ of\ local Maximums}{(M-1)(N-1)dxdy}$$

where δ*x* and δ*y* are the pixel separation distances.

2- Arithmetic mean peak curvature, S_{pc} Arithmetic mean of the principle curvatures of peaks within a definition area. It is defined as:

$$S_{pc} = \frac{-1}{2n}\sum_{i=1}^{n}\left(\frac{d^2 z(x,y)}{d\,x^2}\right)+\left(\frac{d^2 z(x,y)}{d\,y^2}\right)$$

for all local maximums, where δ*x* and δ*y* are the pixel separation distances. This parameter enables to know the mean form of the peaks: either pointed, or rounded, according to the mean value of the curvature of the surface at these points; the higher the value, the more rounded are the peaks.

III. Experiments and AFM Characterization

Thin films of ZnS were deposited on glass substrates by thermal evaporation **[19]**. Using the dynamic mode operation of AFM technique, we have got topographies representing the surface relief. The optical micrographs of 3 different optical thicknesses (300, 200, 50nm thickness respectively), whose sizes are of (500μm*370μm), show, high in-homogeneity of grain sizes. The thicker the sample, the larger the crystallite size is. Fig.2 shows an optical micrograph (500μm*370μm) of zinc sulphide thin film of 300nm thickness, grown on glass substrate. The crystallites are clearly shown, some of which are seen to be united/fused- forming agglomerates.

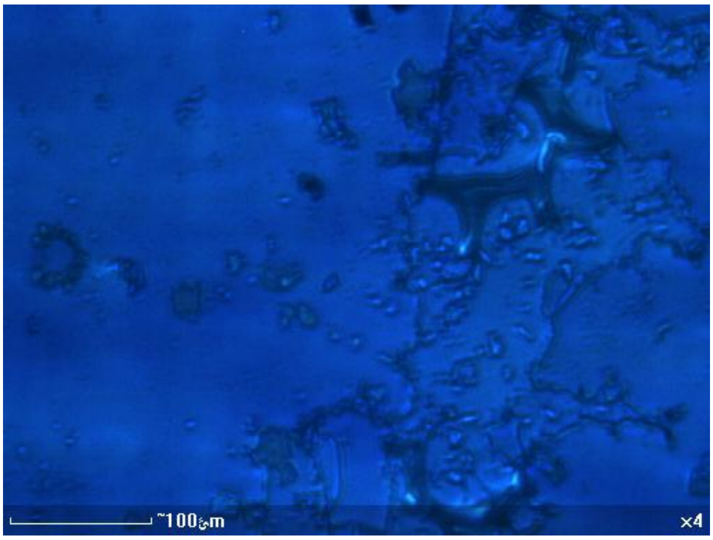


Fig2. An optical micrograph for (500µm*370µm) area of thermally evaporated ZnS film, of 300nm thickness, grown on glass substrate.

In Table I we presented topography and roughness micrographs of three different thicknesses (300, 200, 50nm) of zinc sulphide thin films, their height distribution, height and feature parameters

Topography photographs have been line and column corrected, form removed, denoised and smoothed. Components of the surface relief of long horizontal wavelengths (i.e. waviness) have been removed in order to analyze surface finish (i.e. roughness). As the Form in our samples is coming from a smooth material (thin films), 3nd order polynomial plane fit Form Removal operator has been used for slope correction. **Gaussian Filtering (**with 0.25 µm cut-off wavelength**) has been used to** separate data frequencies (or wavelengths) into two parts, the first one having the long wavelengths (waviness), the other one having the short wavelengths (roughness).

The wavelengths above the cut-off are kept in the waviness whereas the others, smaller, are kept in the roughness.

Table I. Height parameters characterization of 10µm*10µm ZnS micrograph size

<table>
<tr><th></th><th colspan="2">300nm P1, P2, P3, P4</th></tr>
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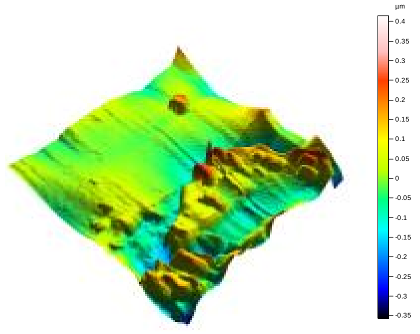
<td>
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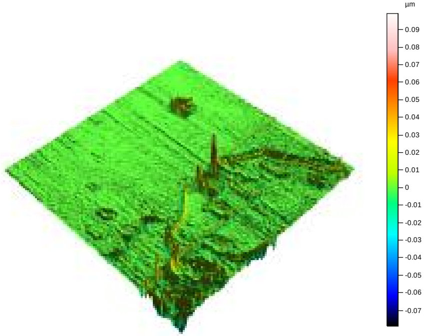
<td>
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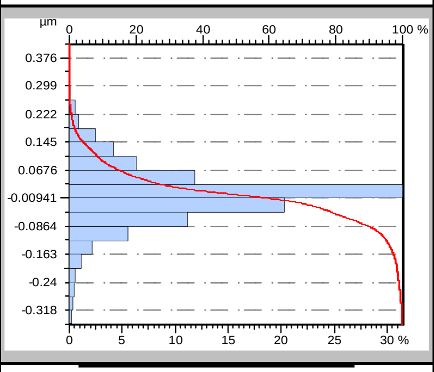
<td>
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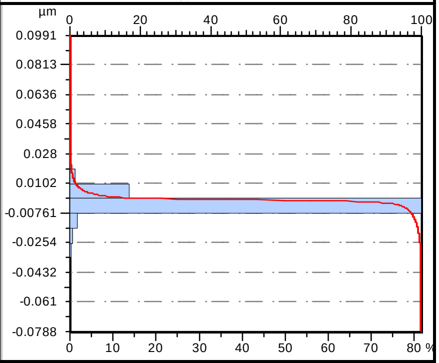
<td>
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<tr><td>Height and feature parameters</td>
<td><table>
<tr><th colspan="3">ISO 25178</th></tr>
<tr><th colspan="3">Height parameters</th></tr>
<tr><td>Sq</td><td>78.2</td><td>nm</td></tr>
<tr><td>Ssk</td><td>-0.162</td><td></td></tr>
<tr><td>Sku</td><td>5.33</td><td></td></tr>
<tr><td>Sp</td><td>415</td><td>nm</td></tr>
<tr><td>Sv</td><td>355</td><td>nm</td></tr>
<tr><td>Sz</td><td>770</td><td>nm</td></tr>
<tr><td>Sa</td><td>54.4</td><td>nm</td></tr>
<tr><th colspan="3">Feature parameters</th></tr>
<tr><td>Spd</td><td>134397</td><td>$1/µm^2$</td></tr>
<tr><td>Spc</td><td>4.29</td><td>1/µm</td></tr>
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<td><table>
<tr><th colspan="3">ISO 25178</th></tr>
<tr><th colspan="3">Height parameters</th></tr>
<tr><td>Sq</td><td>4.73</td><td>nm</td></tr>
<tr><td>Ssk</td><td>-0.624</td><td></td></tr>
<tr><td>Sku</td><td>60.1</td><td></td></tr>
<tr><td>Sp</td><td>99.1</td><td>nm</td></tr>
<tr><td>Sv</td><td>78.8</td><td>nm</td></tr>
<tr><td>Sz</td><td>178</td><td>nm</td></tr>
<tr><td>Sa</td><td>1.92</td><td>nm</td></tr>
<tr><th colspan="3">Feature parameters</th></tr>
<tr><td>Spd</td><td>473195</td><td>$1/µm^2$</td></tr>
<tr><td>Spc</td><td>21.5</td><td>1/µm</td></tr>
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<tr><td></td><td colspan="2">300nm P2</td></tr>
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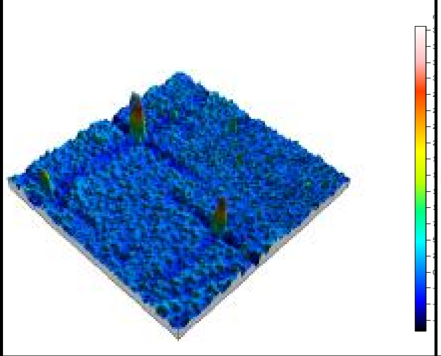
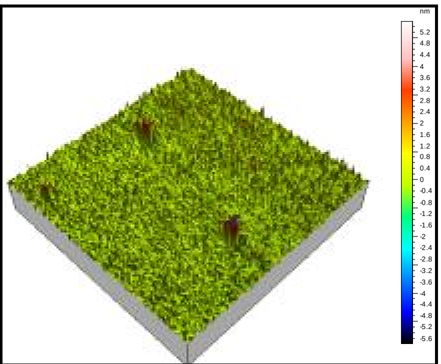
<td>3D photograph</td>
<td></td>
<td>
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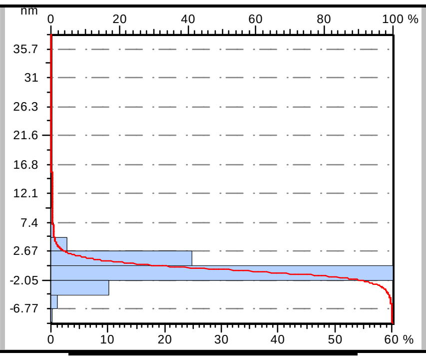
<td>
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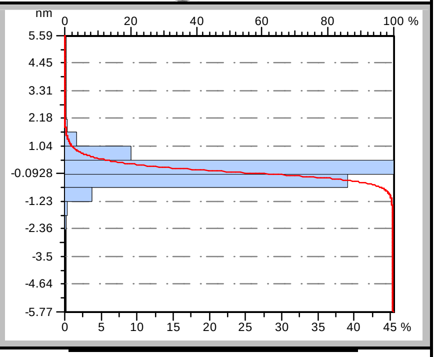
<td>
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<td>Height and feature parameters</td>
<td>
<table>
<tr><th colspan="3">ISO 25178</th></tr>
<tr><th colspan="3">Height parameters</th></tr>
<tr><td>Sq</td><td>2.35</td><td>nm</td></tr>
<tr><td>Ssk</td><td>6.17</td><td></td></tr>
<tr><td>Sku</td><td>79.6</td><td></td></tr>
<tr><td>Sp</td><td>38.4</td><td>nm</td></tr>
<tr><td>Sv</td><td>8.85</td><td>nm</td></tr>
<tr><td>Sz</td><td>47.2</td><td>nm</td></tr>
<tr><td>Sa</td><td>1.3</td><td>nm</td></tr>
<tr><th colspan="3">Feature parameters</th></tr>
<tr><td>Spd</td><td>1420765</td><td>$1/\mu m^2$</td></tr>
<tr><td>Spc</td><td>0.767</td><td>$1/\mu m$</td></tr>
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<tr><th colspan="3">Height parameters</th></tr>
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<tr><td>Ssk</td><td>0.491</td><td></td></tr>
<tr><td>Sku</td><td>17.7</td><td></td></tr>
<tr><td>Sp</td><td>5.59</td><td>nm</td></tr>
<tr><td>Sv</td><td>5.77</td><td>nm</td></tr>
<tr><td>Sz</td><td>11.4</td><td>nm</td></tr>
<tr><td>Sa</td><td>0.319</td><td>nm</td></tr>
<tr><th colspan="3">Feature parameters</th></tr>
<tr><td>Spd</td><td>6946904</td><td>$1/\mu m^2$</td></tr>
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<td colspan="2">300nm P3</td>
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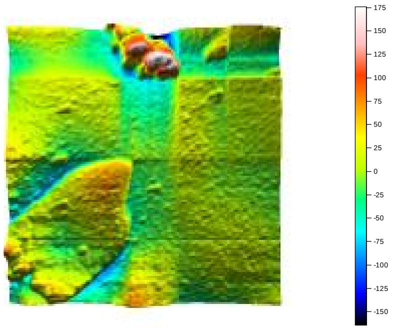
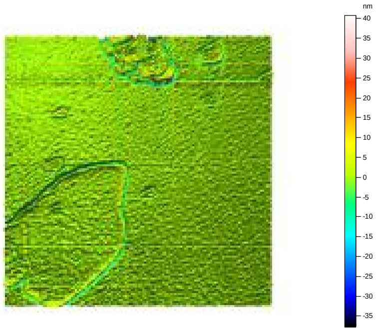
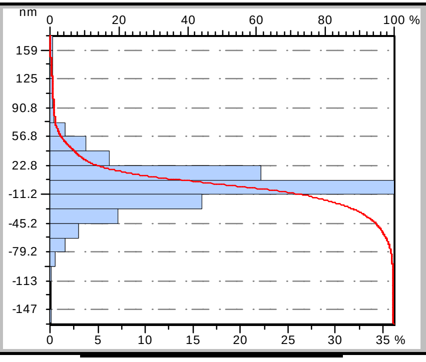
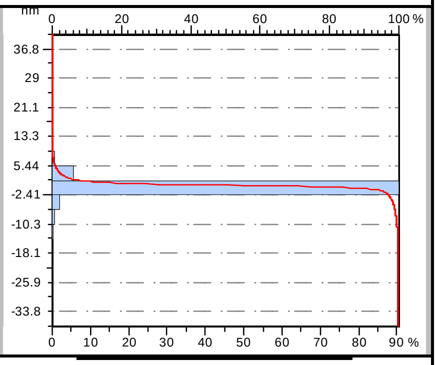
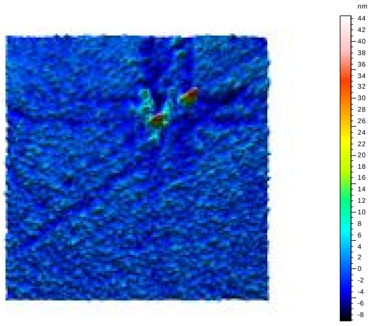
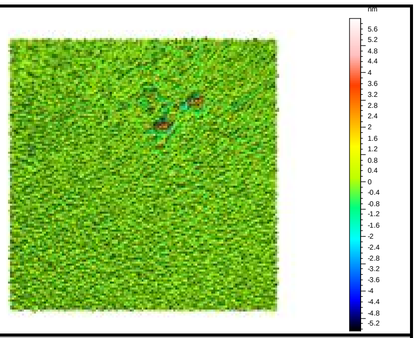
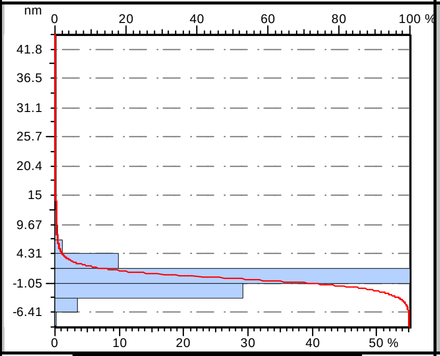
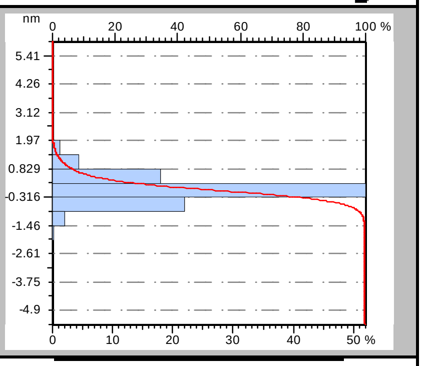
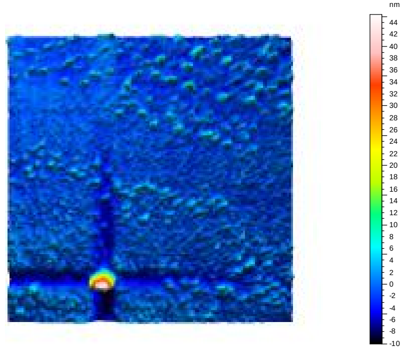
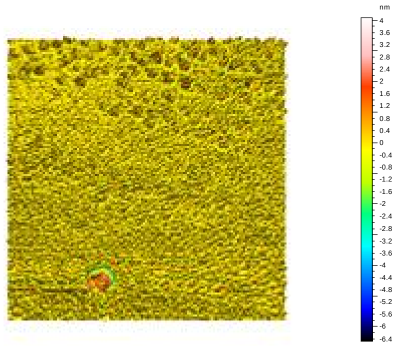
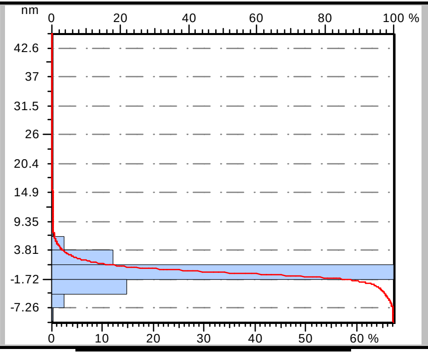
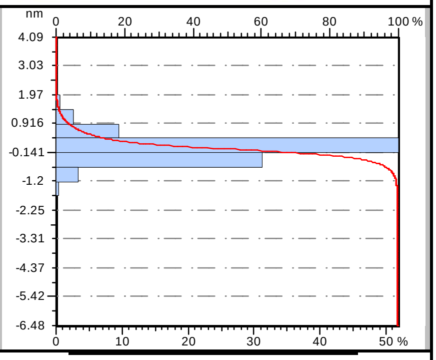
3D photograph		
Height distribution		
Height and feature parameters	**ISO 25178** **Height parameters** **Sq** 30.2 nm **Ssk** 0.735 **Sku** 8.94 **Sp** 176 nm **Sv** 164 nm **Sz** 340 nm **Sa** 20.1 nm **Feature parameters** **Spd** 76798 1/µm^2 **Spc** 0.87 1/µm	**ISO 25178** **Height parameters** **Sq** 1.86 nm **Ssk** -1.26 **Sku** 59.5 **Sp** 40.7 nm **Sv** 37.7 nm **Sz** 78.5 nm **Sa** 0.853 nm **Feature parameters** **Spd** 473195 1/µm^2 **Spc** 7.05 1/µm
	300nm P4	

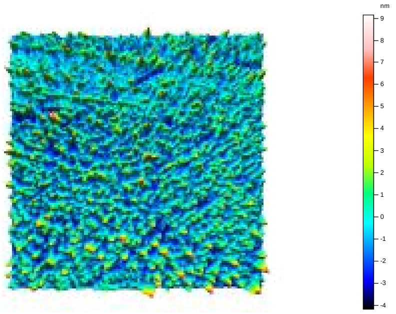
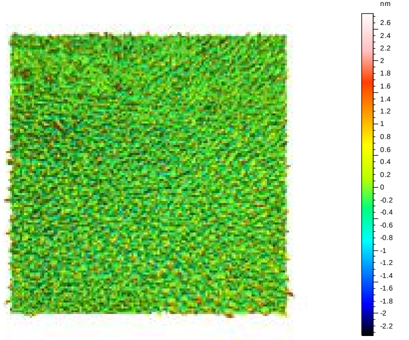
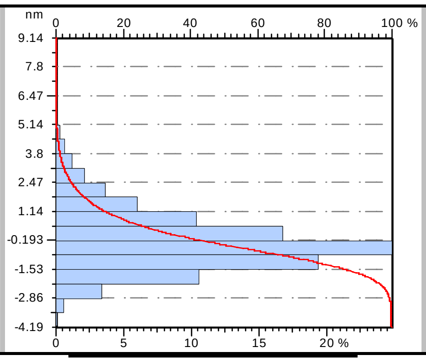
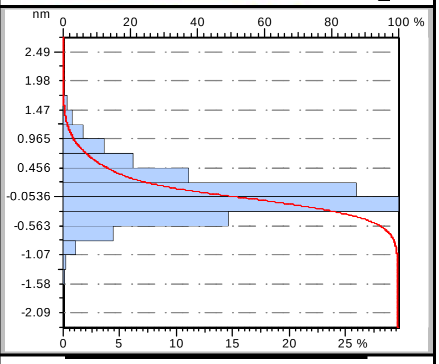
3D photograph		
Height distribution		

Height and feature parameters

	ISO 25178		ISO 25178	
Height parameters				
Sq	2.6	nm	0.517	nm
Ssk	5.9		0.717	
Sku	79		9.81	
Sp	44.7	nm	5.98	nm
Sv	8.85	nm	5.47	nm
Sz	53.6	nm	11.4	nm
Sa	1.5	nm	0.371	nm
Feature parameters				
Spd	1468764	1/µm^2	8718868	1/µm^2
Spc	0.889	1/µm	0.817	1/µm

200nm P1

3D photograph		
Height distribution		
Height and feature parameters	**ISO 25178** **Height parameters** **Sq** 2.78 nm **Ssk** 6.86 **Sku** 95.3 **Sp** 45.7 nm **Sv** 9.7 nm **Sz** 55.4 nm **Sa** 1.44 nm **Feature parameters** **Spd** 929419 1/µm^2 **Spc** 0.791 1/µm	**ISO 25178** **Height parameters** **Sq** 0.445 nm **Ssk** 0.169 **Sku** 14.4 **Sp** 4.09 nm **Sv** 6.48 nm **Sz** 10.6 nm **Sa** 0.298 nm **Feature parameters** **Spd** 5999232 1/µm^2 **Spc** 0.932 1/µm
	200nm P2	

3D photograph		
Height distribution		
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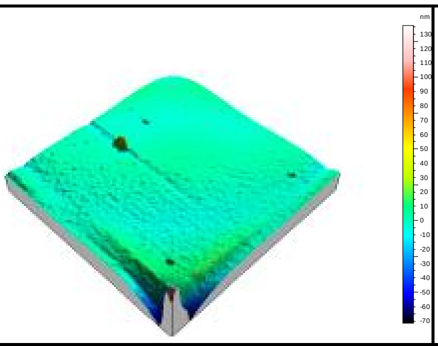
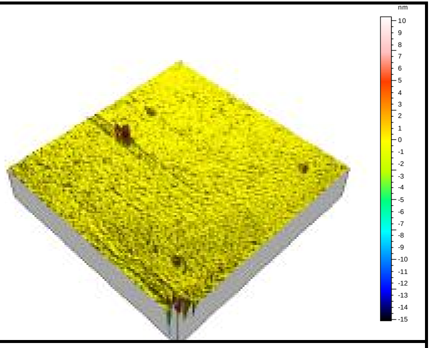
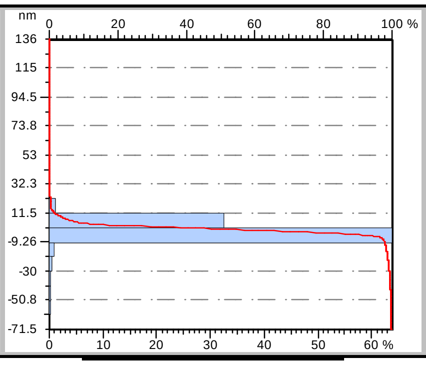
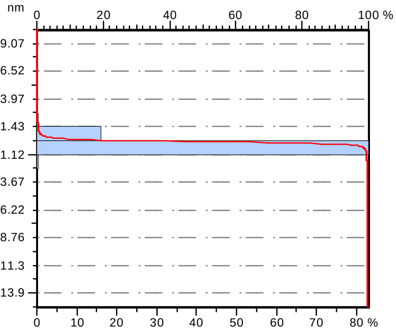
3D photograph		
Height distribution		
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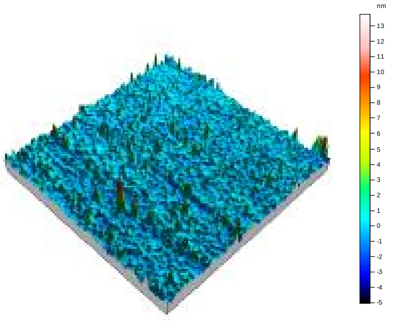
<td>
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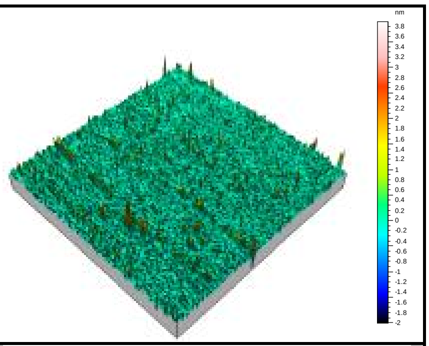
<td>
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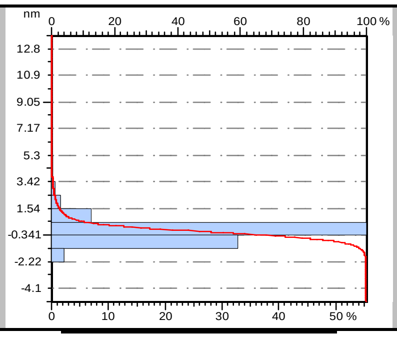
<td>
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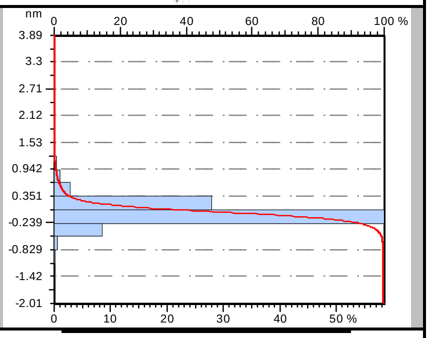
<td>
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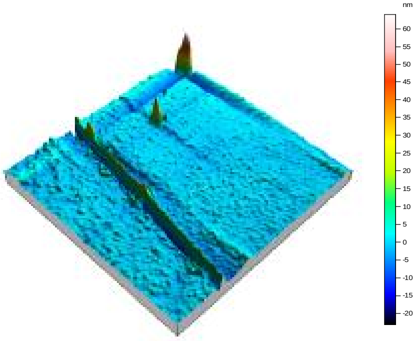
<td>
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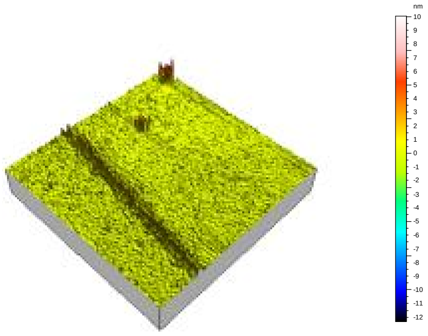
<td>
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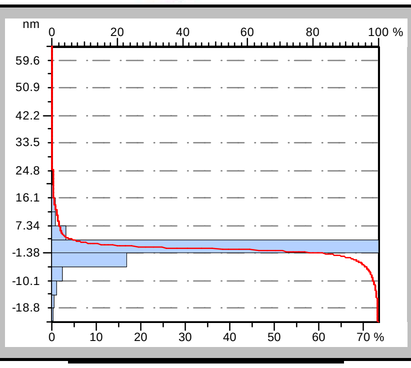
<td>
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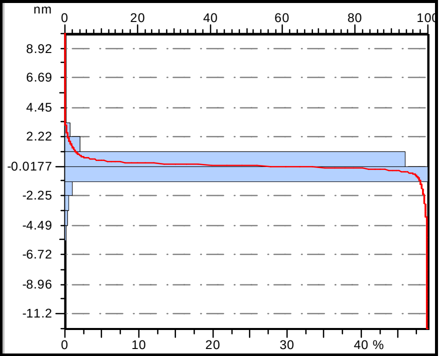
<td>
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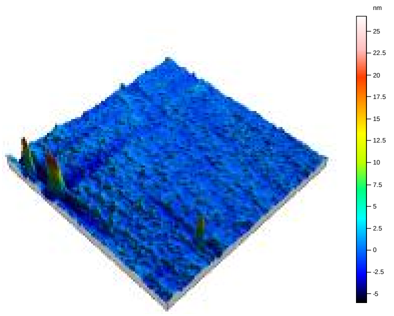
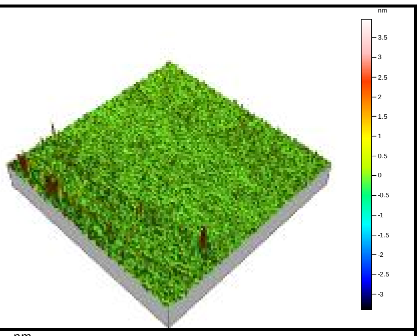
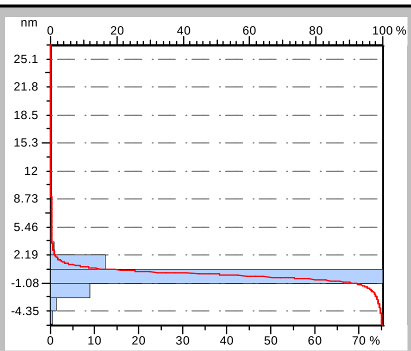
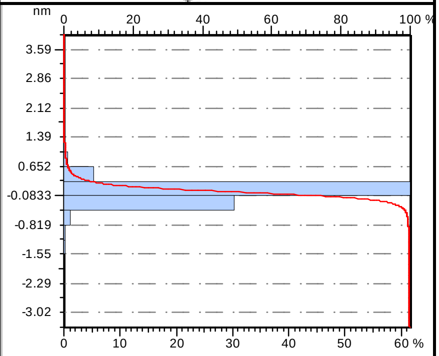
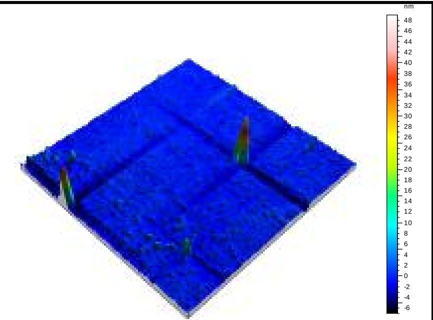
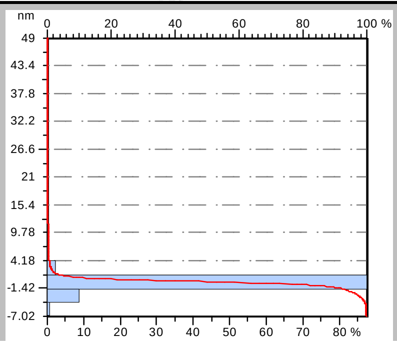
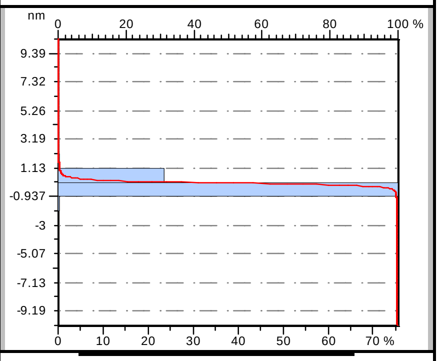
3D photograph		
Height distribution		
Height and feature parameters	**ISO 25178** **Height parameters** **Sq** 1.37 nm **Ssk** 6.25 **Sku** 90.7 **Sp** 26.8 nm **Sv** 5.84 nm **Sz** 32.7 nm **Sa** 0.694 nm **Feature parameters** **Spd** 402429 1/µm^2 **Spc** 0.519 1/µm	**ISO 25178** **Height parameters** **Sq** 0.247 nm **Ssk** 1.4 **Sku** 32.9 **Sp** 3.96 nm **Sv** 3.39 nm **Sz** 7.35 nm **Sa** 0.152 nm **Feature parameters** **Spd** 4130125 1/µm^2 **Spc** 0.509 1/µm
	50nm P3	

3D photograph		
Height distribution		
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IV. Results & Discussion

The study consisted of 4 points (P1, P2, P3 and P4) of 300nm thickness film, 4 points (P1, P2, P3 and P4) of 200 nm thickness film and 3 points (P1, P2 and P3) of 50nm thickness film.

Considering the most important parameters of the study, i.e : skewness and kurtosis of the height distribution, density of peaks and the arithmetic mean peak curvature, we find from table II, containing the values of these parameters, that :

(a) The skewness describing the asymmetry of height distribution of topography and roughness of a surface, varies from one film to another, even from one point to another on the same film, but the dominance of positive values in both topography and roughness (with very few exceptions) reveals the domination of peaks on both of them. The values greater than 1 in most of the points such as 2 and 4 of 300nm topography indicate extreme peaks on the surface as it is seen on the micrographs of table I and peak density values from table II.

Table II. Showing values of S_{sk}, S_{ku}, S_{pc} and S_{pd} for topography and roughness in different points of 300, 200, 50nm thickness of ZnS thin films respectively

		Topography				**Roughness**				
		P1	P2	P3	P4	P1	P2	P3	P4	
300nm	S_{sk}	-0.122	6.17	0.735	5.9	-0.624	0.491	-1.26	0.717	
	S_{ku}	4.9	79.6	8.94	79	60.1	17.7	59.5	9.81	
	S_{pc}	24.5	0.767	0.87	0.889	21.5	0.795	7.05	0.817	1/µm
	S_{pd}	95998	1420765	76798	1468764	473195	6946904	473195	8718868	1/µm^2
200nm	S_{sk}	6.86	1.07	4.01	2.83	0.169	0.845	-0.378	1.47	
	S_{ku}	95.3	5.32	107	30.4	14.4	4.9	188	14.8	
	S_{pc}	0.791	0.597	1.41	0.508	0.932	0.695	1.71	0.471	1/µm
	S_{pd}	929419	2941562	38327	958164	5999232	12259738	124561	5195315	1/µm^2
50nm	S_{sk}	4.15	6.25	12.5	-	-1.42	1.4	0.278	-	
	S_{ku}	55.4	90.7	227	-	47.4	32.9	222	-	
	S_{pc}	0.922	0.519	0.569	-	1.53	0.314	2.38	-	1/µm
	S_{pd}	124561	402429	76653		1075239	4130125	160783		1/µm^2

(b) The kurtosis describing the flatness of height distribution in topography and roughness of a surface varies as well from one point to another, but its values in all points examined are greater than 3 indicating sharper peaks and longer fatter tails in general, a fact confirmed by small values of arithmetic mean peak curvature (micrographs of table I and numerical values of table II).

(c) The negative skewness value of height distribution in some points, such as P1 of 300nm film (S_{sk} = -0.122 in topography surface, -0.624 in roughness), accompanied with the presence of peaks (S_{pd} = 95998 1/µm^2 in topography, 473195 1/µm^2 in roughness) indicates that surface is composed of peaks and holes on a plane.

(d)The dislocation density (Lines/nm^2) calculated, based on the density of peaks varies from one point to another (table III). It is situated between:

0.5 10^{-2}-2.2 for 300nm
0.15 10^{-2}- 8.65 for 200nm thickness
0.5 10^{-2}-0.16 for 50nm thickness

These values for dislocation density are different from X-Rays Diffraction results [19] showing that the dislocation density Is inversely proportional to the thickness.

Table III. Showing Dislocation density calculated for tested points

		P1	P2	P3	P4	
300nm	S_{pd}	95998	1420765	76798	1468764	$1/\mu m^2$
	δ	0.92 10^{-2}	2.0	0.5 10^{-2}	2.2	L/nm^2
200nm	S_{pd}	929419	2941562	38327	958164	$1/\mu m^2$
	δ	0.86	8.65	0.15 10^{-2}	0.9	L/nm^2
50nm	S_{pd}	124561	402429	76653	-	$1/\mu m^2$
	δ	1.5 10^{-2}	0.16	0.5 10^{-2}	-	L/nm^2

It results in that values of Height and Feature parameters of ZnS thin films should be controlled according to the application sought after, transmission, reflection or fluid retention.

شكر وتقدير

أتوجه بخالص شكري للزميل العزيز الدكتور حسان العاقل رئيس قسم الإحصاء في كلية العلوم بجامعة دمشق لإيضاحه مفهوم العزم الإحصائي ذي المرتبة الثالثة الذي يصف انحراف توزع ارتفاعات السطح عن التناظر ومفهوم العزم الإحصائي ذي المرتبة الرابعة الذي يصف تفلطح توزع ارتفاعات السطح.

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