

# **Study of Evolution of Surface Roughness of Cu Thin Films**

A thesis submitted  
in partial fulfilment of the requirement for the award of the degree in

**MASTER OF SCIENCE  
IN  
PHYSICS**



**THAPAR INSTITUTE**  
OF ENGINEERING & TECHNOLOGY  
(Deemed to be University)

*Submitted by:*

**Vaibhavi Pandey**  
Roll No: 301604041

*Under the guidance of:*

**Dr. Bhaskar Chandra Mohanty**

**Associate Professor**

*School of Physics and Materials Science*

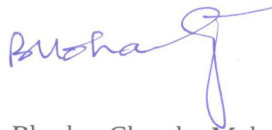
**Thapar Institute of Engineering and Technology, Patiala**

*Punjab-147004*

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## CERTIFICATE

This is to certify that the thesis titled “**Study of evolution of surface roughness of Cu thin films**” submitted by **Vaibhavi Pandey, Roll no. 301604041** in partial fulfilment of requirements for the award of the degree **M.Sc. in Physics** from the School of Physics and Materials Science, Thapar Institute of Engineering and Technology, Patiala is a record of candidate’s own work carried out by her under my supervision and guidance. The matter embodied in this report has not been submitted in part or full to any other institute for award of any degree.



Dr. Bhaskar Chandra Mohanty

Associate Professor

School of Physics and Materials Science

Thapar Institute of Engineering and Technology, Patiala

## DECLARATION

I hereby declare that the work been presented in this thesis report entitled “**Study of evolution of surface roughness of Cu thin films**” by me in partial fulfilment of the requirements for the award of degree of **Masters of Science in Physics**, Thapar Institute of Engineering and Technology, Patiala is an authentic award record on my work carried out under the supervision of **Dr. Bhaskar Chandra Mohanty**, Associate Professor, School of Physics and Materials Science, Thapar Institute of Engineering and Technology, Patiala. The matter presented in this report has not been submitted in any other university/institute for award of master and science or any other degree.

Vaibhavi

Vaibhavi Pandey

Roll no: 301604041

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## CONTENTS

<b>Chapter</b>	<b>Title</b>	<b>Page no.</b>
	<b>ABSTRACT</b>	<b>1</b>
<b>1</b>	<b>INTRODUCTION</b>	<b>2-7</b>
1.1	Scaling Theory	2
1.2	Literature Review	5
1.3	Motivation and objective	7
<b>2</b>	<b>EXPERIMENTAL TECHNIQUES</b>	<b>8-10</b>
<b>3</b>	<b>RESULTS AND DISCUSSION</b>	<b>11-15</b>
3.1	XRD Analysis	11
3.2	AFM Analysis	11
3.3	DST Analysis	13
	<b>CONCLUSION</b>	<b>16</b>
	<b>REFERENCES</b>	<b>17-19</b>

## List of figures

<b>Figure</b>	<b>Title</b>	<b>Page Number</b>
<b>1.1</b>	Surface growth as temporal evolution of $h(\vec{r}, t)$	<b>3</b>
<b>2.1</b>	Schematic Diagram of DC Sputtering	<b>8</b>
<b>2.2</b>	(a) Plasma during the deposition process and (b) The sputtering machine used in this work.	<b>9</b>
<b>2.3</b>	Cu thin film deposited at (a) 5 sec, (b) 30 sec, (c) 1 min, (d) 2 min, (e) 15 min	<b>9</b>
<b>3.1</b>	Typical XRD patterns of Cu thin films of different deposition time	<b>11</b>
<b>3.2</b>	Representative AFM images ( $2 \times 2 \mu\text{m}^2$ ) of surface morphology of films of deposition time.	<b>12</b>
<b>3.3</b>	Line profiles across the sample surface of films of different growth times.	<b>12</b>
<b>3.4</b>	Log-log plot of power spectral density	<b>13</b>
<b>3.5</b>	Log-log plot of height- height distribution	<b>14</b>
<b>3.6</b>	Log-log plot of local surface width and saturated width	<b>14</b>

## List of tables

<b>Table</b>	<b>Title</b>	<b>Page Number</b>
<b>1.1</b>	Class of surface scaling and characteristics	<b>5</b>
<b>1.2</b>	The observed roughness and growth exponents for several sputter deposited experiments	<b>6</b>

## ABSTRACT

Thin film technology is the foundation of wondrous technological advances made in the fields of optoelectronic, photonic, magnetic and solid state electronic devices. Surface morphology of deposited thin films and the method used in depositing thin films are of great interest. The surface roughness plays a critical role in many applications and properties such as contact mechanics, sealing, friction, adhesion, optics and electrical conductivity. The Dynamic Scaling Theory was used to study the surface roughness and scaling behavior of Cu thin films grown on glass substrate by DC magnetron sputtering at 75W power at varying time. The phase of resulting thin films was characterized by X-ray diffraction (XRD), and growth of surface with increasing time was studied by using Atomic Force Microscopy. Determination of the PSDF (Power Spectral Density Function) and HHCF (Height-Height Correlation Function) of the films deposited for different times were used to evaluate details of the kinetic roughening. From the measured exponents, in conjunction with the computed ones, determination of the growth mechanisms were carried out and the processes was classified within certain universality classes. Detailed scaling analysis of surface fluctuation in real and fourier space yielded characteristic exponents  $\alpha(= \alpha_s) = 1.09 \pm 0.40$ ,  $\beta = 0.12 \pm 0.02$ ,  $\alpha_{local} = 1$ ,  $\beta_{local} = 0.63 \pm 0.05$  and  $\alpha_s = 1.47 \pm 0.02$  indicate the presence of super-roughening on the line of anomalous scaling and *not* the conventional scaling obeying Family–Vicsek equation.

# CHAPTER 1

## INTRODUCTION

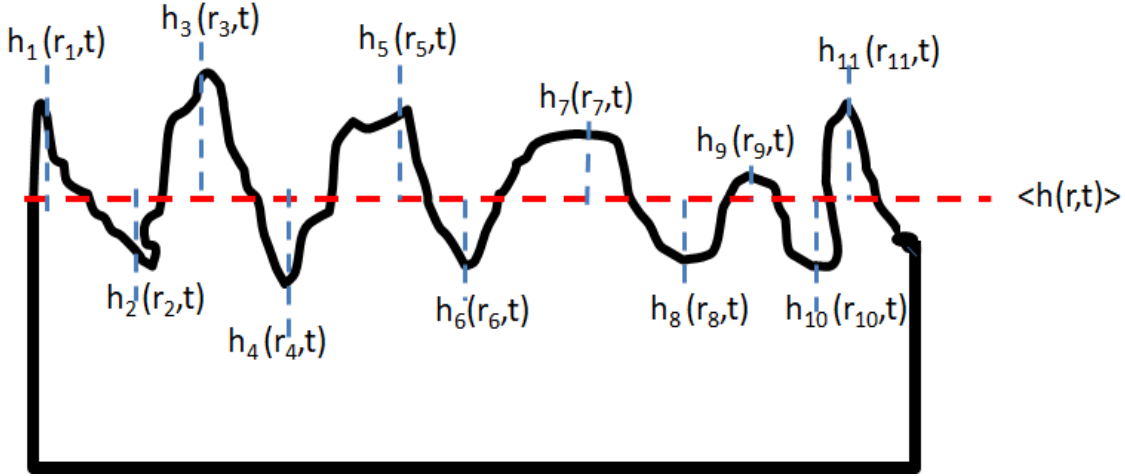
Over the years, thin film technology has been the key to the significant advances made in the fields of optoelectronic, photonic, magnetic and solid state electronic devices.<sup>[1]</sup> The basis of this progress has been the unusual properties of thin films, distinct from their bulk counterparts, due to reduced dimensions leading to a higher surface to volume ratio. While miniaturization of devices has become increasingly popular, the surface of thin films has become more of research interest. The surface roughness plays a critical role in properties such as contact mechanics, sealing, friction, adhesion, optics and electrical conductivity, and hence performance of the devices.<sup>[2]</sup> For example, in semiconductors, rougher surfaces lead to more recombination of charge carriers, which can be sometimes undesirable. On the other hand, rough surface means higher exposed area, and hence better sensing capability. Thus, surfaces become important especially in nano scale devices in which the state of surface governs the behavior of optical and electrical performance. Besides to control the properties of thin films, studying the roughness of the evolving surfaces has been of research interest over several decades as it signals growth of surfaces far from equilibrium that mimics many naturally occurring phenomena.

Significant advancement in studying evolution of surface roughness has been made through the frame work of dynamic scaling theory (DST).<sup>[3]</sup> As per this theory, with the evolution of invaring framework of time and space, the roughness is defined by set of scaling factors namely global roughness and growth exponents,  $\alpha$  and  $\beta$ , respectively. These exponents are directly related to the mechanisms that govern the evolution of roughness, often given by the Kardar-Parisi-Zhang (KPZ) universality class, mathematically.<sup>[4]</sup> From the application point of view, one can measure these exponents, and compare with the theoretically predicted ones to identify the mechanism of growth and to categorise methodology under definite universality classes.

### 1.1 Scaling Theory

The scaling behavior arises due to self-affine nature appearing at the interface in a limited order because of balance various experimental factors such as deposition time, moment of

depositing species on the surface, release of deposited surface from the surface, deposition angle, shadowing effect etc.<sup>[2]</sup> These factors regulate morphology of surface.



**Fig 1.1** Surface growth as a function of space and time  $h(\vec{r}, t)$

The surface growth could be presented as a function of space and time  $h(\vec{r}, t)$  around its mean value about the surface width as shown in fig 1.1,

$$w(r, t) = \langle (h(\vec{r}, t) - \langle h(\vec{r}, t) \rangle)^2 \rangle_r^{1/2}$$

where  $\langle \dots \rangle_r$  is the average in a system of size  $L$  and where  $r \leq L$ . For  $r \ll L$ ,  $w(r, t)$  is the local width whereas for  $r = L$  is known as global width  $\sigma(L, t) = w(r, t)$ .<sup>4, 5</sup> This explains in the kinetic roughening of surface under conventional DST, where the Family–Vicsek equation is followed by both the local width as well as by the global width :

$$w(r, t) = t^\beta f\left(\frac{r}{t^{\beta/\alpha}}\right) \quad (1)$$

$\alpha$  and  $\beta$  are independent exponents. Where  $\alpha$  is roughness exponent and  $\beta$  growth exponent that characterise the surface growth in different universality classes. Behavior of the scaling function is given by:

$$f(U) \sim \begin{cases} U^\alpha & U \ll 1 \\ \text{constant} & U \gg 1 \end{cases}$$

In this regard, for smaller values of  $r$  (i.e.,  $r \ll t^{\alpha/\beta}$ ),  $w$  is independent of deposition time,  $t$ , and scales as  $r^\alpha$  whereas for large  $r$ ,  $w$  is independent of  $r$  and scales as  $t^\beta$ . This cross of both the nature appears at  $r = r_c$ , where  $r_c$  is known as lateral correlation length. The measure of  $r_c$  denotes distance whereupon there is no relation with the surface features. It measures as  $t^{1/z}$ ,

which is known as dynamic exponent and is given as  $z = \alpha/\beta$ . Hence, equation (1) can be given as

$$w(r, t) = \begin{cases} t^\beta, & \text{if } t \ll r^{\alpha/\beta} \\ r^\alpha, & \text{if } r^{\alpha/\beta} \ll t \ll L^{\alpha/\beta} \end{cases}$$

The scaling behavior is studied often from the height-height correlation function (HHCF)  $G(r, t)$  or power spectral density (PSD) of surface fluctuation in reciprocal space. The HHCF is given by  $G(r, t) = \langle [h(\vec{r}_2, t) - h(\vec{r}_1, t)]^2 \rangle$  while PSD  $(k, t)$  defined as  $\text{PSD}(k, t) = \langle H(k, t) H(-k, t) \rangle$ .

In this system the given conventional scaling pattern (i.e., Family–Vicsek relation) , the two functions shows the nature as that of self-affine nature at the interfaces:

$$G(r, t) \sim \begin{cases} r^{2\alpha} & \text{for } r \ll r_c \\ \text{constant} & \text{for } r \gg r_c \end{cases}$$

The power law behavior is shown by PSD as :

$$\text{PSD}(k, t) \sim \begin{cases} k^{-\gamma} & \text{for } k \gg r_c^{-1} \\ k - \text{independent} & \text{for } k \ll r_c^{-1} \end{cases}$$

The  $\alpha$ , is obtained from  $\gamma$  as  $\alpha = (\gamma - d)/2$  where  $d$  is the dimension of the surface. Even though with beneficial application of conventional DST in order to understand the growth of thin film surface grown by various deposition techniques, there are indications which exhibits deviance from the Family–Vicsek equation, theoretically and experimentally.<sup>[10]-[14]</sup> These are called anomalous roughening. Global width is still follows Family -Vicsek behavior while the scaling analysis of the local width is given by:

$$w(r, t) = \begin{cases} t^\beta, & \text{if } t \ll r^{\frac{\alpha}{\beta}} \\ t^{\beta^*} r^{\alpha_{loc}}, & \text{if } r^{\frac{\alpha}{\beta}} \ll t \ll L^{\frac{\alpha}{\beta}} \end{cases} \quad (2)$$

Where  $\alpha_{local}$  and  $\beta^*$  are exponents known as the local roughness exponent the anomalous growth exponent respectively. The significant inference is that for small  $r$ ,  $w$  shows power-law dependence on both  $t$  and  $r$ , while in conventional scaling for small  $r$  it is independent of  $t$ . The experimental existence of the anomalous behavior is being considered in HHCF and PSDF. By using general forms of the scaling functions, Ramasco *et al.*<sup>[4]</sup> in order to determine the theory of dynamic scaling, it indicates the PSD for (2+1) dimensional system as:

$$\text{PSD}(k, t) \sim \begin{cases} t^{(2\alpha+2)/z} & \text{for } kt \ll 1 \\ k^{-(2\alpha_s+2)} t^{2(\alpha-\alpha_s)/z} & \text{for } kt \gg 1 \end{cases} \quad (3)$$

where  $\alpha_s$  is known as the spectral roughness exponent.

For different values of  $\alpha$ ,  $\alpha_{local}$  and  $\alpha_s$  up to four scaling behavior are predicted, as outlined in **Table 1.1.** <sup>[4]</sup>

**Table 1.1:** Class of surface scaling along with characteristics

<b>Class of scaling</b>	<b>Characteristics</b>
Normal Scaling	$\alpha = \alpha_{local} = \alpha_s < 1$
Intrinsic Anomalous Scaling	$\alpha \neq \alpha_{local} = \alpha_s \leq 1$
Super Roughening	$\alpha_{local} = 1$ and $\alpha = \alpha_s > 1$
New Class of Roughening	$\alpha_{local} = 1$ and $\alpha \neq \alpha_s > 1$

Numerous studies of the experimental and theoretical analysis declares the scaling relations and presence of the kinetic roughening in thin films, on the outline of dynamic scaling theory (DST). Still there are many systems in which the theoretical models could not explain the obtained exponents. Thus, in order for detailed evaluation of experimental system, a new model closer to actual experimental conditions is important.

Among the varied method of thin film growth, sputtering method has been conceived as one of the elemental user-friendly and industrially-relevant ones. However, no consensus has been developed in ascribing the growth mechanisms. In this work, scaling of surface roughness of copper thin films grown by DC magnetron sputtering have been studied. The choice of Cu is based on its excellent conductivity, which is highly appropriate for applications as conducting electrode in optoelectronic devices.

## 1.2 Literature Review

Roughening in sputter deposited thin films have been found in number of experimental studies. As shown in **Table 1.2**, a large range of scaling exponents has been observed for different materials, and the observed exponents seem to build upon experimental conditions as well as the deposited species. Although much research work has been done, it has not been possible to determine a standard set of scaling exponents corresponding to a universality class. Jefferies et al. found that roughness exponent  $\alpha = 0.90$  and growth exponent  $\beta = 0.26$  for sputtered deposited Pt at room temperature <sup>[9]</sup> whereas Sharma et al. obtained  $\alpha = 0.89$  and  $\beta =$

0.42 for sputtered deposited Al at room temperature.<sup>[15]</sup> Interestingly, Liu et al. reported  $\alpha = 0.79$  and  $\beta = 0.16$  for sputtered deposited Al at room temperature.<sup>[16]</sup>

**Table 1.2:** The observed roughness and growth exponents for several sputter deposited thin films

System	Temperature	Roughness exponent ( $\alpha$ )	Growth exponent ( $\beta$ )	Reference
ZnO:Al film on glass	RT	$\alpha = 1.5 \pm 0.2$ $\alpha_l = 1.0$	$\beta = 1.03 \pm 0.01$ $\beta_l = 0.67 \pm 0.05$	[5]
TiN film on Si		0.98	0.28	[6]
Pt film on glass	RT	$0.90 \pm 0.02$	$0.26 \pm 0.03$	[10]
TiN film on Si (100)	$< 70^\circ\text{C}$	$\alpha = 1.7$ $\alpha_L = 1.0$	$\beta = 0.67$ $\beta_L = 0.85$	[11]
Al film on Si (111)	RT	$0.89 \pm 0.05$	0.42	[15]
Al film on Ti (100)	RT	$0.79 \pm 0.05$	$0.16 \pm 0.01$	[16]
Au film on Si (111)	220 K	0.42	0.42	[17]
	300 K	0.40	0.40	
Ni film on ITO glass	423 K	$0.97 \pm 0.02$	$0.53 \pm 0.02$	[18]
	473K	$0.97 \pm 0.02$	$0.84 \pm 0.06$	
W film on polished Si	RT	$0.18 \pm 0.02$	$0.06 \pm 0.01$	[19]
Poly crystalline Al film on thermally oxidized Si	$\approx 25^\circ\text{C}$	0.35		[20]
Amorphous Si on flat Si	RT	$0.69 \pm 0.09$	$0.55 \pm 0.09$	[21]
a-Si on Si (100)	$70^\circ - 80^\circ\text{C}$	$0.83 \pm 0.03$	$0.41 \pm 0.01$	[22]
Cu film on Ta covered Si (111)	700 K	$\alpha_l = 0.87$ $\alpha_g = 0.83$	$\beta_l = 0.22$ $\beta_g = 0.85$	[23]
Al film on Si (100)	RT	$\alpha_{3min} = 0.61$	$\beta_1 = 0.73$	[24]
		$\alpha_{5min} = 0.48$		
		$\alpha_{8min} = 0.41$	$\beta_2 = -0.52$	
		$\alpha_{15min} = 0.38$		
Cr (oblique incidence)	300-330 K	1	$0.98 \pm 0.01$	[25]

Si film on	Singular	300 K	$0.85 \pm 0.05$	$0.6 \pm 0.05$	[26]
Si (001)	Miscut	300 K	$0.80 \pm 0.05$	$0.7 \pm 0.05$	

RT: Room temperature

### 1.3 Motivation and objective

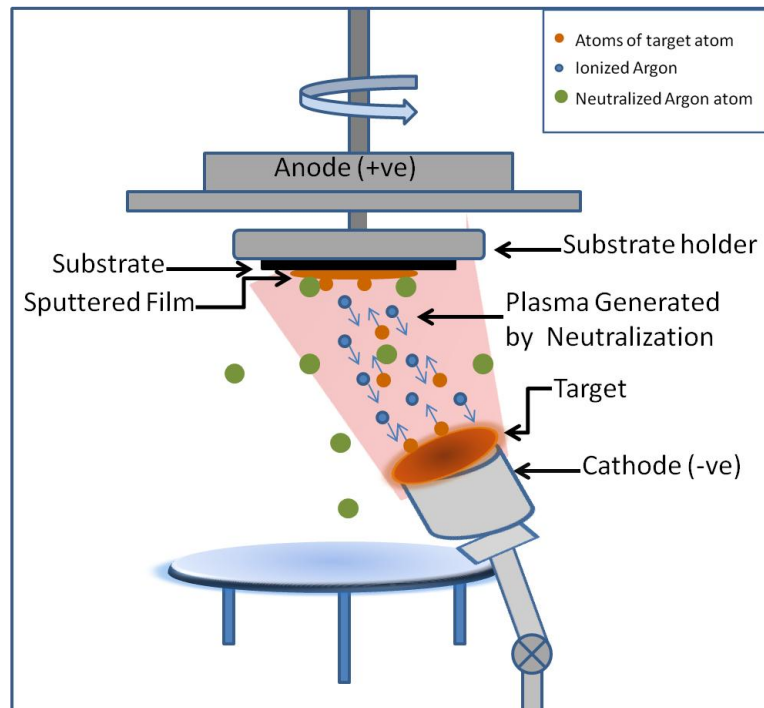
As shown in **Table 1.2**, the wide variation in the exponents indicate that possible variation in the experimental conditions affecting the growth mechanisms. It is also possible that all parameters in the experimental settings have not been included in the existing theoretical models. Hence, theoretical studies have not been successful in explaining the observed indices. At this point of research, thus, it is important to explore the interface thoroughly leading to the establishment of the most complete set of experimental data, which can be potentially integrated into the models and simulations.<sup>[11]</sup> The interest in Cu thin films is due to the diverse characteristics such as high reflection power, high thermal and electrical conductivity, important in scientific and technological applications. In view of this, the objectives of the present work is to grow Cu thin films by an industrially relevant technique such as DC magnetron sputtering and determine the scaling exponents using the dynamic scaling theory.

## CHAPTER 2

### EXPERIMENTAL TECHNIQUE

As mentioned in the previous chapter, among the different methods used for the growth of thin films, sputtering method has been conceived as one of the most user-friendly and industrially-relevant ones. In this work, the Cu films were deposited by the DC magnetron sputtering method. It is a basic and inexpensive route of physical vapor deposition method for metals. It is routinely used in the semiconductor industry, for gold coatings of jewelry, decorative items, and watches. It is also use for non-reflective surface coatings on optical and glass components.

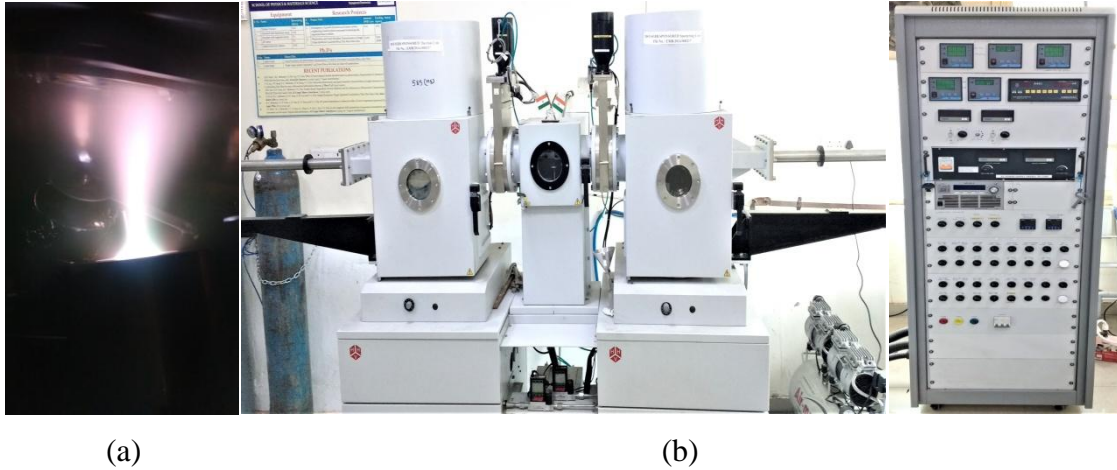
A schematic diagram of the DC magnetron sputtering process is shown in **Fig. 2.1**.



**Fig. 2.1:** Schematic Diagram of the DC Sputtering process

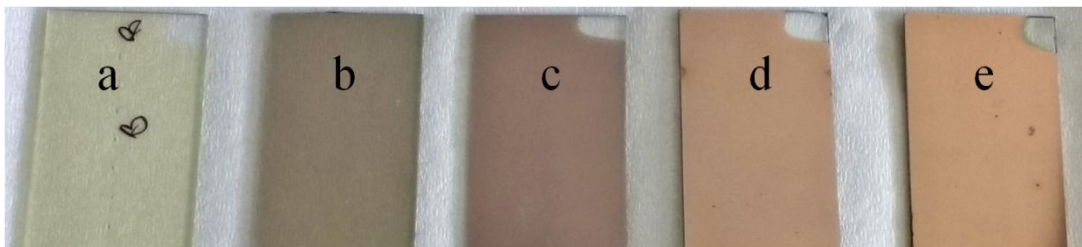
In this process, the target is negatively biased typically in the range of 2 to 5 kV with respect to the common/floating ground (substrate). The electrically neutral argon atoms are firstly ionized due to collision of gas atoms on the surface of the target which is kept at negative potential, as a result of which the plasma is generated consisting of gas ions and electrons which

appears as glowing discharge. The vaporized target atoms move towards the substrate and condense to form a thin layer of coating. Figure 2.2 shows the plasma during the growth of the films and the machine used for the preparation of the Cu films in this work.



**Fig. 2.2:** Photograph of the (a) plasma during the deposition process and (b) the sputtering machine used in this work.

The deposition of Cu films on glass substrates at room temperature is shown in **Fig 2.3**. The system was evacuated to a base pressure of  $5 \times 10^{-6}$  mbar by a turbo-molecular pump backed by a rotary vane pump. Films were deposited at a working pressure of  $2.6 \times 10^{-3}$  mbar, which was achieved by admitting high pure ( $>99.99\%$ ) Ar gas through a mass flow controller. The target-substrate distance was  $\sim 10$  cm. The deposition was carried out using a water-cooled 2-inch diameter high purity Cu target at a DC power of 75W.



**Fig. 2.3:** Cu thin film deposited at (a) 5 sec, (b) 30 sec, (c) 1 min, (d) 2 min, and (e) 15 min

Crystal structure of the films was determined from analysis of the X-ray diffraction (XRD) patterns obtained using Cu  $K_{\alpha}$  radiation ( $\lambda=1.54 \text{ \AA}$ ) in a Bruker D8 advance diffractometer. The surface morphology was evaluated with an atomic force microscope (AFM)

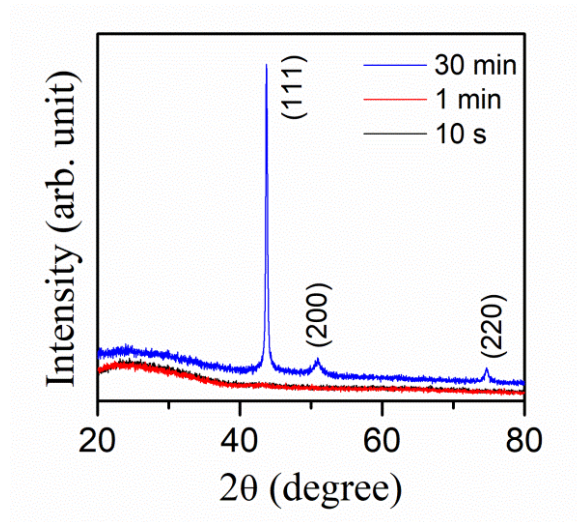
(NT-MDT-INTEGRA) using a single crystal Si tip operated in tapping mode with a radius of 3-50 nm. The scan of  $2 \times 2 \mu\text{m}^2$  wide area were acquired for each sample. The AFM provided height data with a resolution of  $512 \times 512$  pixels. A planar background was subtracted from the data to compensate for tilt of the sample relative to the scanning plane. The results were checked for accuracy by imaging several regions of the same sample.

## CHAPTER 3

### RESULTS AND DISCUSSION

#### 3.1 XRD Analysis

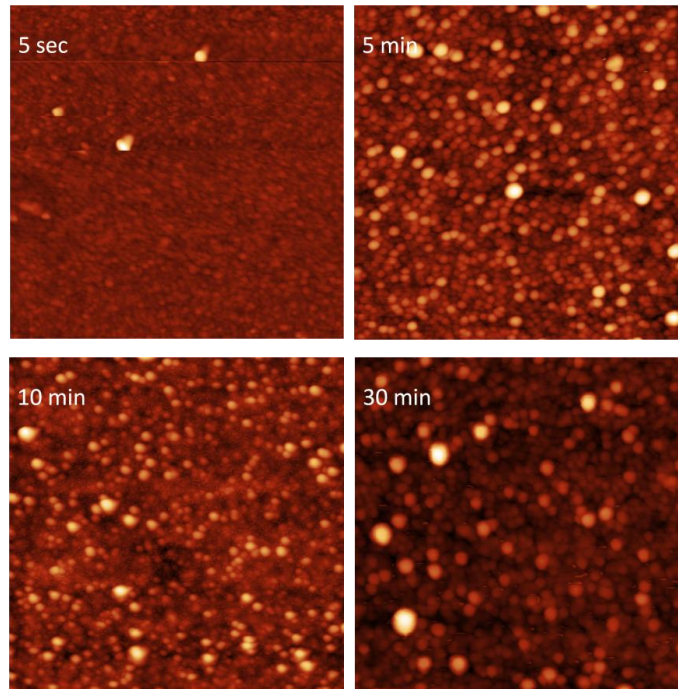
Typical XRD patterns of the films grown for different deposition times are shown in **Fig. 3.1**. For thinner films (i.e., deposition time of 10 and 60 s) no peaks could be observed. However, for relatively thicker film of deposition time of 30 min, three Bragg peaks, namely as  $43.71^\circ$ ,  $50.88^\circ$  and  $74.66^\circ$  were observed. These peaks correspond to (111), (200) and (220) planes of FCC Cu.



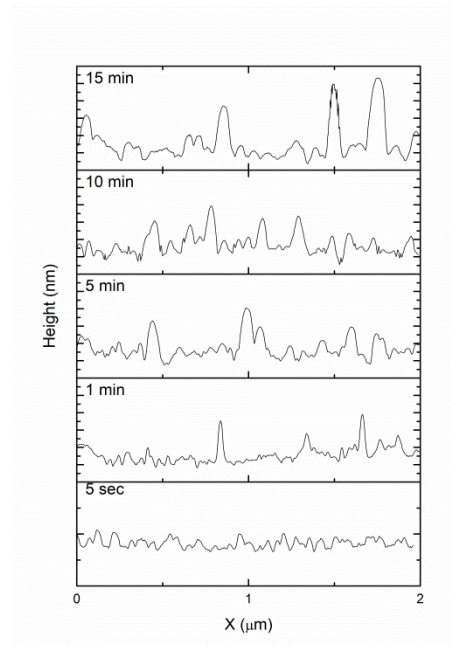
**Fig. 3.1:** Typical XRD patterns of Cu thin films of different deposition time

#### 3.2 AFM Analysis

The evolution of surface morphology with increasing deposition time of the films is shown in **Fig. 3.2**. For all films, granular morphology with uniform distribution of size and shape of the crystallites was observed. The corresponding representative line profiles are shown in **Fig. 3.3**. As seen from Fig. 3.3, with the increase in deposition time, the average height increases, as expected. Also, the lateral and vertical dimensions increased, which corresponds to the size of the crystallites.



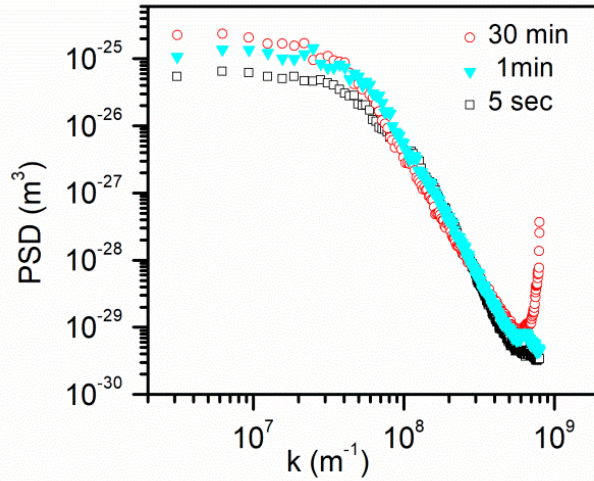
**Fig. 3.2:** Representative AFM images ( $2 \times 2 \mu\text{m}^2$ ) of surface morphology of films of deposition time.



**Fig. 3.3:** Line profiles across the sample surface of films of different growth times.

### 3.3 DST Analysis

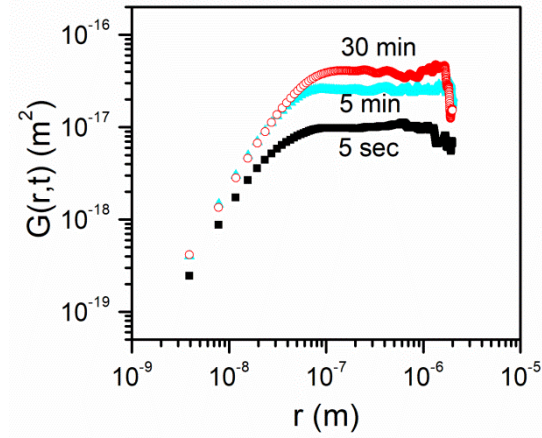
The DST analysis was carried out from the determined PSD and HHCF functions, as described in Section 1.1. The plot of PSD ( $k, t$ ) for all thin film is shown in **Fig. 3.4**. In all of the curves no peaks has been found, which indicates that the surface growth is independent of wavelength as well as there is no significance of Schwoebel barrier.<sup>8</sup> Up to a certain frequency, the PSD curves were independent of  $k$ . For the higher  $k$  values, all PSD curves exhibited a linear dependence on  $k$ , slope  $-\gamma$  from which the exponent  $\alpha_s = (\gamma - d)/2 = 1.477 \pm 0.03$  with  $d = 1$ . The power-law relation of PSD ( $k, t$ ) with  $k$  and the curves are overlapping with  $\alpha_s = 1.47$  indicate the presence of super-roughening (Table 1.1).



**Fig. 3.4:** Log-Log graph of PSD ( $k, t$ ) as a function of  $k$  at different deposition times.

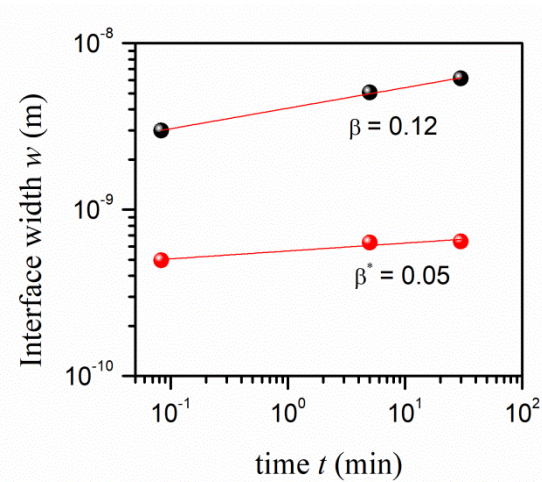
Further analysis of scaling of surface roughness was evaluated from the HHCF function  $G(r, t)$ . The plot of  $G(r, t)$  with  $r$  is presented in **fig. 3.5**. The figure reveals that for smaller values of  $r$  there is linear increase while for larger values it saturates.  $G(r, t)$  displays very important that with time it shifts upwards for the whole order of  $r$ , differently from the trend in the PSD functions. Hence it demonstrates super roughening of thin films. For super roughening, in PSD functions the time dependence vanishes due to  $\alpha = \alpha_s$  as shown in Eq. (3), while  $G(r, t)$  has non-vanishing time dependence in small  $r$  limit.<sup>28</sup> According to Eq. (2), for small  $r$  values from the slope of  $G(r, t)$ , the  $\alpha_{local}$  can be derived. In the present case, we find that  $\alpha_{local} = 0.63 \pm 0.06$ , which is less than the unity value as theoretically predicted.<sup>4,5</sup> However, such a

deviation is not unusual in literature.<sup>5,12</sup> This discrepancy is attributed to the computational limitations.



**Fig 3.5:** Log-Log plot of HHCF as  $G(r, t)$  vs. lateral distance  $r$

As expected from the super-rough scaling behavior, the global and local growth exponents were determined from the HHCF. From the saturated values of HHCF (in the large  $r$ -regime) and the smallest value of the HHCF, the saturated and local surface width was calculated. From the variation of these widths with deposition time (**Fig. 3.6**), the global growth exponent  $\beta$  and the anomalous growth exponent  $\beta^*$  were determined.



**Fig. 3.6:** Log-Log plot of local and saturated surface width in term of deposition time.

We find that saturated width varies as  $t^\beta$  with  $\beta = 0.12 \pm 0.004$ , whereas for the local width is measured as  $t^{\beta^*}$  where  $\beta^* = 0.05 \pm 0.01$ . The local growth exponent is determined to be  $\beta_{\text{local}} = \beta - \beta^* = 0.07 \pm 0.01$ . The observed low value of  $\beta$  suggests that the interface width increases at a relatively slower rate. Similar values were obtained by Liu et al. who have obtained  $\beta = 0.16 \pm 0.01$  for sputtered deposited Al at room temperature.<sup>[16]</sup>

Concluding results, we find that the growth of Cu films in the present growth conditions with the exponents  $\alpha = \alpha_s = 1.47 \pm 0.03$ ,  $\beta = 0.12 \pm 0.004$ ,  $\alpha_{\text{local}} = 1$ ,  $\beta_{\text{local}} = 0.07 \pm 0.01$  depicting super-rough scaling. The observed values are unusual and are not explained based on contemporary theoretical models. Examination of sputtered films, Jefferies et al. found that roughness exponent  $\alpha = 0.90 \pm 0.02$  and growth exponent  $\beta = 0.26 \pm 0.03$  for sputtered deposited platinum at room temperature<sup>[10]</sup> whereas Sharma et al. obtained that roughness exponent  $\alpha = 0.89 \pm 0.05$  and growth exponent  $\beta = 0.42$  for sputtered deposited Al at room temperature.<sup>[15]</sup> As Table 1.2 lists, the present value of  $\alpha$  is much higher than the reported ones.

Further analysis of growth of films with varied process parameters may provide insight to the growth mechanisms. For instance, a variation in substrate temperature or DC power may highlight the ad-atomic movement and correspondingly a different set of exponents may be obtained.

## Conclusion

We have studied evolution of surface morphology and scaling of roughness of Cu thin films grown by DC magnetron sputter deposition on glass substrates at room temperature. The curves are overlapping the PSD ( $k, t$ ) shows a power-law relation with  $k$ . This indicate that there is evolution in the growth modes of the films appearing as the function of deposition time and overlapping of the curves (i.e., independent of deposition time) in coexistence with the set of exponents  $\alpha(= \alpha_s) = 1.09 \pm 0.40 = 0.12 \pm 0.02$ ,  $\alpha_{local} = 1$ ,  $\beta_{local}=0.63\pm0.05$  and  $\alpha_s=1.47 \pm 0.02$  indicate the presence of super-roughening on the line of anomalous scaling and *not* the conventional scaling obeying Family–Vicsek equation.

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