



Surface roughness analysis of SiO₂ for PECVD, PVD and IBD on different substrates

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Received: 22 December 2014 / Accepted: 10 March 2015 / Published online: 22 March 2015
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Abstract This study compares surface roughness of SiO₂ thin layers which are deposited by three different processes (plasma-enhanced chemical vapor deposition, physical vapor deposition and ion beam deposition) on three different substrates (glass, Si and polyethylene naphthalate). Plasma-enhanced chemical vapor deposition (PECVD) processes using a wide range of deposition temperatures from 80 to 300 °C have been applied and compared. It was observed that the nature of the substrate does not influence the surface roughness of the grown layers very much. It is also perceived that the value of the surface roughness keeps on increasing as the deposition temperature of the PECVD process increases. This is due to the increase in the surface diffusion length with the rise in substrate temperature. The layers which have been deposited on Si wafer by ion beam deposition (IBD) process are found to be smoother as compared to the other two techniques. The layers which have been deposited on the glass substrates using PECVD reveal the highest surface roughness values in comparison with the other substrate materials and techniques. Different existing models describing the dynamics of clusters on surfaces are compared and discussed.

Keywords Micro electro mechanical systems · Plasma-enhanced chemical vapor deposition · Physical vapor deposition · Ion beam deposition · Surface roughness · Stylus profilometry · Atomic force microscopy

Introduction

Each MEMS structure, which is electrically operated, requires some kind of insulation. Silicon dioxide (SiO₂) is a very good insulator, which is used in most of the cases because of its transparency and cost-effectiveness.

There are many ways to deposit SiO₂ for these MEMS structures. The best known method for producing SiO₂ is native silicon oxide, in which a silicon surface is exposed to oxygen under ambient conditions (Morita et al. 1990). The most common way to deposit SiO₂ is using plasma-enhanced chemical vapor deposition (PECVD), which is a comparatively low-cost process and operates at low temperatures ranging from 60 °C to approximately 300 °C (Tarraf et al. 2004) and gives a good thickness control (Chen et al. 1993). Physical vapor deposition (PVD) using an electron beam gun (Reichelt and Jiang 1990) is a second method. A third method which is very famous for its smooth surfaces is ion beam sputtering deposition, also known as ion beam deposition (IBD), in which thin films are deposited on a substrate by sputtering the target (McNeil et al. 2002).

In MEMS, SiO₂ layers are mainly used as an electrical insulating layer, as well as structural layer (Chandra and Sudhir 2007). The MEMS devices, which are actuated electrostatically, normally comprise of two electrodes. This SiO₂ layer lies in between those two electrodes and provides the insulation. Micromirror structures are a very good example of electrostatically actuatable MEMS. We investigated the suitability of those different insulation layers for micromirrors which are fabricated for the purpose of daylight guiding and illumination (Viereck et al. 2009; Hillmer et al. 2010). Figure 1 shows schematics of one mirror element.

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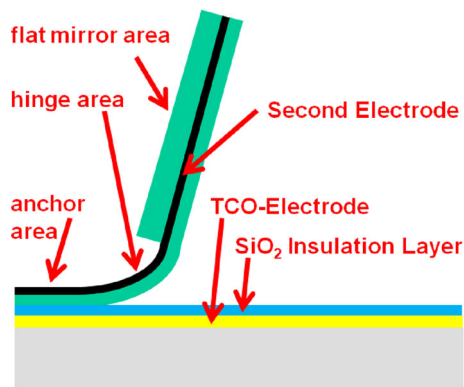


Fig. 1 Schematics of a micromirror element

Such insulation layers have to have good insulating properties between the two electrodes and have to provide good adhesion of structures to the substrate in the anchor area (Jäkel et al. 2010). Furthermore, the surface roughness of the insulation layer plays an important role because it directly affects the actuatability of the structures in terms of stiction when the mirror area is curled down to the insulation layer during the actuation (Tas et al. 1996) and hence becomes an important parameter.

In PECVD processes, the surface roughness depends on the deposition temperatures because the initial cluster which is produced at the start of the deposition varies according to the temperature (Lee and So 2000). When the deposition temperature is high, the initial cluster size is big (Lee and So 2000) because of coalescence of clusters (Ohring 2002) resulting in higher surface roughness and vice versa (Chandra and Sudhir 2007). In IBD processes, the most important parameter on surface roughness (Chandra and Sudhir 2007) is sputtering power used. If the sputtering power is high, the surface roughness will be decreased and vice versa (Chandra and Sudhir 2007). When the RF power is low, ions have low energy and they stay on the surface upon their arrival, thereby resulting in a low surface roughness. Similarly, when the deposition pressure increased, it also increases the surface roughness and vice versa.

Surface roughness may depend on the nature of the substrate. A silicon wafer has a very smooth surface, so the thin films which are deposited on it can be considered as pure layers in terms of surface roughness and can be considered as reference layers to the other substrates. Nowadays, MEMS devices are more and more built using substrate materials like glass or polymers instead of semiconductor materials. So in this work, three different examples for these types of substrates are investigated, namely Si wafers, glass substrates and polyethylene naphthalate (PEN) substrates. In terms of surface roughness, glass substrates should have higher surface roughness

values than silicon wafers (Teichert et al. 1995). The surface roughness value of the PEN substrate should be higher as compared to Si wafer and glass substrate (Klauck et al. 2003).

Experimental work

Silicon oxide layers grown by means of PECVD, IBD and PVD have been grown on crystalline silicon, glass and PEN substrates, and the respective surface roughness has been measured and intercompared.

Definitions

In the following, two types of surface roughness values are recorded: i.e., average surface roughness R_a and root mean square (rms) surface roughness R_q . The average surface roughness in mathematical expression can be defined as (Krizbergs and Kromanis 2006):

$$R_a = \frac{1}{L} \int_0^L |Y(x)| dx \quad (1)$$

where R_a is average surface roughness, Y is total area of scan, and L represents total number of point which can be taken for the calculation of the surface roughness. Similarly, the rms surface roughness can be defined as (Gadelmawla et al. 2003):

$$R_q = \sqrt{\frac{1}{L} \int_0^L \{Y(x)\}^2 dx} \quad (2)$$

Preparation of the substrates

For the measurement of the surface roughness, all three substrate types are prepared to get them free of impurities by rinsing them in isopropyl alcohol and drying them using pure nitrogen. The measurements have been made in a class 10,000 clean room environment.

Deposition of SiO₂

Three different techniques are used for deposition: i.e., plasma-enhanced chemical vapor deposition (PECVD), physical vapor deposition (PVD) and ion beam deposition (IBD). A total thickness of the SiO₂ layer of 150 nm nominally is chosen for all the above-mentioned techniques.

PECVD process

The PECVD process for the intercomparison using different substrate materials has been carried out at a comparatively low temperature of 120 °C (temperature of the substrate holder).

Further on, processes varying the temperature of the substrate holder from 80 to 300 °C have been used. The other parameters of the process are given in Table 1.

PVD process

The PVD process is carried out in high vacuum of about 1×10^{-6} mbar. The other deposition parameters are given in Table 2.

IBD process

For the deposition using IBD, the deposition parameters are given in Table 3.

Surface roughness measurements

Two different methods have been involved for the measurement of surface roughness: one is the stylus profilometry, and the other is atomic force microscopy (AFM). In stylus profilometry, the stylus profiler touches mechanically the surface, and the vertical motion is then

Table 1 PECVD parameters for SiO₂ deposition

Parameters	Values
2 %SiH ₄ N ₂ flow (sccm)	430
NH ₃ flow (sccm)	710
N ₂ O flow (sccm)	0
HF power (W)	20
LF power (W)	20
Pressure (Torr)	1

Table 2 PVD parameters for SiO₂ deposition

Parameters	Values
E-gun voltage	9 kV
Deposition rate	0.3 nm/s
Purity of SiO ₂	99.99 %

Table 3 IBD parameters for SiO₂ deposition

Parameter	Ion source 1	Ion source 2
Gas flow	Ar 6 sccm	Ar 2 sccm
	Xe 0 sccm	O ₂ 11 sccm
Power	220 W	200 W
Beam	800 V (74 mA)	100 V (40 mA)
Voltage	100 V (2.5 mA)	100 V (1.1 mA)
Pulsing	1 kHz (80 %)	10 kHz (60 %)
E-current	0 eV	100 eV (80 mA)

transferred into electrical signal which represents the surface topography (Vorburger et al. 2007). Since there is a mechanical contact between surface and stylus, so it can cause damage to the surface of the sample (Lindroos et al. 2010; Vorburger et al. 2007).

The chosen scanning length of stylus profilometer is 1 mm, the scan speed is 0.05 mm/s, and the stylus force is 0.20 mg.

While recording through AFM, different modes, namely contact mode, non-contact mode and tapping mode, are possible. Taping mode (TM) is used because there is a very common problem of adhesion and shear forces between the tip and the deposited layer lie in the contact mode (Peng et al. 2001). An area of $1.0 \times 1.0 \mu\text{m}$ is used in TM, and the scan speed is 0.75 Hz on 256 lines. The software which has been used for evaluation of AFM images and measurements is named as Gwyddion. For all the measurements, i.e., for profilometer and AFM, two values, i.e., average roughness (R_a) and rms roughness (R_q) have been calculated through the software.

Results

Surface roughness of the plain substrates

Before measuring the values of surface roughness of the deposited layer, it is very important to investigate the surface roughness of the respective substrate itself on which the layer will be deposited because it may affect surface roughness of the deposited layers. Table 4 shows the substrate's surface roughness values of the pre-deposition measurements recorded by profilometer and AFM. From Table 4, it is very clear that the glass substrate shows the highest surface roughness, while Si wafer is quite smooth having the lowest value of surface roughness, i.e., 0.7 nm (avg). The PEN substrate has got a surface roughness almost equal to glass substrate, i.e., 1.9 nm (avg).

PECVD layer analysis on glass substrate

Figure 2 shows an overview of the surface roughness values of PECVD layer on all three substrates accordingly which has been grown at 120 °C. (Only avg values of

Table 4 Surface roughness (in nm) of different substrates

	Glass		PEN		Silicon	
	avg	rms	avg	rms	avg	rms
Profilometer	2.3	2.9	3.1	3.9	0.8	1.0
AFM	2.4	2.9	1.9	2.3	0.7	0.9

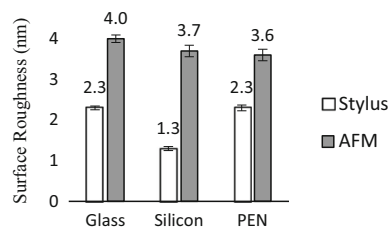


Fig. 2 Substrate vs PECVD surface roughness (avg)

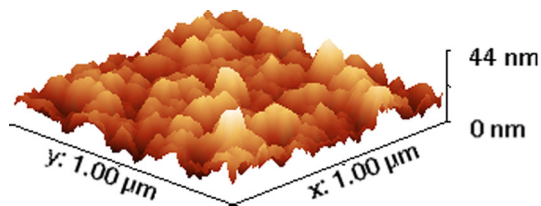


Fig. 3 AFM image of PECVD layer of SiO₂ at 120 °C

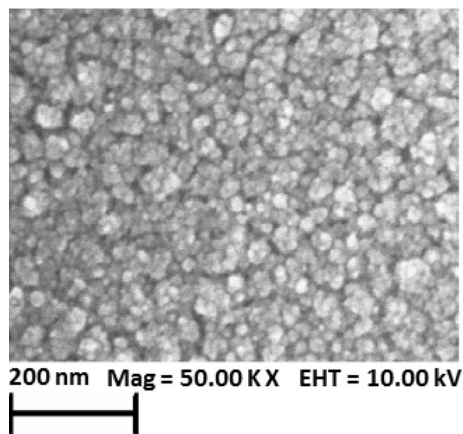


Fig. 4 SEM image of PECVD layer of SiO₂

surface roughness from AFM and profilometer are used for this plot).

In addition, Fig. 3 shows the AFM image of such a PECVD grown layer which has been deposited on a glass substrate and has a value of 4.0 nm (avg).

Figure 4 shows an SEM image of a PECVD layer on glass substrate, in which the grain sizes can be clearly observed, and it is noticed that the surface looks quite rough. The grain size is clearly visible in the picture which is having the size approximately in between 20 and 40 nm. The dark shadow which is present in the picture is an artifact caused by surface charging during the SEM imaging.

PECVD layer analysis on Si and PEN substrates

While observing the PECVD layer on the other two substrates, i.e., Si and PEN, it is observed by AFM and SEM

imaging that the roughness values of the respective layers are similar compared to the glass substrate. Hence, it can be assumed that the nature of the substrate will not affect the surface roughness values considerably. This is due to the fact that the thin film near the interface between substrate and thin film itself, influenced by the surface profile of the substrate till some considerable thickness and after that layer follows a growth mode, i.e., island growth or layer by layer growth (Mattox 2010). In our experiments, the thickness is 150 nm which is considered to be quite thick, so the nature of the substrate becomes irrelevant. However, there are some minor changes in the values of surface roughness; i.e., it varies in between 3.6 and 4.0 nm, but the layer itself resembles to the layer on glass substrate (Figs. 3, 4).

Effect of substrate holder temperature

One important parameter, which can be changed during the deposition, is the temperature of the substrate holder. In PECVD system, there is an option to change the temperature of the substrate holder from 20 to 300 °C. In our experiments, the substrate temperature has been changed between 80 and 300 °C always in steps of 40 °C, and it has been found that the surface roughness keeps on increasing as the substrate temperature is increased. While working with PVD and IBD process, it was not possible to change the substrate holder temperature.

During the PECVD deposition process, usually, the Si_xO_y clusters are loosely attached to the surface of the substrate and then they migrate on the substrate surface if the temperature is high enough. This movement of clusters on the substrate surface is called surface migration (Bose 2014). The surface diffusion length $l = \sqrt{D\tau}$ (Orr et al. 1992), during surface migration, increases with increasing temperature due to an increase in the diffusion constant D , which is a material-dependant property, and increases with the increase in temperature (Ohring 2002); τ is the time to deposit the equivalent of one layer. Also, the Monte Carlo simulation model (Orr et al. 1992) implementing the solid-on-solid model shows that by increasing the diffusion length, a non-uniform surface of the grown layer is produced. Another possible reason for an increase in the surface roughness is the initial cluster size, which is formed at the beginning of deposition on the substrate. The PECVD deposition can be considered as an island or Volmer Weber growth process (Dudeck et al. 2007) in which the cluster size increases with the increase in substrate temperature (Lee and So 2000; Battistona et al. 2000), which causes increase in surface roughness. This is due to the coalescence mechanism, in which two clusters collide with each other and form a one big cluster, when they are in a random motion which is proportional to the substrate temperature (Ohring 2002).

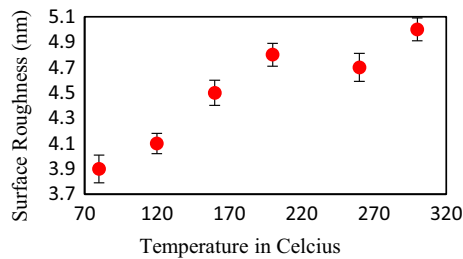


Fig. 5 Surface roughness (avg) vs temperature

However, this relationship varies according to the technique and the material which is going to be deposited. Some studies show that the surface roughness decreases with increasing substrate temperature, e.g., for amorphous ZnO/Al₂O₃ on Si substrates (Elam et al. 2002) and for crystalline GaInAs/InP layers on InP substrates (Cotta et al. 1993). In the past, the variation of surface roughness with temperature has been extensively studied in molecular beam epitaxy and metalorganic chemical vapor phase epitaxy. A good example for these detailed studies is the work of Morkoç et al. (1982), in which decreasing surface roughness was observed with increasing temperature.

Coming back to the materials and substrates studied in this work in combination with a deposition using PECVD, Fig. 5 depicts the trend of the surface roughness against the temperature (avg value of surface roughness by AFM is used).

Conclusion

In this study, it has been perceived that the surface roughness of PECVD layer is approximately three times larger as compared to the other two techniques. The main reason behind this phenomenon is that the PECVD process is very fast as compared to other processes. Chemical action takes place in the presence of plasma, and clusters of Si_xO_y form. Due to the mobility of these clusters, the surface diffusion length increases, which increases the surface roughness (Orr et al. 1992). Also, the initial cluster size which depends on the substrate temperature (Lee and So 2000) is large because of coalescence phenomenon of clusters, thereby resulting in a high surface roughness value.

PVD layer analysis on Si substrate

The AFM image of PVD layer is shown in Fig. 6. It has been observed that the layer which is deposited by PVD process looks smoother than the PECVD layer. Because in PVD deposition, the temperature is approximately 40 °C which is far less than PECVD, thereby preventing the large initial cluster size (Lee and So 2000). The deposition rate is

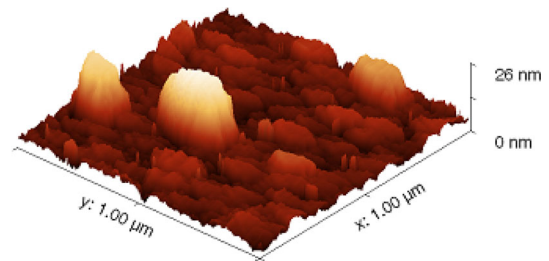


Fig. 6 AFM image of PVD layer of SiO₂

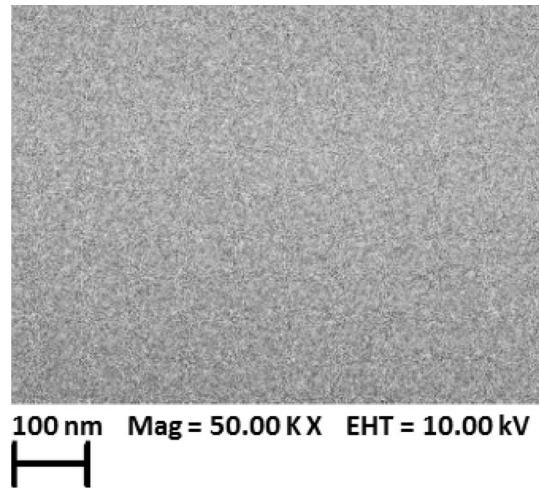


Fig. 7 SEM image of PVD layer of SiO₂

very low in PVD process, which also reduces the initial cluster size (Semaltianos 2001), because at high deposition rate, the number of atoms/molecules arriving per unit time on to the substrate is higher, thereby causing the bigger cluster formation (Bordo and Rubahn 2012).

The SEM image of the same surface is shown in Fig. 7. Generally, surface looks quite smooth, and the values which are recorded for surface roughness are 1.1 nm for profilometer and 1.4 nm for AFM (avg values). The PVD deposition can be considered as island or Volmer Weber growth mode (Dennler et al. 2003). It can be noticed that the surface is quite smooth as compared to the PECVD deposited layer. The grain size is very small as compared to the PECVD layer and hard to analyze through the SEM image.

PVD layer analysis on glass and PEN substrates

Figure 8 shows a comparison between the surface roughness values of the PVD layers on the three substrates.

From Fig. 8, it can be observed that the value of the surface roughness is decreased almost 50 % as compared to the PECVD layer. In terms of the substrate, it can be

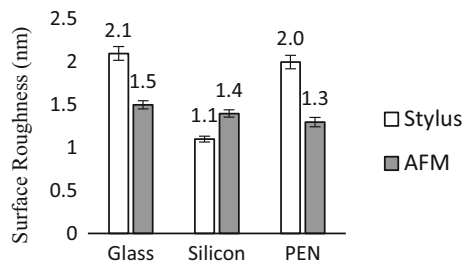


Fig. 8 Substrate vs PVD surface roughness (avg)

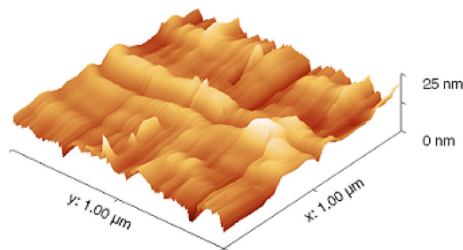


Fig. 9 AFM image of IBD layer of SiO₂

clearly noticed that the values of the surface roughness remain almost the same (with minor differences). So it can be said that the nature of the substrate will not affect considerably the value of the surface roughness of the deposited layer. The measured values for the average surface roughness for glass and PEN substrate are 1.5 and 1.3 nm, respectively.

IBD layer analysis on PEN substrate

The surface profile of SiO₂, which is deposited on the PEN substrate, is shown in Fig. 9. From Fig. 9, the texture of the surface can be well analyzed. Generally, the surface looks quite smooth as compared to the PECVD layer and looks similar to the PVD layer. The main difference in IBD is the deposition process, in which material is ejected from the target by sputtering and then deposited on to the substrate (McNeil et al. 2002), thereby giving a very smooth surface.

The average values of the surface roughness, which are recorded by AFM and profilometer, are 1.0 and 1.8 nm, respectively.

The SEM image of the same layer is shown in Fig. 10.

From Fig. 10, it can be noticed that the grain size, which has been visible in PECVD layer and in PVD layer, has now become even smaller, and it is very difficult to analyze the image. The growth mode is also Volmer Weber or island growth in this deposition (Panomsuwan et al. 2012).

IBD layer analysis on glass and Si substrates

Figure 11 shows a comparison between the values of the surface roughness (avg) of PEN substrate, glass and Si

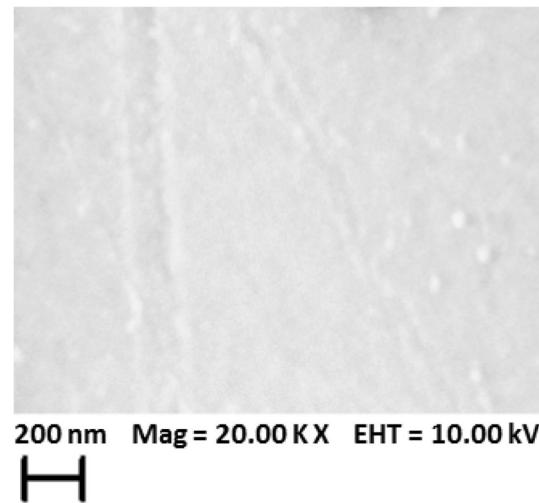


Fig. 10 SEM image of IBD layer of SiO₂

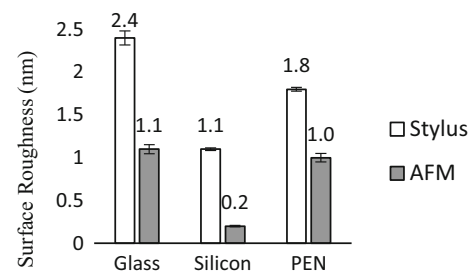


Fig. 11 Substrate vs IBD surface roughness (avg)

Table 5 Surface roughness (in nm by AFM) of substrate against deposition techniques

	Glass	PEN	Silicon
PECVD	4.0	3.7	3.6
PVD	1.5	1.4	1.3
IBSD	1.1	0.2	1.0

wafer. From Fig. 11, it is observed that the values of the surface roughness (avg) for the glass substrate and PEN substrates are almost same, i.e., 1.1 and 1.0 nm, respectively. So the substrate is not playing an important role in this matter, but while looking toward the Si wafer, it is observed that there is a difference in the value of surface roughness as compared to the other two substrates. The reason probably is the abrupt change in the interface between the substrate and the SiO film (crystalline/amorphous) (Lüth 2001) as compared to other substrates, i.e., glass and PEN, which has amorphous/amorphous interface.

So it can be assumed that while depositing through IBD on Si wafer, the initial surface roughness of the substrate

Table 6 Surface roughness (in nm by stylus profilometer) of substrate against deposition techniques

	Glass	PEN	Silicon
PECVD	2.3	1.3	2.3
PVD	2.1	1.1	2.0
IBSD	2.4	1.1	1.8

will make an effect on to the final value of surface roughness because of the abrupt interface change.

Table 5 summarizes a comparison of surface roughness values (recorded by AFM) for all substrates and all deposition techniques for an easy comparison (only average surface roughness values are included).

The average surface roughness values, which have been recorded by stylus profilometer, on all substrates are shown in Table 6.

Conclusion

In this work, SiO₂ is deposited on glass, Si and PEN substrate using different techniques, namely PECVD, PVD and IBD. It has been found that all the three techniques follow the island or Volmer Weber growth mode with different sizes of clusters. After deposition, the surface roughness of the layers is measured using stylus profilometry and AFM. It is noticed that the layer which was deposited by IBD technique on Si substrate reveals the lowest surface roughness value, i.e., 0.2 nm. Similarly, the layer which was deposited by the PECVD technique reveals the highest value of surface roughness, i.e., 4.0 nm. Also, it is noticed that the value of the surface roughness and the surface profile is same on every substrate for each technique. Hence, it can be said that the nature of the substrate is not affecting the final surface roughness value. In this study, the effect of substrate holder temperature in PECVD technique was also analyzed. The substrate holder temperature was varied from 80 to 300 °C, and the surface roughness was changed from 3.8 to 5.0 nm. Hence, it can be said that the surface roughness depends on the substrate holder temperature, while higher surface roughness corresponds with higher substrate holder temperatures. The initial cluster sizes become larger with an increase in substrate temperature, most probably because of coalescence of clusters in island growth mode. Surface diffusion length of clusters also increases as substrate temperature rises, which causes higher surface roughness.

Acknowledgments The authors express their gratitude to all the co-workers of Institute of Nanostructure Technology and Analytics (INA), Universität Kassel, for providing excellent facilities and a very

friendly atmosphere inside and outside the clean room. The underlying work has been done within the NEXT-buildings project. This project has received funding from the European Union's Seventh Framework Programme for research, technological development and demonstration under grant agreement no. ENER/FP7/284533/NEXT-BUILDINGS.

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