Surface scaling behaviour of size-selected Ag-nanocluster film growing under subsequent shadowing process

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Received 2 December 2019, revised 26 March 2020
Accepted for publication 8 April 2020
Published 3 June 2020

Abstract
Surface morphology of size-selected silver nanocluster films grown by dc magnetron sputtering has been investigated by means of an atomic force microscopy (AFM). From the height-height correlation functions obtained from corresponding AFM images, the scaling exponents are calculated and two types of growth regimes have been observed. In the first regime, the growth exponent is found to be \( \beta_1 = 0.26 \pm 0.01 \) close to the Kardar–Parisi–Zhang growth exponent, while in the second growth regime shadowing effect plays dominant role which gives the growth exponent value \( \beta_2 = 0.85 \pm 0.15 \). On the other hand for the whole deposition regime, the roughness exponent value is found to be constant around \( \alpha = 0.76 \pm 0.02 \). UV–Vis spectroscopy measurement suggests how the average reflectance of the film surface changes with different growth times.

Keywords: silver, size-selected clusters, soft-landing deposition, growth dynamics, atomic force microscopy, scanning electron microscopy, shadowing effect

(Some figures may appear in colour only in the online journal)

1. Introduction
The study of kinetic roughening of growing surface under non-equilibrium conditions has been a very active field for many years [1–4]. In the field of materials science, formation of thin film by deposition of nanoclusters has become an interesting topic from fundamental as well as application point of view. For low energy deposition or soft-landing deposition to occur, cluster kinetic energy must be very low. For a growing surface morphology due to low energy cluster beam deposition, there are mainly three factors which play crucial roles and these are deposition, desorption and surface diffusion of clusters. The term desorption is mainly dependent on the kinetic energy of the cluster beam and sticking coefficient, whereas the surface diffusion term mainly depends on the temperature of the substrate surface as well as the size and structure of the deposited cluster [5, 6]. By balancing these three factors a self-affine scaling behaviour of the growing surface can be observed [7]. A detailed study on the growth dynamics of the surface morphology is possible by characterizing the obtained scaling exponents under the framework of dynamic scaling theory [8]. These scaling exponents are basically roughness exponents \( \alpha \), which denotes the degree of surface irregularity for short length scales and growth exponent...
The exponent \( \alpha \) is called the roughness exponent which describes the lateral correlation of the surface roughness and the exponent \( z \) is called the dynamic exponent which describes how the lateral correlation length of the surface scales with time. The scaling function here constitutes two different scaling regimes depending on its argument \( u \equiv (rt)^{1/z} \). For small \( u \), the interface width \( w(r,t) \) varies as \( r^\alpha \). Whereas for \( u \gg 1 \), the interface width \( w(r,t) \) increases as well, and the dependence also follows a power law, i.e., \( w(r,t) \propto r^z \). Here \( \beta = \alpha z \) is called the growth exponent which characterizes the time dependent dynamics of the surface roughening process \([8]\). These two regions are separated by a crossover length at \( r = \xi \), called lateral correlation length which depends on the deposition time as \( t^{1/z} \). Within the lateral correlation length \( \xi \), the heights between two points are considered to be correlated. Apart from the direct computation of the interface width, there are other functions that can be calculated statistically to investigate the scaling properties of the thin film surface. One of the functions is the height-height correlation function (HHCF) calculated in direct space, which measures the lateral auto-correlation of the surface height. The HHCF for a homogeneous, isotropic random surface is given as,

\[
G(r) = \langle [h(r_2,t) - h(r_1,t)]^2 \rangle, \quad r = |r_2 - r_1|
\]

where \( h(r_1,t) \) and \( h(r_2,t) \) are the surface heights at position \( r_2 \) and \( r_1 \) respectively at deposition time \( t \) \([23]\).

For self-affine surface, the time dependent HHCF satisfies the relation,

\[
G(r) = 2[w(t)]^2 f(r/\xi)
\]

with dynamic scaling requirement of \( z = \alpha \beta \).

Thus, HHCF satisfying the above condition shows asymptotic behaviour and is given by \([8]\),

\[
G(r) \propto \begin{cases} 
  r^{2\alpha} & \text{if } r \ll \xi \\
  2^{\alpha} & \text{if } r \gg \xi.
\end{cases}
\]

The other function which manifests the scaling behaviour of random surface is the power spectral density (PSD) function which is the Fourier transform of surface height measured in reciprocal space. The PSD function helps to reveal the periodic surface features that might otherwise appear random by representing the amplitude of surface roughness as a function of spatial frequency of roughness. It is defined as,

\[
PSD(k,t) = \langle |H(k,t)|^2 \rangle
\]

where \( H(k,t) \) is the Fourier transform of surface height in a system of size \( L \) and \( k \) is the spatial frequency in reciprocal space. Statistically, PSD function also follows the Family-Vicsek relation and can be expressed in terms of scaling function \( s(k^{1/z}) \) as;

\[
PSD(k,t) = k^{-(2\alpha+1)} s(k^{1/z})
\]
where the generalized form of scaling function is given by [24, 25],

\[
s(kt^{1/2}) \propto \begin{cases} 
(kt^{1/2})^{2\alpha + 1} & ; (kt^{1/2}) \ll 1 \\
\text{Const} & ; (kt^{1/2}) \gg 1
\end{cases}
\]  \tag{8}

3. Experimental procedure

In this work, we performed deposition of size-selected silver nanoclusters on Si(100) substrates containing native oxide, utilizing a magnetron-based gas aggregation type nanocluster source (Model: NC200U, OAR, UK). The schematic diagram of the experimental set up is shown in figure 1. Before deposition, silicon substrates were properly cleaned in acetone and propanol to remove the organic contamination. To produce size-selected metal nanocluster, a silver target with 99.99% purity is used as a target of the magnetron and sputtered by Ar gas to generate the plasma. After sputtering the target by Ar\textsuperscript+ ion, the sputtered Ag atoms were condensed into clusters of various sizes with the help of buffer gas inside an aggregation chamber in a vacuum environment and then the clusters were swept away by a gas stream from the aggregation chamber into the next chamber through a nozzle and collimated into a cluster beam. In the next chamber, the collimated cluster beam was passed through a quadrupole mass filter (QMF). In QMF a particular mass of clusters from all possible masses of formed clusters are allowed to pass. In this experiment clusters of 4 nm diameter are selected, with a mass \( m = (4/3)\pi r^3 \rho \), where \( r \) is radius of cluster and \( \rho \) is the density of the cluster material. The number of atoms in a cluster of radius \( r \) is given by \( n = (r/r_w)^3 \) where \( r_w \) is the Wigner-Seitz radius. Using this equation, the number of atoms for a silver cluster with diameter 4 nm is found to be \( n = 1750 \), with \( r_w = 0.166 \) nm for silver. Size-selected cluster beam was then directed towards the main deposition chamber where the substrate was placed vertically by an angle of 45° with the cluster beam. A detail elaboration of production of size selected clusters inside the nanocluster system has been reported by Mondal and Bhatcharyya [26]. Using this experimental set-up, it is quite possible to produce clusters of various metals and non-metals. Here the clusters are produced by gas-aggregation method. The unique feature of the size-selected nanocluster facility is the capability to produce clusters of almost equal size or same mass after an intense flux of desired material produced by a magnetron based gas aggregation type source is filtered by the QMF. As a result we can precisely maintain the selected size of the nanoclusters and can easily tune the size from 1 nm to 10 nm range. This gives the novel options to use this facility in the field of nano-technology application point of view, especially in biomedical applications [27].

In this experiment, size-selected Ag nanoclusters with 4 nm diameter were used to be deposited on 10 × 10 mm\textsuperscript{2} Si (100) substrates. The base pressure inside the main deposition chamber and the aggregation chamber were 2 × 10\textsuperscript{-9} mbar & 5 × 10\textsuperscript{-4} mbar respectively and during deposition, pressure rises inside the main chamber and the aggregation chamber to 4.8 × 10\textsuperscript{-4} mbar and 2.5 × 10\textsuperscript{-1} mbar respectively. A series of samples were prepared for different deposition time viz., 5 min, 7 min, 10 min, 12 min, 15 min, 20 min, 23 min, 25 min, 28 min and 30 min. Morphological characterization of the size-selected Ag nanocluster films was carried out by an atomic force microscope in tapping mode (Model: NanoScope IV, make: Veeco, USA) using atomic force microscopy (AFM) tip of nominal tip radius 8 nm and the number of data
Figure 2. Typical (2µm × 2µm) AFM images of size-selected Ag nanocluster film deposited on Si (100) substrates for 5 min, 7 min, 10 min, 15 min, 20 min, 23 min, 25 min, 28 min and 30 min.

4. Experimental result and discussions

Figure 2 shows the AFM images representing morphology of the Ag nanocluster films deposited on Si (100) substrates for all the deposition time. The images reflect the variation in surface morphology with increasing deposition time of Ag nanoclusters. From the corresponding AFM images, it is clearly observed that height of the Ag nanoclusters does not increase much for deposition time up to 20 min, only density of deposited nanoparticles increases. Whereas for deposition above 20 min, i.e. for 23 to 30 min, both height and density of the Ag nanocluster film is found to be higher than that of the previous deposition times.

From the power spectral density (PSD) curve we can determine the kinetic roughening of the films. The log-log plot of PSD (k,t) versus k for all deposition time is shown in figure 3(a). All the PSD curves give two types of variations. Initially for low frequencies (for small k value) the curves are almost saturated which indicates the absence of any kind of lateral correlation in the surface roughness within these range. And for high frequencies (for large k value) all the PSD curves show a linear behaviour with negative slope which indicates the existence of kinetic roughening within that particular length scales. Also the non-occurrence of any kind of peak on the PSD curves reveals the absence of surface periodicity in the growth process. From the saturation region of the PSD curve, it is observed that upto 20 min deposition time all curves overlap and after 20 min of deposition time, a sharp upward shift takes place and then again there is overlapping of the curves. From this type of behaviour of the PSD curves, we can expect a transition in the growth of nanoclusters at the deposition time of 20 min.

Figure 3(b) shows the logarithmic plot of height-height correlation function G(r,t) vs scanning length r for all the samples.
Figure 3. (a) Log-log plot of the Power spectral density (PSD) functions vs wave number $k$ for different deposition time, (b) Logarithmic plot of height-height correlation function $G(r,t)$ vs lateral distance $r$, (c) RMS roughness for different deposition time, (d) Height distribution of Ag nanocluster film for different deposition time.

deposited for various time scales. For all deposition time, it is seen that the HHCF initially shows a linear increase for small value of $r$ and gets saturated for larger value of $r$. It is also observed that there is an upward shift of the graphs with the increase of deposition time for the complete range of $r$. Since HHCF does not show any oscillatory behaviour for the entire range along x-axis, it confirms that deposited surfaces are self-affine. Thus from the slope of the $G(r,t)$ functions for small $r$ value, we can determine the roughness exponent value ($\alpha$) by using the equation $G(r,t) = r^{2\alpha}$. It is found that for all deposition time, the roughness exponent is almost constant and equals to $\alpha = 0.76 \pm 0.02$. Also from the saturation region of the HHCF graph, we can calculate the root mean-square (RMS) roughness ($w$) of the films using the formula $w = \sqrt{G(r,t)/2}$. The variation of RMS roughness with deposition time is shown in figure 3(c) and from the graph, two types of variation of the growth exponent value is clearly seen, initially the RMS roughness increases very slowly with $\beta$ value $0.26 \pm 0.01$ for deposition time 5 min to 20 min and after that the RMS roughness increases sharply with another $\beta$ value $0.85 \pm 0.15$ for deposition time 20 min to 30 min respectively. The dynamic scaling exponent, $z$ is calculated by plotting the lateral correlation length as a function of deposition time in logarithmic scale. From the plot we found two slopes which correspond to two $z$ values, i.e. $1/z_1 = 0.39 \pm 0.09$ and $1/z_2 = 1.15 \pm 0.12$. Computing the value of $\alpha$ using the formula $\alpha = \beta^{*z}$, it is found that the calculated $\alpha$ values are close to the experimental values, which clearly fulfills the criterion of self-affine surface.

When we plot the height distribution function for each sample from the corresponding AFM images, we found that in
all cases height distribution is bimodal in nature, but the intensity falls down after a certain time of deposition as shown in figure 3(d). This can be explained by considering the variation of number density with deposition time. Initially the number of Ag clusters on the surface is very low, so during scanning the surface, AFM tip can detect both the surface as well as large number of clusters distributed on the surface of the substrate. With the increase of deposition time, initially the number density also increases, but after a certain time of deposition the number density starts decreasing, this may be due to the diffusion of clusters on the surface and formation of cluster islands. Due to this possibility, the surface of higher deposition time exhibits low intensity peaks in bimodal height distribution.

In addition, we investigate the UV–Vis reflectance spectra of size selected Ag nanoclusters deposited on Si (100) for different time is shown in figure 4. From the plot, it is found that the average reflectance increases with deposition time and the values of average reflectance for 7min, 20 min and 30 min are 19%, 22% and 26% respectively. With the increase of deposition time surface coverage increases which means more silver nanoclusters are deposited on the substrate. As a result of this, with the increased Ag nanoclusters on the substrates a higher surface reflectance has been found.

The elemental composition of the deposited film was confirmed by the XPS measurement shown in figure 5(a). From the full XPS spectrum, we confirm the presence of silver in our sample. For details analyses, we performed high resolution XPS spectra for the samples having deposition time 30 min, 20 min and 10 min. Figure 5(b) shows the fitted Ag 3d core level XPS spectra of the samples. Binding energies of Ag 3d core level electrons are found as 368 eV, 367.9 eV and 367.75 eV for deposition time of 10 min, 20 min and 30 min respectively. We also observe that with the increase of deposition time, amount of silver nanoparticle on the substrate increases which is reflected from the increase in peak intensity. The observed negative binding energy shift for higher deposition time indicates the formation of large cluster-islands for higher deposition time, which is in agreement with the corresponding SEM and AFM images. In earlier studies, researchers found that the main reason behind the binding energy shifting is due to charging effect on the surface [29], size effect [30] and chemical effects [31]. H.S. Shin et al [32] observed both the chemical as well as size effects of Ag nanoparticles and found that the negative binding energy shift is only due to larger particles. In our case, the only probable reason for binding energy shift is due to size effect as we can completely ignore the surface charge effect as well as the chemical effect on the surface.

Moreover the surface morphology of the deposited Ag nanoclusters is examined using FESEM as shown in figure 6. It is seen that with an increase in deposited time, there is a rise in particle density on the surface. Also from the corresponding size distributions of the deposited Ag nanoclusters for different deposition time shown in right side of figure 6, it is seen that the increased deposition time did not affect much on the mean cluster size of the deposited Ag nanoclusters. Although the size distributions of the deposited nanoclusters broaden towards higher values with the increase of deposition time. It is also noticed that for higher deposition time, number of bigger islands is more compared to that of the lower deposition time. It could be due to the aggregation/coalescences of Ag nanoclusters on the substrate surface. From these SEM images, we can also determine the number density of deposited Ag nanoclusters and their coverage of the surface. In figure 7(a), it is observed that the surface coverage value increases linearly up to a certain value with the increase of deposition time. The number density variation initially follows the trend of coverage variation with deposition time, but later on at higher deposition time the rate is slow and tends to saturate as shown in figure 7(b).

Summarizing the above results, we found two types of growth regimes of the silver films deposited by soft-landing deposition process. The value of β for the two regimes are found to be $0.26 \pm 0.01$ and $0.85 \pm 0.15$ for $t<20$ min and $t>20$ min respectively while the roughness exponent is found.
to be constant $\alpha = 0.76 \pm 0.02$ for all deposition time. In many literature reviews, this type of scaling behaviour has been observed. Buzio et al [33] found self-affine surface of cluster assembled carbon film deposited on Si and Cu substrates at room temperature using a pulsed microplasma source. They found the roughness exponent $\alpha = 0.64 - 0.68$ and growth exponent $\beta = 0.42 - 0.52$ and concluded that these exponents were not influenced by different cluster size but only affected by the presence of large particle in the cluster beam. Some growth dynamic models are there which can properly explain the mechanism during surface growth. One of such model is Kardar–Parisi–Zhang (KPZ) model, in which the dominant relaxation mechanisms are desorption and/or vacancy formation on the surface [10]. According to this model, the value of roughness exponent $\alpha_{KPZ} = 0.387$ and growth exponent $\beta_{KPZ} = 0.25$. Palasantzas et al [34, 35] found that their experimental roughness exponent $\alpha = 0.45 \pm 0.05$ which is close to $\alpha_{KPZ}$, but their experimental growth exponent value ($\beta$) is very much deviated from the KPZ growth exponent value for low energy Cu nanocluster deposited on Si substrates. Since KPZ model did not consider surface diffusion as relaxation mechanism, the surface diffusion that occurred cannot be explained by this model [10]. When surface diffusion is dominant over desorption or vacancy formation for relaxation.
mechanism, the Molecular Beam Epitaxy (MBE) model \[11\] comes into play. In this model the observed value of scaling exponents are \(\alpha_{\text{MBE}} = 0.67\) and \(\beta_{\text{MBE}} = 0.2\).

From the discussions of the above models, we see that our experimental first growth region with a growth exponent value \(\beta_1 = 0.26 \pm 0.01\) follows the KPZ growth exponent value \(\beta_{\text{KPZ}}\), but the roughness exponent value does not agree with that of the KPZ roughness exponent. A similar case is reported by Jeffries \etal\ [36] for Pt film deposited on glass substrates at room temperature using sputtering process. They found their experimental value of roughness exponent as \(\alpha \approx 0.9 \pm 0.02\) and growth exponent as \(\beta \approx 0.26 \pm 0.03\), which they attributed to the linear diffusion. However the probability of cluster diffusion on the surface at room temperature is expected to be insignificant in the present work. So for the first growth regime, interface growth of size-selected Ag nanoclusters can be compared with KPZ growth exponent.

In the second growth regime associated with higher deposition time \((t > 20\text{ min})\), the growth exponent value is found to be \(\beta = 0.85 \pm 0.15\), which indicates very rough surface. Similar to our experimental value, Yang and Xu [37] observed the global surface fluctuation of polycrystalline Cu film at 700 K and their experimental growth exponent is \(\beta = 0.88\). Due to higher substrate temperature they explained the surface growth with the help of bulk diffusion process. In the present case, we can completely deny the bulk diffusion process as the experiment was performed at room temperature. Since none of the well-established theory of scaling can describe the obtained experimental results for the second growth regime, we can think of other non-local effect such as shadowing effect. The shadowing effect mainly depends on the height factor of a surface and it is applicable only for random angular distribution of the incoming flux or tilting the substrate during deposition \[38–40\]. In this process, the surface undulation blocks the incoming flux from reaching the lower lying areas of the surface and thus, the hills receive more incoming particles and the valleys get only fewer particles. This allows the taller surface to grow faster at the expense of valleys. As a result, the overall surface will become much coarse.

A schematic view describing the effect of shadowing and re-emission process is given in figure 8. In the given figure, an incident beam of size selected nanoclusters heading towards the substrates at an oblique incident angle (\(\sim 45^\circ\)) is clearly shown. During early stage of deposition, incoming nanoclusters will randomly distribute over the surface. As a result, the surface will be covered with Ag-nanoclusters as well as nanoclusters aggregates or cluster islands. This self-made cluster islands will form a self shadowed region near the valleys and opposite to the beam direction. As a result no incoming nanocluster can directly reach the shadowed regions. For better explanation of the shadowing and re-emission process, consider A and B as two incoming nanoclusters. During deposition, A will reach the island at an oblique angle and will be captured by the taller surface of the island due to its high sticking co-efficient, where the cluster will be aggregated to the taller surface and the process is known as shadowing process. Due to shadowing effect, the overall roughness of the surface increases. In addition to that, some of the nanoclusters
for example B, cannot be captured by the higher surface of the islands due to its low sticking co-efficient or high kinetic energy. This incoming cluster will either bounce back from its impact point or re-emitted and aggregated in other surface points including valleys of the islands. The remission process basically occurs when the energy of the incoming beam is very high. Since we are dealing with low-energy deposition process, the probability of occurrence of re-emission in our deposition process is almost negligible. So, for a pure shadowing interface growth without any re-emission, the growth exponent value is found to be equal 1, which is also obtained from the simulation result \cite{41, 42}. In the present case, the growth of the size-selected Ag nanocluster film is found to be dominated by the shadowing effect which leads to a growth exponent value $\beta = 0.85 \pm 0.15$ close to 1. During deposition for shorter time, clusters are randomly deposited on the substrate which is found to follow the KPZ growth exponent. As the deposition time increases, the incoming beam clusters get deposited on the already deposited clusters. Here these already deposited clusters act as taller surface features or hills. As the deposition time further increases, the hills will receive more clusters from the incoming cluster beams and form bigger cluster islands at the expense of lower surface. This shadowing effect is also reflected from the height distribution graph shown in figure 3(d), where we observe that with increase of deposition time the height distribution becomes more bimodal in nature. From the particle size distribution, we get large cluster islands due to shadowing phenomena. Furthermore, along with the shadowing phenomenon, there is another factor called steering effect that originates from the attractive forces between incoming clusters and hills, which can influence the trajectory of the arriving clusters and incline them towards the hills on the surface.

In the conclusion, we have reported the growth dynamics of size-selected silver nanoclusters deposited on silicon (100) substrates at room temperature using inert gas condensation technique with a dc magnetron. It is found that, the deposited size-selected Ag clusters shows two types of growth regimes and the surface morphology is self-affine in nature. Two growth regimes are separated by a cross over time of 20 min. In the first growth regime, surface roughness is found very low and the growth exponent value is found close to the KPZ growth model. For the second growth region, shadowing effect is found predominant for the growth of size-selected Ag film. The roughness exponent is found to be constant during all the regimes. The bimodal height distributions of the samples for higher deposition time ($t > 20$ min) prove the shadowing phenomena that affects the surface morphology. Moreover the average reflectance of the surface increases with the increase of surface growth.

**Acknowledgment**

Two of the authors (PB and AD) would like to thank the University Grants Commission (UGC), India for their financial support. The authors gratefully acknowledge Prof. T. K. Chini and Prof. P. M. G. Nambissan for SEM and UV-spectrometry analysis, respectively. The authors also thank Ms. Soma Roy for performing UV-spectroscopy experiment. Finally, the authors thank Mr Debraj Dey for technical support for smoothly conducting the experiment in nanocluster source and XPS facility.

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