Size distribution of sputtered particles from Au nanoislands due to MeV self-ion bombardment

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Abstract

Nanoisland gold films, deposited by vacuum evaporation of gold onto Si(100) substrates, were irradiated with 1.5 MeV Au\(^{2+}\) ions up to a fluence of \(5 \times 10^{14}\) ions cm\(^{-2}\) and at incidence angles up to 60° with respect to the surface normal. The sputtered particles were collected on carbon coated grids (catcher grid) during ion irradiation and were analyzed with transmission electron microscopy and Rutherford backscattering spectrometry. The average sputtered particle size and the areal coverage are determined from transmission electron microscopy measurements, whereas the amount of gold on the substrate is found by Rutherford backscattering spectrometry. The size distributions of larger particles (number of atoms/particle, \(n \geq 1,000\)) show an inverse power-law with an exponent of \(-1\) in broad agreement with a molecular dynamics simulation of ion impact on cluster targets.

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I. INTRODUCTION

Although emission of clusters of atoms during ion sputtering was first observed almost half a century ago\(^1\), this phenomenon has attracted more attention in recent years, both theoretically and experimentally. Recent reviews on this topic are available in the literature.\(^2,3,4\) Various scenarios for cluster emission have been projected; these range from linear-cascade sputtering over surface evaporation to gasification (or volume evaporation).\(^5\)

For cluster emission, it has been shown\(^6,7\) that the size distribution, \(Y(n)\), of the emitted clusters obey an inverse power-law:

\[
Y(n) \propto n^{-\delta}
\]

where \(n\) is the number of atoms in a given cluster. For small emitted clusters \((n \lesssim 40)\) the reported values of \(\delta\) range between 4 and 8.\(^6,7\) For larger clusters \((n \geq 500)\) recently Rehn et al.\(^8\) reported a value of \(\delta = 2\), which is consistent with the model of Bitensky and Parilis.\(^9\) This value of \(\delta = 2\) is consistent with the mechanism that large clusters are produced when shock waves, generated by subsurface displacement cascades, ablate the surface. However, these results are for sputtering either from solid surfaces or from continuous films. For nanodispersed films one may expect different results as the sputtering phenomenon can be expected to be considerably stronger in small finite systems as nanometer sized islands. Kissel and Urbassek investigated sputtering from clusters by molecular-dynamics (MD) computer simulation where they modeled the materials effects occurring in a highly energized nonequilibrium cascade volume (i.e. the effect of energy spike).\(^10\) In this simulation they used 100 keV Au atom bombardment of spherical Au clusters (of 4 nm radius). Their results show a distribution of emitted clusters containing up to 100 atoms and even beyond. Smaller clusters (up to \(n \sim 10\)) show an inverse power-law [Eq. 1] with \(\delta = 2.3\). For larger cluster sizes, the decay appears to become even softer. This fall-off is distinctly slower than that observed for keV-ion bombardment of metal surfaces.\(^11,12\) This means that larger clusters are emitted with a higher probability from cluster targets compared to targets with planar solid surfaces. For the larger sizes \((n > 10)\) of emitted clusters, the value of \(\delta\) was not extracted from the simulation results.

To our knowledge, no experimental results of size distribution of emitted particles in ion bombardment of free clusters are available. However, ion bombardment of clusters
deposited on surfaces has recently been investigated experimentally,\textsuperscript{13,14} although no effort was made to verify the power-law dependence of $Y(n)$ and extract the value of the exponent. In this paper we present an effort to verify the power-law dependence of the large ($n \geq 1,000$) emitted particles from nanodispersed Au on silicon substrates due to MeV Au ion bombardment.

In earlier experiments, where particle ejection from ion-bombed nanoisland films was investigated, cluster ion beams instead of a monomer ion beam was used.\textsuperscript{13} The interaction of a cluster ion beam with solids may be quite different (nonlinear) in comparison to a monomer beam; this happens due to vicinage of atoms in the cluster beam.\textsuperscript{15} Thus the interaction of a cluster ion beam with nanoisland targets can in principle, produce a different distribution of ejected particles compared to the interaction of a monomer ion beam. Here we consider the simpler case of irradiation with a monomer ion beam. Our present study is concerned with the interaction of 1.5 MeV Au\textsuperscript{2+} (monomer) ions with Au nanoisland films.

Our earlier studies have shown that when nanodispersed targets are bombarded by 1.5 MeV Au ions, the size and coverage of sputtered particles collected on a catcher grid are larger compared to thicker continuous film targets.\textsuperscript{14} We observed also higher probability of crater formation in island thin films compared to thick continuous films due to MeV self-ion irradiation and proposed it as experimental evidence of the energy spike confinement in nanoisland targets.\textsuperscript{16,17,18} An energy spike confinement in the nanoislands can result in either a thermal spike or a shock wave spatial confinement.

\section{II. EXPERIMENTAL}

Au films of thickness $1.3 \pm 0.1$ nm were deposited by thermal evaporation under high-vacuum conditions ($\approx 2 \times 10^{-6}$ mbar) on native-oxide covered Si(100) substrates at room temperature (RT). Deposition rate was 0.1 nm/s. Before thin film deposition, substrates were ultrasonically cleaned in acetone, methanol, trichloroethylene, methanol, deionized water and in acetone sequentially. Ion sputtering experiments were performed with 1.5 MeV Au\textsuperscript{2+} ions with fluences ranging from $1 \times 10^{12}$ to $5 \times 10^{14}$ ions cm$^{-2}$ at different angles of incidence with respect to the surface normal. The incident ion current (over a scanned beam area of $1 \times 1 \text{ cm}^2$) was kept between 20 to 40 nA under secondary electron suppressed geometry. During irradiation, the sputtered particles were collected on a catcher grid that
was positioned \( \approx 1 \) cm above the target, the catcher surface making an angle \( \approx 15^\circ \) with respect to the sample surface. Such a large distance of the catcher is to reduce overlap of the collected particles on the catcher grid. During irradiation, \( \approx 2 \times 10^{-7} \) mbar pressure was maintained in the irradiation chamber. Transmission electron microscopy (TEM) measurements were performed (using a JEOL JEM-2010 (UHR) electron microscope operating at 200 kV) on the ion-bombarded samples to study the changes in the morphology of the nanostructures on the substrates and to determine the surface coverage and the size distribution of the sputtered particles on the catcher grids. The effective thickness of the as-deposited Au films on Si substrates has been determined by Rutherford backscattering spectrometry (RBS) using the bulk atomic density of Au and the RUMP simulation package.\(^{19}\)

III. RESULTS AND DISCUSSIONS

The deposited Au grows as islands on native-oxide covered Si substrate. Fig. 1 shows a plan-view TEM micrograph of such a sample and a histogram for the particle size distribution. The distribution is fitted well by a Gaussian function. It is found that the Au islands are isolated with an average particle size of 11.1 \( \pm 0.1 \) nm with a standard deviation, \( \sigma = 5.1 \pm 0.3 \) nm and the surface coverage of islands is \( \sim 26\% \). These values are obtained using several TEM micrographs and the ImageJ analysis package.\(^{20}\)

In the description of the following results, the as-deposited nanoisland Au films that have been ion-bombarded will be called targets. Figs. 2(a) and (b) show TEM images from the target and from the catcher grid, respectively, after irradiation (fluence: \( 1 \times 10^{14} \) ions cm\(^{-2} \)) at 0\(^\circ\)-impact angle (with respect to the surface normal). Fig. 2(c) is a TEM image from the same region as in 2(b) of the catcher grid obtained by some underfocusing of the objective lens. It is evident from Fig. 2(c) that the sputtered Au particles are three-dimensional (spherical in most cases) in nature. The particle size distribution on the catcher grid is shown in Fig. 2(d). The frequency distribution of the particle size in Fig. 2(d) and in the latter figures are obtained by measuring particle size from many TEM images (\( \sim 700 \) particles) from each catcher grid. Following ion irradiation, the surface coverage of islands on the target decreases from 26\% to 7\%.

Figs. 3(a), (b), (c), and (d) show plan-view TEM micrographs from the target after irradiation at 30\(^\circ\)-impact angle with fluences of \( 1 \times 10^{12}, 1 \times 10^{13}, 1 \times 10^{14} \) and \( 5 \times 10^{14} \) ions
cm$^{-2}$, respectively. It appears from the micrographs that in the initial stage of irradiation, particles get agglomerated and elongated. For an ion fluence of $1 \times 10^{12}$ ions cm$^{-2}$, surface coverage of islands increases from 26% to 34% and then it reduces to 18% for a fluence of $1 \times 10^{13}$ ions cm$^{-2}$ and 9% for a fluence of $1 \times 10^{14}$ ions cm$^{-2}$. Finally, at a fluence of $5 \times 10^{14}$ ions cm$^{-2}$, islands are embedded into the Si substrate.

Figs. 3(e), (f), (g), and (h) show plan-view TEM micrographs of sputtered particles collected on catcher grids as a result of irradiating the target to the fluences of $1 \times 10^{12}$, $1 \times 10^{13}$, $1 \times 10^{14}$ and $5 \times 10^{14}$ cm$^{-2}$, respectively. The corresponding surface coverage of islands are 2.6%, 5.1%, 6.2% and 19.5%, respectively. They correspond to the targets in (a), (b), (c), and (d), respectively. The corresponding particle size distributions on the catcher grids are shown in (i), (j), (k), and (l), respectively. The size distribution can be fitted to a log-normal function as described later. The embedding process already starts at a fluence of $1 \times 10^{14}$ ions cm$^{-2}$. The slightly smaller particle size observed for the highest fluence (see Table-1), is perhaps an indication that the probability of emission of smaller particles is higher from the embedded particles on the target.

Figs. 4(a), (b), and (c) show plan-view TEM micrographs from the target after irradiation at 60°-impact angle with fluences of $1 \times 10^{12}$, $1 \times 10^{13}$ and $1 \times 10^{14}$ ions cm$^{-2}$, respectively. For the fluence of $1 \times 10^{12}$ ions cm$^{-2}$, the surface coverage of islands increases from 26% (for as-deposited sample) to 37% and then reduces to 12% for $1 \times 10^{13}$ ions cm$^{-2}$, and finally at a fluence of $1 \times 10^{14}$ ions cm$^{-2}$, islands are embedded into the Si substrate. Compared to 30°-impact angle, here the surface coverage of Au particles on the target reduces more rapidly with ion fluence. The embedding also begins to occur at a smaller fluence for the 60°-impact angle of the ion beam. It appears that embedding has already begun at a fluence of $1 \times 10^{13}$ ions cm$^{-2}$. Figs. 4(d), (e), and (f) show plan-view TEM micrographs of sputtered particles collected on catcher grid. They correspond to the targets in (a), (b), and (c), respectively. The particle size distributions obtained from the catchers are shown in (g), (h), and (i), respectively.

From the cross-sectional TEM images in Fig. 5 it is evident that during ion irradiation the nanoparticles initially (for fluence: $1 \times 10^{12}$ ions cm$^{-2}$) spread over the surface. At a higher fluence ($1 \times 10^{13}$ ions cm$^{-2}$) agglomeration is dominant and larger islands are produced. At a fluence of $1 \times 10^{14}$ ions cm$^{-2}$ almost all the islands get embedded into the Si substrate. All the embedded particles [Fig. 5(d)] appear to be large. However, one small particle is
seen in Fig. 5(d), which is not embedded. It is also evident that at lower fluences when the particle size is small, there is no embedding. This raises a question: is there any critical size of islands on the target so that there is no embedding below this size? This aspect is to be addressed in future investigations.

The size distribution of islands on the as-deposited sample [Fig. 1(b)] is fitted well by a Gaussian function. The size distributions of islands, collected on the catcher grid following ion irradiation of the as-deposited sample, [Figs. 2(d), 3(i)-(l) and 4(g)-(i)] are found to fit a log-normal function:

\[ P(x) = \frac{1}{\sqrt{2\pi}xw} e^{-\left(\frac{\ln(x/x_c)}{\sqrt{2}w}\right)^2} \]

where, \( x_c \) is the most probable size and \( w \) is the width of the size distribution of the particles. By fitting the frequency plot using Eq. 2, we have estimated the most probable particle size \( x_c \) and width \( w \). The results are presented in Table 1. In order to confirm that the collected nanoparticles on the catcher grid are not Si clusters, we irradiated a bare Si substrate (without any deposited Au) to a fluence of \( 1 \times 10^{14} \) ions cm\(^{-2}\). However, no sputtered particle was observed on the catcher grid in this case. In addition, we have carried out lattice imaging by high-resolution TEM to confirm that the sputtered particles are Au nanoclusters.

The sputtered nanoparticles on the catcher grid are usually spherical (as described earlier). For the lowest ion fluence \( (1 \times 10^{12} \) ions cm\(^{-2}\)), the density of islands on the catcher grid [Figs. 3(e) and 4(d)] is low and the island size distribution, although fitted by a log-normal function, actually does not deviate much from a Gaussian function. For higher ion fluences, apparently there is ejection of more larger sized particles [cf. for example, Figs. 3(f), (j) and 4(e), (h)]. This appears to be correlated with the increase of particle size on the target [cf. Figs. 3(b) and 4(b)]. This correlation has earlier been observed by Baranov et al.\(^{13}\), who performed experiments on nanodispersed targets with selected particle sizes. Thus, for ion bombardment at higher fluences the size distributions develop a tail extending into larger sizes and a log-normal function fits the distribution better.

In order to understand the mechanism involved in the ejection of particles in ion-target interaction, many authors have predicted different power-laws. According to Eq. 1, the size distribution, \( Y(n) \), of the sputtered particles obeys an inverse power-law.\(^6,7\) The value of the exponent \( \delta \), in Eq. 1 depends on the mechanism of ion-target interaction. Experimental
determination of the value of $\delta$ exists in the literature only for solid surfaces and continuous film targets. There are conflicting results in the literature. The exponent, $\delta$, was found to correlate with the total sputtering yield, such that higher sputtering yields result in smaller values of $\delta$. Whereas Rehn et al. reported that the value of $\delta$ is independent of sputtering yield. Using mass-spectrometry techniques, the observed values of $\delta$ range between 4 and 8. However, these values are for smaller ejected clusters of $n \lesssim 40$ atoms. A high exponent means that smaller clusters are present with high probabilities in the mass-spectrometry studies. It is suspected that some large clusters get fragmented to increase the number of small clusters. Rehn et al. restricted their studies to large clusters ($n \geq 500$) and obtained a value of $\delta = 2$. Consistent with a theoretical model this value of the exponent indicates that the large clusters are produced when shock waves, generated by subsurface displacement cascades, ablate the surface. The shock wave model proposed by Bitensky and Parilis predicts the inverse-square dependence of $Y(n)$. On the other hand no simple theoretical model has been able to reproduce both the power-law and high values of $\delta$ found from mass-spectrometry techniques. All these results discussed in this paragraph are for solid surfaces and continuous thin film targets.

For cluster targets MD simulation of Kissel and Urbassek predicts a value of $\delta \approx 2.3$ for small clusters ($n \lesssim 10$). For larger clusters ($n > 10$) the decay appears to become softer, although they have not tried to extract the value of $\delta$ from this region. To our knowledge, this is the only theoretical work where cluster ejection in an ion-cluster interaction has been considered. We try to compare our results with these simulation results. We have fitted the larger cluster region ($n > 10$) of the spectrum from Ref. 10 and obtained a value of $\delta \approx 1.15$. The plot of data with the fit is shown in Fig. 6.

Now we present our results of size distributions of the larger clusters ($n \geq 1,000$) in terms of the inverse power-law dependence of $Y(n)$ on $n$. The particle size distribution, $Y(n)$, i.e. the number of clusters containing $n$ atoms, was determined from the catcher grid as follows. The size of each particle was determined using ImageJ software. The measured lateral dimensions were converted into the number of atoms for a given cluster by assuming that the particles are spherical with the density of bulk Au. All visible particles in an area of $\approx 3 \times 10^5 \text{ nm}^2$ (ten frames, each of $\sim 210 \times 160 \text{ nm}^2$) were sized. The particles were grouped into bins having 1 nm steps. The procedure was repeated for each case of irradiation. The size distributions that were obtained in this manner are displayed in Fig. 7 along with a
power-law fit. The values of the exponent $\delta$, although show some variations, broadly are close to that of the theoretical value shown in Fig. 6. Nevertheless, one notices that for the lowest fluence ($1 \times 10^{12}$ ions cm$^{-2}$) where the size distributions of the emitted particles [Fig. 3(i) and 4(g)] are nearly Gaussian and the presence of larger size particles is not prominent, the value of the exponent $\delta$ is larger. One may also note that the particle sizes on the target in these cases are also smaller.

Studies of ejected particle distribution from nanoisland targets must be explored further both theoretically and experimentally. In the simulation of Kissel and Urbassek$^{10}$, electronic stopping of the ions in the target island has been disregarded. The electronic energy loss ($S_e$) and the nuclear energy loss ($S_n$) for 1.5 MeV Au ions in a Au target are 2.41 keV/nm and 9.53 keV/nm respectively, as obtained by using the SRIM 2003 code.$^{22}$ It is important to note that, since $S_n \approx 3.9 S_e$, although the effect of $S_n$ will be dominant, contributions to sputtering from both mechanisms could be possible. Some MD simulations show a dependence of sputtering yield on $S_e$. It is to be investigated how the inclusion of the effects of both $S_n$ and $S_e$ affect the value of $\delta$.

In order to explain the nature of the observed size distributions (Fig. 7), i.e., $Y(n)$ rising at the beginning, following inverse power-law at the middle and then falling faster at the end. We refer to the work of Baranov et al.$^{13}$ They have reported sputtering of nanodispersed targets and shown that the size distributions of ejected clusters are systematically shifted towards smaller sizes in comparison with the island size distributions on the targets. For example average island sizes of 4.5±1.2 nm and 6.7±2.5 nm on the target led to average island sizes of 3.6±1.1 nm and 4.4±1.6 nm, respectively, of the ejected particles. So, experimentally the general trend is that smaller particles on the target produces smaller ejected particles and the larger particles on the target produces larger ejected particles. The simulation of Kissel and Urbassek$^{10}$ shows that for a target particle of a given size, a size distribution of ejected particles is generated. Combining these two results (experimental and theoretical) one would expect that when the target particle is larger a distribution of particles with the same exponent but shifted to a larger average size would be generated. Using this concept we can explain the nature of our observed $Y(n)$ versus $n$ curves. We have illustrated this in Fig. 8. We have generated several plots with the same exponent and then shifted the curves along the $n$-axis to mimic contributions from particles of different sizes, $D$ ($D_1 < D_2 < D_3 < D_4 < D_5$), representing the diameter of the particle on the target. Then
we added all the sets of data. The resultant curve (solid curve) shows the same nature as our experimental curves. This explains why with increasing \( n \) experimental data show the rising nature of \( Y(n) \) at the beginning, power-law fall in the middle and a faster fall at the end. This behavior is prominently seen in Fig. 7(a) \([1 \times 10^{14} \text{ ions cm}^{-2}]\) and in Fig. 7(b) \([1 \times 10^{13} \text{ ions cm}^{-2}]\). Kissel and Uebassek have done the simulation only for a single size particle (4 nm radius). It would be useful to have simulation results for different sizes of target islands.

IV. CONCLUSIONS

We have investigated the distribution of ejected (sputtered) particles from nanoislands in monomer ion interaction with nanodispersed solid targets. The ejected cluster size distribution as a function of number of atoms per ejected particle shows a power-law decay, \( Y(n) \sim n^{-\delta} \), with \( \delta \approx 1 \). The exponent is in broad agreement with a molecular dynamics simulation where the effect of an energy spike in the target islands is considered. In the simulation, as neither serious effort has been made to extract the exponent for larger ejected islands nor the size dependence of target islands has been considered, more theoretical input is required to understand the distribution of sputtered particles and the mechanism of sputtering.

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FIGURE CAPTIONS

FIG. 1: (a) A plan-view TEM image from an as-deposited film (effective thickness: 1.3 nm) showing Au nanoparticles on a Si substrate and (b) histogram showing particle size distribution. The histogram shown in (b) is fitted with a Gaussian distribution to determine the average particle size ($x_c$) and the standard deviation ($\sigma$) in the size distribution.

FIG. 2: Plan-view TEM images: (a) Au particles on the target following 1.5 MeV Au$^{2+}$ (fluence: $1 \times 10^{14}$ ions cm$^{-2}$) bombardment at an impact angle of 0$^\circ$, (b) sputtered particles on the catcher grid, (c) sputtered particles on catcher grid showing spherical nature of the particles after some underfocousing of the objective lens. (d) histogram showing size distribution of sputtered particles.

FIG. 3: Plan-view TEM images from the target (a, b, c, d) following 1.5 MeV Au$^{2+}$ bombardment with an impact angle of 30$^\circ$, from the catcher grid (e, f, g, h) and histograms (i, j, k, l) showing the size distribution of the sputtered particles on the corresponding catcher grids. Each row in the figure corresponds to a given ion fluence: (a), (e), (i) - $1 \times 10^{12}$ ions cm$^{-2}$; (b), (f), (j) - $1 \times 10^{13}$ ions cm$^{-2}$; (c), (g), (k) - $1 \times 10^{14}$ ions cm$^{-2}$; and (d), (h), (l) - $5 \times 10^{14}$ ions cm$^{-2}$.

FIG. 4: Plan-view TEM images from the target (a, b, c) following 1.5 MeV Au$^{2+}$ bombardment at an impact angle of 60$^\circ$, from the catcher grid (d, e, f) and histograms (g, h, i) showing the size distribution of the sputtered particles on the corresponding catcher grids. Each row in the figure corresponds to a given ion fluence: (a), (d), (g) - $1 \times 10^{12}$ ions cm$^{-2}$; (b), (e), (h) - $1 \times 10^{13}$ ions cm$^{-2}$; and (c), (f), (i) - $1 \times 10^{14}$ ions cm$^{-2}$.

FIG. 5: XTEM images from the target for the case of 60$^\circ$-impact angle of ion bombardment: (a) an as-deposited nanodispersed Au film; (b), (c) and (d) from films irradiated with a fluence of $1 \times 10^{12}$ ions cm$^{-2}$, $1 \times 10^{13}$ ions cm$^{-2}$ and $1 \times 10^{14}$ ions cm$^{-2}$, respectively.

FIG. 6: Data taken from Ref.10 and fitted with a power-law given in Eq. 1, to get the exponent ($\delta$) for larger sputtered particles.
FIG. 7: Measured number of collected nanoparticles as a function of nanoparticle size (n) for 1.5 MeV Au ion irradiation: (a) for its 30°-impact with various fluences shown in Fig. 3(e)-(h); (b) for its 60°-impact with various fluences shown in Fig. 4(d)-(f); (c) for a fluence of $1 \times 10^{14}$ ions cm$^{-2}$ for various impact angles.

FIG. 8: Illustration of the nature of the observed distribution of the ejected particles [$Y(n)$ vs. $n$ curves]: Plots of the power-law-generated data for five different particle sizes (diameter) on the target [$D_1 < D_2 < D_3 < D_4 < D_5$]. These five sets of data were added to obtain the resultant yield to explain the nature of our experimental data, i.e. rising nature at the beginning, a power-law decay in the middle and a faster fall at the end (see text for details).
TABLE I: Size distribution of the sputtered particles for different fluences and impact angles

| Fluence, $\phi$ (ions cm$^{-2}$) | Average Size (30°) nm | Average Size (60°) nm |
|-------------------------------|------------------------|------------------------|
| $1 \times 10^{12}$ x | $x_c$: 7.61±0.86 $w$: 0.22±0.17 | $x_c$: 6.28±0.64 $w$: 0.30±0.13 |
| $1 \times 10^{13}$ x | $x_c$: 7.67±0.80 $w$: 0.33±0.14 | $x_c$: 7.00±1.39 $w$: 0.60±0.44 |
| $1 \times 10^{14}$ x | $x_c$: 7.54±0.85 $w$: 0.35±0.15 | $x_c$: 6.24±1.06 $w$: 0.51±0.28 |
| $5 \times 10^{14}$ x | $x_c$: 7.07±0.85 $w$: 0.45±0.14 | —— |

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