



FRONTIERS ARTICLE

Effect of sample rotation on surface roughness with keV C₆₀ bombardment in secondary ion mass spectrometry (SIMS) experimentsBarbara J. Garrison^{a,*}, Zbigniew Postawa^b^a Department of Chemistry, Penn State University, University Park, PA 16802, USA^b Smoluchowski Institute of Physics, Jagiellonian University, ul. Reymonta 4, 30-059 Krakow, Poland

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ABSTRACT

The simplicity of interpreting depth profiling in SIMS experiments is often limited by sample damage and surface roughness that accompany the ion bombardment process. Molecular dynamics simulations are implemented to obtain mechanistic insight into the improvement of depth profiles due to sample rotation during keV C₆₀ bombardment of solids. The simulations show that sample rotation decreases the RMS roughness of the sample compared to a single azimuthal angle of incidence, as observed by experiment. The improvement is most noticeable for near-grazing angles of incidence. Bombardment of the sample at these angles builds up an anisotropic topology which sample rotation at least partially removes.

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1. Introduction

The emergence of energetic cluster beams as erosion sources in secondary ion mass spectrometry (SIMS) experiments has spurred interest in performing molecular depth profiling experiments [1,2]. Although elemental depth profiling has been a mainstay of the SIMS community for decades, obtaining molecular information as a function of depth has not been possible due to beam induced chemical damage. The cluster sources in contrast to atomic ion sources have been shown to remove material from a molecular solid without significant chemical damage accumulation and with a depth resolution of 10–30 nm. These unique properties have expanded applications to the study of organic light emitting diode structures [3], buried lipid bilayers [4] and the localization of metabolomes within a single cell [5], to name a few examples. The ideal scenario is that the material is removed from the substrate in a layer-by-layer fashion. Naturally, this idealized process is not observed and several investigations have been initiated in order to optimize experimental conditions necessary to achieve the best molecular depth profiles. The natural measure of depth profiling quality is the interface width in layered systems. Numerous quantities such as energy of the incident cluster beam, polar angle of incidence, cluster size, and substrate temperature have been shown to influence the apparent interface width [4,6–8]. An intriguing observation is that rotation of the sample during erosion improves the depth resolution for C₆₀ bombardment of Irganox delta layers [9,10] as well as polymer layers [10].

The concept of sample rotation to improve Auger Electron Spectroscopy (AES) depth profiles stems from a study by Zalar who showed that sample rotation improves the depth resolution as measured by interface widths achievable for polycrystalline atomic solids when bombarded by atomic projectiles [11]. Sample rotation enabled a more uniform rate of sputtering that averaged out differential yields to due various exposed microplanes [12]. Subsequently, sample rotation was used in SIMS depth profiles with atomic bombardment [13]. Vastly different yields for various crystal faces, however, are not expected for cluster bombardment. In fact, depth profiles of polycrystalline NiCr layered structures have been performed successfully with C₆₀ bombardment without sample rotation [14,15]. Since sample rotation or the use of C₆₀ cluster beams independently decreases the interface width in depth profiles of NiCr layered structures, it is unclear how sample rotation affects the depth resolution when used in conjunction with cluster beams such as C₆₀.

Over the years, molecular dynamics (MD) computer simulations have been a theoretic partner to the SIMS experiments providing insight even when there is not an exact match between the experimental and computational systems [16,17]. For example, simulations of individual impacts of Ga and C₆₀ bombardment of Ag(1 1 1) almost a decade ago [18] have provided the vision for molecular depth profiling experiments [1]. The simulations demonstrate that the 15 keV Ga projectile penetrates tens of nm into the sample and thus destroys molecular information. The 15 keV C₆₀ projectile, on the other hand, only penetrates 3–4 nm into the solid and only disrupts a depth of 5–7 nm. This limited range of damage suggests that a subsequent C₆₀ impact has the potential to remove undamaged molecules in concurrence with molecular

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depth profiling experiments. Later calculations of C_{60} bombardment of molecular solids [19–21] confirm the predictions of the penetration and damage depth from the simulations on Ag. Even though these simulations of individual impacts of C_{60} bombardment are insightful for molecular depth profiling, the experiments involve repetitive impacts on the surface. Thus, it is desirable to match better the calculation protocol with the experiment one. The computational challenge for repetitive bombardment is that a much larger sample is required, resulting in a significant increase in computational time even for one impact. Recently, however, a divide and conquer strategy has been developed to model repetitive bombardment of solids [22]. Thus, depth profiling calculations are now tractable albeit still for an atomic solid, Ag [22–24]. Since the simulations of an individual impact of C_{60} on Ag provided a vision for molecular depth profiling, it is worth exploring if the depth profiling calculations on Ag give insight into the experimental trends of molecular depth profiling and thus, whether they can give insight into the experimental observation that sample rotation improves the depth resolution.

The first experimental observation is that lowering the kinetic energy (KE) of the C_{60} projectile in the range of 5–80 keV decreases the interface width and thus improves the depth profile in lipid bilayers [6]. Simulations of individual impacts of C_{60} on Ag at different kinetic energies show that the craters formed look quite similar and that the amount of material removed increases with increasing KE [18]. Thus, simulations of individual impacts do not make a direct statement about the quality of the depth profile. Simulations of repetitive bombardment as a function of KE, however, allow for the calculation of RMS roughness of the surface, an indicator from the simulations that is used for monitoring quality of the depth profile [25]. The RMS roughness in the simulations is found to increase with increasing KE. Simplistically, the incident C_{60} projectile creates a crater, removing material from the center and mixing material around the edges. The higher the KE, the more material that is removed and the greater the amount of mixing. Both the bigger crater and more mixing give rise to a larger RMS roughness. Thus, the better quality depth profile should occur at lower incident KE. A similar conclusion has been reached in simulations of large Ar cluster bombardment on an inorganic semiconductor Si [26]. This mechanistic analysis from simulations on an atomic target logically should also be valid for molecular targets, thus validating insights gained from one substrate in the simulation to experiments on a different substrate. As a caveat, both the simulations and experiments only go down to 5 keV incident KE. As the KE is lowered further, the dynamics can change [26]. In particular, for molecular depth profiling, the amount of material removed must remain larger than the amount of damaged layer [27].

The next experimental trend addressed by simulations is that of improving the depth profile by making the angle of incidence more grazing for cholesterol films [7]. This observation is fortunate since the commercial instruments tend to use incident angles between 45° and 75° from the surface normal. Simulations of individual cluster impacts on a benzene target indicates that the craters formed by 75° are shallower with less disruption than with normal incidence [28,29]. Simulations of repetitive bombardment of C_{60} on Ag(1 1 1) find a smaller RMS roughness for 70° rather than 0° incidence [23,24]. Again, the mechanistic analysis for repetitive bombardment on a Ag surface combined with individual impacts on an organic solid give credence to the explanation to the experimental trends for depth profiling on cholesterol.

The third trend to consider is depth profiling by different cluster beams, most notably C_{60} , Au_3 (or Bi_3) and large Ar clusters. Naturally, the choice of beam gets intimately interconnected with which vendor or laboratory has one or another choice of cluster source. First, Au_3/Bi_3 like atomic projectiles, has a range of tens

of nm in organic solids [19]. Thus, this projectile damages molecules deep in the sample and also builds-up implanted Au [30]. Repetitive bombardment simulations of Au_3 , C_{60} and Ar_{872} on Ag(1 1 1) at normal incidence and at an incident KE of 20 keV give that the RMS roughness is smallest for Ar_{872} and largest for Au_3 [24]. Thus, the simulations indicate that the best depth profiles will be obtained with the Ar cluster beams relative to C_{60} and Au_3 . Recent experiments of large Ar cluster depth profiling of the Irganox delta layer system shows clearly that the large Ar clusters give better quality depth profiles than C_{60} clusters at the same incident KE [8].

The successes of using MD simulations for repetitive bombardment of cluster projectiles on an atomic Ag(1 1 1) surface to interpret molecular depth profiles gives us confidence that the reasons behind the improvement of depth profiles with sample rotation can be ascertained. In fairness, it should be pointed out that one experimental trend has not been modeled with MD simulations, namely the observation that reducing the sample temperature improves the quality of the depth profile [4,6,9]. This effect is experimentally quite important for molecular solids. The effect is probably tied to the low cohesive energy of the substrate, making it easy to move material in one impact or the amount of diffusion between impacts. Both effects are difficult or impossible to model with MD simulations.

The aim of this study is to use molecular dynamics (MD) simulations to investigate the effect of sample rotation on depth profiling with energetic C_{60} cluster beams in order to obtain mechanistic insight as to why it might be effective. The cluster C_{60} is chosen rather than Au_3/Bi_3 or Ar_{1000} because it is intermediate in size and is the only cluster beam used to date in SIMS experiments implementing sample rotation. In order to extract only the influence of sample rotation, we choose to use a tightly bound single crystal atomic solid, Ag(1 1 1), at zero Kelvin, instead of a molecular solid, for two specific reasons. First, the atomic solid eliminates the complexity of including a molecular solid that involves chemical effects and reactions. Second, temperature effects in more weakly bound molecular targets also have a pronounced affect on depth resolution [4,6,9]. Even though the Ag target is different than the molecular solids of experimental interest, the dynamics of C_{60} bombardment on a variety of targets is sufficiently similar [16] that prior experimental data have been extensively interpreted using simulations on the Ag system. Finally, these calculations are not applicable to systems where ripples are formed [31].

2. Calculation

The challenge for performing MD simulations of depth profiling is that the sample size required for the simulation is larger than can be used even for one impact. Moreover, the depth profiling simulations require multiple impacts on the surface to achieve a measurable erosion of the sample. To circumvent this dilemma, MD simulations are performed using the divide and conquer approach for depth profiling that has been described previously [22,23]. Briefly, independent impacts are calculated for a subset of the atoms in the system. After each impact is calculated, the atom positions in the master sample are updated. In this manner, it is tractable to perform simulations up to fluences of almost 10^{14} impacts per cm^2 .

A major assumption in these simulations is that the system temperature is zero Kelvin. Each subsystem is quenched before reinsertion into the master sample. For a finite temperature system, all motions that would occur between subsequent impacts by a projectile should be incorporated. For a beam current of 1 nA in a $20 \times 20 \mu m$ spot size, the time between impacts at one crater is on the order of milliseconds. Numerous diffusion

processes can occur in this time frame and these are not included in the simulation. Since chemical damage build-up is greatly reduced at reduced temperature, we assume that our models are most appropriate for this regime.

The system investigated is C_{60} bombarding a Ag(1 1 1) sample of five million atoms with a width of 53 nm. In previous studies, we have used beam conditions that range from 5 to 20 keV and three different incident polar angles, 0° , 45° and 70° [23]. For these studies, 20 keV C_{60} is examined at the three different polar angles. In order to perform the equivalent of sample rotation in experiment, the azimuthal angle of incidence is randomly chosen for each impact.

The effectiveness of a set of conditions in the simulations for depth profiling has been measured with the mean surface position (Z_{mean}) and the RMS roughness, that is the first and second moments of the height distribution [22,23]. These quantities are calculated using a grid of 128×128 points on the surface, resulting in a pixel size of 0.41×0.41 nm. For the analysis here, three additional metrics are employed. The first two are the third and fourth moments of the height distribution, Skewness and Kurtosis, respectively. Both are dimensionless quantities. A Kurtosis value of three implies a Gaussian distribution. The last metric is the number of times that the height crosses the mean surface level in each row or column of the grid. This quantity is measured in crossings per nm. The coordinate system is oriented such that the x -direction is parallel to the incident beam and the y -direction is perpendicular to the incident beam.

3. Results

The average surface position and RMS roughness vs fluence and number of impacts are shown in Figure 1 for 20 keV C_{60} bombardment at three polar angles (0° , 45° and 70°) for a single azimuthal direction and two polar angles (45° and 70°) with sample rotation. The average surface height, Z_{mean} , is approximately linear with fluence above 10^{13} impacts/cm². In this regime the sputtering yield or removal rate is proportional to the slope of the line. The larger the slope of Z_{mean} vs fluence, the larger the sputtering yield. In all cases, the RMS roughness reaches a plateau within $\sim 2 \times 10^{13}$ /cm². The yield for the simulations at 0° and 70° with a single angle of incidence have been reported earlier to be 342 ± 17 and 258 ± 14 atoms per impact, respectively [23].

The effect of a random angle of incidence at 45° and 70° is to increase slightly the value of Z_{mean} (and thus the sputtering yield) and to decrease the RMS roughness as shown in Figure 1. The directions of these changes are consistent with those found in experiment [9,10]. The first step in the analysis is the surface topology for 70° incidence at a fluence of 3.7×10^{13} impacts/cm² for both the single and random azimuth simulations given in Figure 2. Bright orange/red areas are hills and dark blue regions are valleys. The azimuthal angle of incidence is from the left for the single incident angle simulation. It appears that there are longer trenches and valleys parallel to the beam for the single azimuth simulation and that the peaks and valleys are more extended than for the random azimuth simulations. In order to quantitate the differences, additional measures of the surface topology are used.

The height distributions of the surfaces at 3.7×10^{13} impacts/cm² at a polar angle of 70° are shown in Figure 3 for an area extracted from 128×128 pixels. Positive values of height point into the sample and negative values of height point towards the vacuum. As is visually seen in Figure 2, the range of the height distribution (high to low) is greater for the single azimuth than the random azimuth simulation. Sample values of all quantities for a single fluence are given in Table 1. The value of Skewness decreases with the impact angle but in all cases it is small. Sample rotation does not seem to have any consistent effect on this quantity. For both off normal impact angles, the Skewness is negative indicating that the hills in the distribution are slightly higher relative to Z_{mean} than the valleys are deep. The value of Kurtosis increases with the angle of incidence for a non-rotating sample. The value of Kurtosis for the random azimuth simulation is three, indicating a near Gaussian distribution. The values of Skewness and Kurtosis as a function of fluence exhibit a similar onset as shown in Figure 1 for mean surface position and RMS roughness (figure is not shown here).

While the effect of the ion beam on the anisotropy of the vertical distribution of the surface structures is small, a much larger effect can be seen in the anisotropy of the lateral dimensions of the structures created at the surface. The lateral anisotropy is shown in Figure 4 and is represented as the number of the number of times that the height crosses the mean surface level in each row or column of the sampling grid. For the simulations with a single azimuthal direction, the number of crossings in the direction perpendicular to the beam (y -direction) is greater than that in the direction parallel to the beam (x -direction) as shown in Figure 4a and b for 45° and 70° incidence, respectively. This

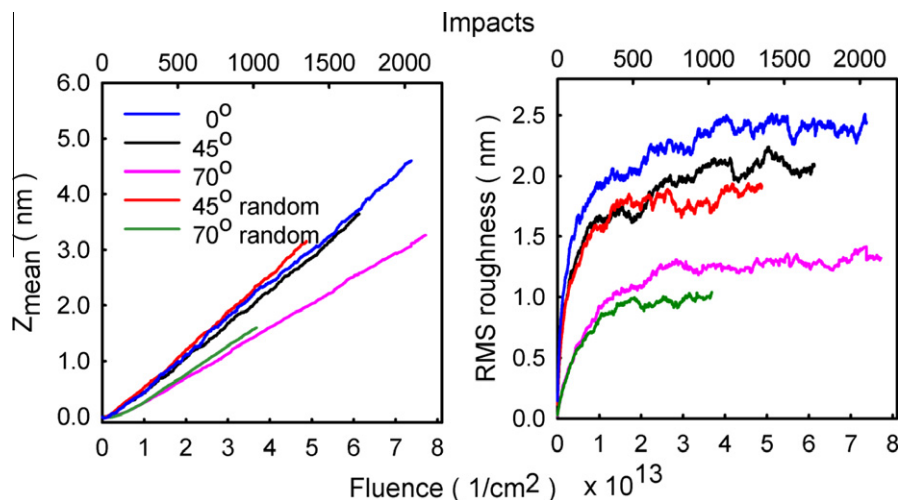


Figure 1. Mean surface height and RMS roughness vs fluence for 20 keV C_{60} bombardment of Ag. Three angles of incidence are used. (a) Mean surface height, Z_{mean} . (b) RMS roughness.

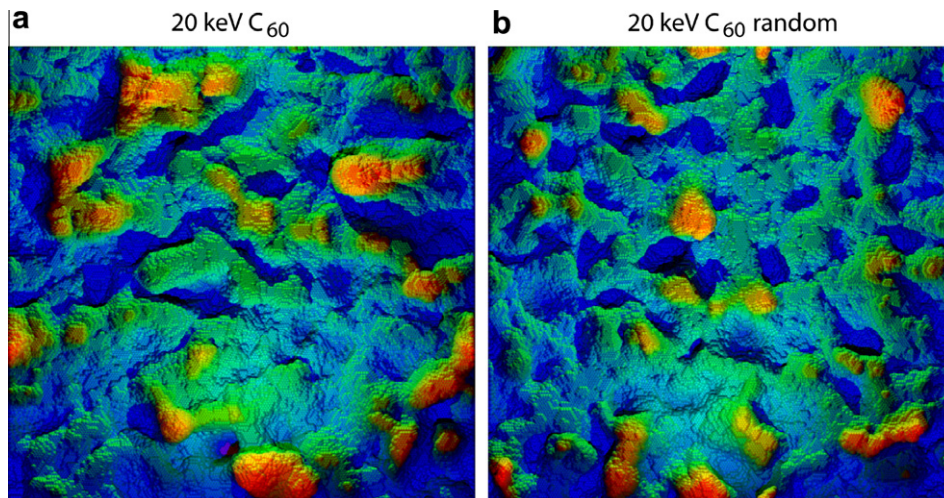


Figure 2. Snapshots of the surface at a fluence of 3.7×10^{13} impacts/cm² for 70° incidence. (a) Single azimuth of impact from the left. (b) Random azimuth of impact.

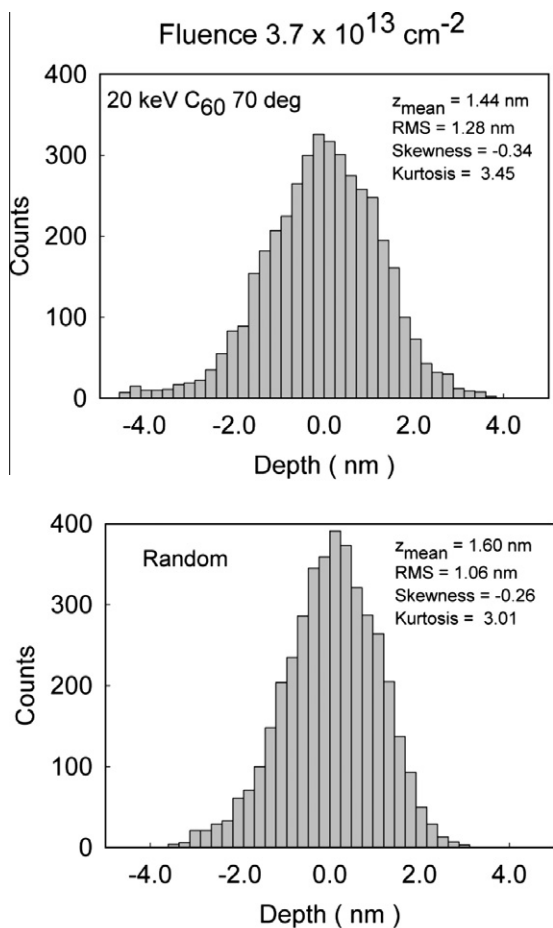


Figure 3. Height distribution at a fluence of 3.7×10^{13} impacts/cm² for 70° incidence. The depth scale is relative to the mean surface height, z_{mean} . Positive values point into the sample and negative values point into the vacuum. (a) Single azimuth of impact from the left. (b) Random azimuth of impact.

confirms the visual observation seen in Figure 2a that trenches and ridges are formed parallel to the beam direction for a single incident azimuthal direction. For the random azimuthal angle simulations, the number of crossings in each direction are the same as seen in Figure 4c and d. Clearly, the effect of sample rotation or random azimuthal direction of incidence is to make the surface

Table 1

Moments of the height distributions. All simulations are 20 keV C₆₀ bombardment of Ag at a fluence of 3.7×10^{13} impacts/cm².

Angle of incidence	z_{mean} (nm)	RMS roughness (nm ²)	Skewness	Kurtosis
0°	2.27	2.39	0.17	2.63
45°, single azimuth	2.06	2.13	-0.03	2.98
45°, random azimuth	2.36	1.71	-0.12	2.93
70°, single azimuth	1.46	1.26	-0.34	3.45
70°, random azimuth	1.60	1.04	-0.26	3.01

smoother by preventing the build-up of the large elongated ridges and valleys on the sample. Impacts parallel to the ridge tend to enhance the ridge, whereas impacts perpendicular to the ridge tend to break it down. The hills shown in Figure 2a are too large to demolish with one impact. This mechanism is clearly different from that for atomic bombardment. Zalar proposed sample rotation as a means to even out sputtering yields due to heterogeneities of the sample [11,12]. For C₆₀ bombardment, the sample rotation obliterates anisotropy developed by the impinging beam at grazing incidence.

A number of different cluster beams are being used in SIMS experiments including Au₃/Bi₃ and large Ar clusters. We have previously implemented the simulation protocol to examine depth profiling with these cluster beams at a single azimuthal direction [23,24,32]. Here we analyze the number of crossings as shown in Figure 5 in order to ascertain if sample rotation might improve the quality of depth profiling with these beams. For Au₃, the number of crossings in the directions perpendicular and parallel to the beam are quite similar (Figure 5a), and thus we would not predict there to be much effect of sample rotation on the depth resolution. The Ar₈₇₂ cluster, on the other hand, creates more anisotropy (Figure 5b) than the C₆₀ cluster, indicating sample rotation will improve the depth resolution in SIMS experiments. Experiments examining topology of Au crystals after bombardment with Ar gas cluster beams (distribution of cluster sizes) show that anisotropic topology is developed at grazing angles of incidence and that sample rotation does smooth the surface [33].

There is a connection between the values of mean height and RMS roughness and the dynamics of the bombardment process. As shown in previous simulations [18,28,29], the craters formed by the C₆₀ cluster impact and thus the amount of material removed are similar for 0° and 45°. Changing the angle of incidence to 70°, however, makes a shallower and more anisotropic crater and thus

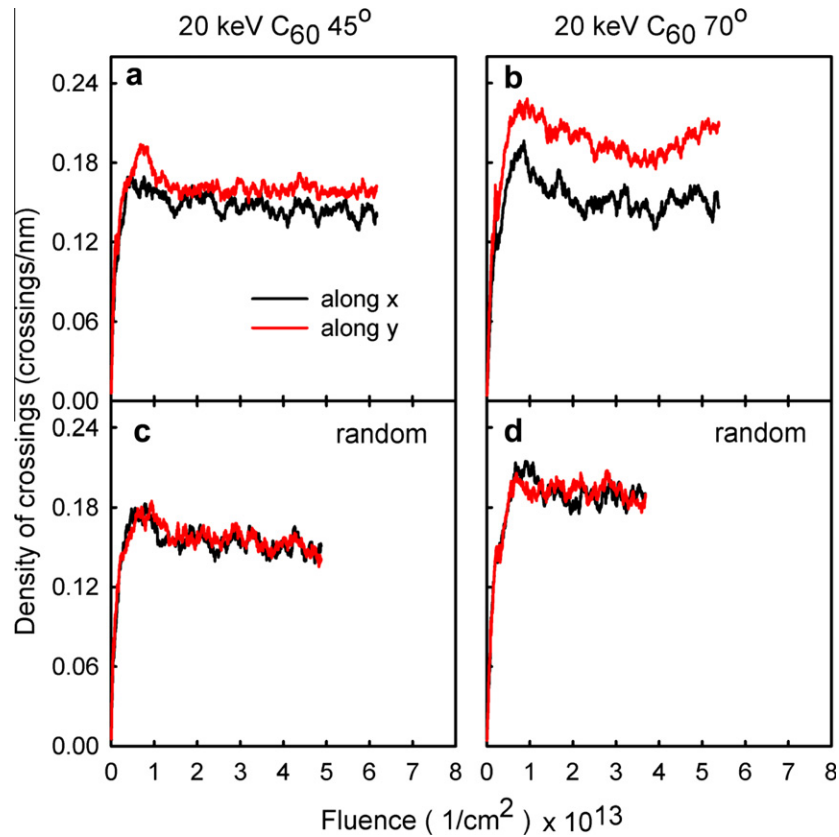


Figure 4. The number of crossings in the direction perpendicular to the beam (y-direction; red or gray lines) and parallel to the beam (x-direction; black line) as a function of fluence. (a) 45° incidence, single azimuth. (b) 70° incidence, single azimuth. (c) 45° incidence, random azimuth. (d) 70° incidence, random azimuth.

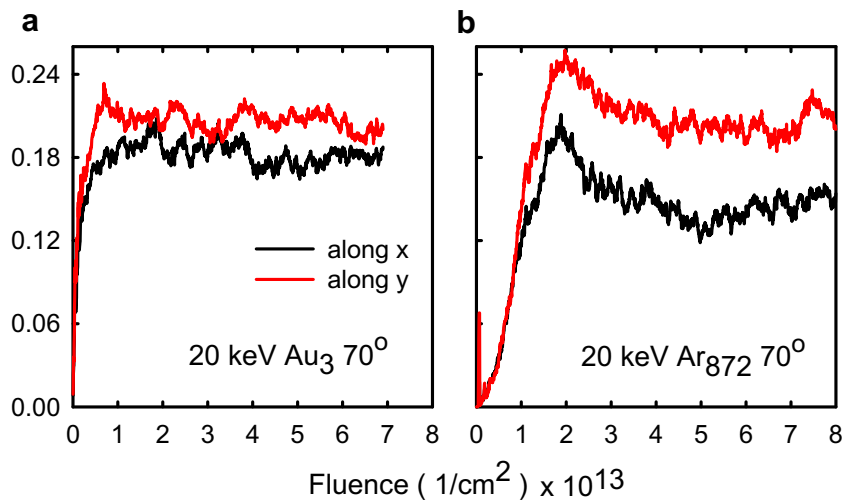


Figure 5. The number of crossings in the direction perpendicular to the beam (y-direction; red or gray lines) and parallel to the beam (x-direction; black line) as a function of fluence for other clusters at a single angle of incidence at 20 keV and 70° incidence. (a) Au₃. (b) Ar₈₇₂.

results in a smaller sputtering yield. Similar observations have been made in experiments of C₆₀ bombardment of cholesterol films [7]. Thus, it is logical that the values of mean height and RMS roughness shown in Figure 1 are more similar for 0° and 45° incidence and less similar for 70° incidence [23,24]. Since the dynamics subsequent to C₆₀ impact at 45° incidence is similar to 0° where there cannot be any effect of sample rotation, it is not surprising that the effect of sample rotation is small as shown in Figures 1 and 4. Changing the angle of incidence to 70°, however, changes the dynamics, thus the effect of sample rotation is greater.

4. Conclusions

The effect of sample rotation of the RMS roughness of a surface due to repetitive keV bombardment by C₆₀ has been investigated with molecular dynamics simulations. Sample rotation decreases the roughness and should improve the quality of the depth profiles as observed in experiment. The improvement is only appreciable at grazing angles of incidence, however. The reason for the improvement is that the incident beam at grazing angles creates an anisotropic surface. Sample rotation reduces the buildup of

elongated valleys and ridges. This mechanism is in contrast to that for atomic bombardment, where it is believed that sample rotation averages out the differential yields for various polycrystalline faces. Estimates of the importance of sample rotation for Au₃/Bi₃ and large Ar cluster bombardment indicate that it will have the most effect for the large clusters, again at grazing incidence.

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