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ABSTRACT

Scanning tunneling microscopy shows that copper deposited at room temperature onto a freshly exfoliated MoS_2 surface forms Cu(111) clusters with periodic preferred heights of 5, 8, and 11 atomic layers. These height intervals correlate with Fermi nesting regions along the necks of the bulk Cu Fermi surface, indicating a connection between physical and electronic structures. Density functional theory calculations of freestanding Cu(111) films support this as well, predicting a lower density of states at the Fermi level for these preferred heights. This is consistent with other noble metals deposited on MoS_2 that exhibit electronic growth, in which the metal films self-assemble as nanostructures minimizing quantum electronic energies. Here, we have discovered that it is critical for the metal deposition to begin on a clean MoS_2 surface. If copper is deposited onto an already Cu coated surface, even if the original film displays electronic growth, the resulting Cu film lacks quantization. Instead, the preferred heights of the Cu clusters simply increase linearly with the amount of Cu deposited upon the surface. We believe this is due to different bonding conditions during the initial stages of growth. Newly deposited copper would bond strongly to the already present copper clusters, rather than the weak bonding, which exists to the van der Waals terminated surface of MoS_2. The stronger bonding with previously deposited clusters hinders additional Cu atoms from reaching their lowest quantum energy state. The interface characteristics of the van der Waals surface enable surface engineering of self-assembled structures to achieve different applications.

Layered crystals with band gaps, such as MoS_2, are of interest due to their potential applications in a variety of devices in both bulk and two-dimensional (2D) forms. MoS_2 has an indirect bandgap of 1.29 eV in the bulk and a direct bandgap of 1.9 eV when reduced to a single monolayer.[1] Notably, field effect transistors derived from MoS_2 semiconductors have demonstrated high electron mobilities and on/off ratios.[2,3] Promising applications also exist in the field of optoelectronic devices such as photodetectors and light emitting diodes,[3–5] with performances similar to Si-based devices.[3,6] Integration of any MoS_2 based device requires metallic interconnects. One potential candidate for metallic interconnects is Cu due to its low resistivity[7] and relative low cost compared to metals such as Au and Ag. Additionally, Cu has a lower Schottky barrier with increased number of surface defects, which are common in MoS_2 crystals.[8,9] This property makes Cu suitable as an interconnect material in devices that require low power loss such as logic based circuits. Aside from the Cu/MoS_2 interface, power loss can occur in the Cu itself due to scattering at grain boundaries. Currently, little work has been done to explore the structural properties of thin Cu films on MoS_2. The limited experiments available indicate that Cu deposited at elevated temperatures form large, isolated clusters that would greatly hinder electron transmission.[10]

Although the Schottky barrier at the Au/MoS_2 interface increases with surface defects, Au forms relatively smooth films on MoS_2 with minimal grain boundaries when deposited at room temperature.[11–13] These films become atomically flat with almost no grain boundaries when followed by a quick annealing step and are conducive to electrical conduction.[14] It is believed that the atomically smooth film of Au that forms on MoS_2 is the result of the increased energetic stability of certain film thicknesses due to quantum size effects.[14,15] Essentially, electron confinement in the Au film results in quantum well states that cause the total energy to oscillate with film thickness. Quantum size effects are typically not seen due to large energy contributions such as surface kinetics or strain due to lattice mismatch. However, recent discoveries show that many metals can experience an electronic growth mode on layered materials due to the reduced strain of the van der Waals interface. This has been observed or suggested on Au/WS_2,[16] Au/MoS_2,[14,15,17] and Ag/MoS_2.[18,19] Given the similarities in electronic and physical properties, it is possible that Cu may follow a similar pattern when grown at non-elevated temperatures. This could potentially give us the ability to grow inexpensive electronic interconnects with minimal crystal defects, high conductivity, and a low Schottky barrier on MoS_2 based devices.

In this paper, we have used scanning tunneling microscopy (STM) to study the surface morphology of Cu(111) grown on MoS_2 at room temperature as a function of coverage and annealing temperature. We find that Cu growth is affected by quantum size effects when initially deposited onto a clean surface of MoS_2 without annealing the sample. Our measurements indicate that Cu clusters have preferred heights of 1.01 ± 0.05, 1.70 ± 0.06, and 2.36 ± 0.05 nm corresponding to 5, 8, and 11 atomic layers, respectively. These preferred heights have a periodicity that strongly correlates with a particular set of nesting vectors on the Fermi surface of Cu. This periodicity is further confirmed by examining the calculated density of states (DOS) of freestanding Cu slabs. Interestingly, we find that quantum size effects can be completely suppressed in Cu clusters formed from successive depositions. In this case, the cluster heights grow at a linear rate with coverage and show no preferred heights. We believe the preexisting Cu clusters from prior depositions hinder additional Cu atoms from reaching their lowest energy state associated with standing electron waves perpendicular to the MoS_2 surface.

Samples were prepared by depositing Cu at room temperature onto the cleaved surface of commercially available MoS_2 (SPI Supplies) in a vacuum chamber with a base pressure of 5×10^{-10} mbar. Deposition was achieved using a mini electron-beam evaporator (MANTIS QUAD-EV) with Cu pieces (99.999% pure) in a molybdenum crucible fitted with an alumina liner. A flux monitor was used to maintain a consistent deposition rate calculated to be approximately 0.2 Å/s from the resulting scanning tunneling microscopy images. Cu was deposited with a nominal thickness ranging from approximately 0.5 to 2.5 nm. Nominal thickness refers to the thickness of the Cu film as it would be if the film were atomically flat. Some samples were annealed using resistive heating while monitoring the temperature using a type K thermocouple in contact with the preparation stage. Annealing was performed at temperature for at least 30 min. This time was found to be sufficient for the surface to reach equilibrium. STM tips were mechanically cut from a 0.25 mm $Pt_{90}Ir_{10}$ wire. Scanning parameters used in this study were relatively consistent. The tunneling bias typically ranged from 0.75 to 1.5 V, and the current set point was typically in the range of 1.0 nA.

Density functional theory (DFT) was used to calculate the DOS of two-dimensional, freestanding Cu(111) slabs. The crystal structure and lattice constants used in this calculation were derived from experimental results.[20] An additional 1.5 nm vacuum spacing was added to the unit cell in the z direction to avoid overlapping wave functions. The Perdew-Burke-Ernzerhof implementation[21] of the generalized gradient approximation (PBE GGA) was used within the ATOMISTIX TOOLKIT package. Convergence with respect to the k-point mesh was relatively slow, requiring a mesh of $70 \times 70 \times 1$.

After room temperature deposition, Cu forms island structures on the surface of MoS_2 with lateral dimensions on the order of 5 nm. A (111) orientation is assumed based on previous experiments on the same system.[10] Additional evidence of the (111) orientation can be seen in the images in Fig. S1. Within the error of the measurement, no change in the island heights or lateral dimensions was observed as the sample was heated from room temperature to 450 K. A typical example of this surface after annealing to 350 K is shown in Fig. 1(a) as well as its corresponding height histogram with a preferred height at approximately 1.80 nm. As the surface is annealed to 500 K, the morphology of the surface begins to change as seen in Fig. 2(b). This image

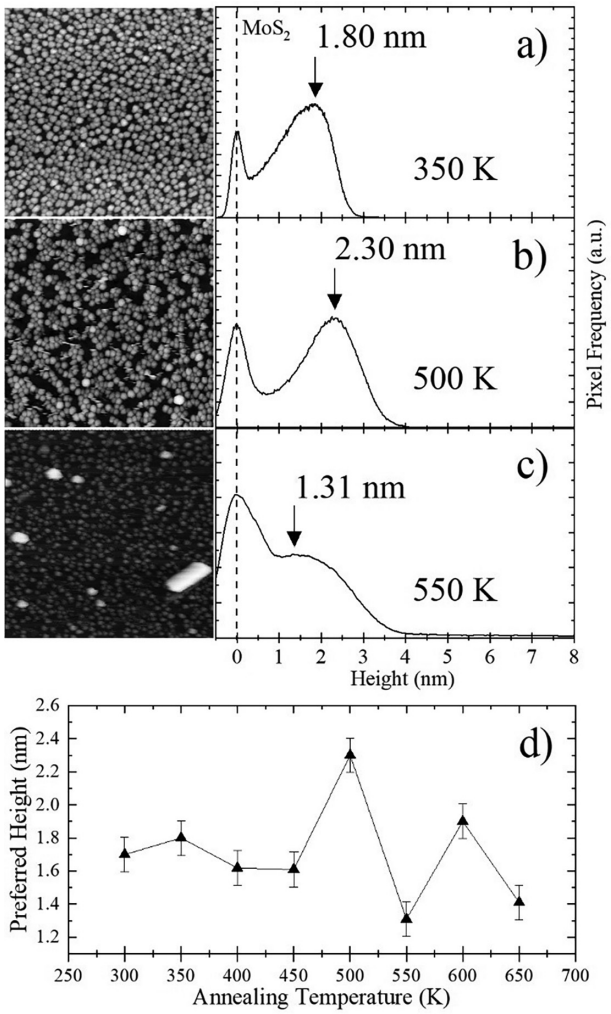

FIG. 1. (a)–(c) Scanning tunneling microscopy images (150 nm × 150 nm) of Cu with a nominal thickness of 1.31 nm deposited on freshly exfoliated MoS_2 and their corresponding height distributions as a function of temperature. These data show the formation of small Cu clusters. The preferred height of these clusters in the distribution plots shows some fluctuations with temperature. This pattern is better seen in (d), the preferred cluster height as a function of annealing temperature. This plot shows little change in the cluster height with temperature before beginning to oscillate above 500 K. In addition to the smaller clusters, the formation of larger Cu clusters begin at approximately 500 K and stop growing at about 550 K as seen in the STM images in (b) and (c), respectively.

shows some of the Cu clusters beginning to aggregate into larger clusters. The height histogram indicates that the preferred height of the Cu clusters has increased by half a nanometer. However, a small number appears to be slightly larger than others. With continued annealing, the larger clusters continue to grow at the expense of the number of smaller clusters as seen in the image in Fig. 1(c). Figure S1 presents magnified sections from the images in Fig. 1. These enlarged images clearly illustrate a reduction in the number of smaller clusters as the larger clusters grow in size. At the same time, the lateral dimensions of the smaller clusters do not appear to change. In some cases, the

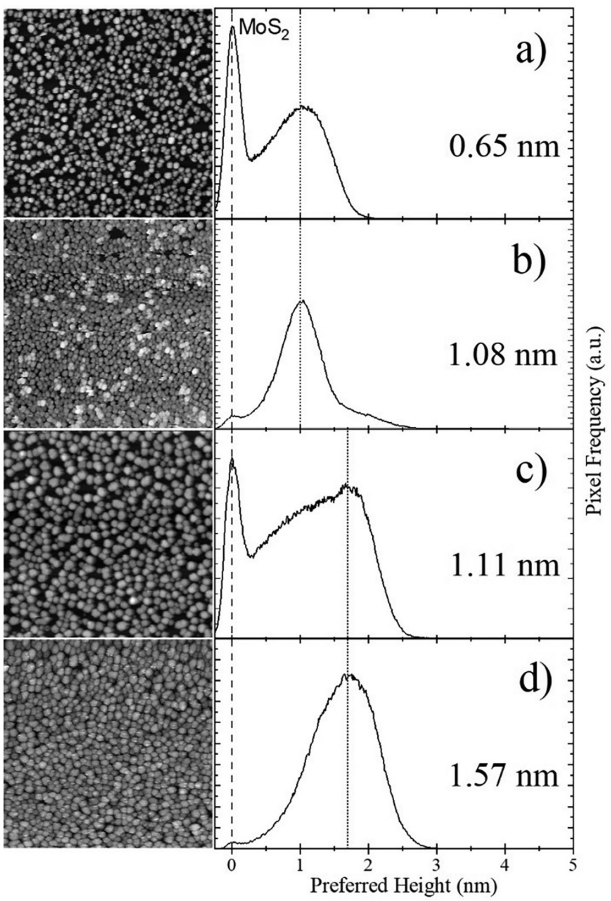

FIG. 2. (a)–(d) Scanning tunneling microscopy images of Cu deposited onto freshly exfoliated MoS_2 with corresponding height histograms as a function of coverage. Comparison of the results in (a) and (b) shows that the density of clusters increases with coverage, while the preferred height remains unchanged at approximately 1.0 nm. This same pattern repeats in (c) and (d). As more Cu is added to the surface, the clusters increase in density in order to maintain a consistent preferred height of 1.7 nm. These data suggest there are preferred heights with increased stability over others. The heights listed in each figure refer to the nominal thickness of the Cu.

dimensions of the larger clusters could exceed 20 nm. The preferred height of the smaller clusters decreases as seen in the histogram of Fig. 1(c). The material from the reduced number of small Cu clusters appears to transfer to the larger clusters. One would expect that the smaller clusters would eventually be entirely absorbed by the larger clusters as the sample temperature increases. Instead, there appears to be a form of bimodal growth occurring in which material is likely passed between the larger and smaller clusters, but has not yet reached equilibrium. This exchange in material can be seen in Fig. 1(d). Higher temperatures would be necessary to test this hypothesis. However, this is beyond the capabilities of our equipment. As the sample temperature increases from 500 to 650 K, we see the preferred height of the smaller clusters oscillates significantly. We note that this does not appear to be the result of diffusion of Cu into the surface as a volume analysis indicates no loss of Cu as a function of temperature. The reason for this is not entirely clear but may have to do with surface defects present on the surface of MoS_2. Defects on the surface may pin certain Cu clusters, preventing them from being completely absorbed by larger clusters. This would explain why the cluster density initially decreases quickly from 500 to 550 K but shows little change at high temperatures.

Figure 2 shows the evolution of the Cu clusters with increasing amounts of Cu after room temperature deposition. We see that as nominal coverage increases from 0.65 to 1.08 nm, the preferred height remains at approximately 1.0 nm as seen in Figs. 2(a) and 2(b), respectively. To maintain the same preferred height with increasing material, the Cu clusters do not increase in lateral size, as is the case with Ag MoS_2.[18] Instead, we find that the density of Cu clusters increases. This is likely due to a relatively larger amount of strain between Cu and MoS_2, which energetically favors a large number of small clusters as opposed to a small number of large clusters, as seen with Ag on MoS_2.[18] This same pattern occurs as coverage increases from 1.11 to 1.57 nm as seen in Figs. 2(c) and 2(d), respectively. Although additional material is added to the surface, the preferred height remains unchanged at approximately 1.7 nm. These data suggest that 1.0 and 1.7 nm are heights of increased stability. This is further reinforced by examining the histograms in Figs. 2(b) and 2(c). Although both histograms have a clear preferred height, both also exhibit secondary heights. Although difficult to determine through peak fitting techniques, these secondary heights appear to occur at approximately 1.0 and 1.7 nm in Figs. 2(b) and 2(c), respectively. It would appear that a critical coverage occurs between 1.08 and 1.11 nm where the preferred height undergoes a sudden shift from 1.0 to 1.7 nm.

The preferred heights with increased stability are more prominently seen as the plateaus in the data of Fig. 3(a). This plot shows the preferred heights as a function of coverage. These data were acquired from an initial deposition of Cu onto a freshly exfoliated surface of MoS_2. These data points were obtained through 12 distinct depositions, with images collected at various locations across the sample surface. The wrinkled nature of the MoS_2 surface results in a small spread of coverages, even for the same deposition, giving multiple data points for each deposition. This plot shows heights of increased stability occurring at 1.01 ± 0.05, 1.70 ± 0.06, and 2.36 ± 0.05 nm. This corresponds to 5, 8, and 11 atomic layers, respectively. The average spacing of stable heights is approximately 0.79 nm or about four atomic layers. The uncertainty in determining nominal thickness based on the STM images is estimated at approximately $\pm 10\%$, primarily attributed to tip convolution effects. Additionally, the error in determining the preferred height is equivalent to that of a single atomic layer on Cu(111), equal to 0.21 nm.[22] In an effort to quickly increase the number of data points, we experimented with skipping the exfoliation step and depositing directly onto surfaces containing Cu from previous depositions. We plot the preferred height as a function of coverage in Fig. 3(b) for data obtained from successive depositions onto unexfoliated surfaces. Under these conditions, we do not observe heights of increased stability as in Fig. 3(a). Instead, the preferred height increases linearly with coverage.

The periodic spacing of the heights with increased stability in Fig. 3(a) suggests that the growth of the Cu clusters is affected by quantum size effects on the electronic structure. This has been observed previously in the surface morphology of Au and Ag on MoS_2,[14–16,18] both metals with an electronic structure similar to Cu. In these metals,

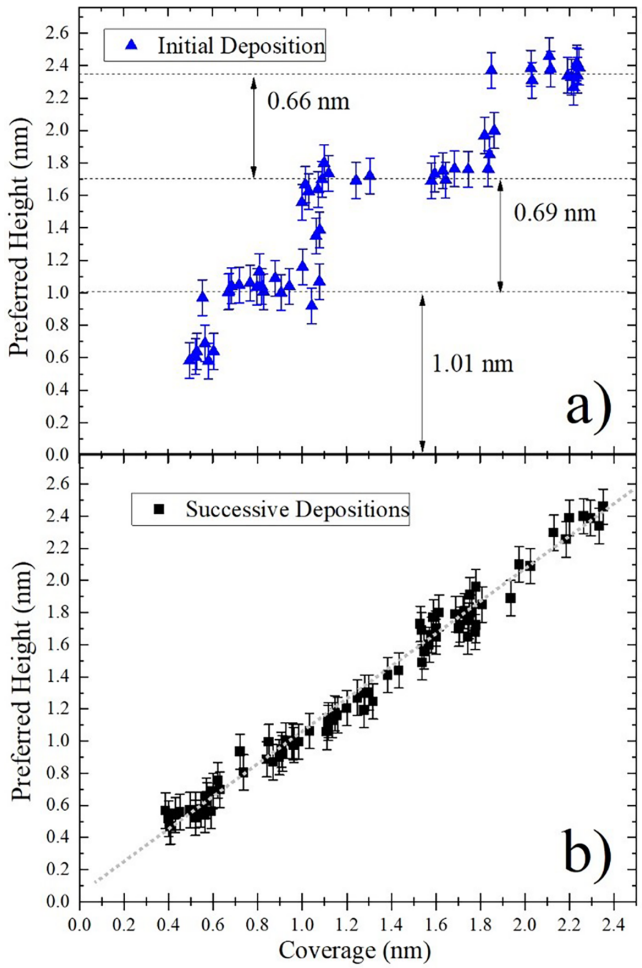

FIG. 3. (a) Plot of the preferred cluster height after the initial deposition on a freshly exfoliated MoS_2 surface. Three distinct preferred heights appear at 1.01, 1.70, and 2.36 nm. (b) Plot of the preferred cluster height after successive depositions on MoS_2 without exfoliating the surface after each deposition. Without a clean surface, the preferred height simply increases linearly with coverage.

quantization of momentum perpendicular to the surface is not possible due to a gap in available states in the [111] direction. However, it is possible to have quantization of states with non-zero parallel momentum. Of particular note is momentum associated with nesting vectors, a set of parallel states found on the Fermi surface.[23] The introduction of periodicity associated with a nesting vector has a disproportionate impact on the Density of States (DOS) and by extension the overall energy. This phenomenon is similar to the connection between nesting vectors and the propensity for charge density wave order in specific materials.[24] Assuming conservation of parallel momentum, only nesting vectors (q) normal to the surface are considered. Contributions to the DOS at E_F are at a maximum when the island height, given as a discrete number of layers (N) with interlayer distance (d), is an integer multiple of $2\pi/q$,

$$Nd = n\frac{2\pi}{q}. \quad (1)$$

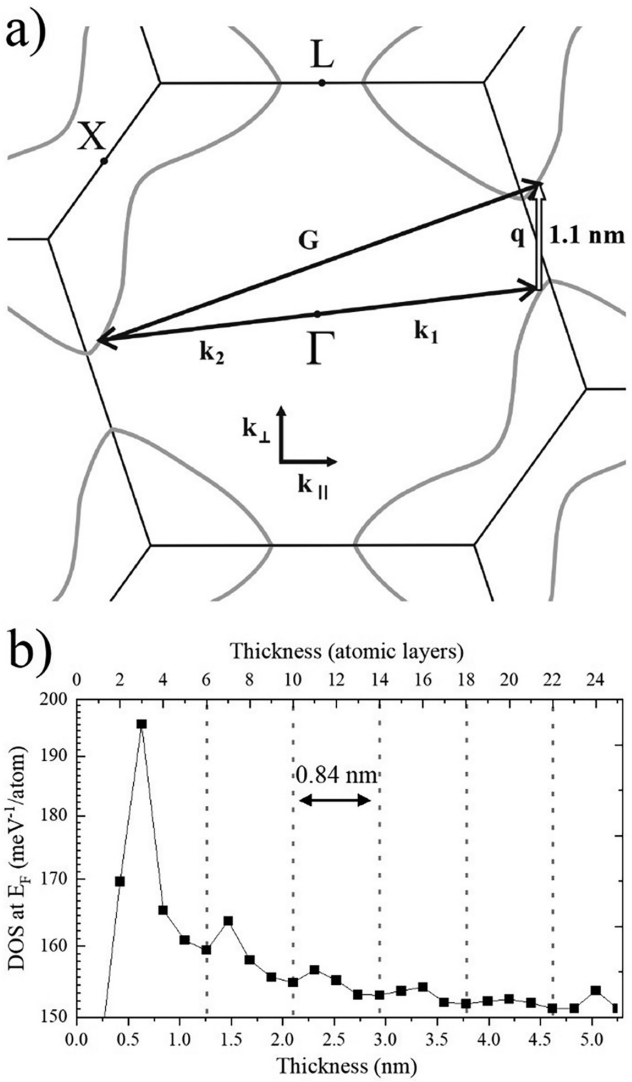

FIG. 4. (a) Cross section of the Fermi surface of Cu in the [111] direction perpendicular to the MoS2 surface. The nesting vector, labeled as **q**, spans the vectors $\mathbf{k}_1$ and $\mathbf{k}_2$ when translated back to the first Brillouin zone. From Eq. (1), the periodicity associated with this vector is 1.1 nm. (b) Calculated DOS of freestanding Cu(111) slabs at the Fermi level as a function of Cu thickness. The spacing between local minima in the DOS is 0.84 nm or four atomic layers.

In Fig. 4(a), we examine a slice of the bulk Cu Fermi surface cut along the [111] direction (adapted from Ref. 25). The only nesting vector we can find that is both perpendicular to the surface and close in size to the experimentally observed periodicity crosses from the first to the second Brillouin zone. In this case, the nesting vector spans the two zones by the reciprocal lattice vector according to the following: $\mathbf{q} = \mathbf{k_1} - (\mathbf{k_2} + \mathbf{G})$. The corresponding periodicity of stable heights expected from this nesting vector is estimated to be approximately 1.1 nm according to Eq. (1), which is slightly larger than our measurements. A better approximation would involve simulating our system to include finite size effects not necessarily present in the bulk

calculation. To do this, we calculate the DOS at E_F of freestanding slabs of Cu (111) using DFT. Although strong enough to affect the lateral shape, we ignore the interactions between the Cu and the MoS_2 surface as they are minimal enough to enable quantum growth. The resulting DOS at E_F is plotted as a function of the thickness of the Cu slab in Fig. 4(b). Periodic dips in the DOS occur at regular intervals of 0.84 nm or 4 atomic layers, in good agreement with our experimental results in Fig. 3(a).

Perhaps just as interesting as the existence of the electronic growth mode is how this mode disappears when we perform successive depositions on the MoS_2 surface without first exfoliating the surface as seen in Fig. 3(b). For these data, each successive deposition occurred at least 24 h after the prior deposition. As discussed in the introduction, energy contributions due to the electronic structure of the Cu clusters are minimal compared to others. We speculate that the crowded structures from the initial deposition (see, for example, images in Fig. 2) are sufficient to hinder subsequent Cu atoms from reaching a new equilibrium associated with the electronic structure. Instead, the Cu clusters simply grow vertically in a linear fashion typically expected in many classic growth scenarios.

We have demonstrated that Cu(111) clusters follow an electronic growth mode on MoS_2 in a similar manner as seen for Au and Ag. This electronic growth mode only occurs when the deposition begins on a freshly exfoliated MoS_2 surface. If the deposition is paused, further deposition of copper follows a standard growth profile where thickness is simply proportional to the deposition amount. In either growth regime, the copper forms a granular structure defined by nanometer scale clusters.

The electronic growth mode causes periodic heights of increased stability in Cu clusters that match thicknesses minimizing the DOS at the Fermi level, resulting in a lower overall electronic energy. These states also correlate with a particular set of nesting vectors located along the necks of the Cu Fermi surface. At room temperature deposition, the lateral size of the clusters varies little with coverage, and instead, the clusters change density in order to maintain preferred heights. This results in certain coverages with gaps between the Cu clusters that are less suitable for electrical conduction and would not be appropriate for use as metallic interconnects on MoS_2 based devices. This knowledge has a number of practical applications that include determining the optimal deposition conditions to maximize the cluster density and optimize conductivity as well as creating highly catalytic surfaces.

See the supplementary material for additional evidence found in the STM images showing the (111) orientation.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Haley A. Harms: Data curation (equal); Investigation (lead); Writing – original draft (supporting). **Connor J. Cunningham:** Data curation (supporting); Investigation (supporting). **Timothy E. Kidd:** Conceptualization (equal); Writing – original draft (supporting). **Andrew J. Stollenwerk:** Conceptualization (lead); Data curation (equal); Investigation (supporting); Methodology (lead); Project administration (lead); Resources (lead); Writing – original draft (lead).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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