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Crystallographic and electronic contribution to the apparent step height in nanometer-thin Pb(111) films grown on Cu(111)

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Abstract. Thermal roughening of Pb(111) films grown on Cu(111) produces three-dimensional (3D) islands of different number of layers allowing the simultaneous and direct measurement by scanning tunneling microscopy (STM) of the step height for different thicknesses in real space. The apparent step heights separating adjacent layers show several oscillations with amplitudes of up to 0.8–1.4 Å around the bulk interlayer distance as a function of film thickness. The oscillations have bilayer periodicity with a superimposed longer beating period that produces a phase slip every eight layers. Based on first-principles calculations of Pb(111) free standing slabs, we can identify the relevant electronic states responsible for these quantum size effects. In addition, we can distinguish between geometric and electronic contributions to the apparent step heights measured on the STM images.

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1. Introduction

Quantum size effects (QSEs) in nanometer-thin metal films, where electrons are confined in the perpendicular direction by band gaps in the substrate and image potential in the vacuum side, were first discussed by Sandomirskii almost 40 years ago [1] and reveal themselves as *oscillations*, in many physical properties, with the film thickness. This is produced by the systematic variation in the density of states (DOS) at the Fermi level due to the periodic crossing by the quantum well states (QWSs) created by the confinement of electrons [2]. Thus, many physical properties of metallic films have been predicted or observed to *oscillate with the thickness* with a periodicity of a few monolayers ($\lambda_F/2$) [3]. Among them we may cite bulk properties, such as electron density [4], electrical resistivity [5, 6], Hall coefficient [7], metallicity [8], or superconducting transition temperature [9, 10], surface properties, such as work function [11, 12], chemical reactivity [13], or activation barrier for surface diffusion [14], and thermodynamic properties, such as equilibrium height distribution of islands [15] or thermal stability of different thicknesses on semiconducting [16, 17] and metallic [18, 19] substrates. Oscillations in surface phonons have also been recently proposed [20].

For thin films of Pb deposited onto substrates that efficiently confine electrons within the Pb film, He atom scattering data indicated the presence of QSE-induced oscillations in the *apparent* step height [21, 22]. Direct observations of oscillations in the interlayer distances from x-ray diffraction data obtained for Pb/Si(111) have been recently reported [23]. The changes in the interlayer distances (typically 0.2–0.3 Å) affect the whole thin film, but are mostly concentrated close to the substrate and the external surface [23]. Scanning tunneling microscopy (STM) images of Pb islands on Si(111) recorded at positive sample bias have shown a pair of oscillations in the step height with an amplitude of 0.3 Å. The measured step heights, however, were always below the bulk value [24].

Here, we report the oscillatory behavior of the apparent step height, as measured by STM, for ultrathin films of Pb deposited at low temperatures on Cu(111). Experiments and first-principles calculations for isolated Pb(111) slabs agree in detail, except for a sign inversion that can be attributed to the interface layer. The observed oscillations in the apparent step height are mostly due to QWS-induced, layer-dependent, changes in the electronic DOS at the Fermi level. The step height oscillations mirror the oscillatory behavior of the roughening temperature

for the same system [19]. The thicknesses that are more *unstable* thermally, as a consequence of their larger DOS at the Fermi level, show *larger* apparent step heights. We also identify the relevant electronic states and the relaxation of the atomic positions due to the quantized electronic structure.

2. Experimental

The experiments were carried out in an ultra high vacuum (UHV) chamber (with a base pressure of 3×10^{-11} Torr) that contains a variable-temperature scanning tunneling microscope (with the capability of evaporating *in situ*, a rear-view low-energy electron diffraction (LEED) optics that is also used for Auger electron spectroscopy), an ion gun and a mass spectrometer. The Cu(111) crystal was cleaned by cycles of Ar⁺ sputtering and annealing and displayed a sharp LEED pattern and atomically resolved high-quality STM images. Pb was evaporated from a Knudsen cell on the Cu(111) substrate, while it was in the microscope at 90 K. The images were recorded in the constant-current mode at temperatures ranging from 60 to 300 K.

3. Results

3.1. Experimental results

Although Pb grows on Cu(111) at 300 K forming three-dimensional (3D), (111)-oriented islands on top of a wetting layer, just 1 monolayer (ML) high [15], depositing Pb below 100 K leads to a kinetically controlled layer-by-layer growth. At 98 K, the surface diffusion is frozen enough so that even films with ‘forbidden’ thicknesses can be stabilized [19]. Figure 1 illustrates this for a nominally 5.1 ML-thick film. Most of the surface is covered by 5 ML, while a certain number of ramified islands 1 ML high cover a small fraction of the surface. There are also ramified holes one atom deep. The islands and holes have a dendritic shape, indicating that at this temperature surface diffusion is essentially frozen. The local thickness of the deposited Pb films can be accurately determined experimentally by comparing the energy position of the corresponding QWS detected by scanning tunneling spectroscopy with those observed on top of the Pb islands of various heights that appear upon deposition on Cu(111) at 300 K [15]. The inset in figure 1 shows the tunneling spectra recorded on top of the regions with local coverages of 4, 5 and 6 ML. Only one QWS is detected on the 5 ML regions, while two QWSs appear in the explored energy window for the 6 ML-thick islands. In the spectra measured in 4 ML there are two peaks, one at +0.65 eV that is present in all the thickness with an even number of atomic layers [5, 25] and another that comes from the fact that some electrons are tunneling from the edge of the steps of the depressions and correspond to the 5 ML thickness.

In order to measure the apparent step heights in the same conditions, an initially flat 5.1 ML-thick Pb(111) film was thermally decomposed by applying a temperature ramp of 1 K min^{-1} , while STM images were continuously recorded in tunneling conditions chosen not to disturb the morphology of the film by electric-field-induced mass transport [26, 27] and to include the effects of the relevant occupied QWSs. As described elsewhere [19], the morphology of the initial film changes upon increasing the temperature, since not all thicknesses are equally stable. In figure 2, we show the evolution with temperature of 5.1 ML of Pb deposited at 98 K on Cu(111). In panel (a) the images show the ramified islands and voids also shown in figure 1. From 100 K up to 200 K the main change in the topography is that the diffusion along the steps

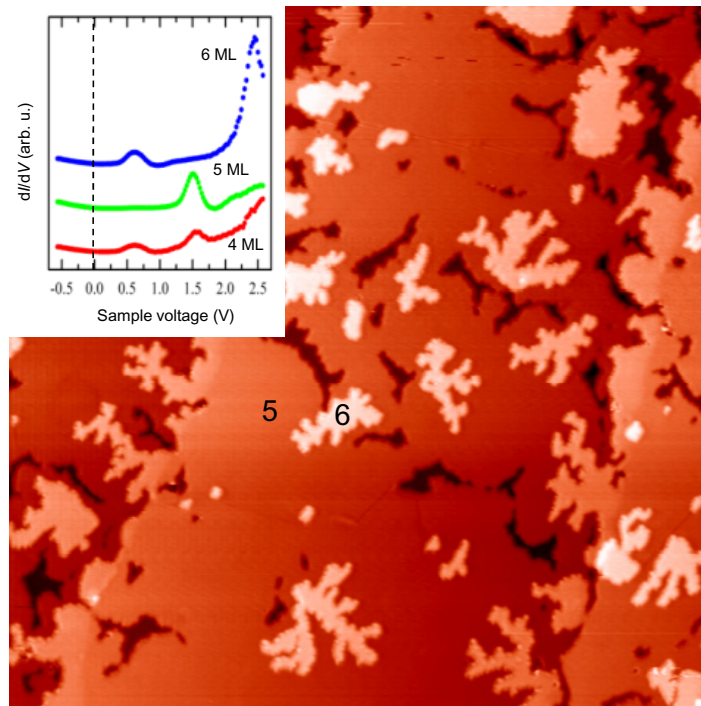


Figure 1. $100 \times 100 \text{ nm}^2$ STM image recorded ($V_s = -0.8 \text{ V}$, $I_t = 0.1 \text{ nA}$) *in situ* after deposition of 5.1 ML of Pb on Cu(111) at 98 K. Inset: tunneling spectra recorded at 98 K on regions of the surface with local thicknesses of 4, 5 and 6 ML of Pb. The tunneling gap was stabilized at a sample voltage of -0.4 V and $I_t = 0.2 \text{ nA}$. The spectra have been shifted vertically for clarity.

sets in and the islands acquire a round shape. Around 200 K mass transport between terraces takes place and a more dramatic change in the morphology of the films starts to take place. For 5.1 ML we stabilized the temperature at 217 K and measured high-resolution images. After a careful subtraction of the plane, we can calculate the histogram of heights present on the image.

Panel (e) of figure 2 reproduces the histogram of heights corresponding to the image of panel (d). From histograms of the heights observed in a large number of STM images, the apparent step height for the N th layer, h_N , can be obtained. In figure 2 it is clear that h_N is not constant. The measured step heights are independent of the tunneling conditions for negative sample biases, where the tunneling current from the occupied states reflects the DOS at the Fermi level. The values change strongly, however, for positive bias voltages, in particular, across the empty QWS in the $+0.6$ to $+3 \text{ V}$ range.

Figure 3 shows a compilation of the apparent step heights measured for the topmost layers of several film thicknesses. It is clear that the apparent step height (h_N) is smaller than the bulk interlayer distance (dashed line) for $N = 3, 4, 6, 8, 10$ and 13 ML , and larger for $N = 2, 5, 7, 9$ and 12 ML . The oscillations have bilayer periodicity with a longer beating period that produces a phase slip every 8 ML, marked with red arrows in figure 2. The typical amplitude of the oscillation is 0.5 \AA , with a maximum of $1.2\text{--}1.4 \text{ \AA}$ for $7\text{--}10 \text{ ML}$. The apparent step height measured by STM in a constant-current topographic image results from scanning the

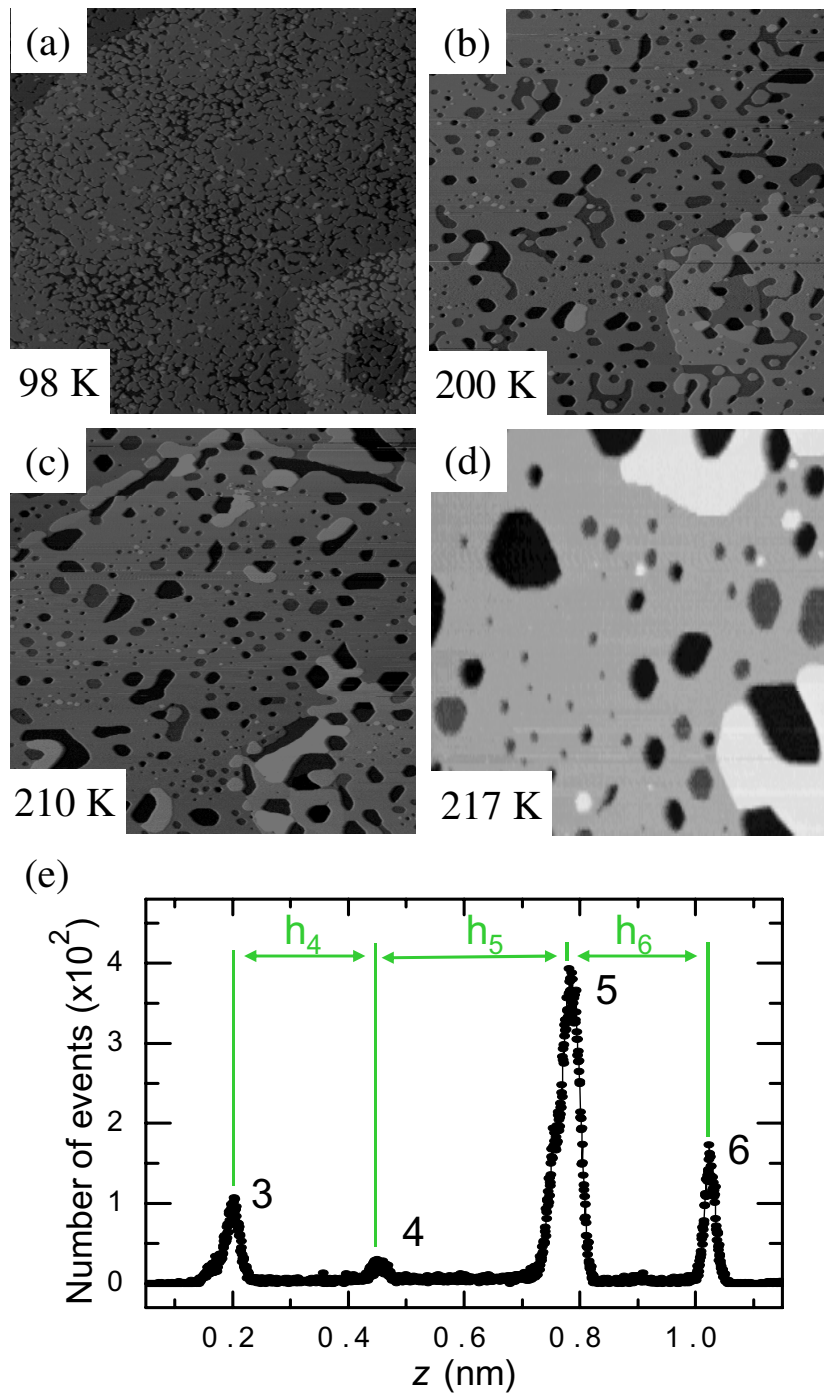


Figure 2. $350 \times 350 \text{ nm}^2$ STM images recorded ($V_s = -0.8 \text{ V}$, $I_t = 0.1 \text{ nA}$) *in situ* after deposition of 5.1 ML of Pb on Cu(111) at 98 K and annealed at 100 K (a), 200 K (b) and 210 K (c). (d) $100 \times 100 \text{ nm}^2$ annealed to 217 K. (e) Histogram obtained after careful subtraction of the plane in the STM image shown in (d) to illustrate the procedure to determine the apparent step heights h_N .

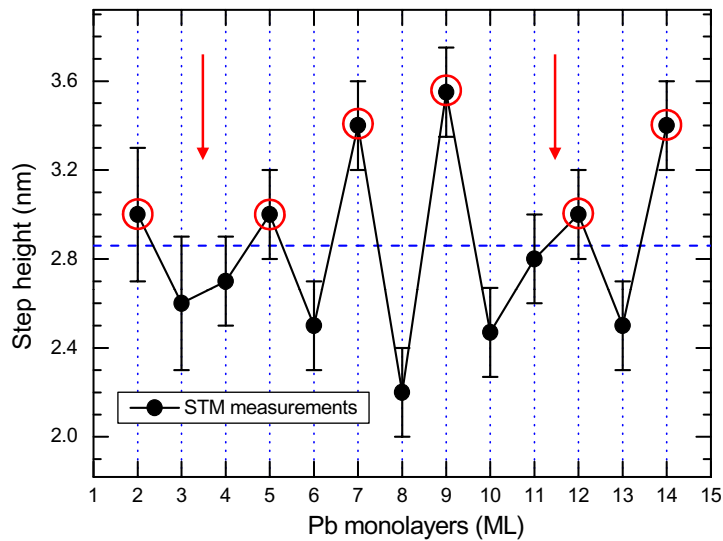


Figure 3. Experimental apparent height differences, h_N , between Pb regions differing by a single atomic layer, as obtained from histograms in STM images, such as figure 2(a). The horizontal line represents the Pb bulk interlayer distance (2.84 Å). The red arrows mark the phase slip in the bilayer oscillation of the step height. The red circles mark the unstable island heights.

tip over constant electron density contours in neighboring atomic terraces and contains both an electronic and a geometric contribution. The electron spillage length into the vacuum for an N -layer film depends on its electronic structure, in particular on the DOS at the Fermi level. The atomic relaxations will also be influenced by the modulation of the DOS inside the film due to the QWSs. In order to elucidate the relative importance of the atomic relaxations and the electronic density at the surface of the film to the apparent step height, we perform total energy and electronic structure calculations.

3.2. Theoretical results

The calculations are based on the density functional theory [29]. To solve the Kohn–Sham self-consistent equations, we use the Vienna *ab initio* simulation package (VASP) [30, 31]. We use the functional of Perdew and Wang [32] to approximate the exchange correlation within the generalized gradient approximation. Ultrasoft pseudopotentials [33] are used such that only s and p electrons are included in the valence band. Inclusion of d electrons in the valence band does not change the results. Special care has been taken to obtain good convergence with respect to the k -points and real space sampling, energy cutoff, self-consistency convergence criteria, etc. Since the DOS varies very rapidly at the Fermi level due to the QWSs, we have used the Methfessel and Paxton [34] smearing method. We have obtained for bulk fcc a lattice constant of 5.03 Å, in reasonable agreement with the 4.95 Å experimental value. We have calculated the electronic structure of Pb(111) slabs with widths ranging from 3 to 30 hexagonal closed packed (111) layers. We do not include the interface between Pb and Cu(111) because in our experiments we did not find any spatial modulation on the intensity or energy position of the QWS due to the atomic structure of the interface as in the case of Pb grown on Si(111) [36]–[38].

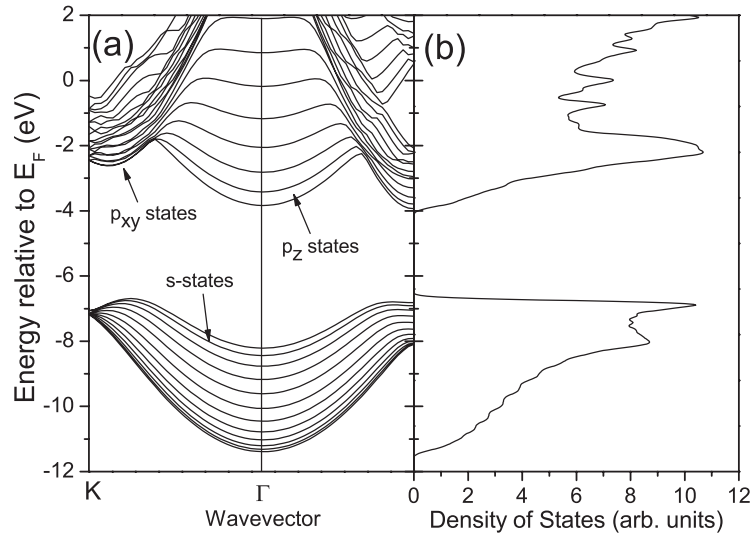


Figure 4. Calculated electronic structure of a free standing 12 layers Pb slab. Panel (a) shows the band structure along the symmetry directions in the 2D Brillouin zone. The different character of the bands is indicated. The z -direction is normal to the surface. The total DOS is shown in panel (b).

The structures have been relaxed with the constraint that the forces acting on all the atoms should be smaller than $0.01 \text{ eV } \text{\AA}^{-1}$.

In figure 4, we show, as an example, the calculated electronic structure of a free standing 12 layer slab. We identify the different character of the bands and we analyze their behavior. s -bands behave like a standard 2D system; the corresponding DOS plotted in figure 4 shows the stepwise form typical of electrons confined in a slab. In contrast, the p_z states normal to the surface, although they behave like free electrons in the direction perpendicular to the surface (the band is not shown) and therefore are quantized in energy, in the direction parallel to the surface around the Γ point, are dispersionless in the vicinity of the Fermi energy. This gives rise to well-defined peaks in the DOS (see figure 4(b)) and therefore in the STM spectra (see figure 1(b)). The dispersionless behavior of these bands in the vicinity of the Fermi level is a consequence of the special shape of the Pb bulk Fermi surface [35].

4. Discussion

In the so-called ‘topographic’ mode, the STM images correspond to the profile of constant LDOS at a certain distance from the atomic nuclei. To identify the origin of the apparent step height oscillations measured by STM on Pb films, we calculate the electronic charge DOS for free standing Pb(111) slabs for the QWS in the energy range between the Fermi level and -5 eV (see figure 5). The red crosses indicate the surface charge density contour used to calculate the electronic contribution to the apparent step heights separating adjacent layers. We use the value of $5 \times 10^{-4} \text{ electrons } \text{\AA}^{-3}$, close to the value explored by STM in the conditions employed [28]. From this calculation we obtained a maximum value for the change in the apparent step heights, due to the different spill-over of electrons for the different layers, of

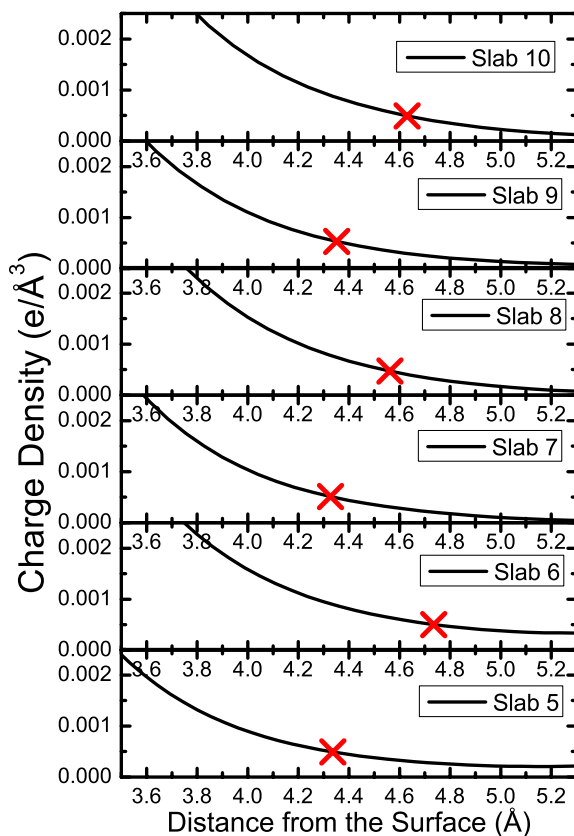


Figure 5. Calculated charge density outside the different Pb slabs for the QWS in the energy range between the Fermi level and -5 eV. The red crosses indicate the DOS explored by the STM. The distance refers to the distance from the relaxed outermost atomic surface layer.

the order of 0.5 \AA . This value is smaller than the value found on the STM experiments (see figure 3); this lack of qualitative agreement is expected due to the limitations of the theoretical method used to describe the effective potential and the corresponding electron density away from the surface.

As mentioned before, the calculations allow atomic relaxations until the forces in all the atoms of the slab are smaller than 0.01 eV \AA^{-1} . The calculations indicate that interlayer distances, the positions of the ion cores, oscillate with film thickness. For the positions of the ion cores we obtained displacements of the order of 0.2 \AA in each layer. Figure 6(a) shows the calculated slab structural width difference between slabs of a consecutive number of layers. The height difference shown on that graph for a layer N is defined as the difference in width between slabs with N layers and the slab with $N - 1$. The width of the slab *oscillates* with N , the number of Pb layers, around the ideal (111) interlayer distance in bulk Pb (dashed line). As can be seen in figure 6(a) the oscillation in height difference has a maximum of 0.5 \AA . The oscillations have bilayer periodicity with a longer beating period that produces a phase slip. The next question to answer is if the spill-over of electrons towards the vacuum is in phase or out-of-phase with the structural expansions and contractions. In figure 6(b) we compare the actual height, related with the movement of the atomic positions on the different slabs, with

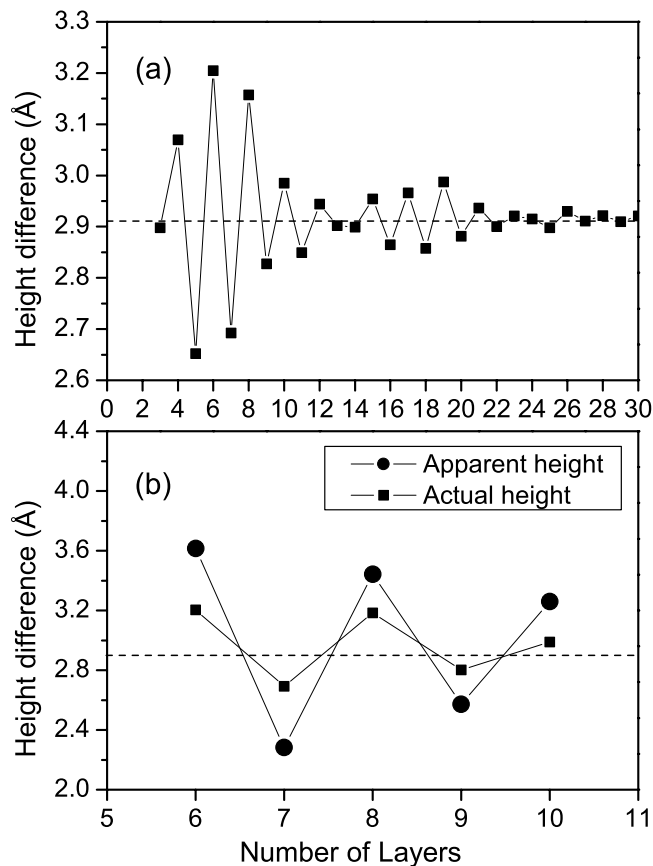


Figure 6. Calculated slab width versus number of layers. Panel (a) shows the calculated slab width difference between slabs of consecutive numbers of layers. The calculated bulk layer separation is 2.91 Å. In panel (b) the calculated actual and apparent heights are indicated by squares and circles, respectively. The apparent height difference includes the vacuum charge spilled off (see figure 5). The bulk interlayer separation is indicated by a broken horizontal line.

the apparent one. In the latter the spill-over of electrons into the vacuum is included and we consider the distance ‘seen’ by STM, as discussed in figure 5. As can be seen in figure 6(b), both contributions supplement each other and the electronic contribution is larger than the geometric contribution.

5. Summary

In summary, we have shown that QSEs produce an oscillatory behavior of the apparent step height for Pb(111) films, with the same periodicity as many other properties of thin Pb films controlled by these QSEs. The step height oscillations with bilayer periodicity and the longer beating period are in good agreement (in the common thickness range) with earlier observations of oscillations in the apparent step height deduced from He atom scattering during the growth of Pb films on Cu(111) at 140 K [22]. The apparent step height oscillations contain a much larger contribution from oscillating quantized electronic states than from interlayer distances. We have

been able to identify the relevant electronic states in this system and their dispersion relation in the vicinity of the Fermi level.

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