Coverage-dependent self-organization: from individual adatoms to adatom superlattices

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Abstract. The coverage-dependent self-organization of Ce-adatoms on a Ag(111) surface is studied by scanning tunnelling microscopy at temperatures ranging from 3.9 to 10 K. At a coverage of 0.03% of a Ce monolayer individual Ce-adatoms and Ce dimers are observed, the mutual interatomic distances of which are clearly related to multiples of the Fermi wavelength $\lambda_F/2$, reflecting surface-state-mediated electronic interactions. At a coverage of 0.2% the formation of chains and small islands with hexagonal structure prevails. At a coverage of 1% a hexagonal superlattice with a periodicity of 3.2 nm is observed. At a coverage of 2% the superlattice of Ce-adatoms is found to be compressed, showing an interatomic distance of 2.2 nm. At higher coverage the number of dimers increases considerably and the superlattice collapses into compact islands. An increase in the temperature towards about 10 K at a coverage of 1% also causes the collapse of the hexagonal Ce superlattice. These experimental findings are rationalized within the electron scattering model of Hyldgard and Persson, which specifically takes into account the electronic surface-state on Ag(111). The experimentally derived two-body interaction potential is able to account for the observed phenomena as a function of concentration and temperature.

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1. Introduction

The interest in nanostructured materials arises from their remarkable variations in fundamental properties. Novel physical, chemical and biological behaviour occurs on the nanometre scale, resulting from the properties, interactions and processing of building blocks containing a finite number of atoms. By varying the size of the units and equally controlling their interactions, the fundamental properties of nanostructured materials synthesized from these building blocks may be tuned. One of the ultimate goals in the realization of nanomaterials in two dimensions consists of the fabrication of ordered arrays of single adatoms or molecules on a surface extending over macroscopic distances.

One route leading towards this goal is the sequential manipulation of single atoms with the tip of a scanning tunnelling microscope (STM) at low temperature [1]–[3]. In this way, atomic-scale-precision nanostructures made of single adatoms have been achieved. A second route, based on self-assembly processes, has been explored for the realization of ordered nanostructures. Colloidal nanocrystals self-organize in large hexagonal domains on flat surfaces [4]–[6]. The quality of ordered materials based on multicomponent nanocrystal assemblies depends on the strength of directional forces between the building blocks and on sufficient mobility of the building blocks during self-assembly.

Until recently, this process was limited to materials consisting of a finite number of atoms, such as nanoparticles, clusters and molecules [7]–[9]. An interesting possibility to achieve long-range self-assembly of individual atoms with interatomic distances of several lattice constants of the supporting substrate is to take advantage of surface-state-mediated adatom interactions [10], which have been found on noble metal (111) surfaces [11]–[13]. While the experiments in [12, 13] displayed preferential adatom–adatom separations illustrating the oscillatory behaviour of the potential of the adatom–adatom interaction mediated by the surface-state
electrons, no large ordered atomic superlattice was produced. This situation changed very recently, as Silly et al reported on the discovery of the formation of a two-dimensional (2D) hexagonal superlattice of Ce adatoms on Ag(111) with a superlattice constant of 3.2 nm. By a careful quantitative analysis of the phase shift of the surface-state electrons scattered at the adatom sites and of the observed adatom mobility around their superlattice sites, the depth of the two-body Hyldgard and Persson interaction potential had been determined to be 0.8 meV [14]. Thus, it was shown that the self-assembly of the adatom superlattice is a consequence of a subtle balance between the sample temperature, the surface diffusion barrier and the concentration-dependent trapping potential generated by the surface-state electrons.

In this paper, we explore the self-assembly of Ce adatoms on Ag(111) as a function of adatom concentration and of temperature. The control of the temperature permits tuning of the adatom mobility on the surface, whereas the control of the adatom concentration influences the adatom–adatom interaction and, most interestingly, the interatomic distance of the adatoms in the superlattice. We show that the surface-state electron scattering at the adatom sites sets the scale of the superlattice constant at a temperature of 4.8 K and a concentration of 1% of a monolayer (ML) of Ce adatoms to an interatomic distance of 3.2 nm. On the other hand, there is some freedom to increase the adatom concentration to 2%, where additional adatoms are squeezed into the superlattice and a soft hexagonal superlattice is maintained at reduced interatomic distances of 2.2 nm. At higher adatom concentration, more and more Ce dimers are found and the superlattice collapses to compact islands. Increasing the temperature to 10 K leads to the collapse of the superlattice. These experimental findings are rationalized within the electron scattering model of Hyldgard and Persson, which specifically takes into account the electronic surface state on Ag(111). The experimentally derived two-body interaction potential is able to account for the observed phenomena as a function of concentration and temperature.

2. Experimental

Ce atoms were deposited from a thoroughly degassed tungsten filament onto a well-prepared Ag(111) surface held inside a low-temperature STM in ultrahigh vacuum (UHV) at 4.8 K. During specific experiments the temperature of the sample is lowered to 3.9 K by pumping on the He bath [15]. We used a cut PtIr wire as STM tip. Bias voltages $U_s$ refer to the sample voltage with respect to the tip.

3. Coverage-dependent self-organization

3.1. Ce adatom coverage of 0.03%

Figure 1 presents the Ag(111) surface at large scale ($210 \times 210$ nm$^2$) covered by 0.03% of a ML of Ce adatoms at 3.9 K. We have analysed the distribution of Ce nearest-neighbour (NN) separations in this image in a similar manner as Wahlström et al and Knorr et al [11, 13] for other adatom systems. The histogram of NN separations (within 12 nm of a given Ce adatom) reveals that the neighbouring Ce adatoms are not randomly distributed on the surface but that preferential separations between adatoms exist. The first preferential distance is about 3 nm, the second one at 7 nm and the third one near 11 nm.
Figure 1. 210 × 210 nm² STM topography of a Ag(111) surface covered by 0.03% of a ML of Ce adatoms (about 120 adatoms in the image) at a temperature of 3.9 K \((U_s = -100 \text{ mV}, I_s = 20 \text{ pA})\). (b) Histogram of nearest-neighbour (NN) adatom–adatom separations.

Figure 2. 105 × 105 nm² STM image of the Ag(111) surface covered by 0.2% of a ML of Ce adatom (about 200 adatoms in the image) at a temperature of 3.9 K \((U_s = -100 \text{ mV}, I_s = 20 \text{ pA})\). See also the movie. Histogram of NN adatom–adatom distances.

3.2. Ce adatom coverage of 0.2%

At a concentration of 0.2% of a ML of Ce adatoms, figure 2 shows that rows of few Ce adatoms and small close-packed island arrangements coexist. The histogram of NN adatom–adatom separation (200 adatoms) shows that the typical adatom–adatom separation is now essentially about 32 Å, whereas the second adatom–adatom separation peak at 70 Å is very weak. The position of the Ce adatoms on this area of the surface was recorded while increasing the temperature from 3.9 to 4.8 K (see movie). This small increase in temperature drastically affects the adatom mobility on the surface. At 3.9 K, the hopping time of Ce adatoms on Ag(111) is estimated to be 150 s [14], i.e. during the time of a line scan over an atom (16 s) the Ce adatom
structures can be considered to be stable. At 4.8 K, the hopping time of Ce adatoms is about 0.3 s, the adatoms become mobile and adatom–adatom arrangements are different in two consecutive images (5 min apart). The typical Ce adatom patterns (closed-packed islands and rows of adatoms) are still visible, displaying clearly an adatom–adatom interaction. Immobile species are also observed on the surface (in green in the movie), which are attributed to Ce dimers (see below). We note that our observations of 1D structures are in excellent agreement with recent findings for Co adatoms on Cu(111) by Stepanyuk et al [17], who showed, using density functional theory (DFT) and STM, that the effect of quantum interference of surface-state electrons on adatom motion leads to the self-assembly of 1D structures on metal surfaces.

3.3. Ce adatom coverage of 1%

Figure 3 shows a STM topograph after deposition of about 1% of a ML of Ce onto Ag(111). The Ce adatoms now self-organize into a large hexagonal domain covering the Ag(111) surface at macroscopic distances [14]. The superlattice is visible on all large-scale STM images but also at different regions of the sample separated by macroscopic distances (2 mm). Such a long-range ordered superlattice has not been observed before, neither with Cu on Cu(111) [11, 12] nor with Co adatoms deposited at the same concentration on Ag(111) [13].

Figure 4 shows a STM topograph after deposition of about 1% of a ML of Ce onto Ag(111) at 3.9 K. The Ce adatoms form a well-ordered hexagonal superlattice covering the Ag(111) surface. The distance between two neighbouring adatoms is determined to be $32 \pm 2$ Å as observed in the histogram of the next-neighbour adatom–adatom distances in the image (520 adatoms).

**Figure 3.** STM topography of a self-assembled superlattice of Ce adatoms deposited on Ag(111) at 4.8 K. Image size: $140 \times 135$ nm$^2$ ($U_s = +200$ mV, $I_s = 100$ pA). The Fourier transform (inset) confirms the 2D hexagonal superlattice structure on a large scale.
Figure 4. $69 \times 69 \text{nm}^2$ STM topography of a Ag(111) surface covered by 1% ML of Ce adatoms (about 520 adatoms in the image) at a temperature of 3.9 K ($U_s = -100 \text{mV}$, $I_s = 10 \text{pA}$). The Fourier transform of this image (inset) confirms the well-ordered 2D hexagonal superlattice structure, the horizontal line indicates the length in reciprocal space and the peak intensity is coded according to the colour scale bar. The histogram of NN adatom–adatom separations displays a typical adatom–adatom distance of about 32 Å.

Step edges are efficient 1D scatterers for 2D surface-state electrons [23, 24]. The standing-wave pattern in the LDOS near a step edge results from the superposition of incident and scattered electron wave functions at the step leading to a pronounced oscillating amplitude of the LDOS very close the step edge. Figure 5 shows that the Ce adatoms are aligned parallel to the step edge. The position of the first Ce adatoms are at a distance of $26 \pm 2 \text{Å}$ perpendicular to the step edge, which corresponds to the first maximum in the LDOS at the Fermi level near a step edge for the Ag(111) surface [20]. This finding confirms previous observations, which suggested that adatoms are preferentially located at positions where they share LDOS standing wave maxima [11].

3.4. Ce adatom coverage of 2%

Figure 6 presents a topography of the Ag(111) surface covered by 2% of a ML of Ce adatoms. At this concentration, the adatoms still self-organize into a hexagonal superlattice, as confirmed by the Fourier transform of the image. The typical interatomic distance is now reduced to about $22 \pm 2 \text{Å}$, as displayed in the histogram of the NN adatom–adatom distances (1090 adatoms). We note that this superlattice shows more disorder than the one at 1% Ce adatom concentration. This fact is also visible in the FFT insets of figures 4 and 6. The reason for more disorder in the superlattice lies in the increase of the number of Ce dimers with increasing adatom concentration.

4. Stability of the adatom superlattice

4.1. Evolution with temperature

High-resolution topographies of the Ce superlattice are presented in figure 7 at two sample temperatures. At 3.9 K the Ce adatoms appear as stable protrusions, whereas already at 4.8 K
Figure 5. (a) 38 × 53 nm² STM topography of Ce adatoms near a step edge on Ag(111) at 4.8 K (Uₜ = −100 mV, Iₜ = 30 pA). (b) Line profile taken perpendicular to step edge (see inset).

Figure 6. 69 × 69 nm² STM topography of a Ag(111) surface covered by 0.02 ML of Ce adatoms (about 1090 adatoms in the image) at a temperature of 3.9 K (Uₜ = −93 mV, Iₜ = 20 pA). The Fourier transform of this image (inset) confirms the 2D hexagonal superlattice structure, the horizontal line indicates the length in reciprocal space and the peak intensity is coded according to the colour scale bar. The histogram of NN adatom–adatom distances displays a typical adatom–adatom separation of about 22 Å.
they display a ‘frizzy’ image indicating a finite adatom mobility. At 3.9 K adatom hopping time from one silver site to another is about 150 s, whereas it is about 0.3 s at 4.8 K [14]. As in our measurement at 4.8 K, one linescan is recorded in 1 s, the available statistical time to perform a hop during one linescan is sufficiently long for the Ce adatom on Ag(111), the adatoms appear ‘frizzy’. The increase of the Ce adatom mobility with temperature in their superlattice sites is clearly visible in the movie. We note that at 4.8 K, Ce adatoms are confined in their superlattice sites and are not diffusing on the surface as observed at lower concentration (see the movie). Fixed species are attributed to Ce dimers. It is probable that the dimers appear to be close to superlattice sites because the surrounding mobile Ce atoms have adjusted their positions to best incorporate the fixed dimers into a superlattice. Additional formation of Ce dimers was not observed at 4.8 K, pointing towards a repulsive interaction between Ce adatoms.

4.2. Adatom displacement

Figure 8 shows a sequence of STM topographies recorded at different tunnel resistances. The superlattice is recorded in figure 8(a), and figure 8(b) shows the same area taken at a bias voltage increased by a factor of 12. The tip seems to repel now locally Ce adatoms, but not dimers, from their superlattice site. Subsequently, the tunnel parameters were reset to those utilized in figure 8(a) and the topography shown in figure 8(c) was obtained. Evidently the hexagonal superlattice is restored. The time between two consecutive images is about 10 min. This reformation of the Ce adatom superlattice underlines the low-diffusion barrier of the Ce adatoms on Ag(111) and, concomitantly, the adatom–adatom interaction strength. Creation of new Ce dimers was not observed during this sequence of scanning processes, indicating a repulsive interaction between Ce adatoms. We note that missing Ce adatoms in the superlattice are not imaged at the same locations in figures 8(a) and (b). This observation seems to indicate that Ce adatoms are displaced by the tip by a distance larger than one Ce–Ce superlattice constant.
Figure 8. Sequence of STM topographies (67 × 67 nm²) taken at identical tunnelling current (Iₜ = 50 pA) but different tunnelling voltages. (a) Uₛ = −100 mV. The superlattice of Ce atoms is visible. (b) Uₛ = −1.2 V. Now only immobile Ce dimers are imaged. (c) Uₛ = −100 mV. The hexagonal superlattice of Ce adatoms is restored.

5. Analysis and discussion

5.1. Phase shift

The origin of adatom–adatom interactions on the (111) noble metal surfaces was predicted long ago [10] and revealed experimentally by recent STM studies of metal-adsorbate systems [11]–[13]. It was found that the surface-state electrons influence the long-range spatial distribution of adatoms via scattering at the adatom sites [21]. The two-body interaction energy decays as 1/ρ², where ρ is the distance between two adatoms, and oscillates with a period of λₚ/2, half of the Fermi wavelength of the surface-state electrons. An important parameter in the model is the scattering phase shift δ₀ of the surface-state electrons at the adatom site, which can be obtained independently from an analysis of the standing-wave patterns. Figure 9 shows a STM topograph of an isolated Ce adatom at 3.9 K measured near the Fermi level at a tunnel bias voltage of −3 mV. The circular electron density oscillations correspond to the well-known standing-wave patterns near adsorbates on the (111) surfaces of noble metal surfaces [23]. The variation in the local density of states (ΔLDOS) at the Fermi level originating from the interference of incident and scattered surface-state electrons at adatom sites is given by [23]

\[
\Delta \text{LDOS}(\rho) \propto \frac{1}{k_F \rho} \left( \cos^2 \left( k_F \rho - \frac{\pi}{4} + \delta_0 \right) - \cos^2 \left( k_F \rho - \frac{\pi}{4} \right) \right),
\]

where \( k_F = \sqrt{2m^*(-E_0)/\hbar^2} \) is the Fermi surface wavevector (0.0813 Å⁻¹) with the surface-state band edge \( E_0 = -63 \) meV for Ag(111) [24], \( \rho \) is the distance from the position of the centre of an adatom and \( m^* = 0.40m_e \) [25] is the effective mass with \( m_e \) the free electron mass. The periodicity of the standing-wave oscillations at \( E_F \) is given by \( \lambda_F/2 = 38 \) Å. However, the position of the first maximum of the LDOS from the adatom is determined by the scattering properties of the adatom. For a perfect scatterer, \( \delta_0 = \pi/2 \), the position of the first maximum of the LDOS oscillations at \( E_F \) would be at 38 Å. Our analysis, shown in figure 9, yields a value of \( \delta_0 = (0.37 \pm 0.05)\pi \), similar to the phase shift that Knorr et al found for Co on Ag(111) [13]. Consequently, the scattering properties of the Ce adatoms determine the position of the first
maximum in the electron density oscillations at $E_F$ to be at a distance of 32 Å, instead of 38 Å from the adatom.

5.2. Interaction potential

The interaction energy $\Delta E_{int}$ mediated by a Shockley surface-state band between two adatoms at a distance $\rho$ on Ag(111) is given by [21]

$$\Delta E_{int}(\rho) \simeq -AE_0 \left( \frac{2 \sin \delta_0}{\pi} \right)^2 \frac{\sin(2k_F \rho + 2\delta_0)}{(k_F \rho)^2}.$$  \hspace{1cm} (2)

where $A$ is a scattering amplitude as defined in [13] that expresses scattering from surface into bulk states. Previous experiments show that $A = 0.27$ in the case of Ce/Ag(111) [14]. The phase shift $\delta_0$ obtained for Ce/Ag(111) imposes the position of the minimum in $\Delta E_{int}$. Theory predicts that, with $\delta_0 = 0.37 \pi$, $\Delta E_{int}$ has its deepest minimum at the measured Ce adatom distance of 32 Å and that the subsequent minima are located at distances which are multiples of $\lambda_F/2 = 38$ Å. Consequently, the first three preferential adatom–adatom separations are 32, 70 and 108 Å, in excellent agreement with the experimental results presented in figures 1 and 2. In addition, the analysis of the standing-wave pattern and of the interaction potential shown in figure 9 clearly demonstrates that the first maximum of the LDOS coincides with the first minimum of the interaction potential, i.e. with the position of the NN adatom. Evidently, a surface location with a high LDOS constitutes a preferred binding site for the Ce adatoms.

Applying the above theoretical model, we are now able to rationalize the conditions for formation and stability of the 2D hexagonal Ce adatom superlattice. The Ce adatom–adatom interaction potential, shown in figure 9, exhibits a maximum at a distance of 20 Å from the Ce...
Figure 10. Superposition (full line) of the diffusion potential for Ce on Ag(111) and the long-range potential mediated by the surface-state electrons (broken line): (a) hexagonal superlattice well, (b) two-body potential. The height of the diffusion potential barrier is 10 meV.

adatom at the origin, which in principle should prevent dimer formation. Consequently, the first and deepest minimum, located at 32 Å from the Ce adatom, governs ‘naturally’ the superlattice parameter. The depth of this two-body potential well has been determined to be 0.8 meV by an analysis of the Ce adatom motion in their superlattice sites [14]. The deposited Ce adatoms must have sufficient thermal energy to overcome by diffusion the outer potential barriers located at 5 and 9 nm from the Ce adatom at the origin and to lock-in ‘their’ prescribed superlattice site at 3.2 nm. Consequently, the sample temperature has to be chosen carefully in order to allow for a match between the thermal excitation of the Ce adatom with the surface diffusion barrier of Ce on Ag(111). In addition, the depth of the first adatom–adatom potential well at 3.2 nm should be of the same order of magnitude in order to allow for Ce adatom diffusion within the well (extending over 11 Ag surface atom distances) but prevent the trapped Ce adatoms from escaping from the well or to form Ce dimers. This situation is illustrated in figure 10(a), where the full line represents the sum of diffusion potential and long-range potential. The diffusion potential barrier for Ce on Ag(111) has been estimated from the observed hopping time of 0.3 s at 4.8 K. The energy barrier $E_D$ is then deduced by using the Arrhenius law

$$\nu = \nu_0 \exp \left( -\frac{E_D}{k_B T} \right) .$$

(3)

Assuming an attempt frequency $\nu_0 = 10^{12} \text{s}^{-1}$, we deduce $E_D$ to be about 10 meV. This is of the same order of magnitude (11 meV) as the trapping potential felt by each Ce adatom in the superlattice [14]. Figure 10(b) shows that already a low-potential barrier as found for Ce on Ag(111) is predominant with respect to the long-range potential. If higher temperatures are required to allow the adatom diffusion (as for Cu/Cu(111) [12] or Co/Ag(111) [13]) it is possible that the long-range potential is too weak to be relevant with respect to the diffusion potential. For the system Ce on Ag(111) the peculiar energetic balance between diffusion barrier height and potential well depth, necessary for successful superlattice formation, is realized. In our case of Ce on Ag(111), the temperature of 4.8 K fulfils these conditions as can be observed in the movie of figure 2.
The increase in the Ce adatom concentration on the surface has two consequences, (i) the amplitude of the trapping potential (due to NN interactions) will increase and (ii) the adatom motion will be confined to the first minimum of the adatom–adatom potential with the concomitant increase in adatom–adatom repulsion. Figures 3 and 5 illustrate that a temperature of 4.8 K and a concentration of 1% of a ML of Ce adatoms lead to the formation of a stable Ce adatom superlattice. Cooling of the sample to 3.9 K ‘freezes’ the Ce adatoms in their superlattice site and leads to a well-ordered hexagonal superlattice as observed in figure 4. At higher Ce adatom concentration, as all NN sites in the adatom superlattice are already occupied, the additional Ce adatoms have to ‘climb-up’ the inner potential well (see figure 9) and to reduce their adatom–adatom separation. For example, in the case of 2% of a ML shown in figure 6, the hexagonal superlattice still survives; however its superlattice constant is reduced to 22 Å. This finding shows an interesting additional way to further tune the superlattice constant.

6. Summary

To summarize, by increasing the Ce adatom concentration on Ag(111) from the dilute limit to about 0.02 ML we have followed in detail the 2D self-assembly of a hexagonal superlattice of metallic Ce adatoms. We have shown that the long-range order is mediated by the electrons in the 2D surface-state band of Ag(111). Moreover, we have established that a subtle balance between sample temperature, adatom diffusion barrier, and concentration-dependent adatom interaction potential is at the origin of the superlattice formation. Variation of support and of adatom element as well as adatom concentration may allow us to tune the lattice constant of the superlattice over a wide range. In the case of magnetic adatoms, we can envisage forming ferromagnetic or antiferromagnetic adatom pairs at different separations. Such a 2D Kondo lattice may be of interest for electron-spin-based quantum computing. The ability to control the long-range order of metallic adatoms opens new possibilities for the investigation and development of atomic-scale devices.

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