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Step contrast reversal in LEEM during Pb deposition on W(110)

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Abstract

Anomalous step contrast in low energy electron microscopy (LEEM) images observed during Pb deposition on a W(110) surface is discussed. The steps are dark on the clean surface, and become bright by Pb deposition at about 200 °C. The contrast reversal is related to the presence of a two-dimensional (2D) Pb gas on the surface and its atomic density distribution. Upon further deposition the steps become dark again and show an anomalous intensity profile. This change is attributed to the 2D crystallization process.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Low energy electron microscopy (LEEM) is a useful technique for the investigation of surface phenomena. Two types of contrast are seen in LEEM images in general [1]. In most cases diffraction governs the LEEM contrast. The energy-dependent contrast between different surface structures is a typical case. Bright and dark field images can be observed by selecting different diffraction spot with the contrast aperture. The other contrast is interference contrast, which arises from the interference of electron waves reflected from two different planes. Quantum size contrast, which is the result of the interference between waves reflected on the top of the thin film and at the interface, has been modeled with the Kronig–Penney model as a function of film thickness [2]. Step contrast, which results from the interference between waves reflected from the bordering terraces, has been simulated by Fresnel diffracted waves from two apertures, which model the upper and lower terraces [3].

Diffraction contrast is strongly influenced by disorder on the surface, for example by randomly distributed adatoms on the surface, which cause diffuse scattering, thus weakening the intensity of the (00) beam that is used in general for imaging. Random adatom distributions form during adsorption or deposition at temperatures at which the adsorbed atoms do not form ordered structures. de la Figuera *et al* have recently used this phenomenon to determine the adatom

concentration in the two-dimensional (2D) Ag gas that forms during deposition at high temperatures on W(110) before the onset of 2D crystallization [4]. If the concentration of adatoms near steps is different from that on the terraces then step contrast is expected to be affected too.

Step contrast is generally observed as a dark line in the bright surrounding terraces. Here we report on anomalous step contrast reversal from dark to bright and back again to dark during Pb deposition on a W(110) surface.

2. Experimental details

The W(110) surface was heated repeatedly for 10 min at about 1000 °C in O₂ at a pressure of about 5×10^{-7} Torr to remove carbon. After this treatment, the sample was flashed to about 1800 °C to remove the surface oxygen. More than 100 cycles were used to produce a clean W(110) surface as judged by low energy electron diffraction (LEED). The heating of the crystal was done by electron bombardment from the backside of the specimen. The temperature was measured with a thermocouple attached to the sample cartridge, which was calibrated with the melting point of three-dimensional (3D) Pb islands on the surface.

Pb was deposited from an Mo crucible using a commercial electron bombardment evaporator. The deposition rate was about 0.01 ML min⁻¹, where 1 ML is the atomic density of the

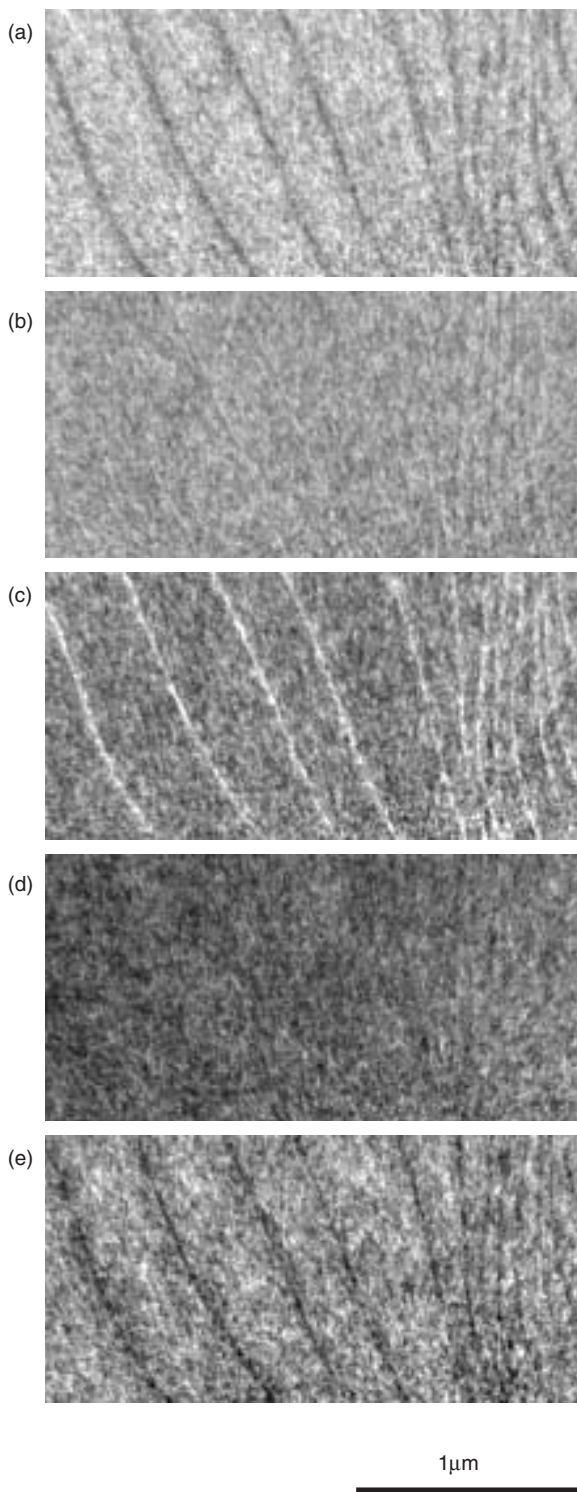


Figure 1. LEEM images during Pb deposition. (a) Clean W(110) surface. Relative coverages: (b) $\theta^* = 0.09$, (c) $\theta^* = 0.19$, (d) $\theta^* = 0.64$, (e) $\theta^* = 0.84$. The electron energy is 7.5 eV.

W(110) surface. During deposition the substrate temperature was kept at about 200 °C.

The growth process was monitored with LEEM and LEED. The details of our apparatus have been described elsewhere [5]. The LEEM images shown here are bright field images taken with the (00) beam.

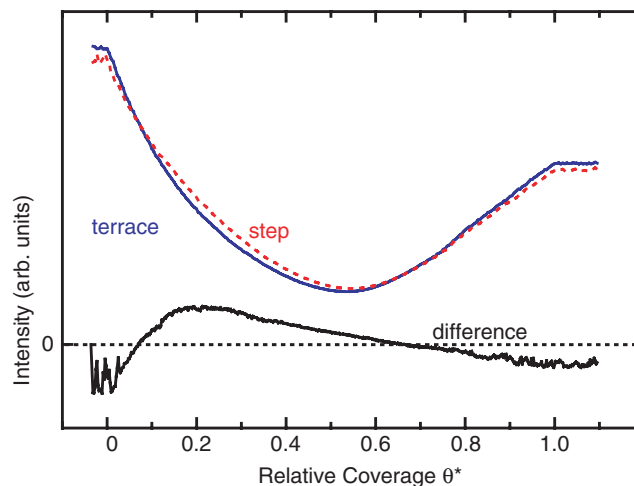


Figure 2. Variation of the LEEM intensity on terraces and along steps as a function of relative coverage.

3. Results and discussion

Figure 1 shows a series of LEEM images taken during Pb deposition. The electron energy is 7.5 eV. Mono-atomic steps on the clean surface are dark as shown in figure 1(a). With Pb deposition, the step contrast disappears as shown in figure 1(b). Steps appear again with bright contrast (figure 1(c)) and smear out again as shown in figure 1(d). With further increasing coverage, dark step contrast develops again (figure 1(e)) and finally 3D Pb islands start to form. The Pb coverage is represented here as relative coverage θ^* , where $\theta^* = 1$ is defined by the start of the 3D island formation.

The width of the step contrast is almost constant within the resolution limit of our LEEM in the first contrast reversal from dark to bright, therefore step decoration by Pb can be ruled out. In order to see how the contrast changes in detail, the LEEM intensity along steps and on terraces is measured as a function of coverage as shown in figure 2. The difference between the intensity along steps and on terraces is also shown in the figure. Since on the clean surface ($\theta^* = 0$) the intensity on terraces is larger than that along steps, steps appear with dark contrast in the LEEM image. Both intensities decrease with coverage, however the decrease of the intensity along steps is slower than on terraces. Therefore the intensity along steps becomes larger than that on terraces at around $\theta^* = 0.08$, so that the step contrast reverses from dark to bright. The intensity shows a minimum around $\theta^* = 0.55$ and then increases again with coverage. A second contrast reversal, now from bright to dark, can be seen at around $\theta^* = 0.66$. Therefore the different dependence of the intensity on coverage along steps and on terraces determines the step contrast reversal. de la Figuera *et al* showed that the LEEM intensity decreases linearly with coverage while depositing Ag on W(110) surface at 400 °C. At higher temperature the decrease is nonlinear due to the competition between incoming and desorbing flux [4]. The nonlinear decrease of the LEEM intensity shown in figure 2 is not caused by desorption but rather by the increasing overlap of the scattering cross-sections.

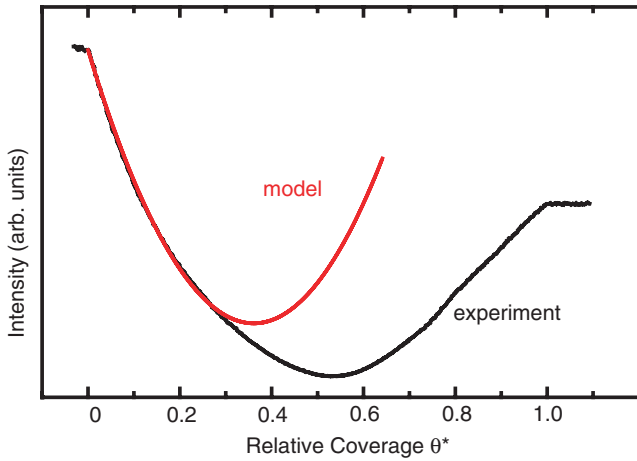


Figure 3. Model fit of the experimental intensity on terraces.

Now, we consider the decrease of LEEM intensity on the terraces, which is equivalent to the intensity of the (00) diffraction spot. The growth process of Pb on a W(110) surface at room temperature has been observed with LEED and Auger electron spectroscopy (AES) by Bauer *et al* [6]. They showed that the background in the LEED pattern gradually increases with Pb coverage and that clear LEED spots from the condensed Pb layer do not appear until around 0.5 ML. This observation implies that in the initial stage of the growth the surface is covered by a 2D Pb gas. Taking the contribution from the substrate and individual Pb atoms in the gas into consideration, the diffraction intensity can be expressed as

$$I = I_m - (I_m - I_0) \left[1 - \frac{\theta}{\theta_m} \right]^n,$$

where I_0 is the initial diffraction intensity before deposition, I_m the minimum intensity, θ_m the coverage at which $I = I_m$. The experimental intensity variation on terraces can be fitted with this equation as shown in figure 3, where I_0 , I_m and θ_m are taken from the experiment and n is a fitting parameter. As seen in the figure, the experimental curve is well fitted with $n = 2$ up to around $\theta_c^* = 0.28$. Any other value of n could not reproduce the experimental curve.

θ_c^* is the limit of the 2D Pb gas phase on the surface at this temperature, and it is expected that condensation starts beyond

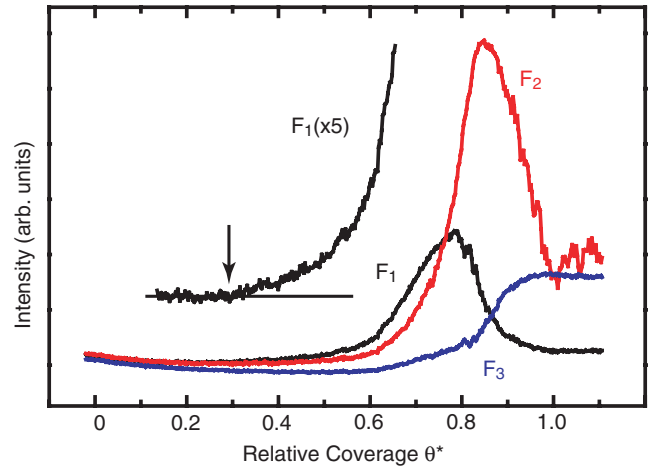


Figure 5. Change of the intensities of the LEED spots F_1 , F_2 and F_3 indicated by circles in figure 4.

θ_c^* . This could be confirmed by LEED. Figure 4 shows several LEED patterns observed during Pb deposition. In figure 4(a), a pair of spots, in which one of the spots is indicated by F_1 , can be seen, and this surface structure is called ‘structure I’ hereafter. These two spots move closer to each other with increasing coverage and form a single spot F_2 as shown in figure 4(b) (structure II). These spots move along the $[1\bar{1}0]$ direction and finally the LEED pattern shown in figure 4(c) is observed at $\theta^* = 1$ (structure III). The sequence of LEED patterns observed here is the same as that reported by Bauer *et al* [6]. They proposed the atomic arrangement corresponding to these LEED patterns. More precisely, the LEED pattern of structure III observed by Bauer *et al* differs slightly from that shown in figure 4(c). They observed almost a 4×1 structure, whereas the spots indicated by the arrow in figure 4(c) do not merge. This difference is caused by the different substrate temperature. The present substrate temperature is rather high so that fewer atoms are incorporated in structure III before 3D crystals nucleate. The change of the position of the LEED spots from structure II to structure III as a function of coverage can be easily derived using the structure model. For the LEED pattern shown in figure 4(c), the absolute atomic density at the saturation ($\theta^* = 1$) is estimated to be about 10.3×10^{14} atoms cm^{-2} , which corresponds to 0.72 ML.

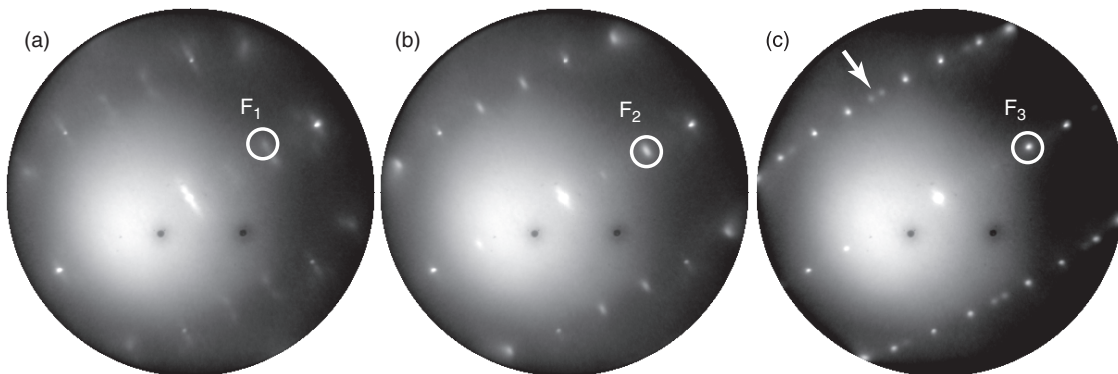


Figure 4. LEED patterns during Pb deposition. The electron energy is 44 eV.

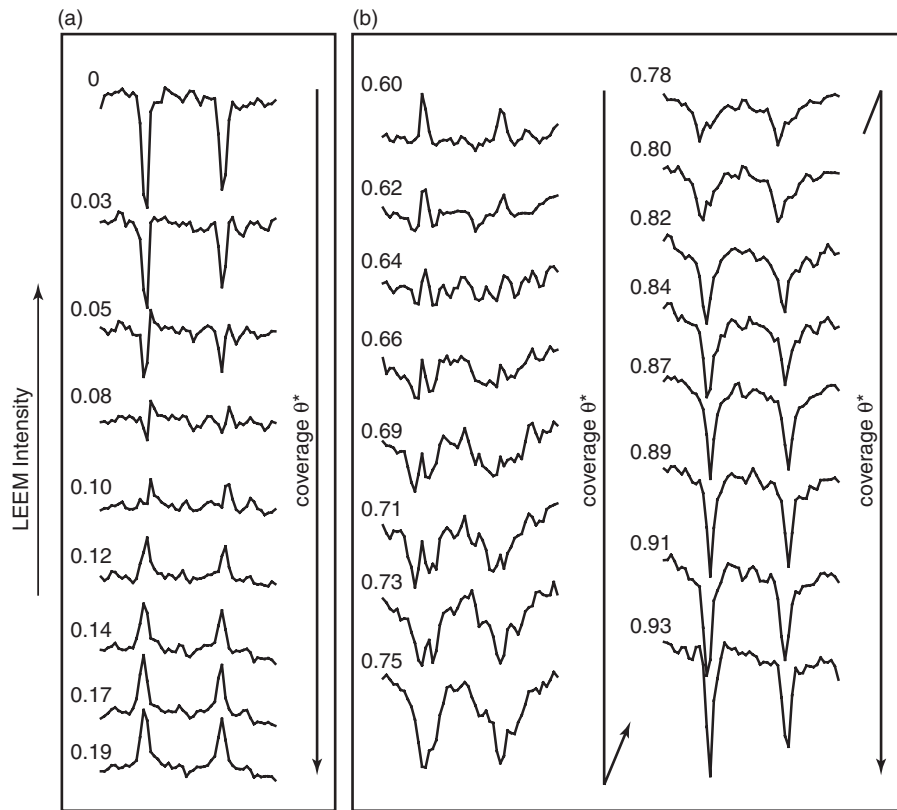


Figure 6. Change of the LEEM intensity profile across steps. (a) The first contrast reversal and (b) the second reversal. The numbers shown are the relative coverages. From the bottom of the center panel to the top of the right panel the scale was changed by a factor of 0.4.

The condensation of Pb from the 2D gas phase was monitored by measuring LEED spot intensities. Figure 5 shows the dependence of the intensities of the spots F_1 , F_2 and F_3 indicated in figure 4 upon coverage. The intensity of F_1 becomes strong at around $\theta^* = 0.6$, and has a maximum at about $\theta^* = 0.79$. Then spot F_2 develops because the spots F_1 merge into spot F_2 . After the intensity maximum of spot F_2 , the spot moves along the $[1\bar{1}0]$ direction toward spot F_3 , therefore the intensity of spot F_2 decreases and instead the intensity of spot F_3 becomes large. An important point is the onset of spot F_1 at about $\theta^* = 0.28$ indicated by the arrow. The onset of this spot agrees well with the limit of the 2D gas phase seen in figure 3. This clearly indicates that the 2D Pb gas starts to condense into 2D crystals at around $\theta_c^* = 0.28$.

Initially the LEEM intensity on terraces decreases due to the formation of the 2D gas phase. However, the intensity still continues to decrease up to around $\theta^* = 0.55$. As seen in figure 5, the intensity of spot F_1 increases rapidly at about $\theta^* = 0.6$. This suggests that up to around $\theta^* = 0.55$ the crystals are very small and contain imperfections, which induces the continuous decrease of the (00) beam intensity. With the rapid increase of the intensity of spot F_1 above about $\theta^* = 0.6$, the LEEM intensity also recovers.

The intensity variation along the steps shows a different behavior, which is the origin of the step contrast reversal. As mentioned before, step contrast is caused by the interference of electrons reflected on the upper and lower terraces [1]. Therefore the intensity along the steps should depend on the

intensity on terraces and consequently the intensity variation along steps should be similar to that on the terraces. A plausible explanation of the intensity difference between steps and terraces is a difference of the atomic concentration of 2D gas. Because of the large difference between the atomic radii of Pb and W the atomic density around steps is likely to be lower than that on terraces so that the LEEM intensity along steps is less reduced than on terraces.

Finally we look at the intensity profiles across steps to see the step contrast reversal process in more detail. Figure 6 shows the cross-sectional intensity distribution across steps as a function of coverage. Figure 6(a) shows the change of the profile during the first contrast reversal. The initially dark step contrast gradually smears out and changes to the opposite direction. The width of the profile in this stage is almost constant. On the contrary, the change of the profile in the second reversal process shown in figure 6(b) shows some peculiarities. Both sides of the step become darker than the surrounding terraces, initially with bright contrast within the center, which decreases with increasing coverage while the dark side bands become broader than on the clean surface in figure 6(a) and stronger. With further deposition, the dark contrast increases more and becomes narrower. At $\theta^* = 1$, the width of the step contrast is comparable with the initial step width on the clean surface.

The first contrast reversal was explained by the lower atomic concentration of the 2D gas near the steps as described above. The second contrast reversal is closely related to the

crystallization process of the 2D Pb layer. Because of the large lattice mismatch between Pb and W, crystallization is expected to occur first on the terraces and spread towards the steps. This leaves initially bright contrast directly at the steps and causes the slow growth of the dark side bands. As long as the 2D crystals are not very close to the steps, the distances between them and the steps will fluctuate, causing the broad dark regions. With increasing coverage of the terraces by 2D crystals these distances decrease, narrowing the dark regions until at full coverage the line width observed on the clean surface is recovered. A more quantitative analysis of these intriguing changes requires less noisy profiles, which could not be achieved in the present study.

4. Summary

The step contrast observed with LEEM during Pb deposition on W(110) at about 200 °C shows an anomalous contrast reversal. The first reversal from dark to bright contrast is explained by the presence of a 2D Pb gas on the surface and its atomic density difference between near steps and on terraces. With

increasing coverage the 2D gas condenses into 2D crystals, which cause a second contrast reversal. A change of the width of the step contrast is observed and is attributed to the details of the crystallization process.

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