Defects on the Pt(100) surface and their influence on surface reactions—A scanning tunneling microscopy study

by W. Hösler
R. J. Behm
E. Ritter

Structural differences between a clean, reconstructed Pt(100) surface and one exhibiting chemical or structural irregularities have been identified by means of a scanning tunneling microscope. The (temperature-dependent) defect structure of a surface which had undergone a phase transition involving mass transport was characterized and compared to results obtained using other techniques. The catalytic activity of surface step sites was probed by the thermal decomposition of ethylene. The resulting surface roughening and buildup of carbon, which could be resolved in STM images, clearly showed that the decomposition proceeds from terrace edges.

1. Introduction

Surface defects are often considered as being active sites for many surface processes such as adsorption, dissociation/decomposition, associative reactions, etc. Therefore their characterization plays an important role in the understanding of these reactions—even on well-annealed, clean single-crystal surfaces, where such defects are expected to exist only in very low concentrations. Defects in this respect are generally understood as chemical contaminations or geometric irregularities such as steps and kinks. The work described here has been aimed at developing an

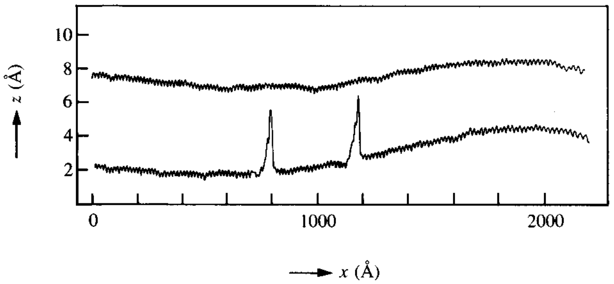

Figure 1

One-dimensional scans along the [011] direction of a well-annealed clean Pt(100) surface, exhibiting surface corrugation due to hexagonal reconstruction.

understanding of the topography and activity of such defects. In this paper, we take advantage of the specific ability of the scanning tunneling microscope (STM) to image local surface areas. Use of the STM for this purpose seems to be an improvement over methods based on diffraction phenomena.

In the first part of the paper we compare the topography of the clean reconstructed Pt(100) surface with that of a surface containing carbon ("chemical contamination") and with that of one which has been subjected to a brief ion bombardment ("geometric defects"). In the next part we focus on the topography of a surface which has undergone a surface phase transformation, including transport of surface atoms. In that case, the defects are introduced by the reaction of the surface with the adsorbate itself; i.e., the substrate-adsorbate interactions are strong enough to cause a rearrangement of the metallic surface. This effect—the adsorbate-induced reconstruction or removal of a reconstruction—is well known for a variety of adsorption systems [1]. We describe our studies for the case of the (temperature-dependent) surface roughening caused by CO adsorption on the reconstructed Pt(100) surface.

In the last part of this study we discuss the characterization of the activity of defect sites. Platinum is well known for its catalytic activity in the conversion of hydrocarbons [2]. The distinct effect of its surface steps and kinks ("geometric defects") has been especially noted [2, 3]. We have therefore used the decomposition of ethylene on a Pt surface as a test case. The resulting buildup of carbon could be directly resolved with an STM. We could thus determine the activity of certain sites by direct observation and spatial resolution of the increase in carbon coverage.

2. Experimental conditions

The STM used was fully UHV compatible. Its base pressure following bake-out was about 1×10^{-10} torr, and it contained standard facilities for surface cleaning (Ar^+ ion sputtering) and characterization [auger electron spectroscopy (AES), low-energy electron diffraction (LEED)]. Gases of high purity were admitted via bakeable leak valves. Their purity could be monitored using a quadrupole mass spectrometer.

The STM was based on concepts similar to those which applied to the STM developed by Binnig et al. [4]. Vibrational decoupling was achieved by two stages of spring systems and eddy current damping. The use of viton spacers and connectors at several points led to additional damping of high-frequency vibrations. The sample was brought close to the tip by a "louse" [4], which consisted of a piezoelectric body and could be electrostatically clamped to a fixed support platform. The louse was moved by alternating contraction and expansion. For movements over relatively large distances, the upper part of the louse (which carried the sample) could be picked up by a manipulator. The entire setup—frame system with support platform plus manipulator—could be rotated via a differentially pumped rotatable flange. The support of platform plus sample could thus be directed toward portions of the system where facilities for sample characterization and cleaning were located. The sample was rigidly clamped to a tantalum housing and could be indirectly heated during STM imaging, etc. by means of a filament contained therein. Sample temperature was determined by means of a Ni-CrNi thermocouple which was spot-welded to the sample holder.

Tunneling experiments were performed using voltages between 0.01 and 0.8 V which were applied to the sample while the tip was virtually grounded. The tunneling current—typically around 5 nA—was measured between tip and ground using a shielded connection. The tunneling tip consisted of a tungsten rod (1-mm diameter) which was ground to an angle of 90°. It was moved by three massive piezoelectric rods mounted normal to one another and connected to the support platform. The calibration of the rods was based on the measured height of well-defined steps on the surface of the sample. The feedback loop which was used to control the *z*-movement (normal to the surface) included a controller having a variable cutoff frequency for its proportional part [5].

The surface was prepared following standard cleaning procedures [6] and was subsequently annealed to 1100 K in order to achieve a well-ordered state which exhibited the LEED pattern of the hexagonally reconstructed Pt(100) surface [7–9]. Experiments were performed mostly at temperatures near 500 K to inhibit adsorption of the residual gas species and to reduce the time needed to achieve sufficient thermal stability after the annealing.

3. Results and discussion

- *Characterization of defects*

A well-annealed clean Pt(100) surface is characterized by flat terraces, several hundred to several thousand Å in diameter, which are separated by mostly mono- and bi-atomic steps.

Macroscopic defects such as etch pits, etc., insufficient annealing following ion bombardment, or persistent chemical contamination lead to a microscopically rough surface; terrace formation is no longer detectable. The well-annealed clean surface is well known to be reconstructed in its thermodynamically most stable state. The metastable (1×1) structure undergoes an irreversible reconstruction at $T > 420$ K [10, 11]. Its LEED pattern exhibits extra spots because of the presence of a (5×25) or "hexagonal" structure, and, upon annealing to $T > 1100$ K, of a "rotated" hexagonal structure. The corresponding real-space structures have been interpreted in terms of a hexagonal Pt topmost layer, which is rotated by 4.6° with respect to the substrate [7–9]. This structure model has recently also been confirmed by an STM study [12].

The flat portions of the surface exhibit a corrugation which is characteristic of the hexagonal reconstructed surface and which has been attributed to the mismatch between the underlying (1×1) bulk region and the (reconstructed) hexagonal topmost layer [9, 10]. The corrugation amplitude has been found to be ~0.4 Å for $0.02\ \text{V} < V_t < 1\ \text{V}$; the (one-dimensional) corrugation length has been found to be 14 Å, which corresponds exactly to the fivefold periodicity of the (5×25) or "hexagonal" structure [7–9, 12]. The lateral extension of the corrugated domains is demonstrated in **Figure 1**, which shows two scans over a reconstructed terrace ~2000 Å in width. One of the scans indicates that the surface is completely flat (except for the corrugation); the other contains two irregular features. The latter can be ascribed to small islands of "adsorbed" Pt which are—in [100] spacings—two layers high. Deviations from a linear baseline are due to a slight distortion effect (<0.1%) in the particular piezoelectric drive system and to nonlinearities in the thermal drift.

In **Figure 2** a model of the reconstructed surface is shown in two different aspects. It was constructed from a set of scans which covered an area of 500×340 Å. The images show the presence of two terraces separated by a monoatomic step and containing corrugations which are almost orthogonal to each other.

These "clean" structures contrast with those depicted in **Figures 3** and **4**, which were recorded at a similar sample temperature. Illustratively, geometrical defects induced by ion bombardment at 550 K are indicated in Figure 3. While long bombardment cycles result in a microscopically rough surface, brief bombardment ($t \sim 1$ min, $I_{\text{Ar}^+} \sim 1\ \mu\text{A}$, $E_{\text{Ar}^+} \sim 1$ kV) at 550 K, starting from a well-annealed surface, leads to the indicated surface structure. It is interesting to note that the surface is not rough in terms of point defects but exhibits a large number of steps and small terraces. This can be judged as a first indication of the (short-range) mobility of the Pt surface atoms at this temperature, since the bombardment (and associated sputtering) process itself is expected to create only a microscopically rough surface.

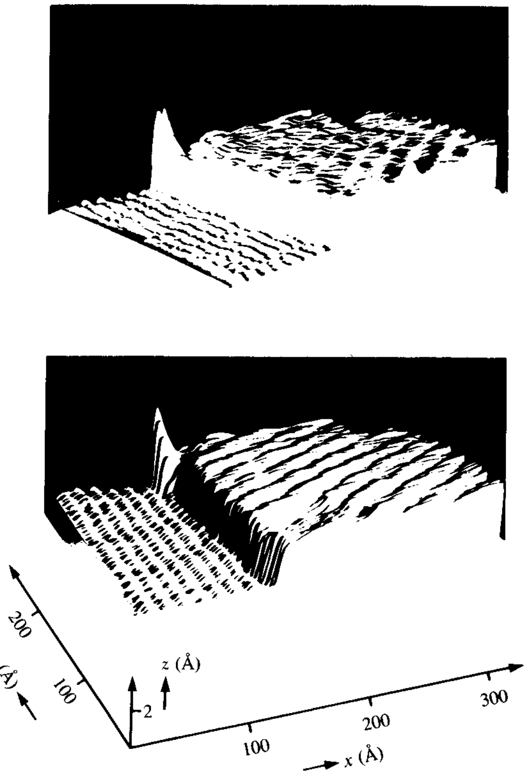

Figure 2

Images of a reconstructed Pt(100) surface, obtained from set of original scans.

The correlation between chemical contamination and structural variation is illustrated in Figure 4. Shown in that figure is a typical image from a sample which was annealed to 1100 K with small amounts of carbon present on its surface. The front central region is entirely clean and exhibits the corrugation of the reconstructed phase. It is surrounded by a monostep and a large number of narrow "tips." These "tips" correspond to small islands which, however, are several layers high (in [100] spacings); they represent a characteristic feature of carbon-contaminated surfaces after annealing. They cannot be removed by annealing alone, but only by additional oxidation. In the area between these islands, noncorrugated (therefore nonreconstructed) flat areas are seen. We attribute the "tips" to Pt clusters which are pinned together by carbon. They also exist predominantly close to or at step edges. This figure indicates that chemical contaminations are not necessarily only passively adsorbed on a Pt surface but may, after a high-temperature treatment, also lead to a massive rearrangement of the Pt atoms themselves.

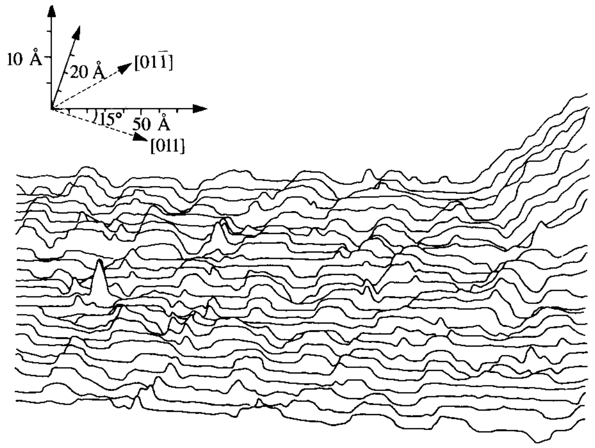

Figure 3

Two dimensional array of scans on a Pt(100) surface after annealing to 1100 K and a brief Ar$^+$ ion bombardment at 550 K (t = 1 min. I_{Ar^+} = 1 μA, E_{Ar^+} = 1 ke V).

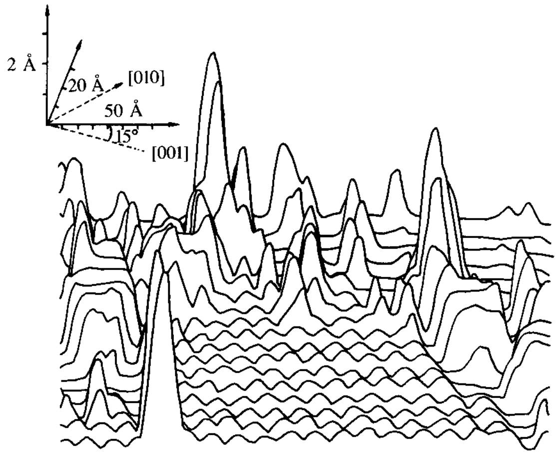

Figure 4

Two-dimensional array of scans on carbon-contaminated Pt(100) surface, following annealing at 1100 K.

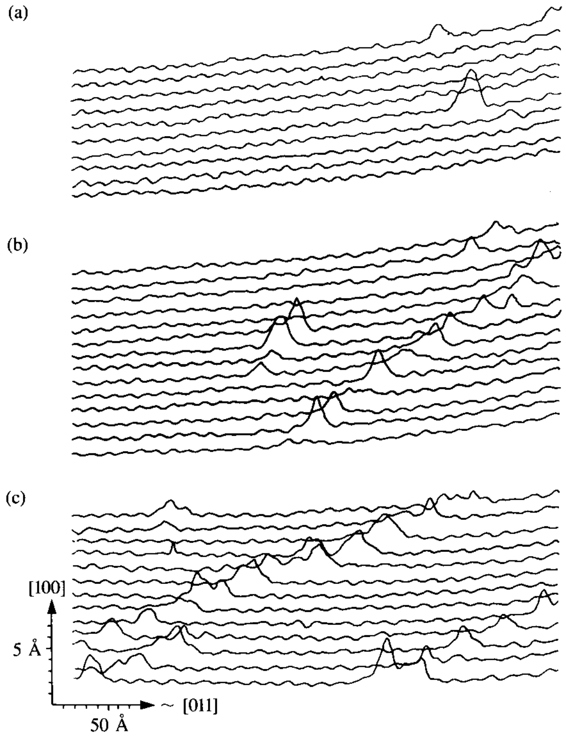

Figure 5

Exposure of reconstructed Pt(100) surface to CO at 460 K: (a) Two-dimensional array of scans of clean surface; (b) Sequence of scans during exposure at p_{CO} = 1 × 10^{-8} torr; (c) Sequence of scans during exposure at p_{CO} = 4 × 10^{-8} torr.

- *Topographical changes induced by surface reactions*

Surface reactions are not limited to adsorbates but may involve the substrate atoms as well. This process is often connected to a change in the density of the topmost metal layer. Because of the rather low temperatures at which these transformations can proceed, the question of mass transport has been raised repeatedly, and specific surface structure models have been proposed to avoid this difficulty [13]. We have investigated the transformation from the reconstructed Pt(100) hexagonal surface phase into the bulklike (1 × 1) structure which leads to a density decrease of ~20% in the topmost layer. The transformation can be facilitated by several adsorbates such as O, H, NO, or CO [6, 12, 14–16]; we have used CO here. In order to separate out the influence of adsorbed CO, we have also performed a set of experiments at temperatures above which CO adsorption is possible only in equilibrium with an ambient pressure; subsequent reduction of the CO partial pressure causes a rapid decrease in the adsorbate coverage. Any changes in the surface topology which persist at the end of this procedure must be due not to adsorbed CO molecules but rather to a rearrangement of the topmost Pt atoms caused by the intermittent interaction with adsorbed CO. The temperature was chosen to be 460 K, which just fulfills these conditions in the range of CO pressures experimentally accessible [6].

Figure 5(a) shows an array of scans of a 430 × 150-Å portion of a large flat area prior to CO exposure. The array exhibits the corrugation of the clean reconstructed surface. A set of line scans taken during exposure to a CO pressure (p_{CO}) of 1 × 10^{-8} torr is shown in **Figure 5(b)**. When the CO

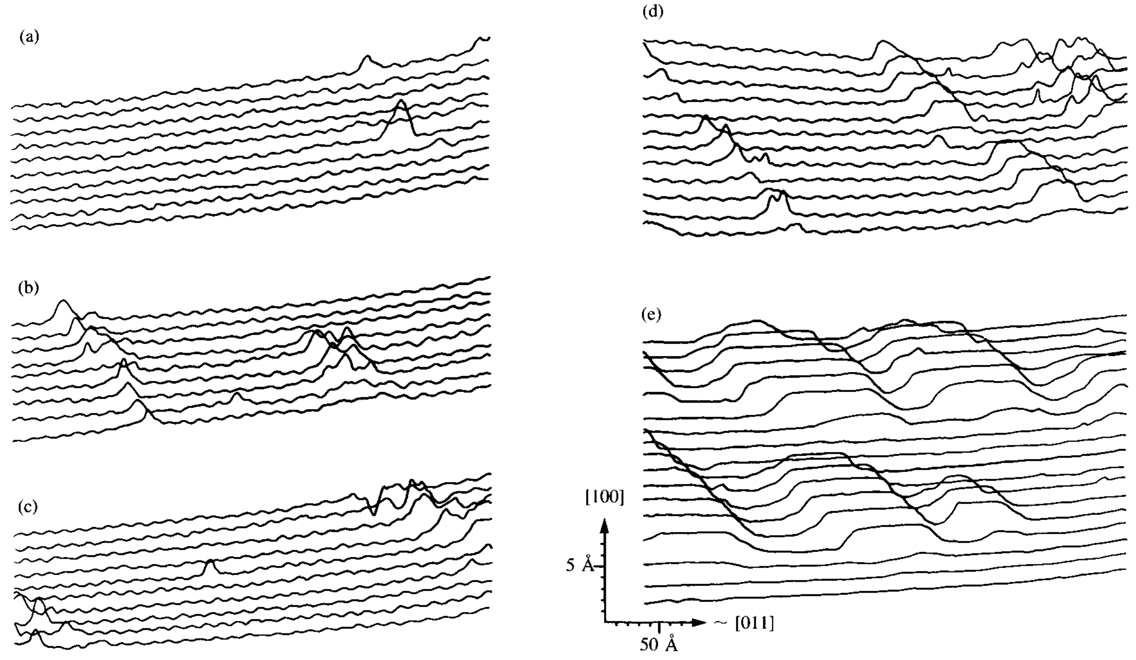

Figure 6

Reconstructed Pt(100) surface following CO exposure at $T = 460\mathrm{K}$: (a) Two-dimensional array of scans of clean surface; (b)–(e) Two-dimensional array of scans following subsequent exposure at $p_{\mathrm{CO}} = 1 \times 10^{-7}$, 2×10^{-7}, 1×10^{-6}, and 5×10^{-6} torr, respectively.

pressure is increased to this level, thermal drift effects lead to the observed shifts in the array, as indicated by the movement of the defects to the left. Except for this effect, no significant change is evident; e.g., the corrugation remains intact. The same applies for a sequence of line scans taken at $p_{\mathrm{CO}} \sim 4 \times 10^{-8}$ torr, as shown in **Figure 5(c)**, where the thermal drift effects are more pronounced.

If the pressure is further increased, the thermal drift effects become too pronounced to permit continuous measurements during exposure. We have therefore imaged the surface following CO exposure at a given pressure and subsequent pumpdown. The surface area scanned in these experiments was identical to that depicted in Figure 5. **Figure 6(a)** was obtained from the clean surface, and **Figures 6(b–d)** were obtained following CO exposure to $p_{\mathrm{CO}} = 1 \times 10^{-7}$, 2×10^{-7}, and 1×10^{-6} torr, respectively. In agreement with the results of Figure 5, none of the latter arrays were significantly different from that obtained from the clean surface. The density of defects seemed to be slightly greater, but large areas exhibiting the corrugation of the reconstructed surface remained visible. A distinct change did, however, appear when the CO pressure was raised to about 5×10^{-6} torr for 150 s [**Figure 6(e)**]. Most obvious was the loss of the corrugation from the flat portions of the surface. In addition a variety of mono-atomic steps appeared, suggesting that additional islands of Pt atoms were adsorbed on a smooth base. The widths of the terraces were typically between 50 Å and 100 Å, but larger flat areas were also detected. Following this experiment, we observed the LEED pattern of a (1 × 1) structure, without any indications of the extra beams related to surface reconstruction.

These changes are clearly related to the intermittent adsorption and desorption of CO. A closer look at the adsorption characteristic of CO on Pt(100) reveals that the sudden transition in the surface topography is not surprising. The adsorption isotherms of CO on Pt(100) indeed rise very steeply beyond a certain amount of coverage. It can be shown that this coverage also represents the critical coverage θ_{crit} (on the reconstructed surface) which is required to initiate the phase transformation into the (1 × 1) structure [6]. CO coverages below θ_{crit} reversibly desorb from the reconstructed surface, while higher coverages lead to a rapid and complete transformation into the (1 × 1) phase ("nucleation and trapping" mechanism [6]). The critical coverage apparently was exceeded when p_{CO} was increased to 5×10^{-6} torr. Because of the rapid CO desorption after the pressure was subsequently decreased, the structure in Figure 6(e) is interpreted as reflecting the rearrangement of the Pt

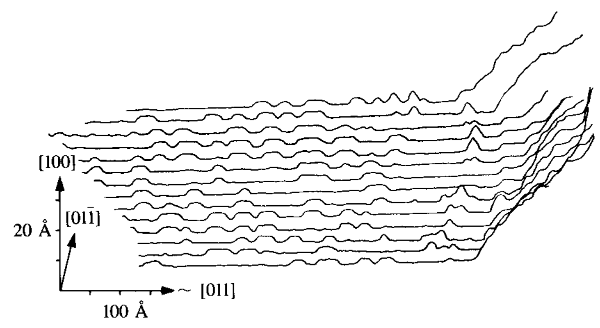

Figure 7

Two-dimensional array of scans of Pt(100) surface during exposure to CO at $T = 415$ K and $p_{CO} = 8 \times 10^{-8}$ torr.

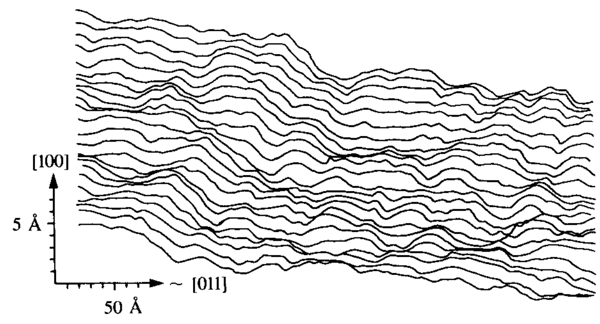

Figure 8

Two-dimensional array of line scans of Pt(100) surface following exposure to CO at $T = 350$ K.

itself; i.e., it does not show islands of CO_{ad} on a smooth surface. The Pt substrate does not immediately reconvert into a (well-ordered) reconstructed phase, since at this temperature the reconstruction rate is too slow (due to its activation barrier [10, 11]).

The influence of the adsorption temperature was investigated by examining the effects of CO exposure at a lower temperature, $T \sim 415$ K. While the pressure was increased stepwise, no significant change was observed until it reached $p_{CO} = 1 \times 10^{-8}$ torr. At $p = 4 \times 10^{-8}$ torr, however, a change like that depicted in Figure 6(e) occurred. A two-dimensional array of scans taken at $p_{CO} = 8 \times 10^{-8}$ torr (**Figure 7**) illustrates the resulting surface topography—viz., a flat surface plus strictly mono-atomic steps to again form small "islands" of Pt on a smooth substrate and the complete loss of surface corrugation. Neither the latter pressure increase ($4 \rightarrow 8 \times 10^{-8}$ torr) nor any subsequent pressure increase (to $p_{CO} = 5 \times 10^{-6}$ torr) caused a change in the characteristics of this pattern. While the features of the surface are comparable to those in Figure 6(e), a smaller island size is seen at lower temperatures. This effect is even more striking for adsorption at $T = 350$ K. Due to a lower resolution of the STM, the islands in this figure are not resolved as flat terraces, but appear as a roughening of the surface (**Figure 8**).

The structural changes of the surface are caused by the different densities of the topmost layers of the two Pt(100) phases which are involved; viz., $n = 1.3 \times 10^{15}$ atoms/cm^2 and $n = 1.56 \times 10^{15}$ atoms/cm^2 for the (1 × 1) and the rotated hexagonal reconstructed phase, respectively. For the CO-induced transition [rotated hexagonal → (1 × 1) structure], this leads to a surplus of ~0.2 monolayers of Pt atoms. These atoms are forced upward into the next higher layer. By surface diffusion, they can coalesce into islands, which are expected to increase in size with temperature because of the higher mobility of the Pt atoms. This mechanism also provides a straightforward explanation for the observed appearance only of monosteps; viz., the excess atoms are easily accommodated in one additional layer, and, since they originate from different locations of the surface, there is no reason for the formation of double steps.

These observations are in excellent agreement with results of a previous LEED study of this phase transition [6], where the measured increase of order in the CO adlayer with annealing temperature was attributed to the higher order of the substrate. Because of the well-known rather high mobility of adsorbed CO, the substrate was assumed to limit the ordering. At 380 K, only diffuse extra beams of the CO adlayer were reported. The island size was estimated to be about 25 Å [6]. Upon annealing to about 460 K in an equilibrium pressure of CO, the island size increased to 70 Å—in quantitative agreement with the STM results.

The question of the detection of the adsorbed CO has not yet been addressed. While for Figures 6(b–e) no adsorbed CO was present during the STM measurements, the images of Figures 7 and 8 were taken in the presence of a CO adlayer. Neither during CO exposure nor in measurements on the CO-covered surface could any distinct features be resolved which might have been correlated to the presence of the CO adlayer.

- *Defect-induced changes in surface reactivity*

The role of steps on the catalytic activity of the Pt(100) surface is elucidated by utilizing the decomposition of ethylene as a probe. We used a more densely stepped area for this experiment. As depicted in **Figure 9(a)**, the clean surface consisted of terraces of 30 Å to 100 Å in width; a total area of 500 Å × 500 Å was scanned. Characteristic of this surface area were its multiple steps and differently sized terraces. Its general shape was conserved during the subsequent ethylene decomposition.

Exposure to approximately one liter (1 L = 10^{-6} torr·s) of ethylene at 550 K did not lead to a significant change, as is evident from the scans in **Figure 9(b)**. During subsequent exposure to 10 L, however, characteristic alterations in the traces became visible, as indicated in **Figure 9(c)**. The steps in the center of the scans developed increased roughening which soon spread to the adjacent, rather small terraces. At the left portion of the topmost three scans, the growth of a rather flat "island" is indicated. The small step height indicates that this could not consist of an extra layer of Pt atoms, pointing instead to a carbonaceous overlayer and a possible surface roughening. Clearly the development of roughness, indicative of the carbon buildup, started from the steps on both sides of and between the two major flat terraces. Soon, the entire left terrace exhibited a similarly rough surface.

Following this dose, as seen in **Figure 9(d)**, the surface was scanned over a larger area, confirming the previous observation and indicating that all of the smaller terraces were covered with a rather rough overlayer; only the large terrace at the right was preserved as a clean, smooth (Pt) surface.

During further exposure, to 25 L of the ethylene, as can be followed in **Figure 9(e)**, that terrace, too, became completely covered with a rough overlayer. The two-dimensional array of scans in **Figure 9(f)** further supports this observation. While the gross step structure of the originally clean surface was preserved over the scanned area, essentially no flat regions remained. The entire surface seemed to have become uniformly covered by a microscopically rough overlayer. A subsequent AES spectrum [shown in **Figure 9(g)**] confirmed that we had grown a graphitic adlayer on the surface.

To our knowledge, this was the first time that the spatial progress of a surface reaction had been monitored *in situ* on an atomic scale. For the thermal decomposition of ethylene, we could clearly verify that the surface steps act as active centers: The growth of the carbonaceous adlayer proceeds from both sides of the steps of a terrace towards its center. These results are in complete agreement with observations for, e.g., the fragmentation of hydrocarbons. Somorjai and coworkers had investigated a series of differently stepped surfaces and found that in general those surfaces exhibiting a higher step or kink density also were the more active ones. Thus, a higher activity was assigned to these sites [3]. The importance of defects has also been reported for CO oxidation. In that case, however, the total effect is smaller, since the increase of the sticking coefficient of oxygen at defects is counteracted by its lower reactivity at these sites [15, 17].

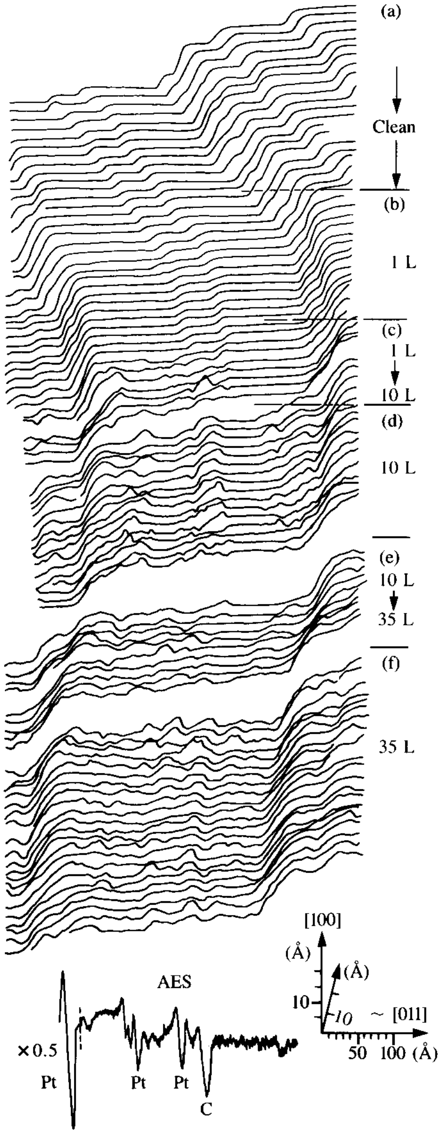

Figure 9

Decomposition of ethylene on Pt(100) surface at 550 K: (a) Two-dimensional array of scans of clean surface; (b) Two-dimensional array of scans following subsequent exposure to ~1 L of ethylene; (c) Sequence of scans during exposure to 10 L of ethylene; (d) Two-dimensional array of subsequent scans; (e) Sequence of scans during subsequent exposure to 25 L of ethylene; (f) Two-dimensional array of subsequent scans; (g) Subsequent Auger spectrum, indicating presence of graphitic layer on surface.

4. Conclusions

We have identified the structural variations induced on a clean, reconstructed Pt(100) surface by carbon contamination and by brief ion-bombardment cycles. In the presence of carbon contamination, the surface exhibits—after annealing—many small, several-layer-thick Pt islands, which are regarded as metal clusters pinned together by carbon atoms. The appearance of a Pt(100) surface subjected to ion bombardment at 550 K has indicated that the surface atoms are mobile enough at that temperature to lead to the formation of small terraces rather than a multitude of point defects. Furthermore, we have found that the (metal) mass transport necessary during the CO-induced surface phase transformation is possible at rather low temperatures. Small terraces having smooth surfaces are formed at temperatures as low as 415 K to accommodate the extra Pt atoms. The terraces were found to be larger at higher temperatures, indicating the influence of the surface mobility of Pt atoms. Finally, we have, to our knowledge, for the first time, observed the role of steps as active sites in a surface reaction: The buildup of carbon associated with the thermal decomposition of ethylene was imaged and was shown to proceed from the step edges of a Pt(100) surface onto its flat terraces.

Acknowledgments

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Wolfgang Hösler *Institute for Crystallography, University of Munich, Theresienstrasse 41, D-8000 Munich 2, Federal Republic of Germany.* Dr. Hösler received a Diploma degree in physics in 1977 from the Technical University and a Ph.D. degree in 1982 from Ludwig Maximilians University in Munich. Since 1983 he has worked on a project funded by the Deutsche Forschungsgemeinschaft at the University of Munich on fundamental aspects of surface physics and chemistry. His research interests include scanning tunneling microscopy and its application to the analysis of surface structure, defects on surfaces, and growth phenomena.

R. Jürgen Behm *Institute for Physical Chemistry, University of Munich, Sophienstrasse 11, D-8000 Munich 2, Federal Republic of Germany.* Dr. Behm has been with the University of Munich since 1980. He received his Ph.D. in physical chemistry from the University of Munich in 1980. During 1982/83 he worked as a visiting scientist at the IBM Research laboratory in San Jose on photoelectron spectroscopy of surfaces. His research interests include structural properties of surfaces and their activity on surface processes. He is currently engaged in the application of the scanning tunneling microscope to these problems.

Eckard Ritter *Institute for Physical Chemistry, University of Munich, Sophienstrasse 11, D-8000 Munich 2, Federal Republic of Germany.* Mr. Ritter received a diploma in physics from the University of Munich in 1984. His Diploma thesis dealt with the investigation of metastable de-excitation spectroscopy on semiconductor surfaces. He is currently working toward his Ph.D. degree in connection with studies of surface reactions using the scanning tunneling microscope.