Reconstructed Au (100) Surface Imaged with Scanning Tunneling Microscopy in Air

T. Schilling, B. Tesche, and G. Lehmpfuhl

Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4–6, W-1000 Berlin 33, Federal Republic of Germany

Z. Naturforsch. 47 a, 1187–1190 (1992); received September 19, 1992

Scanning tunneling microscopy (STM) of Au (100) was performed in air with a NanoScope II. Regions of 150×150 nm with atomatically flat areas fragmented by atomic steps were observed. The reconstructed (100) surface, deformed to a twisted hexagon with an interatomic distance of 0.27 ± 0.02 nm, could be seen and a corrugation of about 0.05 nm depth was measured. These results are in good agreement with Reflection Electron Microscopy measurements and STM investigations in UHV.

1. Introduction

The reconstruction of Au single-crystal surfaces has been studied with scanning tunneling microscopes (STM) in ultrahigh vacuum (UHV) by many investigators [1–3]. Recently, the reconstruction of Au (100) was observed by STM also in an electrolytic cell [4, 5] induced by a characteristic potential [6]. But in air, STM experiments on the low-indexed Au (100) surface with flat areas and atomic steps have – to our knowledge – never shown the reconstruction, although it is known from RHEED and LEED experiments that this reconstruction is stable in atmosphere and even in aqueous electrolyte under special potential conditions [6].

The Au (100) surface is reconstructed after cleaning in UHV, forming a pseudo-hexagonal arrangement, known as (5×20) superstructure. A more detailed analysis by Van Hove et al. [7] explains this reconstruction as a $c(28 \times 68)$ structure. The analysis is based on electron diffraction experiments, in which results are obtained as an average over a large surface area.

Until recently, the reconstruction of Au(100) has been found to be not twisted. However, Melle and Menzel [8] observed on a stepped Au(100) surface a twisted structure, which can be described by the matrix $\begin{pmatrix} 14 & 1 \\ -1 & 5 \end{pmatrix}$. By reflection electron microscopy (REM) experiments the influence of the (100) reconstruction on the formation of steps was found to be the same for Pt and Au [9], providing indirect confirmation of the twisted "hex" reconstruction on Au (100) observed by Melle and Menzel [8]. By REM, the different possibilities of reconstruction on Au (100) could be localized [10]. Our REM images of the reconstruction of Au(100) show clearly a twisted structure of the type $\begin{pmatrix} 27 & 2 \\ -1 & 10 \end{pmatrix}$ as a refinement of the $\begin{pmatrix} 14 & 1 \\ -1 & 5 \end{pmatrix}$ structure.

It is the goal of this work to show that the reconstruction of Au (100) can be observed with an STM in air after a special preparation procedure [4]. The tunneling data obtained under atmospheric conditions permit the determination of the atomic structure of the gold surface.

2. Experimental Methods

The reconstruction of the Au single-crystal surface can be produced by a simple flame treatment [11] and subsequent dipping in high-purity (pyrolytic) water [12]. This dipping is necessary in order to avoid reactions of the surface with the atmosphere during cooling down. The reconstructed surface of Au after such a treatment was shown by LEED and RHEED [12]. Triply distilled water is not clean enough, as could be shown in REM images from Au single-crystal spheres after rinsing [13]. Rinsing with pyrolytic water, however, did produce a clean surface [14].

In the present investigation a reconstructed Au (100) surface was prepared by this flame treatment. The STM images were recorded with a commercial NanoScope II, which is protected against low-frequency and acoustic vibrations. For scanning, the constant-current mode was used with the current set

Reprint requests to T. Schilling, Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4–6, W-1000 Berlin 33.

0932-0784 / 92 / 1200-1187 \$ 01.30/0. – Please order a reprint rather than making your own copy.

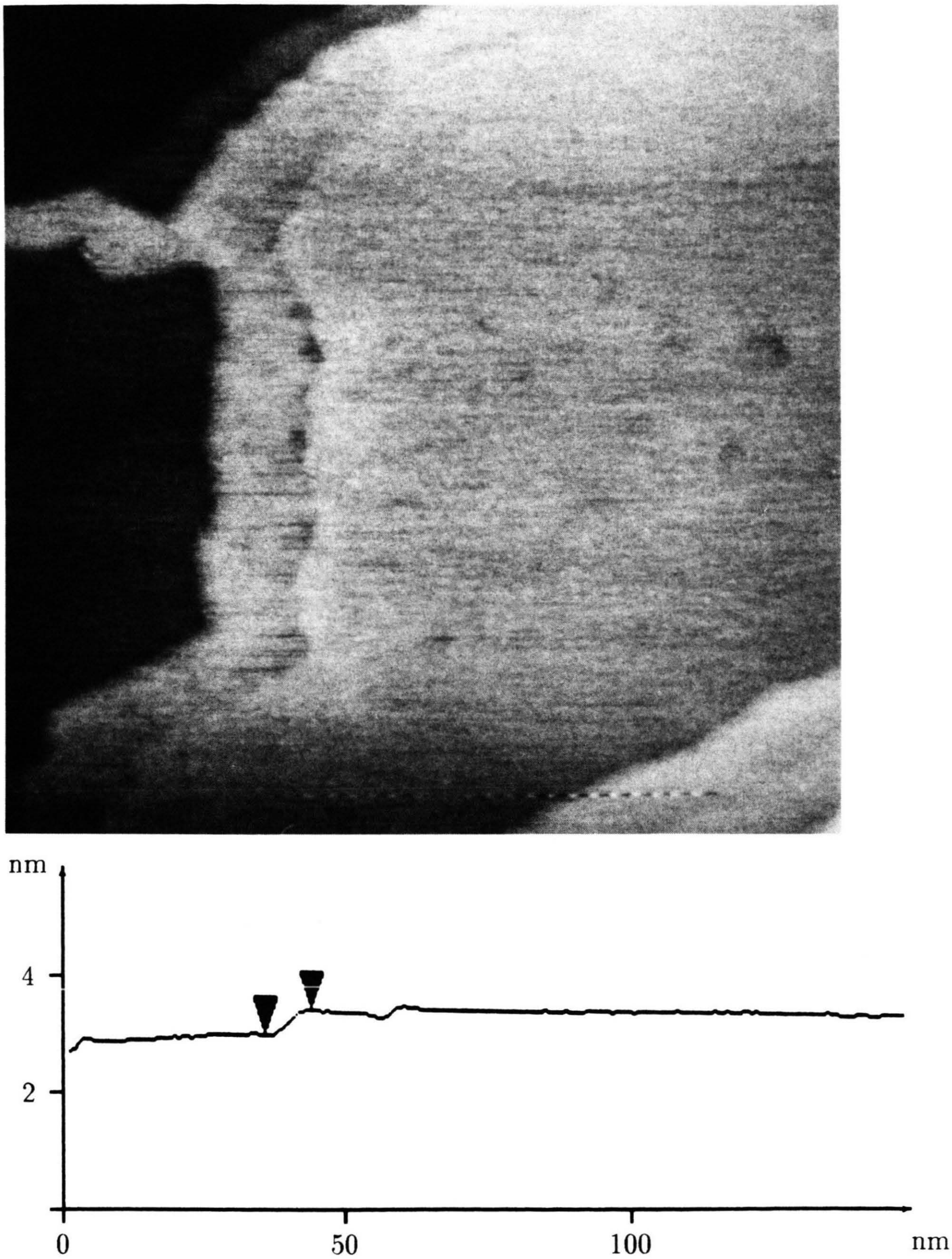

Fig. 1. STM image of a 150 × 150 nm field, with atomic steps. Height profile along the x direction.

to 1 nA; thus during the image formation it was possible to control any kind of disturbance concerning the tip by observing the current with an oscilloscope. The scan rates were adjusted to the width of the scanned region from 1 Hz for small scans to 4.3 Hz for wide scans.

3. Results

Figure 1 shows a typical STM image of the Au(100) surface with steps at low magnification ($8.3 \cdot 10^5$). Also a horizontal linescan is presented, where the height profile of the surface can be seen. The difference at the

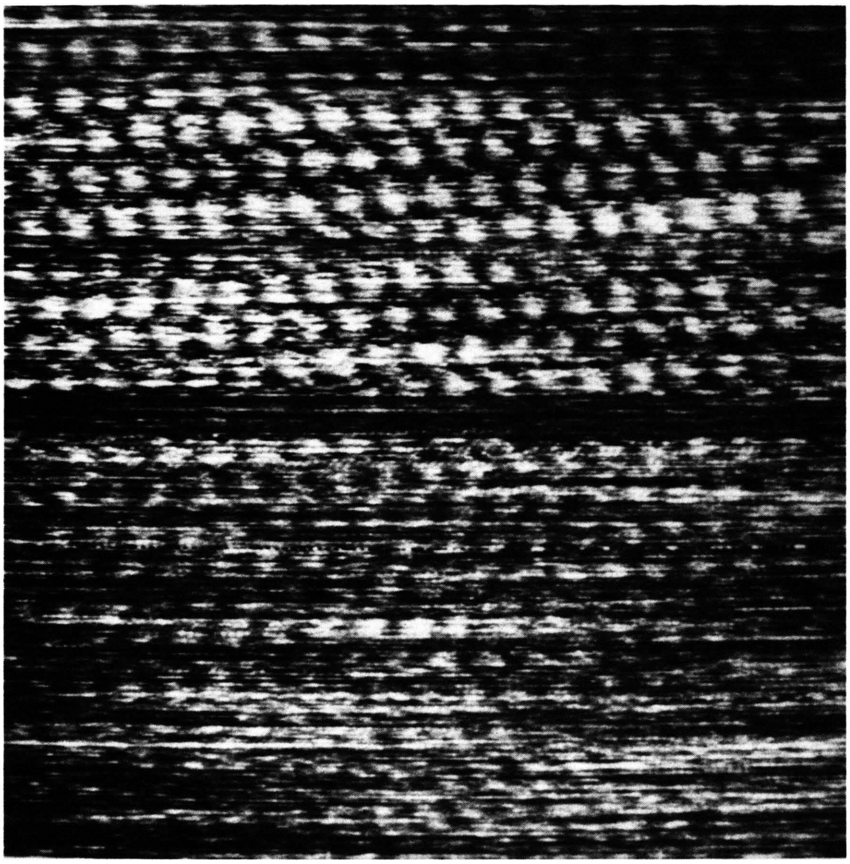

Fig. 2. Atomic resolution STM image of the reconstructed Au (100) surface, without any kind of filtering (5.0 × 5.0 nm).

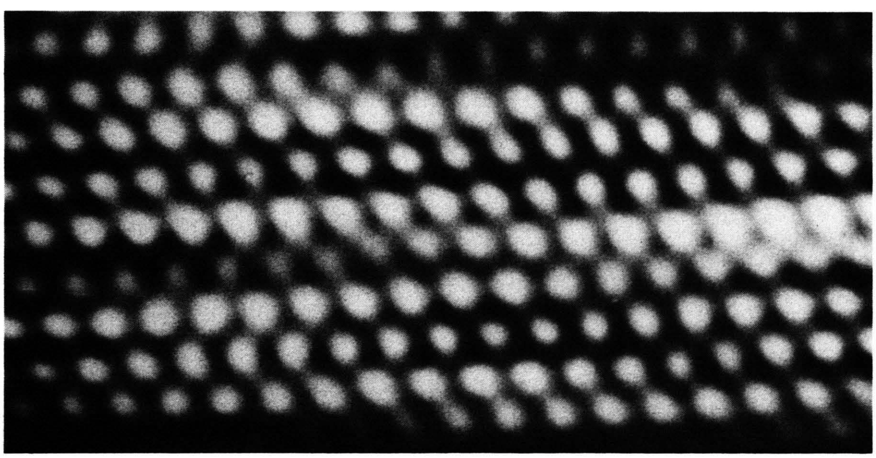

Fig. 3. High-resolution STM image of the reconstructed Au (100) surface, after cutting off the high frequencies (5.0 × 2.5 nm).

step indicates a height of 0.42 nm which means that the step is of double-atomic height.

Figure 2 shows an unfiltered image of the same surface of 5 nm × 5 nm dimension with atomic resolution. Here, the "hex" reconstruction can clearly be seen, but no quadratic arrangement of atoms as one would expect from the non-reconstructed Au(100) surface. The recorded area is not large enough nor is the resolution over the whole scan area good enough to show the complete unit-cell of the reconstruction, but the pseudo-hexagonal arrangement of the atoms can be seen. The modulation in the intensity along one row of atoms, caused by the corrugation of the compressed and twisted overlayer is visible. The modulation of intensity along one row of atoms and also along neighbouring rows is typical for a twisted overlayer. A non-twisted overlayer, as a simple 5 × 1 superstructure, would show constant intensity along one atom row or only a slight modulation along this row due to compression in this direction. But this "noisy" STM image shows that the overlayer is twisted.

After filtering the image (cutting off the high frequencies) the modulation in intensity can be seen more clearly. This is presented in Fig. 3, and a detailed analysis of the image should allow one to determine the position of the atoms of the surface lattice with respect to the bulk lattice, especially the position of the coincidence lattice at bridge positions or at hollow or top positions. These two positions lead to different corrugations of the surface layer.

Gao et al. observed coincidence at bridge positions without twist by STM investigations in an electrolyte where the reconstruction of the Au(100) surface was induced at room temperature Fig. 2f in [6]. In our experiment, however, the reconstruction was formed by flame treatment at elevated temperature of 700 to 800 °C and preserved by quenching in pyrolitic water. Under these conditions a twisted reconstruction may have been formed and preserved.

4. Conclusion

This investigation shows that, using special specimen treatment, tunneling in air can image the atomic structure of the Au(100) surface and demonstrate the possibility to investigate the complicated reconstruction on Au(100) under atmospheric conditions by STM.

The good agreement concerning the modulation in intensity and corrugation supports the correctness of our investigation.

Acknowledgement

We would like to acknowledge the continued support of Professor E. Zeitler in this work and thank him for many helpful discussions.

[1] G. K. Binnig, H. Rohrer, Ch. Gerber, and E. Stall, Surface Sci. 144, 321 (1984).
[2] J. V. Barth, H. Brune, G. Ertl, and R. J. Behm, Phys. Rev. B **42**, 9307 (1990).
[3] O. M. Magnussen, J. Hotlos, D. M. Kolb, and R. J. Behm, Phys. Rev. Letters **64**, 2929 (1990).
[4] X. Gao, A. Hamelin, and M. J. Weaver, Phys. Rev. L. **67**, 618 (1991).
[5] M. S. Zei, G. Lehmpfuhl, and D. M. Kolb, Surface Sci. **221**, 23 (1989).
[6] D. M. Kolb, G. Lehmpfuhl, and M. S. Zei, in: Spectroscopic and Diffraction Techniques in Interfacial Electrochemistry", Kluwe Academic Publishers 1990, p. 361–382, page 372.
[7] M. A. VanHove, R. J. Koestner, P. C. Stair, J. B. Biberian, L. L. Kesmodel, I. Bartos, and G. A. Somorjai, Surface Sci. **103**, 189 (1981).
[8] H. Melle and E. Menzel, Z. Naturforsch. **33a**, 282 (1978).
[9] G. Lehmpfuhl and Y. Uchida, Surface Sci. **235**, 295 (1990).
[10] N. Wang, Y. Uchida, and G. Lehmpfuhl, Ultramicroscopy submitted.
[11] J. Clavilier, R. Faure, G. Guinet, and R. Durand, Journal of Electroanl. Chem. **107**, 205 (1980).
[12] D. M. Kolb and J. Schneider, Electrochimica Acta **31**, 929 (1986).
[13] Y. Uchida, G. Lehmpfuhl, and J. Jäger, Ultramicroscopy **15**, 119 (1984).
[14] Y. Uchida and G. Lehmpfuhl, not yet published.