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A SCANNING TUNNELING MICROSCOPY STUDY OF THE
AU(334) SURFACE IN AIR

by

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Abstract

High resolution images of a Au(334) surface in air have been obtained by scanning tunneling microscopy. The surfaces were prepared in UHV and produced clean Auger spectra and ordered stepped LEED patterns. Exposure to air caused only a small carbon contamination. The STM images in air revealed a structure of parallel steps of monoatomic height grouped in domains that are several hundred Angstroms wide and a few tens of Angstroms high. These large domains are part of a surface that is smooth on the scale of several thousands of Angstroms.

The field of Scanning Tunneling Microscopy (STM) is expanding rapidly to several scientific and technological areas that involve surfaces and interfaces.¹ The ability of STM to produce topographic as well as spectroscopic images at the atomic resolution level is well proven. While there have been several atomic resolution topographic images of surfaces in vacuum¹, to our knowledge only highly oriented pyrolytic graphite has been observed in air^{2,3} and graphite, TaS₂ and TaSe₂ in liquid environments^{4,5} with this resolution. Since one of the big advantages of STM is that it is capable of imaging in air, it is of interest to find additional test surfaces where this is possible. In this communication we report that the stepped structure of the Au(334) surface is stable in air and can be imaged with atomic resolution.

We have recently developed an STM instrument that utilizes the tripod piezo configuration⁶ supported on a rigid stainless steel block. The approach of the sample to the tip is accomplished by means of two micrometer screws whose motions are arranged to nonlinearly bend a stainless steel beam supporting the sample. Vibration isolation was achieved by suspending the instrument by means of viton o-rings. Permanent magnets provided eddy current damping. In our studies the STM instrument was protected from background sound noise by a glass bell jar. Additional isolation from building vibrations was obtained by mounting the instrument on a laser table equipped with legs that are air cushioned. As a check of the instrument performance, atomic resolution images of graphite in air were obtained with a noise level below 0.4Å in the z-direction (perpendicular to the surface). The microscope is

sturdy and rigid enough that topographic images of gold with a noise level below 2Å could be obtained with the instrument sitting on a lab stool with only a 1 cm thick rubber cushion for vibration isolation. The details of the mechanical design as well as the feedback and control electronics, and computer interfacing, will be described elsewhere.⁷

In order to test the stability of stepped Au(111) single crystal surfaces in an air environment, we chose to study Au(334) where the surface structure is composed of (111) terraces separated by steps. Au(334) can be described as a 7(111)x(100) surface if the terraces are assumed to be separated by a single monoatomic step. The crystal was prepared in an Ultra High Vacuum chamber (UHV) until a clean and well ordered surface was obtained. This was achieved after several cycles of Ar-ion sputtering and annealing to near the crystal melting point. After this treatment a clean surface was obtained as checked by Auger Spectroscopy (less than 1% monolayer of impurities). This surface produced a Low Energy Electron Diffraction (LEED) pattern of hexagonal symmetry with doublet spots at certain energies, which is characteristic of stepped surfaces.⁸ This LEED pattern is shown in figure 1. The background in the doublet direction is higher than in the rest of the pattern. This indicates that the step structure is not perfect and probably forms small domains.

The Au crystal was then exposed to air at one atmosphere for ten minutes and examined again in UHV after pumpdown. The only effect of air exposure is the irreversible deposition of carbon as revealed by Auger spectroscopy. The estimated carbon-coverage was approximately 0.3

monolayers. This carbon contamination is probably the result of organic molecules trapped on the surface as a result of exposure to the atmosphere. The carbon contamination is probably inhomogeneously distributed as indicated by the very small attenuation of the main Au Auger peak at 70 eV, as well as by our STM results where in certain areas of the crystal the images were found to be less noisy and more reproducible than in other areas. The LEED pattern after this air exposure was similar to that of figure 1 with a higher background and some blurring of the spots. The above evidence indicates that although there is some contamination of the surface in air, there are patches on the surface where imaging of the clean surface can be done.

The Au surface was imaged in air with the STM in the topographic mode, that is plotting the feedback z-piezo voltage as a function of the x and y scanning voltages while keeping the current constant. These voltages are then converted into distances by means of calibration values for the piezos.⁹ Typical scan times for images varied between 4 and 8 minutes. In scans of a few thousand Angstroms width, the observed topography reveals a smooth surface with plateaus of a few hundred Angstroms width and a few tens of Angstroms high, elongated in the $[01\bar{1}]$ direction, i.e. parallel to the atomic steps. At close range, the flat tops of these large terraces reveal a multitude of monoatomic height steps parallel to each other, as shown in figure 1. The orientation of the steps is coincident with that expected from the LEED pattern (within the experimental accuracy of $\pm 10^\circ$). The average distance between steps in this figure and others is approximately 22Å and the step height

is between 2 to 4Å. This compares to a terrace size of 18Å and a step height of 2.3Å for a bulk terminated Au(334) surface that has the 7(111)x(100) structure. The difference in the values is within the accuracy of our piezo calibration.⁹ The quality of our LEED pictures was not sufficient to extract the surface periodicity of the steps. The step separation in the STM images in Figure 1 becomes smaller and finally unresolved near the left edge of the large plateaus, while near the right edge the separation becomes larger. The relatively short domain size of the periodic steps, as well as their different spacing near the edges of the large plateau, is consistent with the diffuse background in the LEED pattern along the spot doublet direction.

Heating the crystal in air to between 550°C and 630°C produced a modification of the step structure with the formation of large (111) plateaus that have an average size of a few hundred angstroms, similar to the size of the stepped domains observed prior to annealing. Atomic height steps could not be resolved on these plateaus. These results and a more extensive account of our work on stepped surfaces will be reported elsewhere.¹⁰ In summary, it has been shown that clean and well ordered stepped Au crystals prepared in UHV can be imaged by STM in air with atomic resolution. This indicates the stability of stepped Au surfaces in an air environment.

Acknowledgments

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10. M. Salmeron, D.S. Kaufman, B. Marchon, and S. Ferrer, to be published.

Fig. 1 STM images of a Au(334) surface in air. The sequence of images from top left to bottom right illustrate the surface topography at various scales from 1000Å to 200Å. The monoatomic height step structure starts to be visible in the x2.5 image along the [011] direction, and is fully visible in the higher magnification image. The vertical scale is only indicated in this last image but scales up as indicated by the multiplying factors in the top images. The LEED pattern at 70 eV on the left reveals the doublet structure characteristic of stepped (111) surfaces. The surface is oriented in the same way ($\pm 10^\circ$) as in the STM images.

1000x 1000 A

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Figure 1

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