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Surface Science 331–333 (1995) 1070–1076

surface science

Structural studies of the Au(110) surface with scanning tunneling microscopy and RHEED

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Received 1 August 1994; accepted for publication 3 November 1994

Abstract

The Au(110) surface has been studied by STM and RHEED (reflection high energy electron diffraction). The surface structure was varied by different annealing procedures. Very fast cooling can preserve part of the disordered state from above the phase transition temperature $T_c = 650$ K. In that state the surface is disordered, but develops local (1 × 3) structures. The well annealed surface develops the usual (1 × 2) structure in combination with the mesoscopic "fish scale" pattern.

Keywords: Gold; Low index single crystal surfaces; Reflection high-energy electron diffraction (RHEED); Scanning tunneling microscopy; Surface structure

1. Introduction

The Au(110) surface has been studied in great detail since the discovery of the (1 × 2) reconstruction [1]. The structure is called the "missing row" structure, because every second of the $\langle 1\bar{1}0 \rangle$ surface chains is missing. Another view is that $\frac{1}{2}$ a monolayer of Au atoms is adsorbed on the (110) surface such that only every second $\langle 1\bar{1}0 \rangle$ surface is occupied. The (1 × 2) structure on Au(110) is formed after the usual surface cleaning procedures, i.e. sputtering and annealing cycles, by keeping the surface above $T_c = 650$ K for some minutes and cooling the surface to room temperature at a rate of about 1 K/min. During the annealing the surface develops a mesoscopic "fish scale" structure as found by STM studies [2–4; therein are also references to earlier LEED, ion scattering, etc. work on Au(110)]. In the present work we report first results from an almost in situ study of Au(110) using STM and RHEED.

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2. Experiment

For the experiments we use an Omicron STM system. We obtain atomic resolution on metal surfaces routinely. STM is performed at room temperature only. RHEED patterns have been obtained up to 700 K. For the RHEED observation the crystal is mounted on a standard two-axes manipulator with X, Y, Z movement. The sputtering is performed in another chamber to which the crystals are moved without breaking the vacuum. This "preparation chamber" can be separated from the STM-RHEED part by a valve.

SSDI 0039-6028(95)00159-X

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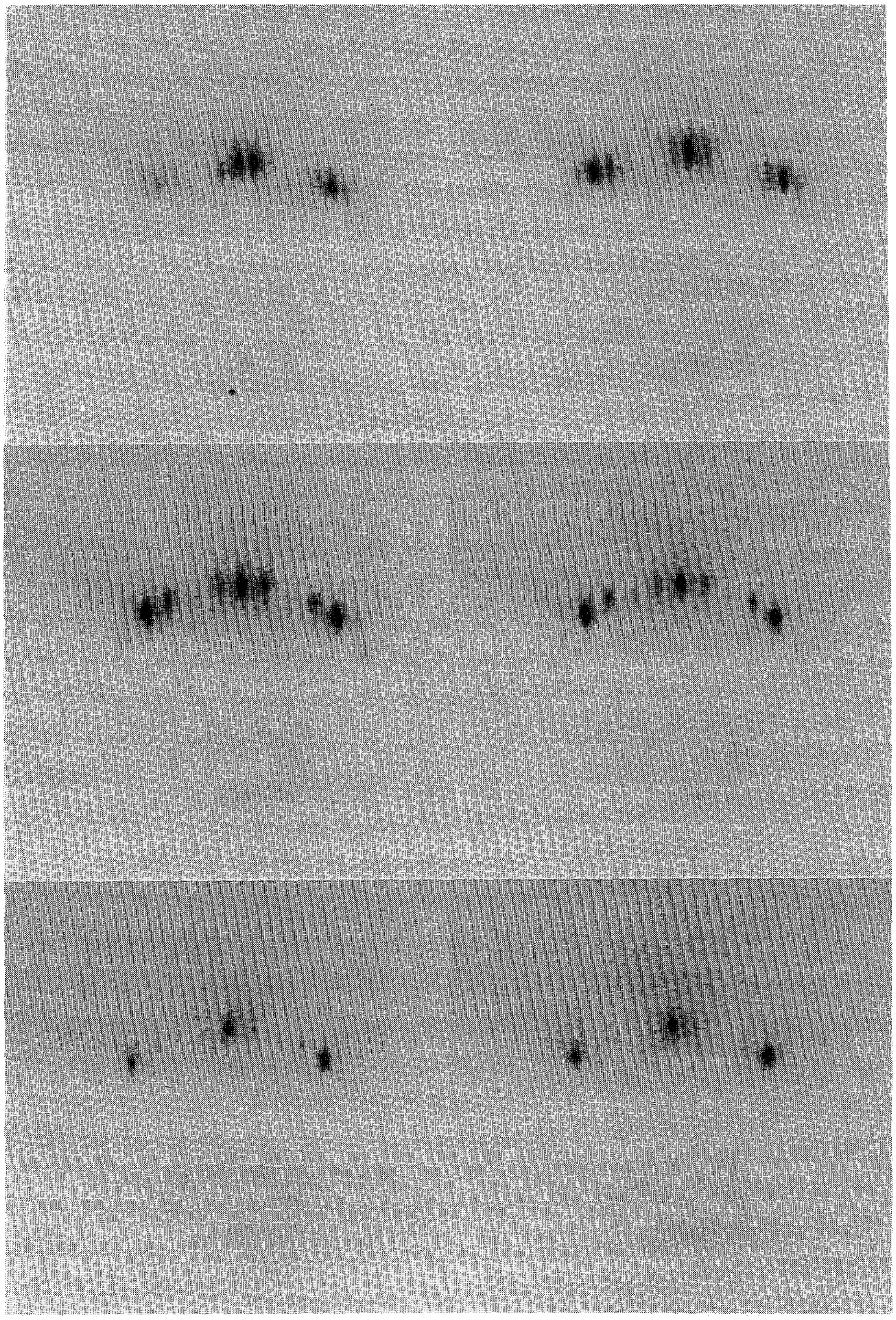

Fig. 1. RHEED analysis of an annealing procedure of a sputtered Au(110) surface. (a) 1 h after sputtering (approximately room temperature), rough, disordered, weak evidence for (1 × 3); (b) 14 min, 400 K, disordered, evidence for (1 × 3); (c) 24 min, 500 K, ordered (1 × 2); (d) 34 min, 530 K, ordered (1 × 2); (e) 43 min, 660 K evidence for disorder (1 × 2); (f) 48 min, 680 K, disordered (1 × 1).

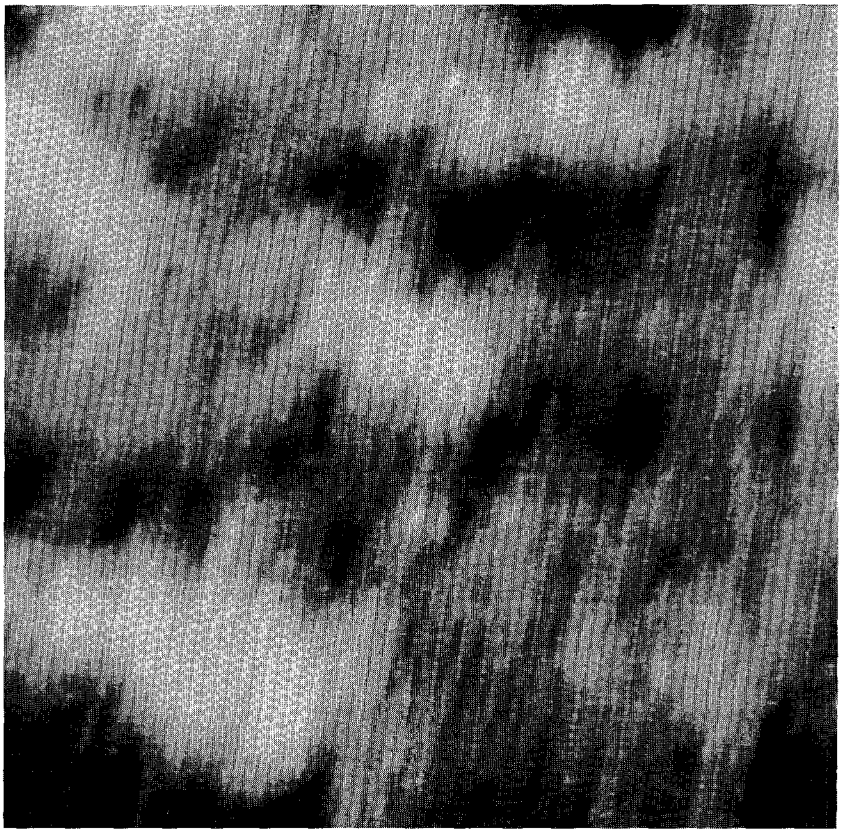

Fig. 2. STM result from the sputtered, nonannealed Au(110) surface (1000 Å)^2. The surface is in the same state as in (a). Visible full grey scale corresponds to 10 Å.

3. Results

The targets are transferred after sputtering to the STM-RHEED chamber. STM scans without annealing of the targets show very rough surfaces without any well defined steps. Fig. 1 shows an annealing process monitored by RHEED. The initially sputtered surface (Fig. 1a) shows roughness – streaky intensity – and some evidence for (1×3) reconstruction. Fig. 2 shows the status of the sputtered surface in STM, i.e. rows with a typical length of 100 Å. The distance between the rows is three times the lattice constant and the roughness is about 10 Å. The first annealing step (Fig. 1b) reduces the roughness to $\approx$ 8 Å. The lower curvature of the pattern formed by the diffraction spots in Fig. 1a compared to Fig. 1b is due to contributions from transmission through the rough surface. At 500 K the pattern has changed to (1×2). Especially at 530 K the diffraction spots are well defined and form a good Laue circle. At 660 K just above T_c = 650 K the half-order spots become weaker and all spots become streaky again. Well above T_c at 680 K the surface is both obviously disordered and rough. After fast cooling the pattern remains (1×2).

Since our STM does not allow measurements at elevated temperature, we tried to "shock cool" the sample by grabbing the sample holder with the wobble stick. The infrared pyrometer we use to measure the sample temperature shows a temperature decrease at a rate of 50 K/min. Obviously some of the initial roughness is preserved, as seen in RHEED and now also in STM (Fig. 3). Slow cooling, on the other hand, leads to large area (1×2) terraces in the shape of "fish scales" [2–4]. The predominant structures on the shock cooled surface are irregular arranged short $\langle 1\bar{1}0 \rangle$ chains on a rough surface. The average distance be-

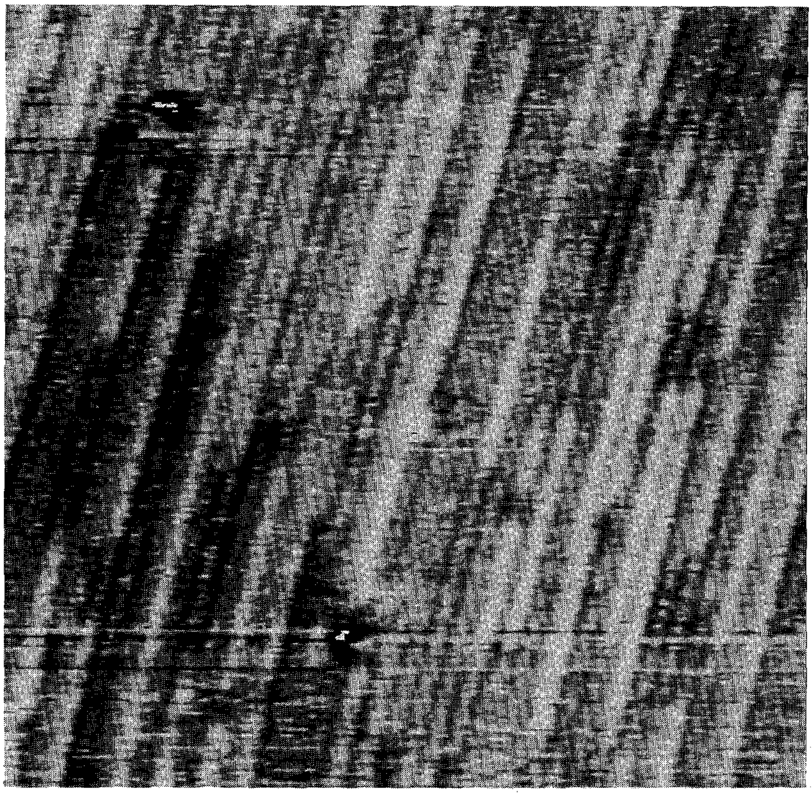

Fig. 3. "Shock-cooled" Au(110) surface in a (500 Å)^2 STM scan. The $\langle 1\bar{1}0 \rangle$ direction is parallel to the clearly visible rows. The roughness is about 4 Å.

tween those chains corresponds to (1×3) rather than to (1×2). The roughness extends over several layers and hence no well defined step structures are found directly after shock cooling. After one day at room temperature the roughness is reduced and extends to two or three layers only (Fig. 4). The mesoscopic structure is formed by (1×3) islands (light or higher areas) and darker "clearings" (dark or deeper areas) which are predominantly (1×2) reconstructed. A Fourier analysis of the surface structure reveals (1×3), (1×2) and (1×5) structure elements (Fig. 5). Besides the dominant (1×3) intensity peak, a distance of 5.5 lattice constants is found which corresponds to a simple step in a (1×3) structure. The series of STM frames of Fig. 4 shows also a high mobility of the Au atoms on the surface, i.e. the lengths of the $\langle 1\bar{1}0 \rangle$ chains varies considerably. After some further changes which could be followed over several days with the resolution of the chains [5], we find on the fourth day after the shock cooling the "fish scale" pattern (Fig. 6) with predominantly (1×2) reconstruction. A few $\langle 1\bar{1}0 \rangle$ strings (white stripes) remain on the terraces. They can be removed by a mild annealing (100°C).

4. Discussion

Comparison of the RHEED and STM results on the freshly sputtered surface shows that a visual inspection of RHEED patterns may be misleading. The deviations from the Laue circle and the streakiness of the diffraction spots are more important than the existence of diffraction spots, including the weak higher-order spots which may lead us to believe that there is some long-range order on the surface. The STM result shows that any long-range order information is fortuitous at best. The surface roughness extends over several layers. The RHEED information is useful, though, for

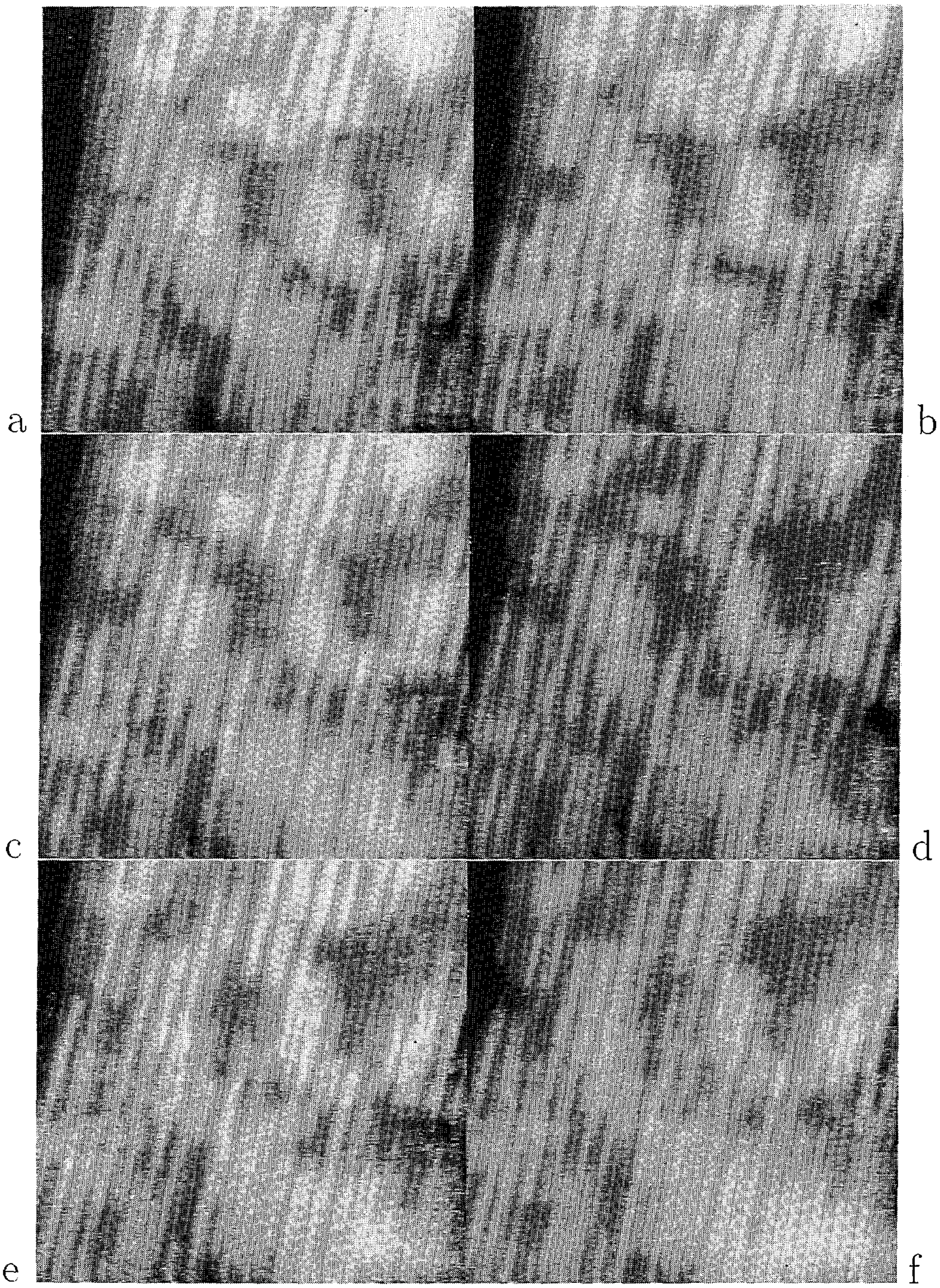

Fig. 4. Series of STM (500 Å)^2 scans one day after the shock cooling. Light (high) areas (1 × 3), dark (low) areas (1 × 2) reconstructed. Time interval between scans: 3 min. Full grey scale corresponds to 3 Å.

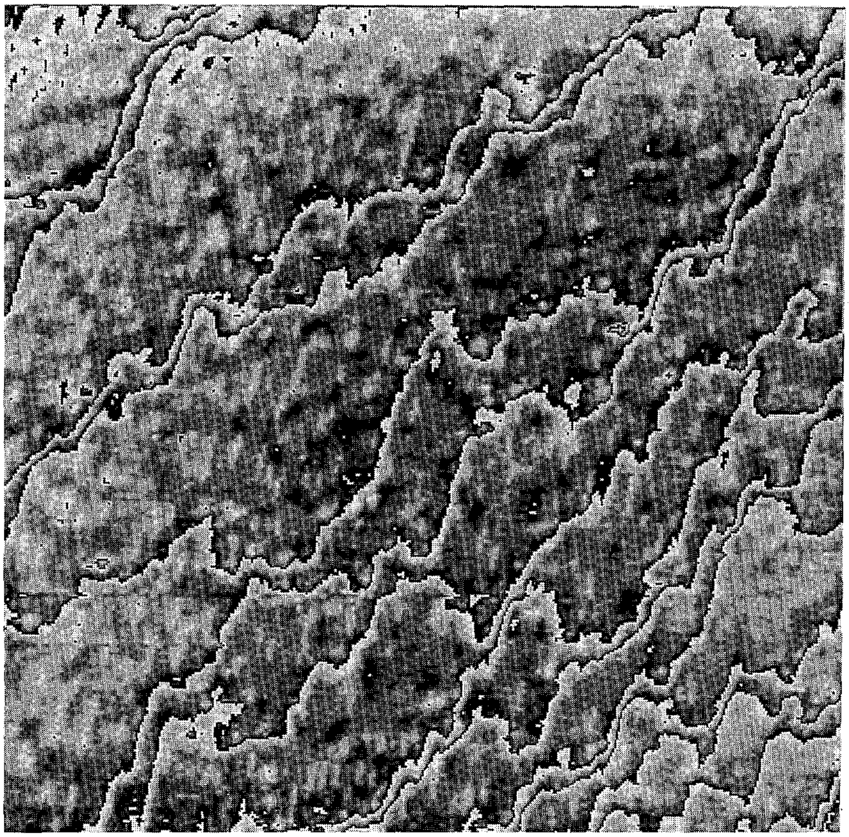

Fig. 6. (2000 Å)^2 STM scan of Au(110) four days after the "shock cooling". The highest terrace is in the upper left hand corner. An intensity "plane" is subtracted to improve the contrast of the step structure and the remaining "disorder" on the terraces.

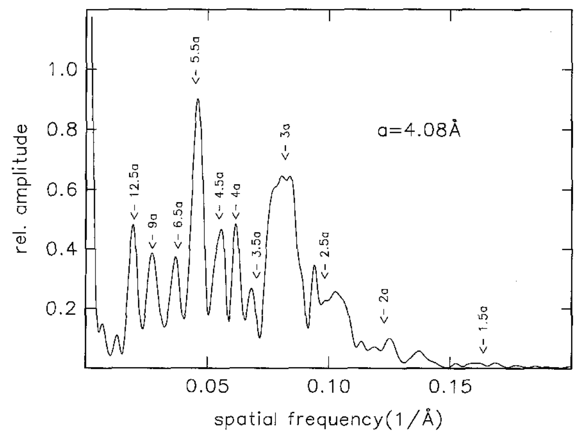

Fig. 5. Fourier amplitudes of an STM scan. The Fourier analysis is made after summing up the real space intensities along the ⟨1$\bar{1}$0⟩ chains.

the annealing process. The "quality" of the diffraction spots is at least a qualitative measure of the decreasing roughness of the surface. In principle, it is possible to evaluate the RHEED data quantitatively, especially if "rocking curves" are measured [6]. The much slower annealing at room temperature which was followed by STM allows some estimates of the activation energy for the formation of the (1×2) structure. Combined with the information that the (1×2) structure is formed during a slower cooling down procedure [4,5] within hours we obtain for the activation energy a value of about 0.25 eV. The activation energy includes obviously not only the energy needed for the local positioning of the Au atoms but also the energy for the formation of the "fish scale" structure. The "driving force" for this structure is the fact that (1×2) reconstructed terraces will naturally be limited by (111) and (331) type steps. The energy of the

(331) type is however higher than that of the (111) type steps. By forming the fish scale pattern, antiphase boundaries can be introduced and the number of (331) terraces is reduced [2,3]. Another observation from the STM results is the preference for the formation of $\langle 1\bar{1}0 \rangle$ chains compared to any other low index direction. This is certainly a condition for the formation of the final (1×2) missing row structure. However, in the early, not well annealed state of the surface rather large parts of the surface have distances between the chains which correspond to the (1×3) structure. It is interesting to note that in one of the early LEED studies [7] the occasional finding of (1×3) structures mixed with (1×2) was reported, depending on the annealing and cooling conditions.

Acknowledgement

This work is supported by the Deutsche Forschungsgemeinschaft.

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