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Nucleation and growth during faceting of the platinum covered W(111) surface

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Abstract

Low energy electron microscopy (LEEM) and scanning tunneling microscopy (STM) have been used to investigate the faceting of W(111) as induced by Pt. The atomically rough W(111) surface, when fully covered with a monolayer film of Pt and annealed to temperatures higher than ~750 K, experiences a significant morphological restructuring: the initially planar surface undergoes a faceting transition and forms three-sided pyramids with {211} faces. When Pt is dosed onto the heated surface, the transition from planar to faceted structure proceeds through the nucleation and growth of spatially separated faceted regions, as shown by LEEM. STM reveals the atomic structure of the partially faceted surface, with large planar regions, dotted by clusters of pyramids of various sizes.

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DISCLAIMER

1. Introduction

The faceting of W(111) and Mo(111) surfaces as induced by thin metallic adsorbate films[1-3] is one of the most intriguing phenomena in large scale morphological transformations of metal surfaces. In addition to their academic interest, the thin film covered bcc(111) metal surfaces are model bimetallic catalysts of key importance, as they provide insights into the effect of surface structure on reactivity and selectivity[4, 5]. Many aspects of the faceting transition have been studied in detail. Facets have been identified as three-sided pyramids with {211} sides, using both low energy electron diffraction[2, 6, 7] and atomic resolution STM[8]. It was also shown that the pyramids are composed of tungsten, with a thin adsorbate film cover, rather than being pure adsorbate clusters[9, 10].

A number of metals have been investigated as adsorbates on W(111) and Mo (111); among them Pt, Pd, Au, Rh and Ir are found to induce faceting[2, 3, 7]. It has been established that there is a minimum film thickness necessary for the surface to be completely faceted, and this critical adsorbate coverage is one "physical" monolayer ($\sim 1.7\times10^{15}$ atoms/cm^2), which, in case of the atomically rough bcc(111) surface, is equivalent to three "geometrical" monolayers using the conventional definition (figure 1). A large anisotropy in surface free energy has been identified as the driving force for faceting by first principles calculations[11], and the same calculations also verify the strong coverage dependence. It was also found that the faceting transition does not occur spontaneously at room temperature, due to kinetic limitations. In order to overcome the kinetic barrier and induce faceting the surface has to be annealed above a threshold temperature of ~750 K. The average pyramid size is also found to increase with annealing temperature, from less than 100 Å at low temperatures, up to ~700 Å for Pt/W(111) annealed to 1400 K[12, 13].

There are scientific issues, however, that are yet to be addressed in more detail in connection with metal overlayer induced faceting. These include the nature of facet

nucleation, coexistence of faceted and planar surfaces, facet growth (with annealing time), and a high temperature *reversible* transition from faceted to planar surface[14].

In the present work we use a scanning tunneling microscope and a low energy electron microscope[15] to provide new insights into the Pt induced faceting of W(111). LEEM can distinguish between planar and faceted surfaces based on the different types of diffraction of low energy electrons on surfaces with different morphologies, with up to ~70 Å spatial resolution.

Under certain conditions a coexistence of faceted and planar regions has been observed using LEED and STM for the Pt/W(111)[1], Pd/Mo(111) and Au/Mo(111) systems[14]. LEEM is capable of providing information on the spatial distribution of such coexistence phases. Most important, it enables real time observation of structural and morphological changes, such as the transitions from planar to coexistence to faceted systems. Our main objective is to conduct real time observations of the nucleation and growth of Pt induced faceting of W(111) using LEEM, and compare our results to high resolution STM measurements on the same system.

2. Low Energy Electron Microscopy

2.1. Experiments

The LEEM experiments were performed at Sandia National Laboratories using a commercial Elmitec microscope[15] (STM experiments — performed at Rutgers — are discussed in section 3). The LEEM apparatus is housed in a conventional stainless steel ultra-high vacuum (UHV) system with an average base pressure of 2×10^{-10} torr. LEEM images and LEED patterns are formed on a channel plate intensifier, and imaged by a phosphor screen. The images are recorded onto video cassettes using a CCD camera. A video image-grabbing software also enables the storage of LEEM pictures on a computer for further analysis.

Attached to the main LEEM chamber are two separate preparation chambers: one for sample cleaning, and the other equipped with a cylindrical mirror analyzer and a concentric electron gun for Auger electron spectroscopy (AES). We use AES to check sample cleanliness and to determine the approximate Pt coverage, using the calibration of Pt to W Auger peak ratio versus coverage of previous works[1, 16].

The W(111) sample is cleaned in an auxiliary chamber by flashing to temperatures above 2400 K using electron bombardment heating, while lower temperature anneals (up to 1500 K) are performed in the LEEM chamber *in situ*. The sample temperature is measured using both an infrared pyrometer and a matched filament pyrometer.

The chamber is equipped with a Pt evaporation source for dosing the W(111) sample *in situ*, allowing the observation of the surface while it is being dosed. Ultrathin Pt films are deposited from a commercial metal evaporator, in which a Mo rod holding a small Pt ball is heated by electron bombardment. The dosing rate is automatically kept constant by monitoring the beam flux. For the experiments outlined below very low deposition rates were desirable: the estimated effective deposition rate at the sample surface is approximately 3×10^{11} atoms/cm^2/sec, which results in a full physical monolayer coverage in ~90 minutes.

As described below, we aimed to observe the fully or partially faceted surfaces in real space. However, the most important advantage of LEEM is its capability of imaging a surface in real time, even surfaces at high temperatures, or surfaces as they are being dosed. The main objective of the LEEM experiments, therefore, was to conduct real time observations of the nucleation and growth of Pt induced faceting of W(111), that is, observing the sample kept at a constant elevated temperature while Pt is being slowly deposited onto the surface.

2.2. Results

The transition from a planar to fully faceted Pt/W(111) surface is confirmed by using the microscope in LEED mode, when the Pt coverage exceeds a critical coverage of one

physical monolayer (i.e. every W surface atom is covered by a Pt atom, ~1.7×10^{15} atoms/cm^2) and the sample is annealed between 750 and 1200 K. The identification of faceting of a W(111) surface using LEED has been well described before[1, 6, 7]. The three sided pyramids of the faceted W(111) surface have equivalent {211} faces: (211), (121) and (112). Therefore, the LEED pattern of the faceted W(111) surface is a superposition of three distinct rotated and tilted {211} LEED patterns.

At coverages between 0.7 and 1 physical monolayer, the Pt/W(111) surface produces a superposition of planar and faceted LEED patterns (figure 2), indicating a partially faceted surface structure. The fact that planar W(111) and faceted {211} regions give rise to different diffraction beams in LEED may be exploited in LEEM, by applying a small aperture to one of the diffraction beams only, and forming a real-space image of the surface using this single beam. In the so called "bright field" imaging mode the selected beam is the specular (0,0) beam from the planar surface, and as a result planar regions appear bright, while faceted regions appear dark on the LEEM screen (figure 3a).

The experiments aimed at observing the nucleation and growth of Pt induced faceting of W(111) yielded results such as the one presented in figure 3[17]. The figure shows a sequence of bright field images of the surface at 1050 K, as the surface is being dosed at a rate of ~1 physical monolayer of Pt per 90 minutes. LEEM detects no change in the surface structure for the first 60 minutes, up to a coverage of 2/3 physical monolayer. After ~60 minutes well defined small islands of faceted regions (dark circular spots) become observable (figure 3a). After ~90 minutes the surface appears uniformly dark, i.e. fully faceted (figure 3f). When compared to STM data presented in section 3, it is obvious that even the small dark areas in figure 3a, approximately 1000 to 3000 Å in diameter, do not correspond to individual facets, but rather to faceted *regions* of the surface. It is also interesting to note that, aside from the initial nuclei on figure 3a, there is no further nucleation observable during growth.

We have also attempted to observe the nucleation and growth of faceted features when Pt is dosed onto the W(111) substrate at room temperature (in excess of one physical monolayer), and then the sample is gradually heated to high temperatures (up to 1200 K) while observing the surface with LEEM. However, a nucleation and growth type transition can not be observed in this case, due to the uniform roughening of the entire W(111) surface that is believed to be the initial stage of faceting, as shown by STM (see section 3.2).

3. Scanning Tunneling Microscopy

3.1 Experiments

The STM is operated in a conventional stainless steel ultra-high vacuum (UHV) system with an average base pressure below 2×10^{-10} torr. Besides a McAllister STM, the chamber is equipped with a single pass cylindrical mirror analyzer and a concentric electron gun used to obtain Auger spectra for determination of surface cleanliness and surface composition. A conventional four grid reverse-view LEED optics is used to obtain surface structural information.

The sample is a W(111) single crystal disk identical to that used in the LEEM experiments. The sample is supported by a Ta wire (0.5 mm diameter) loop, spotwelded to the circumference of the sample at several points, which is in turn spotwelded to a molybdenum cylinder that serves as the structural support during STM scans. Electron beam heating is used to clean the sample of surface contaminants by flashing the surface to high temperatures (up to 2500 K). Similar to the LEEM system setup, the sample is positioned in front of a spiral W filament that is heated resistively to incandescence, while the sample is kept at high positive potential (1500 V). A second spiral W filament is part of the sample holder assembly which is usually inserted into the back of the molybdenum cylinder (except while scanning) for sample manipulation. This second filament is used for e-beam heating when annealing the sample at low temperatures (up to

1400 K). This arrangement with the heating filament mounted behind the sample enables the use of an infrared pyrometer to measure the temperature during annealing.

Ultrathin Pt films are deposited from a water cooled metal evaporator, consisting of a piece of high purity Pt foil (0.1 mm thick, 1×5 mm in size) spotwelded to a W wire (0.5 mm in diameter) which is heated resistively. Outgassing loops enable a thorough degassing of the source without loss of material, and the pressure in the chamber remains below 4×10^{-10} even while dosing Pt at a source temperature of ~1700 K. In order to minimize sample contamination during overlayer deposition, faster evaporation rates are used than in the LEEM experiment, on the order of one physical monolayer per ten minutes.

Long time high temperature anneal of the sample, and therefore of the molybdenum support cylinder, would result in unacceptable levels of outgassing and sample contamination. STM measurements, therefore, are a modified version of the above described LEEM experiments. The sample is initially deposited with a desired Pt coverage, then annealed at high temperatures to induce faceting. After Pt deposition and annealing the sample is allowed to cool. Faceted or partially faceted surface structures are identified using LEED observations made at room temperature with electron energies between 50 and 250 eV; then the sample is transferred to the STM for scanning.

3.2 Results

When the W(111) surface is covered with a thin Pt film slightly in excess of one physical monolayer, a fully faceted LEED pattern is observed (figure 2b) and STM reveals a fully faceted surface, composed of three-sided pyramids and adjacent pits with sharp edges. Figure 4 shows one such surface with ~1.1 physical monolayer Pt coverage, annealed at 1200 K (all STM images presented in the paper are displayed in XSLOPE mode, which gives a 3D top view perspective of the pyramids, with an apparent lightsource to the right). Annealing at this temperature results in facets having dimensions ranging from 200 to 500 Å.

When the surface is deposited with Pt in the 0.7 to 1 physical monolayer coverage range and annealed above threshold temperatures (>750 K), a partially faceted surface results. LEED shows a superposition of planar and faceted patterns (figure 2c). STM shows large planar areas with scattered clusters of pyramids. Figures 5 and 6 are scans of a partially faceted surface, with Pt coverage ~0.8 physical monolayers and an annealing temperature of 1200 K.

Figure 5 depicts the overall structure of the partially faceted surface in a 5000Å by 5000Å scan, with clusters of pyramids. There are two important observations: first, relatively large pyramids (200 to 700 Å in size) are scattered on the otherwise smooth planar surface, sometimes standing individually, sometimes forming large clusters that are several thousand Ångstroms in extent. Some pyramids have neighboring pits (figure 6 center), indicating short distance direct mass transport; some are surrounded by planar regions only, indicating long range mass transport from planar regions to form large scale facets. Second, every large pyramid appears to be surrounded by a large number of satellite pyramids. Near-atomic resolution scans reveal that these are indeed small pyramids rather than random clusters, as they possess the structure of faceted pyramids, with {211} oriented sides.

As mentioned previously, it is important to note that the pyramids are not Pt clusters; rather, they are composed of tungsten and covered with a thin film of Pt[9, 10]. In order to generate pure Pt pyramids of the size and density shown in figure 5, an average Pt coverage of ~4 physical monolayer would be required; this is much greater than the 0.8 physical monolayer used here.

We also mentioned in section 2.2 that LEEM does not show a nucleation and growth type transition in experiments where the W(111) substrate is first dosed with Pt in excess of one physical monolayer at room temperature, then the substrate is gradually annealed to higher temperatures to induce faceting. We attribute the lack of observable nucleation and growth to the uniform roughening of the W(111) surface towards a faceted geometry.

Figure 7 shows STM scans of a W(111) surface covered with ~1.2 physical monolayers of Pt, and annealed to 800 K (near the onset of the faceting transition) for 1 minute. These scans reveal the rough surface that is believed to represent the initial stages of faceting. Note that the observed structure is composed of very small pyramid-like bumps with {211} sides, and short ridges running parallel to the surface in either of the [1$\bar{1}$0], [10$\bar{1}$] or [01$\bar{1}$] directions. Based on geometrical arguments, these ridges are believed to be composed of (110)+(112), (101)+(121) or (011)+(211) faces on opposite sides (figure 7.c), respectively, or highly stepped (vicinal) surfaces thereof. This surface gives a somewhat fuzzy {211} faceted LEED pattern with high diffuse background (indicating disorder on the surface). The fuzzy facet LEED spots are attributed to the small pyramidal bumps[1] visible on figure 7.a. The dimensions of the sides of the ridges (10-20 Å) is less than the transfer width of the low energy electron beam; this is consistent with the absence of {110} spots.

Note that ridges running in the three different directions form triangular structures that are oriented the same way as the {211} pyramids. When the same surface is annealed to higher temperatures (1000 K), the ridges are replaced by {211} pyramids (figure 7.d). This observation lends further support to our hypothesis that the ridges of figure 7.c represent the initial stages of faceting at constant overlayer coverage.

4. Discussion

An interesting conclusion based on our LEEM experiments is that the fully faceted surface is achieved through a nucleation and subsequent growth of a relatively few faceted regions. This implies that the Pt film itself grows via a nucleation/growth process.

The processes of film growth during deposition of atoms on surfaces, including the nucleation, growth and coalescence of adsorbate islands, are among the best studied phenomena in surface science. Experimental methods range from diffraction techniques (such as SPA-LEED[18] and thermal energy helium-atom scattering[18-20]) to real space

observations using STM[21-26], transmission electron microscopy (TEM) and LEEM[27]; theoretical predictions and models also abound[28-33].

Metal adsorbates, when deposited onto metal surfaces at temperatures ≥ 300 K, often have high mobility and condense into 2-D or 3-D islands[23, 24]. These islands may be nucleated either at terrace edges or other surface defects such as screw dislocations (heterogeneous nucleation)[23, 27], or in the middle of wide terraces, if the adsorbate density is high enough for the spontaneous creation of nuclei because the number of atoms exceeds a critical value (homogenous nucleation)[24, 26], or both[21, 25].

One physical monolayer on the W(111) surface comprises three different layers, also referred to as three geometrical monolayers. Based on synchrotron soft X-ray photoemission spectroscopy (SXPS) measurements[10], refractory metals, including Pt, are believed to grow layer-by-layer on W before the completion of the first physical monolayer. This suggests that the first two geometrical monolayers (2/3 of a physical monolayer) are completed before the third layer nucleates and spreads via island growth. This suggests that while Pt adatoms in the first two geometrical monolayers remain relatively stable on the W(111) surface, the Pt atoms deposited on top of this surface readily diffuse around until they bond to an already growing island.

While regions covered with 2/3 of a physical monolayer remain planar, faceting begins under Pt islands where the critical coverage has been reached. It is important to note that as the faceted regions grow, there appear to be no new regions nucleated on the remaining planar surface (figure 3). This may be explained by the rapid diffusion of Pt atoms at the elevated temperature of 1050 K, and the very low impingement flux. The combination of these two factors results in a low probability of island nucleation as compared to the growth rate of existing islands.

Note that the formation of overlayer islands in the third geometrical monolayer must depend on a delicate energetic balance, since — due to the atomically rough structure of the bcc(111) surface — atoms within this layer interact via second nearest-neighbor

interactions only. It is possible, however, that overlayer islands that reach the critical thickness of 1 physical monolayer thickness are not only responsible for inducing the faceting transition of the tungsten substrate, they may also be stabilized energetically by the faceting process. On the faceted surface the overlayer atoms essentially form a physical monolayer coverage on W(211) faces, which may provide stronger overlayer-to-substrate bonding than on planar W(111). As a result, Pt adatoms diffusing on the surface may stick preferentially to already faceted regions. This question clearly calls for a detailed theoretical investigation.

The STM data confirm the LEEM observations that a partially faceted surface is a combination of large planar regions, with scattered faceted regions. The faceted regions include clusters of pyramids of two distinct size distributions: large individual pyramids or clusters of large pyramids, surrounded by smaller size satellite pyramids. The latter are consistent with the relatively round shape of the overlayer Pt islands that induce the faceting (figure 3a): the role of the small satellite pyramids is to fill in the rounded outline imposed by the island.

STM and LEEM experiments are also in agreement concerning the initial stages of faceting at constant overlayer coverage. LEEM indicates the lack of a nucleation and growth type transition from the planar to a faceted structure, and suggests the uniform roughening of the surface instead. STM reveals the rough atomic structure of the initial stages of faceting, with microfaceted structures (ridges) that are believed to evolve into {211} facets upon further annealing.

5. Conclusions

We have demonstrated using real-time LEEM observations that when Pt is deposited onto a heated W(111) substrate, the well known faceting process progresses through the nucleation and growth of fully faceted regions distributed over the otherwise planar

surface. STM reveals that each faceted region is a cluster of pyramidal facets of various sizes.

LEEM and STM prove to be excellent complementary techniques in the study of faceting. STM provides structural information down to the atomic scale. The resolution of LEEM does not match that of an STM; however, it does have several advantages that make it a most useful tool in studying the kinetics of large scale morphological transformations: convenience, the capability of imaging surfaces at very high temperatures, and the fact that the surface is easily accessible to observation during metal deposition.

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Figure captions

figure 1. Top and side views of the W(111) surface demonstrate that atoms from the three outermost layers are exposed to the surface. When a metal adlayer is grown epitaxially on W(111), three different atomic layers are needed to cover every W atom — these three "geometrical" monolayers together form one "physical" monolayer.

figure 2. LEED patterns of three W(111) surfaces: a: planar (clean W), b: fully faceted (~1.1 physical ML of Pt, annealed at 1200 K), and c: partially faceted (~0.8 physical ML of Pt annealed at 1200 K).

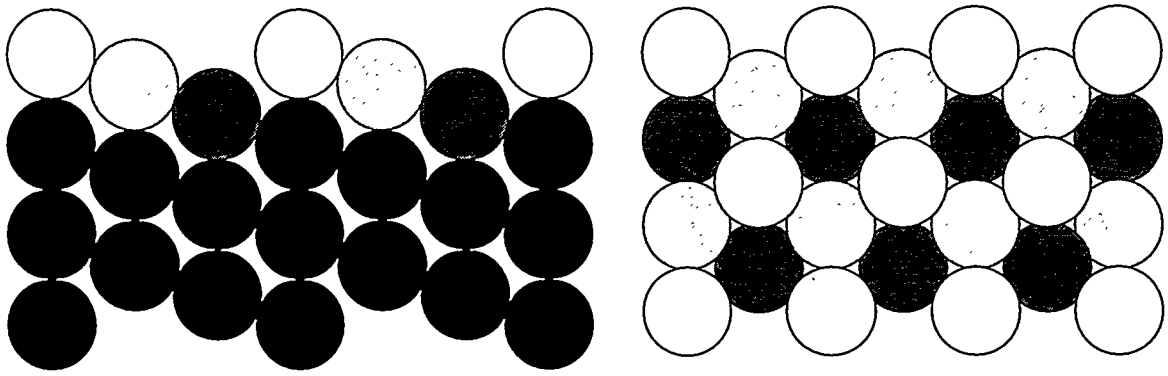
figure 3. Bright field LEEM image of nucleation and growth of faceted regions on Pt/W(111) at constant temperature (~1050 K) and constant Pt flux. The dosing times are 63, 69, 75, 81, 87 and 93 minutes for images 6a through 6f, respectively. Field of view is ~5 μm.

figure 4. STM image of fully faceted W(111) surface: Pt coverage is ~1.1 physical ML, annealed at 1200 K for 1 minute. Field of view is 2000Å × 2000 Å. Sample bias: +1.5 V, tunneling current: 1.5 nA. All STM images are displayed in XSLOPE mode, which gives a 3D top view perspective of the pyramids.

figure 5. STM image (5000 Å × 5000 Å) of partially faceted surface (a mosaic of several 3000 Å × 3000 Å scans). Pt coverage is ~0.8 physical ML, annealed at 1200 K for 1 minute. Sample bias: +1.5 V, tunneling current: 1.5 nA.

figure 6. STM image (1400 Å × 1400 Å) of the same partially faceted surface as figure 5, under identical scanning parameters. Center of image shows a pyramid-pit pair, while lower right part displays two stand-alone pyramids with their satellites.

Figure 7. (a-c) STM images of Pt covered W(111) surface annealed at ~800 K for 1 minute. Initial stages of faceting are observable: coexistence of small {211} pyramids (arrows on figure a) and {110}+{211} ridges (marked on figure c). Image sizes are 1000 Å, 500 Å and 300 Å, respectively. (d) is a 1000 Å scan of the same surface annealed at ~1000 K for 1 minute; the surface is fully faceted into {211} pyramids. The azimuthal orientation of all images is the same.



side view top view

Figure 1.
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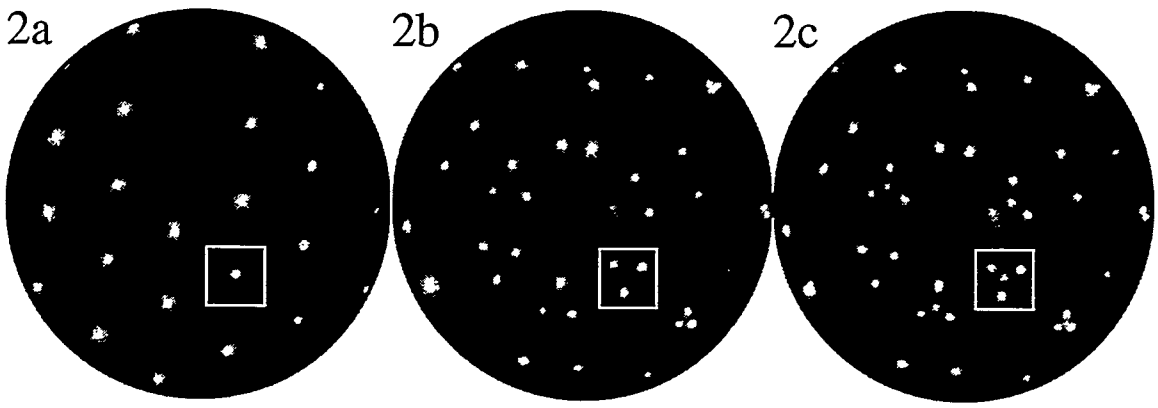

Figure 2.
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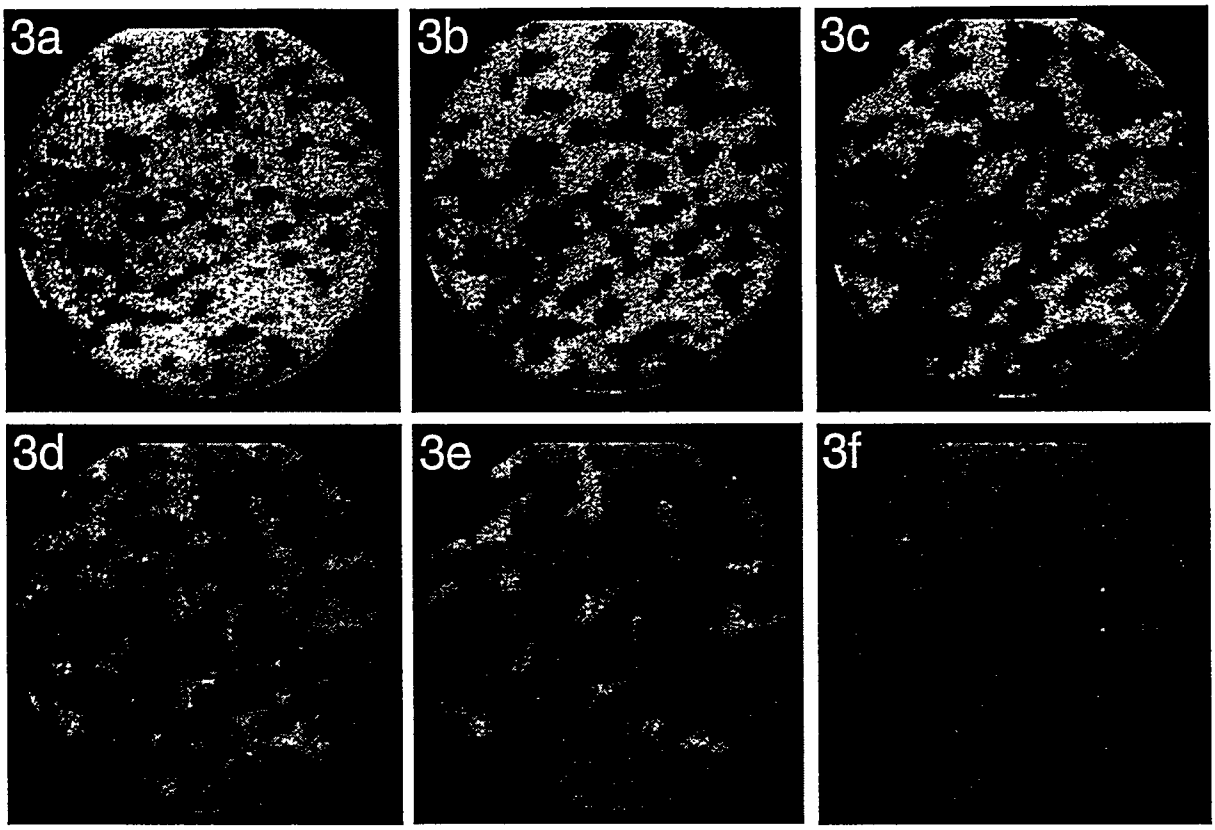

Figure 3.
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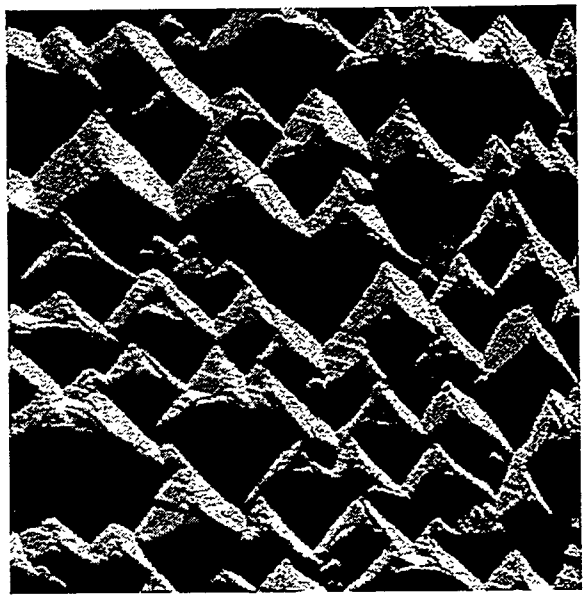

Figure 4.
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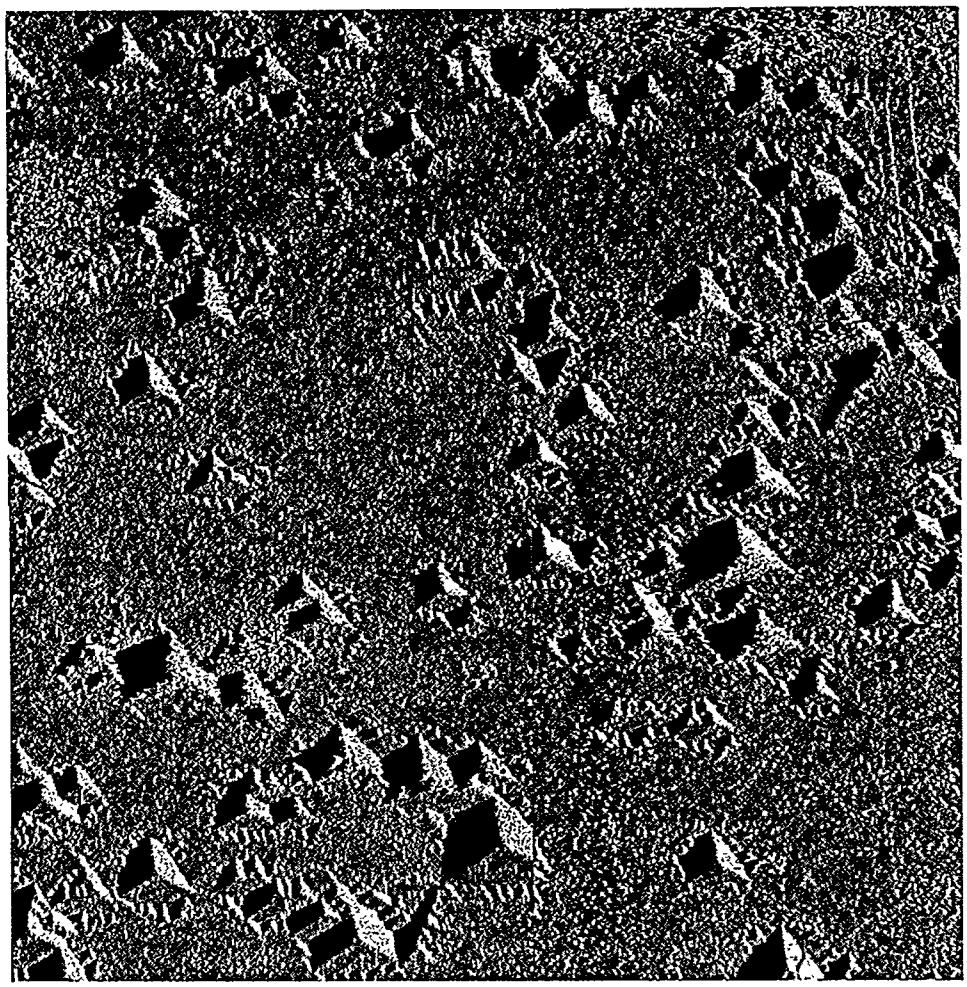

Figure 5.
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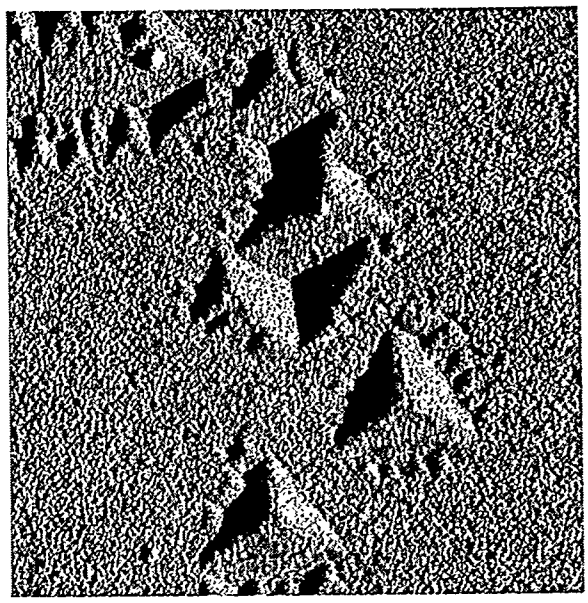

Figure 6.
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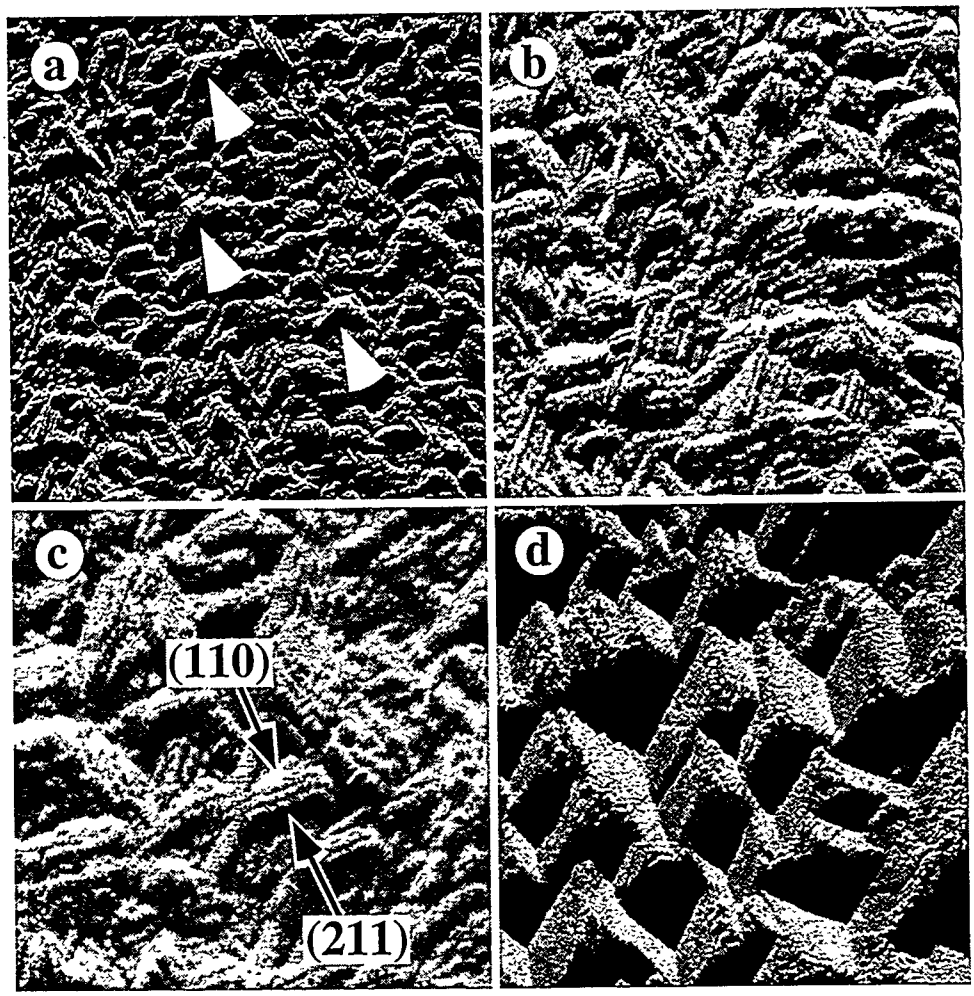

Figure 7.
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