Calibration and evaluation of scanning-force-microscopy probes

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It is demonstrated that a stepped (305) surface of a $SrTiO_3$ crystal can be used routinely to evaluate the probing profile of scanning-force-microscopy probes. This provides a means to select optimal surface probes, and to evaluate possible image distortions within the range of the atomic and nanometer scale. The scope and limitations of the resolution of structural defects are discussed as a criterion for a true atomic resolution.

Scanning force microscopy (SFM), which is often also referred to as atomic force microscopy,[1] currently is intensively used in various fields of material science in order to study surface interactions and surface structures. However, the image information often remains vague or ambiguous, due to the lack of insight of the actual imaging process.

The resolution of a scanning microscope depends on the effective size of the probe which in the case of scanning force microscopy is determined by the probe geometry and the forces involved in the probe-surface interaction. Theoretical calculations so far are based on the assumption of an idealized defined geometry of the probe. This assumption is used in microscopic approaches for the calculation of the probe-surface interaction,[2] in molecular-dynamic simulations[3] of the scanning process, and in the application of macroscopic theories.[4–6] Good qualitative agreement between theory and experiment can be achieved but quantitative agreement is lacking. Deviations from the ideal geometry of

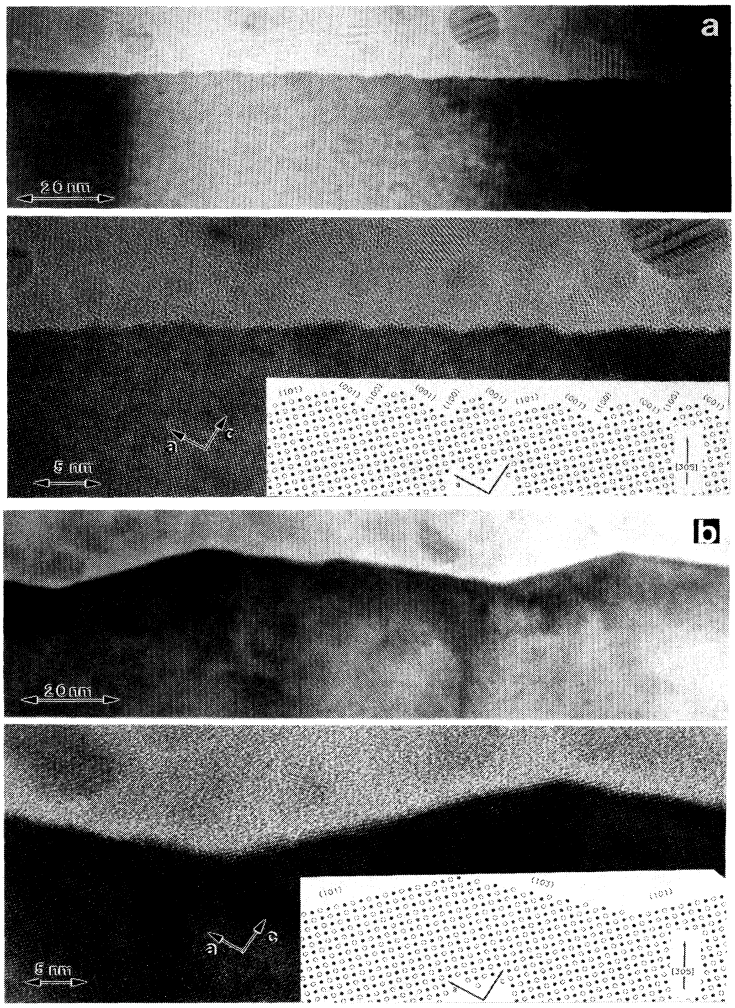

FIG. 1. High-resolution transmission electron micrographs of a (010) cross section through a $SrTiO_3$ crystal after the (305) surface was mechanically polished and chemically etched (a) and after the crystal was annealed for 20 h at 1100 °C under oxygen (b).

the tip become essential when the irregularities are more than 10–20 % of the surface-probe distance.[7] Thus, measurements of nonretarded forces at small distances should take into account the corrugations of the atomic scale. Variations in the probe size and shape affect the interaction and therefore also the image resolution. If the surface corrugations are comparable with the probe apex shape, mesoscopic artifacts (structural details in the range below 100 nm will be denoted as mesoscopic) are produced in the SFM images.[8]

In order to evaluate SFM images for the actual distance and force value and to recognize artifacts unambiguously, routine determination of the exact shape of the probe apex is essential. Such a mesoscopic characterization of the SFM probe can be realized by electron microscopy.[8] However, the required resolution needs sophisticated experiments and sample preparation techniques. Although the development of SFM probes with high aspect ratio and improved sharpness is progressing rapidly,[9–12] direct control of the probe will remain important. In the present paper we describe a simple and reliable method how to evaluate the mesoscopic profile of the probe apex.

Based on the following considerations, we propose a gauge which allows direct calibration of a SFM probe: The gauge should have a macroscopically regular surface with a well-defined atomic structure *and* superstructure. This superstructure should contain atomically sharp elevations varying more or less periodically within a few nanometers in the z direction. The surface must be rigid and invariant with time at ambient conditions. In order to check the image distortions for a particular tip and probing mode *in situ,* recording of an image of such a test surface must be possible as a routine operation prior to or after an unknown surface is monitored by SFM.

The proposed gauge is a specially treated surface of a perovskite-type crystal of $SrTiO_3$.[13] This substrate has

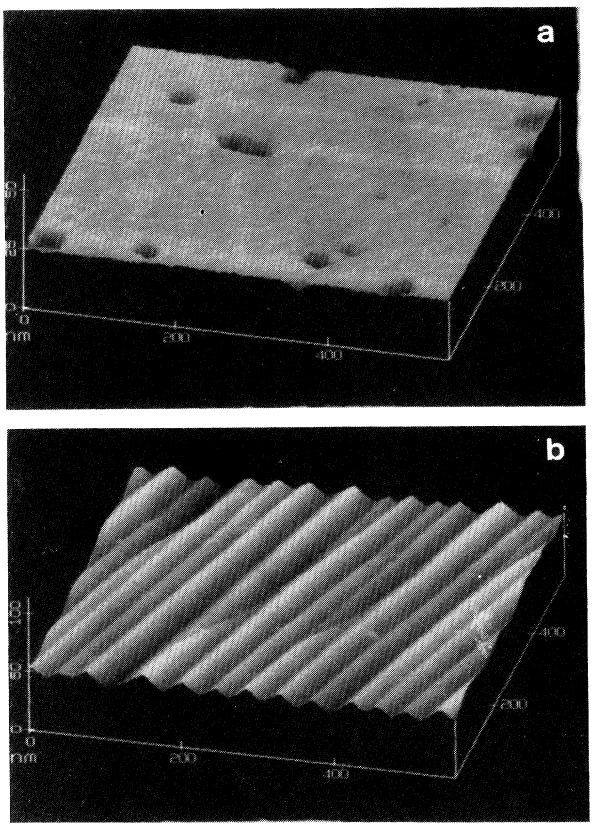

FIG. 2. SFM micrographs of the (305) surface of a $SrTiO_3$ crystal. (a) The sample was mechanically polished and chemically etched. (b) The sample was annealed for 20 h at 1100 °C under oxygen.

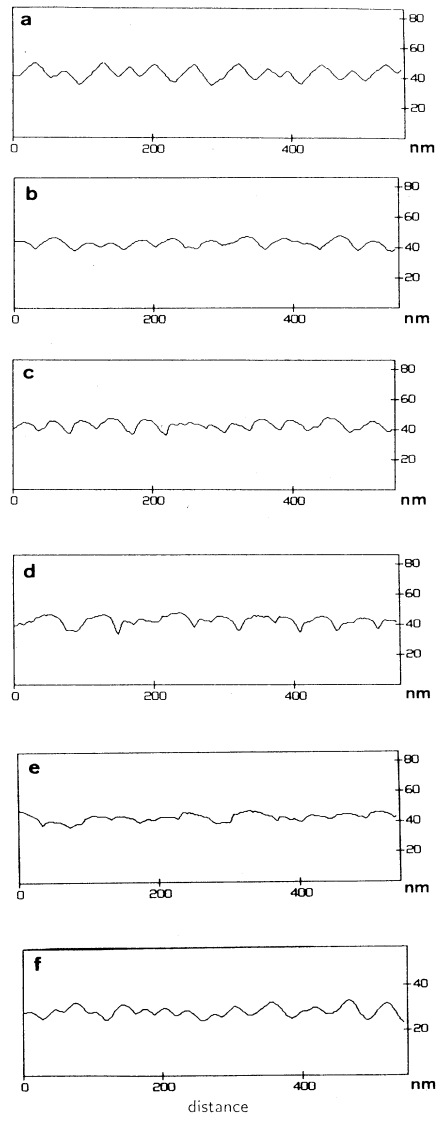

FIG. 3. A series of SFM 2D profiles which were recorded by different commercial Si_3N_4 tips from the same indented $SrTiO_3$ surface from which an example is shown in Fig. 1(b). Only (a) gives a good reproduction of the surface. The images impart a wrong impression about the actual structure as the distortion of the needle becomes more severe from (b)–(f).

been used for epitaxial growth of high-temperature superconductors.[14,15] The crystals had been cut parallel to the (305) orientation and the (305) surface was mechanically chemically polished.[14] The high-resolution transmission electron micrographs in Fig. 1(a) show the (010) section of the resulting surface. The surface profile can be described as alternating (100) and (001) steps piled up from not more than a few atoms. The crystals were annealed for 20 h at 1100 °C in flowing oxygen. The treatment resulted in a rearrangement of the atoms in the surface and the formation of larger terraces. This is shown in Fig. 1(b) where the surface consists of alternating (101) and (103) crystal planes. The transmission electron microscopy (TEM) micrograph reveals the defined inclinations with respect to the surface plane (305), i.e., $+14°$ and $-11.6°$, respectively. This was found all over the entire crystal surface.

Figure 2 shows scanning-force-microscopy images of such a surface before and after the annealing, which were recorded with a selected tip as will be described below. The surface in Fig. 2(b) was scanned by different commercial Si_3N_4 tips. SFM micrographs were recorded at ambient conditions with a Nanoscope 2 (Digital Instruments, Inc., Santa Barbara, CA). Commercial pyramidal Si_3N_4 tips mounted on a V-shaped cantilever with a force constant of 0.06 N/m (Nanoprobes^{TM}) were employed.[12] Applied forces were varied in the range of 5–50 nN, not indicated directly in the figure captions. Forces in this range can be recommended for the evaluation of the tip quality at ambient conditions. Supplementary measurements were done with a "super tip" which was prepared according to Keller and Ching-Chung and has a higher aspect (length/diameter) ratio.[9] The corresponding two-dimensional (2D) profiles are presented in Fig. 3. Within the range of 5–50 nN for the applied force the scanning process was stable and the recorded images did not change in dependence on the force significantly.

Obviously, most of the profiles do not represent real true-to-scale reproductions. A nearly perfect surface image was obtained only with one tip selected out of 20 (which had been used also for the images in Fig. 2). Figure 3(a) shows the true step structure of the $SrTiO_3$ crystal with correct and rather sharp angles.

Figures 3(b)–3(e) show the same $SrTiO_3$ surface recorded with less perfect Si_3N_4 tips. The tip used for Fig. 3(b) reproduced the shape of the steps with strongly smoothed edges. Grooves were disguised due to the multiple imaging. The radius of the probe apex can directly be estimated from Fig. 3(b) to be around 20 nm.

Figure 3(c) gives an image which has been recorded with a tip having a truncated pyramidal peak. Still, a corrugated profile is monitored, however details of actual structure are significantly misrepresented. From the truncations of the peaks in the surface profile, it can be estimated that the tip had a flat plateau of roughly 10×10 nm^2.

Another example of a needle with a truncated peak is given in Fig. 3(d). A strongly distorted image resulted and the apparent profile does not resemble the actual topology. However, using this probe the surface atomic arrangement of different crystals was monitored reproducibly and with excellent quality. From the profile, it can be concluded that the probe is not only flattened but it apparently possesses several small protrusions which can be in contact with different points of the surface simultaneously.

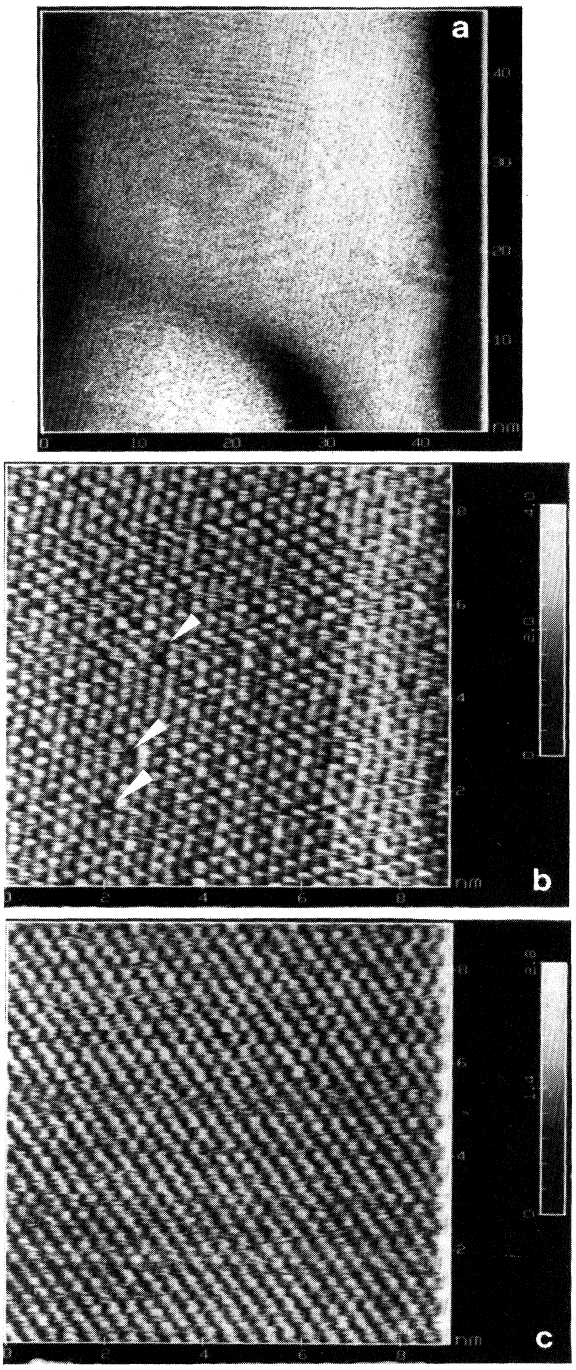

FIG. 4. Selected examples of AFM images which show a real near-to-atomic resolution. All images were recorded with a first-class probe like the one used for Fig. 3(a) at 20 nN repulsive force: (a) a gold film sputtered at room temperature on glass, (111) terraces of a width of 6 Å can still be resolved; (b) image of a TaS_2 crystal with several point defects; (c) image of the TaS_2 crystal recorded with the less sharp probe.

Another tip which is completely useless for mesoscopic surface characterization is demonstrated in Fig. 3(e). Surface corrugations are masked, flattened, and also their periodicity is strongly misrepresented. No straightforward conclusions of the malformation of the tip can be drawn in this case.

Figure 3(f) gives the image of the same surface as it was monitored with a "super tip."[9] The step structure is observed correctly but the sharp edges were imaged with considerable smoothing. The radius of the curvature of the round edges in the image is directly related to the radius of the probe apex which can thus be evaluated to 10 nm. A similar profile was obtained with a silicon tip which recently became available.[11]

From theoretical considerations it is not excluded to obtain images with a true atomic resolution, provided that the investigated surface is flat, rigid, and probed by an atomically sharp tip with a nondamaging force.[17] Figures 4(a) and 4(b) demonstrate examples of high resolution where the selected tip from Fig. 3(a) and selected samples were used. Because of the nearly perfect shape, the contact between this tip and the surface is highly localized and the basis for visualization of single atomic details is rather optimum.

Figure 4(a) shows small gold particles which were deposited on mica in vacuum at 50 °C. Terraces from different (111) layers which can be considered as linear defects were observed even when their width was only 6 Å.

While the visualization of such linear defects as terraces, grain boundaries, and dislocations is in agreement with other recent SFM studies,[18] atomic point defects could not be exposed clearly by SFM.[19] Whether this becomes possible by using a carefully selected tip is discussed when considering the example of surface images from TaS_2 crystals. According to scanning tunneling microscopy data, layered crystals of TaS_2 possess a large number of point defects on their surface.[20,21] Figure 4(b) gives the SFM image of the (0001) surface of a TaS_2 crystal which was not filtered or otherwise processed. Several point defects can be observed which are marked by white arrows. In many cases these vacancies were reproduced in successive measurements. Mostly when they were located at the lower or upper edge of the image which can be monitored twice within a short time interval (~ 0.1 s). An atomic structure was also resolved when the same crystal was monitored with a less sharp tip [Fig. 4(c)], however, no vacancies were depicted. Based on a large number of similar experiments, we believe that the images in Figs. 4(a) and 4(b) give examples of true atomic resolution, while Fig. 4(c) results from multiple contacts of the tip with the regular surface and represents only the regular periodicity of the surface.

In summary, the high-temperature treated (305) surfaces of a $SrTiO_3$ crystal provide a helpful means to select the most perfect tips and to evaluate possible image distortions. Because of the crystallographically defined angles, the gauge can also be used for the height calibration of the SFM scanner. The images in Figs. 2 and 3 demonstrate the quality of the experimental data on which a deconvolution of the tip shape can be performed. On this basis the calibration will be helpful for approaching true atomic resolution in SFM, 3D deconvolution of the surface structure, nanoindentation, evaluation of interaction forces, and development of peculiar tips. However, the calibration gauge provides a necessary but not a sufficient condition in order to attain a maximal resolution. In a particular experiment, the nature of the interaction and micromechanical characteristics of the sample and the tip are as important as the geometry of the probe.

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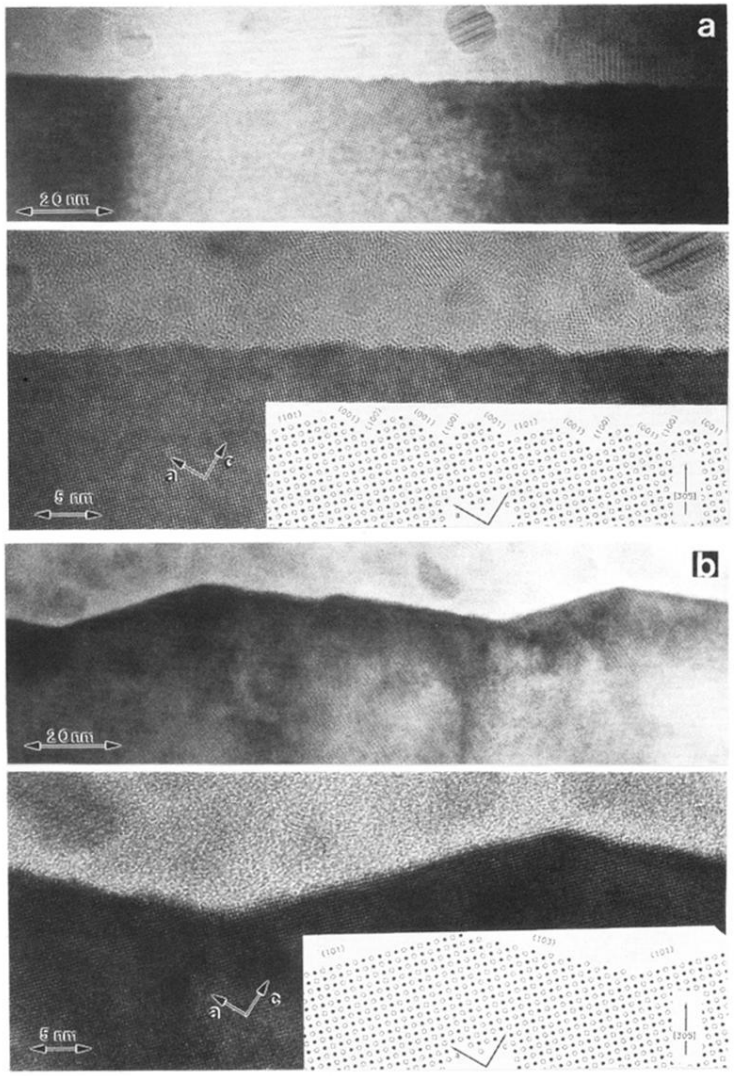

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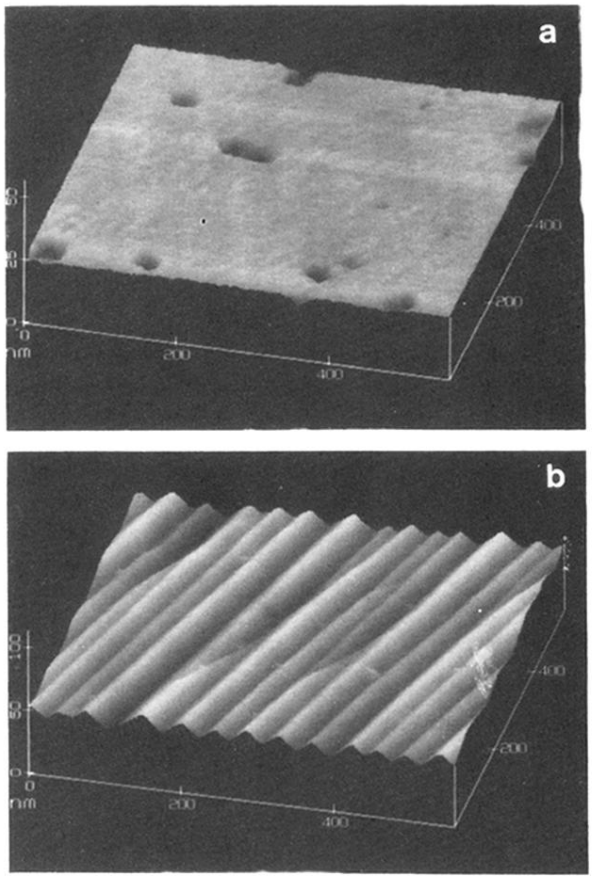

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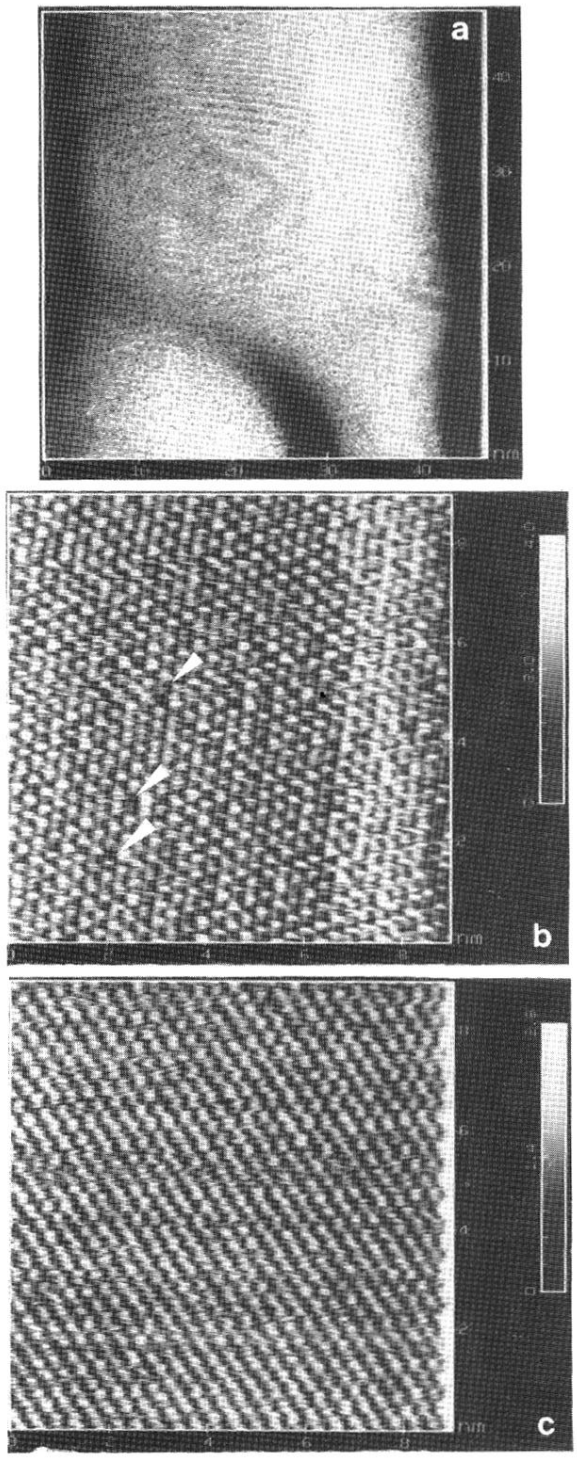

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