

preprint from:

S. Morita, R. Wiesendanger, E. Meyer (eds.)

Noncontact Atomic Force Microscopy

Springer Verlag, Berlin to be published June 2002

6 Atomic resolution imaging on fluorides

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Fluorides are an important class of inorganic materials that have been intensively studied in their bulk properties for several decades [1]. Among crystals with the fluorite structure, CaF_2 is the most prominent prototype crystal that recently gained tremendous interest as a vacuum ultraviolet optical material. Components made of CaF_2 are necessary for the development of next generations of laser lithography that is a key technology for the semiconductor industry [2]. Laser lithography optics requires materials with structural perfection and utmost purity of bulk and surface. In this context, highest resolution and sensitivity imaging on fluorides for structural characterization and surface defect detection became an important issue and is a major driving force behind the development of dynamic force microscopy for highest resolution imaging on fluorides.

Beside this specific application, fluorides are important test materials for the development of atomic resolution force microscopy on insulators. Surfaces of fluorite type crystals prepared by cleavage along the (111) plane are atomically flat over large areas and very stable under ultra-high vacuum conditions. Their structure is simple but bears more details than that of cubic halides (see Chapter 5) and these details provide an excellent test for the resolution power of the scanning force probe but also for theoretical predictions on scanning results. As will be discussed in Chapter 17, scanning force imaging on the $\text{CaF}_2(111)$ surface has been modelled by theoretical simulations and this is presently the best understood insulator surface in terms of a quantitative interpretation of atomic scale contrast formation. Both, the ease of preparation and the detailed understanding of atomic contrast predestines the $\text{CaF}_2(111)$ surface as a standard for the calibration of atomic resolution scanning force measurements and the calibration of tips.

In this chapter we review the state of the art in atomic resolution imaging of fluoride surfaces and describe main features and peculiarities while all aspects of quantitative imaging are discussed in Chapter 17. In the following sections we will briefly introduce details of our experimental techniques, then address the important issue of tip structure and tip instability and finally discuss atomic contrast on flat surfaces and at step edges and kinks. An important feature of experiments introduced here is that measurements are often not operated in the standard *topography mode* where the cantilever resonance frequency detuning is kept constant but in the *constant height mode* where the detuning is the primary output signal. Results obtained in the two modes are contrasted and it is discussed what kind of information and details can best be deduced from either method.

6.1 Experimental techniques

All experiments reported here were performed with a scanning force microscope based on the design by Howald et al. [3] operating in an ultra-high vacuum (UHV) system with a base pressure in the low 10^{-8} Pa range. The principles of operation of this instrument are those outlined in Section 2.3 and a schematic representation of the electronics for cantilever self-excitation and measurement is shown in Fig. 6.1. The cantilever is excited to vibration at its resonance frequency of typically 75 kHz with an **amplitude A** stabilized to a pre-set value between 20 nm and 100 nm (peak to peak) by a positive feedback loop where the error signal of the amplitude loop yields a measure for the per-cycle dissipated energy during cantilever oscillation and is therefore called **damping G** . The cantilever resonance frequency **detuning Δf** is detected by a PLL demodulator and used as an input for the distance control loop where the error signal of this Δf loop yields the **topography z** . The extent to what the tip follows the contour of the pre-set Δf value is determined by the gain of the topography (Δf) loop. In standard *topography mode* imaging, the loop gain is high, Δf virtually vanishes and z represents the full topographic contrast. In the *constant height mode*, the Δf loop is switched off so that the tip scans effectively at constant height above the surface and ideally the topography signal z vanishes. In practice, the loop is often not switched off but operated at very low gain so that a minor inclination between the surface and the scanning plane is compensated to maintain a constant tip-surface distance while the cantilever response to small surface features and especially atomic corrugation is not influenced by the distance control. In this case, the detuning signal Δf is the primary output signal carrying information on the tip-surface interaction. During scanning, all four output signals are recorded for forward and backward scanlines and thus each image point can be characterized by a set of eight measured values. If relevant, the scanning direction is indicated in images and graphs by arrows for

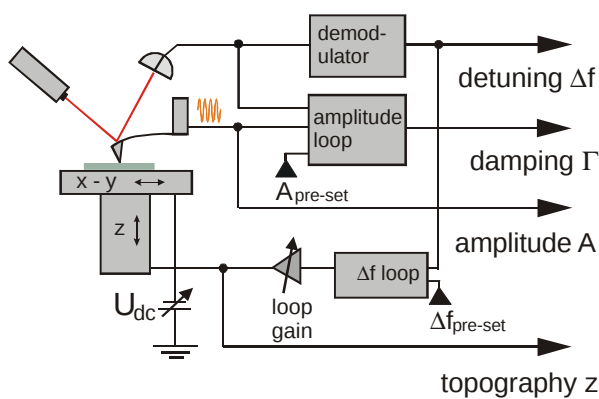


Fig. 6.1.

Experimental setup for dynamic mode SFM with self-driven cantilever oscillation. In *topography mode* imaging, the topography signal z is recorded at constant detuning Δf while the primary output signal is the detuning Δf when operating in the *constant height mode* at fixed tip-surface distance

forward ($\Rightarrow$) and backward ($\Leftarrow$) scanlines, respectively. Limitations in image quality are imposed by noise from the detection electronics rather than physical properties of the cantilever set-up (see 2.5). For the better visualization of their detailed features, images are often low-pass Fourier filtered. Imaging results also depend on the speed of scanning used for image acquisition. When scanning at low speed (5 to 20 nm/s), atomic features can be recorded in all signal channels. When scanning with high speed (30 to 60 nm/s), however, atomic contrast generally vanishes in the damping signal since in this case, the oscillation amplitude is not primarily stabilized by the amplitude feedback loop but properties of the high-Q cantilever oscillation [4].

Cantilevers are p-doped ($1.5 \Omega\text{cm}^{-1}$) silicon cantilevers with a spring constant of typically 5 N/m. Tips are not sputtered and, therefore, coated by oxide, hydroxide and possibly other molecules of unknown composition. We found that it is most favourable for atomic resolution imaging on fluorides to bring the tip in slight contact with the surface and slowly retract it while scanning an area of a few nm^2 prior to taking images. For this method of tip preparation it is likely that not silicon is the material at the tip end but the tip picks up surface constituents and the sensing cluster yielding atomic contrast is a polar group enhancing the interaction with the ionic surface [5]. Although, tip preparation is of utmost importance for atomic resolution scanning, no general recipe can be given for a procedure to prepare a tip with a well defined, stable structure yielding optimum results. However, as discussed in Section 17.4.1, some tip properties can be deduced from force spectroscopy experiments with the help of theoretical simulation and in the next section we describe how atomic scale tip instabilities are detected.

Samples are taken from commercial fluoride single crystals of highest available quality (vacuum-ultraviolet grade) and surfaces are prepared by cleavage in UHV along the (111) surface as the natural cleavage plane. For stability it is important to select a scanning area with a small density of macroscopic steps. To minimize electrostatic forces due to residual surface charging, sometimes samples are heated to about 350 K for some hours and in all measurements, a tip bias voltage U_{dc} of several volts is applied. The appropriate charge compensating voltage is determined by a method similar to that described in Ref. [6].

6.2 Tip instabilities

Atomic resolution imaging crucially depends on the composition and structure of the sensing cluster at the tip end. Although, there have been careful investigations of the atomic composition and efforts for manipulating the tip end [7, 8], to date there is no experimental method that would allow to determine which atom yields the atomic contrast observed in a given image. Neither it is possible to prepare a tip where a specific atom or atomic configuration can deliberately be placed at the tip end. Furthermore, once prepared, the tip is rarely found to be stable on the atomic scale but changes frequently, often several times within recording a single frame. The possibility of tip changes has been known for a long time, however, it

has often been ignored or not recognised since changes in the atomic tip structure are frequently not observed in images of topography and detuning. It was an important discovery that tip changes can most sensitively be detected in the damping signal and this lead to a much better understanding of subtleties in the interaction between the sensing tip atom and surface atoms and their dynamic instabilities.

This is demonstrated for the examples shown in Fig. 6.2. where contrast features in topography and damping signals are compared to each other. In the measurement shown in frames (a) and (b), there appears clear atomic contrast in the topography while the damping attains a constant value over many scanlines with atomic features as a faint superposition only. The most prominent feature, however, is an abrupt change occurring after having acquired more than half of the image that appears as a shift in atomic rows in the topography and as a 50 % change in the damping signal. We recognize that a shift in atomic rows is not only present when the damping signal changes drastically but also upon very subtle variations amounting to only a few percent of the damping signal. Our interpretation of these findings is illustrated in frame (c) of Fig. 6.2. We propose that there is a correlation between details of the atomic structure and relaxation at the tip end and the level of energy dissipation. Weakly bound tip atoms can strongly interact with the surface and couple energy into many degrees of freedom during each period of cantilever oscillation, effectively yielding a high level of energy dissipation. A compact cluster at the tip end with a highly coordinated terminating atom is expected to yield much less interaction and dissipation. Therefore, we associate strong changes in the dissipation signal with the appearance or disappearance of weakly bound atoms or the pick up and drop off of atoms. The

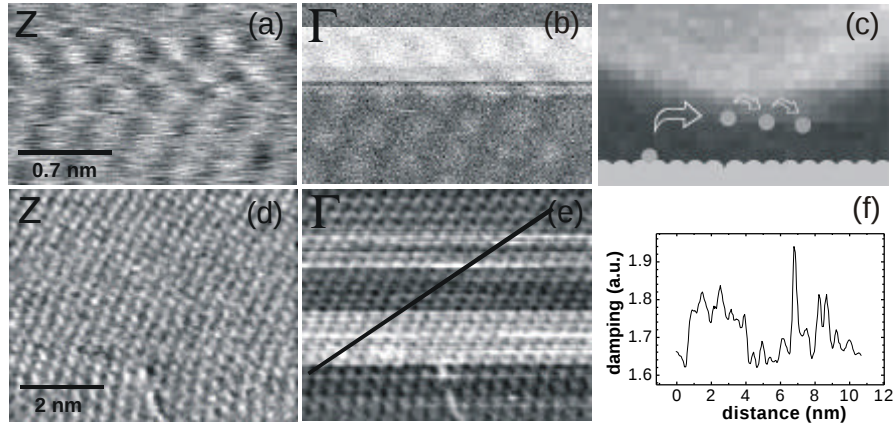


Fig. 6.2. Tip instabilities apparent during imaging $\text{SrF}_2(111)$ in the topography mode. Frames (a), (b) and (d), (e) represent topography and damping signals for two measurements with different tip structures. Graph (f) is a cross-section of the damping signal along the line shown in frame (e). Sketch (c) visualizes atomic instability events at the tip apex

more subtle variations are movements on the tip apex from one local energetic minimum to another where the observed shift in atomic rows associated with such events directly reflects the atomic displacement. One might expect that such atomic movement will stop as soon as the atom reaches a deep energetic minimum. However, the strong chemical interactions between the tip atom and surface atoms present in each cantilever oscillation cycle lead to strong atomic displacements and thus provides much activation energy for further jumps even into shallower energy minima. This is the reason for the inherent instability in atomic resolution force imaging and the great challenge for further improvement of the technique aiming for the preparation of perfectly stable tips with a highly coordinated terminating atom at the end that is subject only to weak displacement. On the other hand, low coordination of tip atoms may yield additional atomic scale information due to a strong enhancement in damping contrast. This is demonstrated in Section 6.4 where the low coordination of atoms at step edges is detected by the interaction with weakly bound tip atoms.

Figure 6.2. (d) and (e) are illustrations for another class of dissipation phenomena related to tip instability. In this measurement, we find strong atomic contrast in both, the detuning and damping images. However, while tip changes are clearly visible as frequent changes in average dissipation, they are almost absent in the detuning image. The cross-section of the damping image shown in frame (f) reveals that there appear, in fact, variations in the damping signal level comparable to those from the previous example and that there seem to be several preferred levels that may indicate specific configurations. The precise nature of such configurations is not clear presently but we speculate that these involve bistability in the atomic position and a weakly bound atom flips from one position to another excited by each cycle of cantilever oscillation and the amount of energy dissipated in these flips contributes to dissipation. The fact that tip changes are not recognized in the detuning image points to separate mechanisms of contrast formation in detuning and damping images. While atomic contrast in the detuning signal is formed by a stable atom terminating the tip, the weakly bound atom is at another position on the tip further away from the surface but flips position each time the tip approaches the surface. Thus, its motion dissipates oscillation energy but the atom does not contribute strongly to the primary atomic detuning contrast.

So far, the discussion of tip instabilities remained rather general and contrast changes could not be associated with specific atomic events. However, occasionally this appears to be possible and one can directly observe a change in polarity of the tip terminating atom as is the case in the measurement shown in Fig. 6.3. During acquiring this image in the constant height mode, the tip changed one time where the atomic contrast in the detuning image was altered from one characteristic appearance to another and this change was associated with a change in the average level of the damping signal by 6 %. These two characteristic contrast patterns will be introduced in the following section and analysed in detail by theoretical simulation in section 17.4.1. The clear outcome of this analysis is that the measurement discussed here has been performed by a tip that was initially terminated by a negative species that changed to a positive one by the event of instability. A simulation of dynamic instability suggests that the tip change event

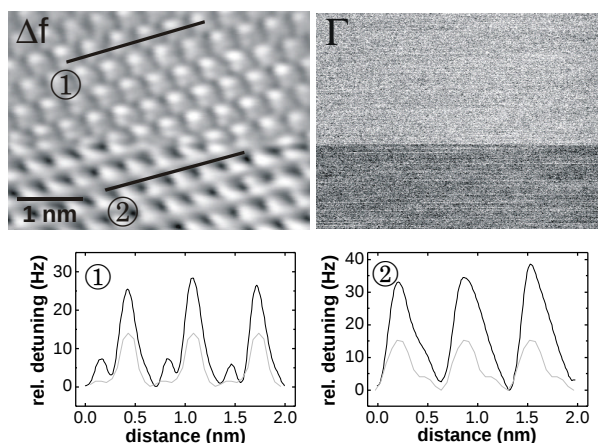


Fig. 6.3.

Tip change during scanning on $\text{CaF}_2(111)$ observed as a change in atomic contrast in the detuning signal and simultaneously as a change in the damping level. Graphs ① and ② are cross-sections taken from the images as indicated above. Grey lines are simulated profiles described in Section 17.4.1

could likely be triggered by the pick up of a positive ion from the surface, however, it could also be solely a re-arrangement of atoms on the tip terminating cluster where the ion closest to the surface changed from a negative to a positive one. As the change in damping is rather small, we assume that the latter was the case, however, there is no evidence for this assumption.

In conclusion, we can claim to have understood some of the basic mechanisms of tip instability and their influence on atomic resolution contrast formation but details of dissipation need to be explored. Tip changes often appear as an obstacle for stable imaging but also bear the potential of being useful for understanding and possibly manipulating chemical bonds and atomic scale structures on the surface.

6.3 Flat surfaces

First atomic resolution images on the $\text{CaF}_2(111)$ surface were obtained in the topography mode and revealed the ionic structure expected for the bulk terminated surface. On the clean surface, in a large number of images with various frame sizes up to $10 \times 10 \text{ nm}^2$, perfect atomic periodicity was found while atomic scale defects could never be detected [9]. We attribute this to the very high surface mobility of point defects anticipating that such defects are certainly present in large numbers at surfaces prepared by cleavage. Indirect evidence for this assumption could be obtained from a study of the interaction of oxygen with the clean surface [9]. Upon oxygen dosage, stable, atomic size defects could clearly be identified and from the slow kinetics of defect formation in conjunction with further evidence from studies of surfaces exposed to higher dosages [10], one can conclude that defects present on the surface prior to gas dosage act as reactive centres and initiate the formation of a chemisorbed species most probably in the form of hydroxyl groups and clusters. As these studies clearly established atomic resolution force imaging on this surface, they could not yet yield quantitative information on atomic corrugation.

To obtain such information, it turned out to be necessary to refine the experimental technique and specifically to very carefully adjust the loop gain and regulation characteristics of the Δf loop. An example for a measurement performed under conditions of full distance regulation is shown in Fig. 6.4. While images of topography and damping clearly exhibit atomic corrugation, there is not any contrast in the detuning signal demonstrating the perfect action of the distance control loop. The change in detuning appearing after half completion of the frame is due to a deliberate reduction of the pre-set Δf value to slightly retract the tip from the surface. Note also that considerable tip instability is evident from the damping image, however, this does not affect the topography image. The graph in Fig. 6.4. shows a cross-section through the topography image along the indicated line. From this trace we deduce a peak-to-peak corrugation of 60 pm and this is a typical value obtained on fluoride surfaces and alkali halide surfaces (see 5.2.2). To date it is not possible to associate a specific corrugation with a specific fluoride material since there are large variations from one measurement to another when working with the same material. For $\text{CaF}_2(111)$ as an example, we determine corrugation values ranging from 30 pm to 100 pm. These differences cannot systematically be associated with experimental parameters that could be controlled but are most probably due to a variation in tip structure from one measurement to another. A clarification of this point will only be possible by theoretical simulation on the basis of various tip models.

In topography mode imaging, surface ions always appear as spherical caps without any significant variation in appearance other than by noise, drift or other image distortions. In an effort to reveal the surface structure in its most subtle details, we introduced scanning in the constant height mode where Δf is the primary signal and this signal is entirely independent from the action of a distance control loop that potentially leads to distortion of image features. Indeed, it was possible to refine this technique and reach a level where significantly more information can be obtained from atomic contrast patterns.

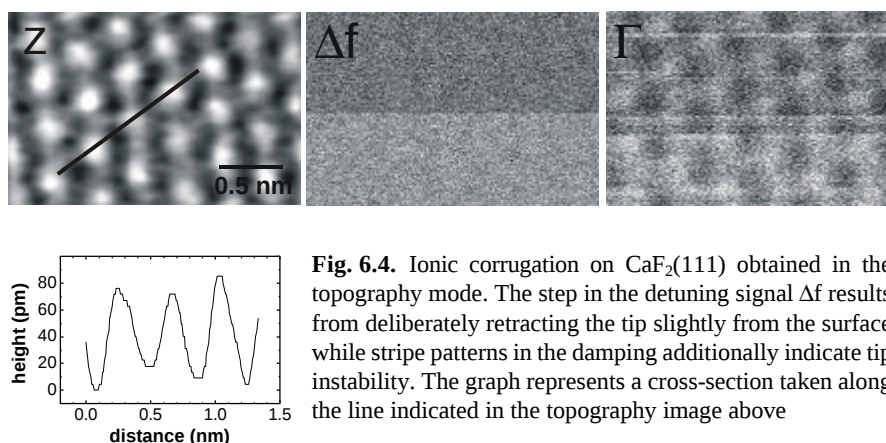


Fig. 6.4. Ionic corrugation on $\text{CaF}_2(111)$ obtained in the topography mode. The step in the detuning signal Δf results from deliberately retracting the tip slightly from the surface while stripe patterns in the damping additionally indicate tip instability. The graph represents a cross-section taken along the line indicated in the topography image above

It is found that contrast features in detuning images can be grouped in two categories, one where ions appear as triangles and the other where the appearance is similar to the contrast obtained in topography mode imaging; respective examples are shown in Figs. 6.5. and 6.6. together with cross-sections along the $[\bar{2}11]$ direction that reveal further details. As discussed in detail in Section 17.4.1, by extended theoretical simulation, the two different contrast patterns can be explained as a result of imaging with tips of different polarity where the triangular shape appears when scanning with a positive tip. For imaging with a positive tip, each ionic position is split into two peaks where the large one results from the attractive interaction of the sensing cation with a fluorine ion from the topmost layer while the smaller peak appears at the position of a fluorine in the sub-lattice beneath. When imaging with a negative tip, the main periodicity is created by calcium ions from the layer in between the fluorine layers and the latter contribute to image contrast with a small peak between the large ones. Such measurements establish that imaging in the constant height mode allows to unambiguously identify contrast from different sub-lattices but also yield information about the polarity of the terminating tip atom [11].

However, as shown in Fig. 17.7. and discussed in conjunction with theory, these simple contrast patterns appear only in a certain range of tip-surface distance and more complex processes of contrast formation arise when the tip approaches the surface more closely. This figure also demonstrates another experimental feature that is frequently observed, namely that scanning in forward and backward directions does not yield identical results. The details of this phenomenon are not yet well understood, however, from many pieces of experimental evidence it is clear that it is related to the details of the structure at the tip end. For the same reason, one also frequently observes that contrast patterns along equivalent directions on the surface are not identical. This, of course, is a severe obstacle for quantitative surface analysis but can partly be overcome by extended image analysis as described in Ref. [12]. A better understanding of the associated phenomena can be expected from theoretical modelling of more complicated tip configurations, however, the most desirable solution would be the development of reliable preparation techniques routinely yielding tips that are perfect on the atomic scale. Occasionally, the experimentalist is lucky enough to prepare such a perfect tip as was the case for the measurement shown in Fig. 6.6. This scan yielded identical results for forward and backward scanning and also systematic deviations in the cross-sections taken along equivalent directions are as small as in no other scan. As can be deduced from Fig. 6.6., cross-sections in directions ①

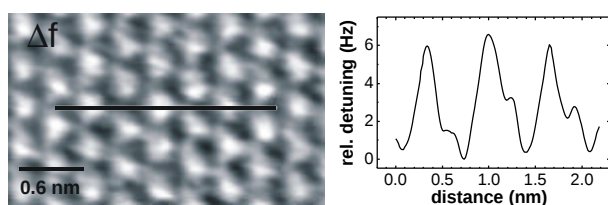


Fig. 6.5. Ionic corrugation on $\text{CaF}_2(111)$ obtained in the constant height mode. The graph is a cross-section along the $[\bar{2}11]$ direction indicated in the detuning image. From Ref. [11]

and ③ are identical and only in the averaged line representing direction ②, a significant deviation is found. For a quantitative analysis, precision is only limited by the statistical error introduced by the noise. In these measurements, the noise level (see 2.5) is mainly determined by the data acquisition electronics and, hence, further improvement of precision in data analysis appears to be possible.

This example demonstrates that $\text{CaF}_2(111)$ is an excellent surface for calibrating tips on the atomic level. This surface can easily be prepared by cleavage and under UHV-conditions it remains clean for several days. Atomic resolution contrast in the constant height mode is straightforward to obtain but imaging the detailed structure as described here is an excellent test for the tip. The polarity of the tip terminating atom can be determined from the basic contrast pattern observed. As the surface structure is highly symmetric, irregularities in the tip structure are easily detected. Once a tip has been prepared yielding results similar to those shown in Fig. 6.6., it can be used as a calibrated tip for imaging unknown surfaces. However, in doing so, utmost care has to be taken not to alter the tip in transfer from one sample to the next. Furthermore, the distance dependent significant changes in detuning contrast (Fig. 17.7.) and their understanding by theory provide a unique possibility to calibrate the absolute value of tip-surface distance on the sub-nanometer scale.

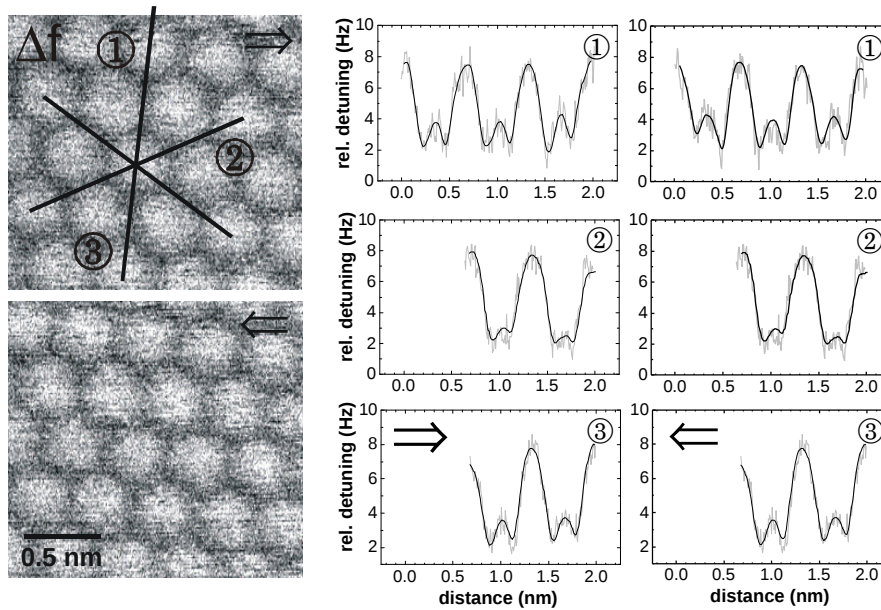


Fig. 6.6. Ionic corrugation on $\text{CaF}_2(111)$ obtained in the constant height mode. Images represent scanning results for forward and backward scanlines, respectively, while the graphs are cross-sections along equivalent surface directions as indicated in the images

6.4 Step edges

Step edges are well defined atomic scale structures and well suited to test atomic resolution force microscopy for its use in the investigation of insulating nanostructures. Steps form naturally on the surface of fluoride crystals upon cleavage and a large number of them have the smallest possible step height of one F-Me-F triple-layer (F: fluorine, Me: Ca, Sr or Ba). A step is a test for the stability of the Δf control loop in response to strong variations in the input signal but also for atomic tip stability during interaction with atomic scale irregularities and low coordinated ions at the step edge. It is still rather difficult to image a step edge with atomic resolution on both, the top and the bottom terrace and often this is achieved in the immediate vicinity of the step edge only as is the case in the image shown in Fig. 6.7. This can be due to the response of the feedback loop on shifting the balance between chemical and background forces when the tip apex crosses the step edge but may also be the result of an inclination between the plane of scanning and the surface plane or a combination of both. Except for this restriction, Fig. 6.7. is an example for stable and regular imaging of a step edge where individual ions and vacancies can be identified as a topographic contrast [13]. Due to rapid scanning, the noise level is high in this measurement and the atomic corrugation can only be estimated to be 30 pm. There appears no or only extremely small contrast in any of the other signal channels evidencing the proper action of amplitude and distance control loops. Consequently, topography is also well reproduced when crossing the step edge and the apparent step height of 340 pm is well in agreement with the expected value of 334 pm within the experimental error.

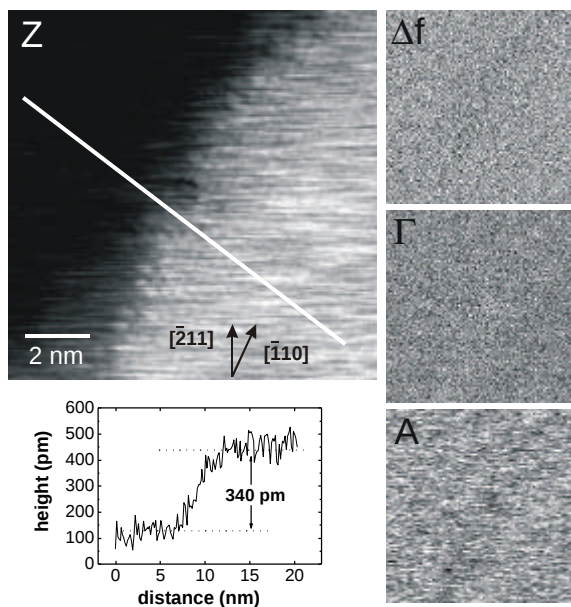


Fig. 6.7.

Step edge on SrF₂(111) measured in the topography mode. The large frame displays the topographic contrast while the other frames show simultaneously recorded data for detuning, damping and cantilever excitation amplitude. The graph is a cross-section taken along the line indicated in the topography image and allows a determination of the 340 pm step height. From Ref. [13]

A very different result is obtained in the measurement shown in Fig. 6.8. that was also recorded in the topography mode. In this case, Δf could also be kept constant within the noise level, however, there appears a strong contrast in the damping and amplitude signals. This is testament of extremely strong dissipative interactions on the atomic scale that cannot be compensated by the action of the amplitude control loop. Dissipative interaction appears to be specifically strong at step edges and there is a tendency for tip instability expressed in horizontal bands of altered signal contrast in topography, dissipation (see arrows) and amplitude but also single events of a local burst in contrast. These observations point to an atomic tip structure where an atom is weakly bound and, thus, strongly interacts with defects and low coordinated ions at step edges. Similar effects have been observed on other halide surfaces (see 5.3.2 and 5.5.2) and there, the apparent enhanced topographic contrast at edge ions was interpreted as a dynamic effect of transient displacement upon interaction of the step ion with the sensing tip atom (see 17.5). The mechanisms of energy dissipation by such processes have, however, not yet been explored in detail. We conclude from our measurements that the strong contrast in both, the topography and dissipation signals is closely related to tip instability in the form of periodic position flips of the weakly bound atom as proposed in Section 6.2. This view is based on the observation that in a multitude of measurements, we find a broad range of edge enhanced contrast features ranging from the extreme cases of no enhancement and extremely strong enhancement accompanied by instability represented by the examples discussed above.

The last example in this series displayed in Fig. 6.9. is intermediate in strength of edge enhancement but exceptional for its stability in imaging. When recording

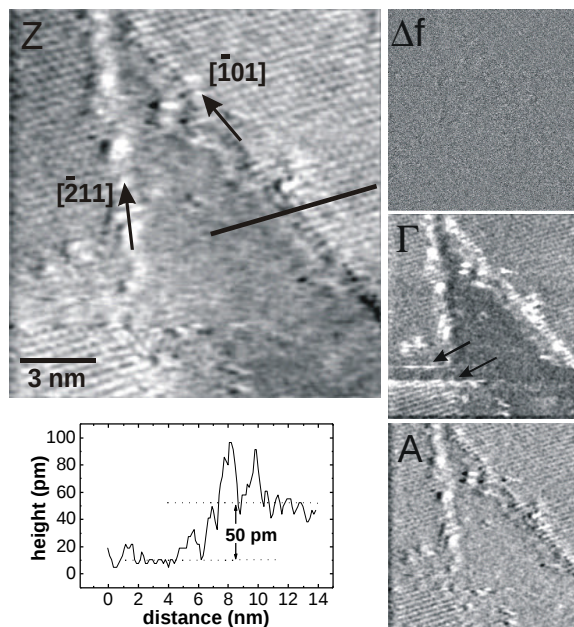


Fig. 6.8. Step edge on $\text{BaF}_2(111)$ scanned in topography mode. The images and the graph represent the same data as those from Fig. 6.7. Here, the enhanced contrast at step edges, stripes with changed contrast (see arrows in detuning image) and single bursts of contrast are due to tip instability. The expected step height of 358 pm cannot be reproduced since the width of the cleavage tip is smaller than the radius of the tip apex preventing the tip end from reaching the bottom plane

this series of images, the loop gain for distance control was reduced so that parallel to the noisy topographic image, clear images of detuning contrast could be recorded. Effectively, this was a scan under imaging conditions between topography and constant height modes that is not well suited for quantitative analysis but for a study of the relation between the atomic structure and an enhanced contrast. Edge ions are enhanced in contrast and appear like pearls on a chain except for a few irregularities. One of the irregularities is indicated in frame (c) by an arrow marking a position where apparently two ions are missing or for an unknown reason do not exhibit an enhanced contrast. The contrast drop marked by an arrow in frame (a), however, is one of several similar features found in this image and can well be explained in terms of coordination. In many cleavage tip studies we find that there is a preferential orientation of step edges along the $[\bar{2}11]$ direction but they are occasionally broken by small sections with a direction along $[\bar{1}10]$ and $[\bar{1}01]$. Edge ions in sections with the latter orientation have a higher coordination than those incorporated in $[\bar{2}11]$ edges. Hence, their interaction with the sensing tip atom is less pronounced and the respective contrast feature is missing at exactly the site of the ion with higher coordination.

In summary, we find that scanning across step edges on fluoride surfaces with atomic resolution appears to be possible but it is rather difficult to obtain atomic resolution simultaneously on both, upper and lower terrace. An enhanced contrast at defects and low coordinated ions at step edges is a phenomenon frequently observed and most probably related to tip instabilities and dissipative processes of weakly bound tip atoms. Hence, step edges and even more kinks are excellent objects for testing tip stability.

Acknowledgments The authors are grateful to T. Arai, R. Bennewitz, A. S. Foster, F. Giessibl, S. Gritschneider, F. Himpsel, L. Kantorovich, C. Loppacher, A. L. Shluger and L. Tröger for most stimulating discussions and support during the preparation of this contribution.

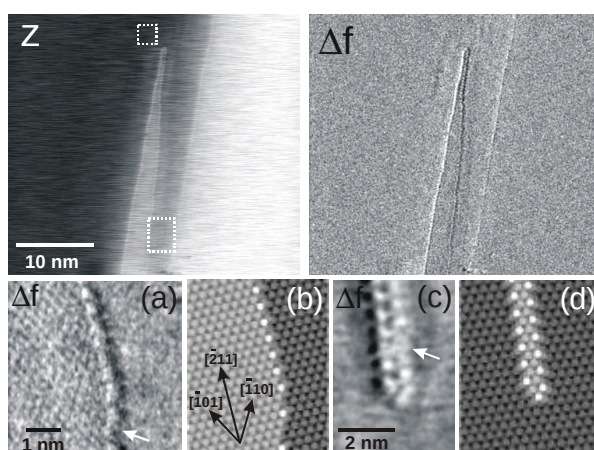


Fig. 6.9. Step edge on $\text{CaF}_2(111)$ obtained in topography mode at reduced loop gain for the Δf control loop. In this mode, topographic contrast and contrast in the detuning image can be observed simultaneously. The lower frames show details of the ionic structure of the cleavage tip as appearing in the detuning contrast and schematic representations.

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