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EVIDENCE FOR ATOMIC-SCALE RESOLUTION IN ATOMIC-FORCE MICROSCOPY OF LAYER SILICATES

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ABSTRACT

We have recorded atomic force microscopy (AFM) images of the surfaces of kaolinite and lizardite. Typically, the AFM images of the rings of basal-O atoms of these structures have two-fold rotational symmetry, and there is an apparent difference in height between adjacent tetrahedra. These are anomalous features not found in the known structures, and comparison with calculated images of graphite suggest that both features are due to the interaction of a double-atom AFM tip with the atoms of the surface of the sample. However, in some cases we have recorded the first images of the O-atoms in the basal-O plane of sheet silicates at atomic-scale resolution. Currently, there are two views of such AFM images: (1) they show arrays of individual atoms; (2) they show arrays of averaged atom positions. There are two interpretations for the second view. The first relates to the use of two-dimensional fast-Fourier transforms (2DFFT) to enhance detail in the image, the argument being that the use of 2DFFT introduces spurious features *and* averages the atomic positions. The other interpretation is that the periodic repeat-units of the structure being scanned set up harmonic vibrations in the cantilever and produce an image of an average structure. We have recorded a *single* image that shows two *different* atomic repeat-units in different parts of the raw image. Enhancement using 2DFFT significantly increases the resolution of each part of the image but maintains the two discrete images of the different structural elements; it does not average the separate parts of the image. In another image, we have recorded atoms that are displaced from the expected atomic position, a feature that would not be recorded if averaging took place. We conclude that these images represents individual atoms on the surface.

Keywords: lizardite, kaolinite, atomic-force microscopy, atomic-scale resolution, two-dimensional fast-Fourier transforms.

SOMMAIRE

Nous avons enregistré, par microscopie en force atomique (AFM), des images de la surface de la kaolinite et de la lizardite. Il est typique pour ces images AFM des atomes d'oxygène des anneaux à la base du feuillet de montrer une symétrie bilatérale, et il semble y avoir une différence dans l'élévation des tétraèdres adjacents. Ce sont des caractéristiques anomales, non documentées dans les structures connues. Une comparaison avec les images calculées de la graphite fait penser que dans les deux cas, il s'agit de résultats dus à l'interaction d'un stylus AFM à atomes doubles avec les atomes de la surface de l'échantillon. Dans certains cas, nous avons enregistré les premières images d'atomes d'oxygène du plan de base des phyllosilicates à résolution atomique. Il y a deux façons d'interpréter de telles images: (1) elles montrent bien des agencements d'atomes individuels; (2) elles montrent la moyenne des agencements de positions d'atomes. Il y a deux interprétations de ce deuxième point de vue. La première implique l'utilisation de transformations rapides de Fourier en deux dimensions afin d'améliorer les détails de l'image, et veut qu'un tel logiciel introduise artificiellement de telles anomalies *et* montre la moyenne des positions atomiques. L'autre interprétation veut que la périodicité de la structure induise pendant le balayage des vibrations harmoniques dans le cantilever et

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produise une image moyenne de la structure. Nous avons enregistré dans une seule image deux périodicités distinctes dans différentes portions de l'image non traitée. L'utilisation du logiciel 2DFFT améliore de façon importante la résolution de chaque partie de l'image, mais sans voiler les images des domaines séparés, aux deux éléments structuraux distincts. De plus, ce logiciel ne produit pas un agencement moyen représentatif des deux parties de l'image. Dans une autre image, nous avons enregistré des atomes qui sont déplacés par rapport aux positions idéales, ce qui ne pourrait résulter du logiciel si celui-ci faisait la moyenne des positions. Nous pensons que ces images représentent vraiment des atomes individuels à la surface.

(Traduit par la Rédaction)

Mots-clés: lizardite, kaolinite, microscopie en force atomique, résolution d'atomes, transformations rapides de Fourier en deux dimensions.

INTRODUCTION

There is current controversy as to whether "atomic scale" images produced with the atomic force microscope (AFM) are (1) arrays of individual atoms recorded at atomic-scale resolution or (2) arrays of averaged positions of atoms. Unfortunately, much of the criticism we encountered against atomic-scale resolution occurred in referee's reports of manuscripts submitted to journals. The interpretation that the images represent average atomic positions is made particularly where two-dimensional fast-Fourier transforms (2DFFT) have been applied to remove spurious noise in an image. At other times, reviewers suggested that the periodicity of a structure may set up a harmonic response in the cantilever, so that the gross structure is reproduced, whereas individual atoms are not. It is important to resolve this problem. In the first case, we can discuss the behavior of individual atoms making up, or attached to, a mineral structure; in the second case, we can only discuss the behavior of an average structure. Obviously, the goal of high-resolution AFM studies is to illustrate individual atoms, as this is an objective that cannot otherwise be attained in many cases. In the course of our work on layer silicates, we have recorded images that, in our opinion, show atomic-scale resolution. We are currently doing more extensive modeling and simulation studies to further understand this phenomenon, but we present our atomic-scale images here to contribute further to this debate.

PREVIOUS WORK

Atomic resolution of individual O and Ca atoms has been recorded in water by Ohnesorge & Binnig (1993) on the $(10\bar{1}4)$ cleavage plane of calcite with separations of 4 to 10 Å between the calcite surface and the AFM tip. At these distances, physical tip–surface interactions are eliminated, and the tip senses long-range forces of the surface atoms. That study first illustrated the importance of non-contact AFM, in contrast to the usual contact AFM, and has led to the rapid development of the non-contact technique (Morita & Tsukada 1999). We have done numerical simulations of non-contact AFM (Sokolov *et al.* 1997a, 1999a), and developed a pseudo-non-contact technique using a surface surfactant (Sokolov *et al.* 1997b) to obtain atomic-scale resolution (Sokolov *et al.* 1999b).

Single-atom defects in the silicon $\{111\}$ 7×7 surface have been recorded by AFM in ultra-high vacuum by Giessibl (1995) and Uchihashi *et al.* (1997). Previously, the absence of point defects in AFM images has been cited as evidence of a lack of atomic resolution. It is of critical importance to realize (Sokolov *et al.* 1999b), however, that for many materials there are no vacancies to record when AFM is operated in air or water. Under these conditions, individual point defects, particularly in silicate surfaces, will be filled by a H_2O molecule or a OH group (Wicks *et al.* 1993) and thus are not there to be imaged. In this situation, the lack of point defects is a function of the operating conditions (non-vacuum), not the resolution of the instrument.

These non-contact and ultra-high-vacuum studies demonstrate atomic-scale resolution, but our objective here is to discuss atomic-scale resolution obtained in our studies in which the tip was in contact with, or interacting with, the surface (contact AFM). The contact AFM study of calcite by Rachlin *et al.* (1992) was confirmed by the non-contact AFM atomic resolution images of Ohnesorge & Binnig (1993). Both studies show that the O atoms projecting out of the cleavage plane are preferentially imaged with respect to the Ca atoms. Wicks *et al.* (1992) and Vrdoljak *et al.* (1994) obtained atomic-scale-resolution images of the OH groups and Mg atoms that make up the sheet of octahedra in lizardite and the interlayer sheet of octahedra in clinochlore. Wicks *et al.* (1993) obtained atomic-scale images of the internal (O, OH) plane that is common to the sheets of tetrahedra and octahedra in lizardite by using a high tracking-force (325 nN) on the cantilever to remove the basal O and Si atoms of the sheet of tetrahedra. Similar results have been obtained by Eby *et al.* (1993) for muscovite and by Vrdoljak *et al.* (1994) for clinochlore. Although obtaining atomic-scale resolution of OH groups was relatively straightforward, we did not obtain atomic-scale resolution in scans of the hexagonal rings of basal O-atoms forming the $\{001\}$ surface of the sheets of tetrahedra. However, the hexagonal rings were more clearly defined than in earlier studies (Drake *et al.* 1989, Hartman *et al.* 1990), in which each hexagonal ring appears as a single light spot (Hartman *et al.* 1990, Figs. 2 and 3).

During our experiments using AFM to remove surface atoms and to reveal the internal structure, we recorded atomic-scale-resolution images of the basal O-atoms of the sheet of tetrahedra of lizardite. These images of basal O-atoms, and the possible importance of ambient H_2O in the mechanics of tip–surface interaction, are discussed in Wicks *et al.* (1993). In this paper, we use some of these earlier images to emphasize important evidence that we believe demonstrates atomic-scale resolution in contact AFM.

Experimental

The lizardite crystal (ROM #M45037) used in this study has been described by Wicks *et al.* (1992), and was used in the AFM study of internal structure by Wicks *et al.* (1993). The kaolinite sample consisted of fine euhedral crystals up to 200 nm on edge, used as a coating on a paper product. AFM scans were done in air on the {001} surface of kaolinite on the surface of the sheets of paper using a Nanoscope II Atomic-Force Microscope produced by Digital Instruments, Inc. A number of image-processing software routines are provided by Digital Instruments, including low-pass filtering to remove low-frequency noise, and 2DFFT to more selectively remove spurious signals. The effect of 2DFFT, low-pass filtering and other image filtering software on raw images has been discussed, with examples, by Wicks *et al.* (1994, Figs. 1, 2, 3 and 4).

Results

We have recorded images of what seem to be basal-O atoms (Figs. 1, 2 and 3). The image of kaolinite in Figure 1 is typical of the results obtained from the basal-O atoms of layer silicates using a contact force of 100 nN. The images of lizardite in Figures 2 and 3 were recorded using a higher tracking force of 325 nN to re-

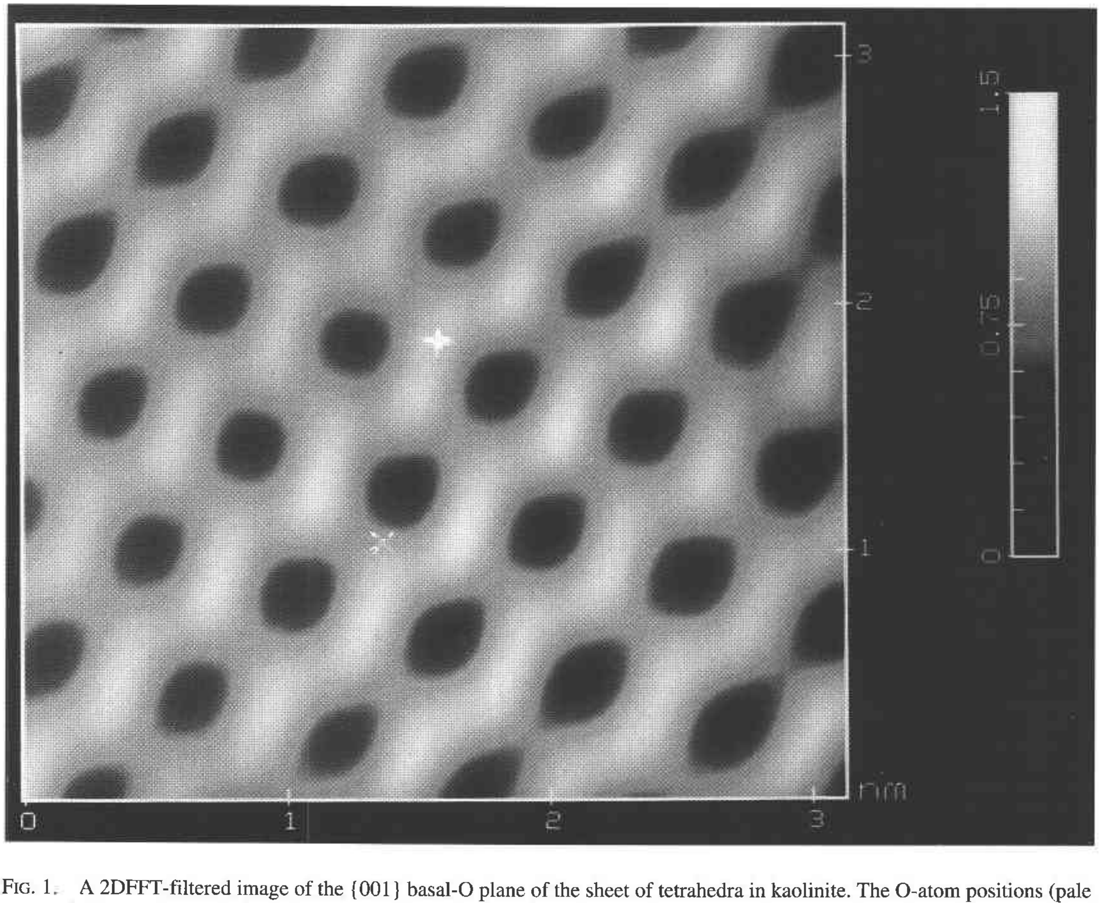

Fig. 1. A 2DFFT-filtered image of the {001} basal-O plane of the sheet of tetrahedra in kaolinite. The O-atom positions (pale grey) show an apparent 2-fold symmetry produced by tip–sample interaction, rather than the expected ditrigonal symmetry. All divisions are in nanometers in all figures.

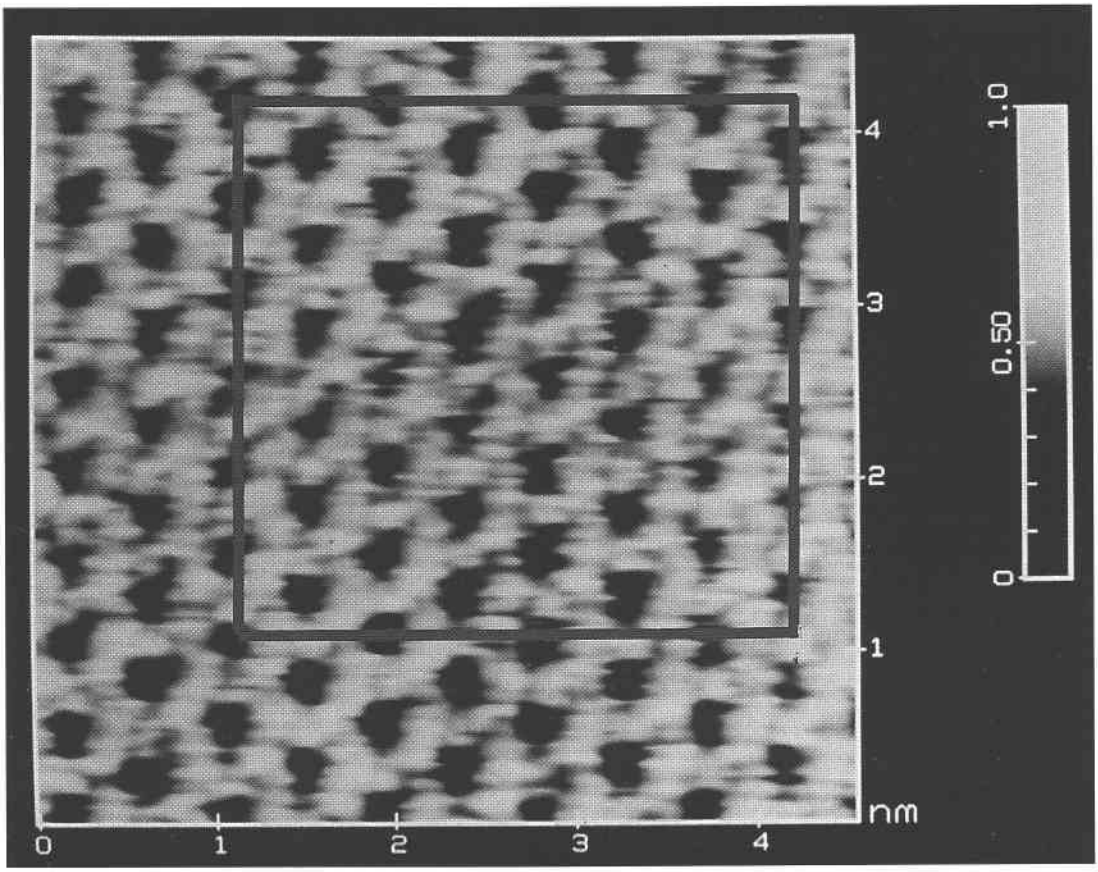

FIG. 2. An unfiltered image of the {001} basal-O plane of the sheet of tetrahedra in lizardite. The image is particularly noisy, as it was collected during the removal of the basal-O and Si atoms by high-force scanning. The area outlined is enlarged in Figure 3 (Fig. 5a, Wicks *et al.* 1994).

move basal-O and Si atoms of the sheet of tetrahedra from the surface of lizardite, and to reveal O, OH and Mg atoms in the interior of the structure. However, before the O and Si atoms were completely removed, the image in Figure 2 was recorded. This image lasted only for a few minutes before it deteriorated as the basal-O and Si atoms were swept away. The initial image (Fig. 2) is particularly noisy, as it was recorded under nonideal conditions (*i.e.,* while the surface, and presumably the tip, were being deliberately disrupted). Under these extreme conditions, there is little choice but to use 2DFFT to produce a cleaner image that contains considerable detail (Fig. 3).

DISCUSSION

Although the basal rings of oxygen atoms are commonly referred to as "hexagonal", detailed studies of lizardite (Mellini & Zanazzi 1987), clinochlore (Rule & Bailey 1987), muscovite (Bailey 1984) and kaolinite (Giese 1988) show they have a ditrigonal aspect due to slight rotation of the tetrahedra (Bailey 1988). Molecular-orbital calculations suggest that this is the preferred configuration for layer silicates (Peterson *et al.* 1979, Wicks & O'Hanley 1988). However, AFM images of the basal-O plane rarely display either a ditrigonal or even a hexagonal aspect. Our images of the basal-O atoms in kaolinite (Fig. 1) and lizardite (Wicks *et al.* 1992, Fig. 5) display 2-fold, as opposed to 3- or 6-fold, rotational symmetry. In addition, there is an apparent difference in height between alternate tetrahedra in AFM images, every other tetrahedron appearing displaced downward relative to its neighboring tetrahedra.

Comparison with calculated images of graphite

Gould *et al.* (1989) have modeled the atom–atom interactions between graphite and a scanning silicon

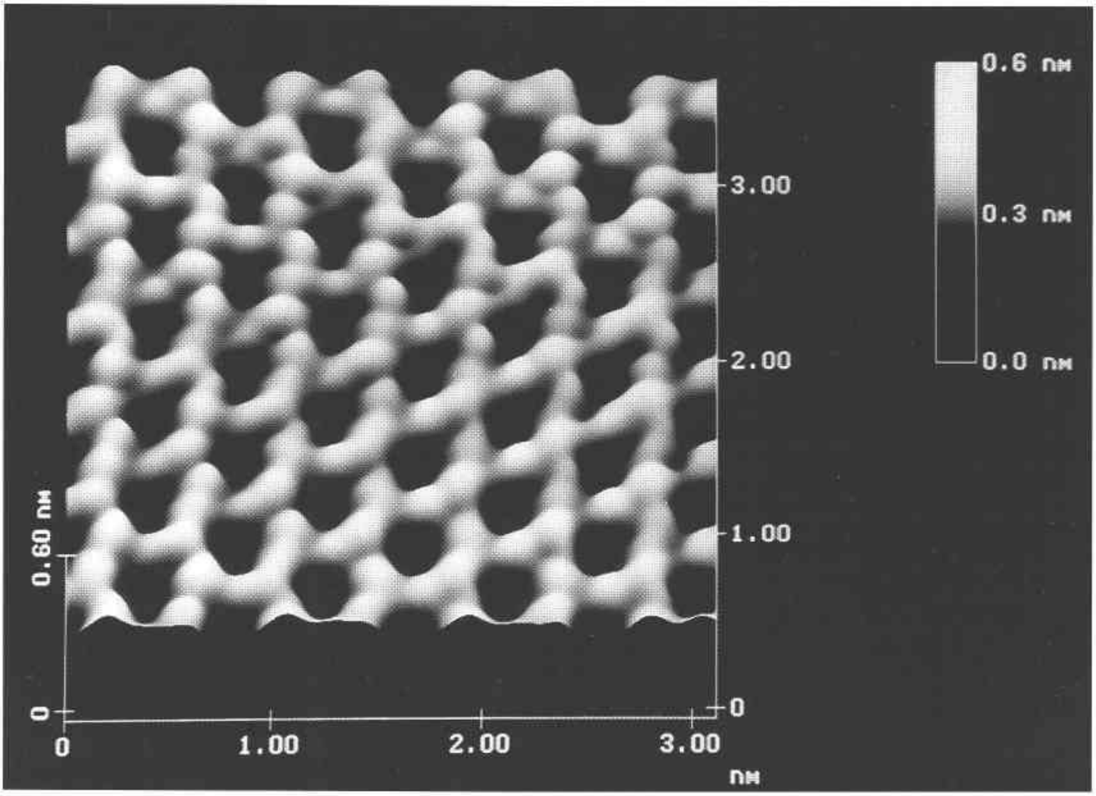

FIG. 3. An oblique view of the image in Figure 2 after 2DFFT filtering. The spherical shapes represent oxygen atoms, and show the distortion produced by the high tracking-force used. If this were an image of an average structure, the atoms in anomalous positions would not be recorded. A few minutes later, the basal-O and Si atoms were scraped away (Fig. 5c, Wicks *et al.* 1994).

nitride tip, and their results provide a basis for interpreting our results. Graphite has a ditrigonal ring-geometry similar to that of the layer silicates. Gould *et al.* (1989) have shown that anomalous images of the {001} surface of graphite can be reproduced from theoretical calculations involving a double-atom tip. By changing the relationship of the double-atom tip to the surface atoms, they produced calculated images that match the published images of graphite (Gould *et al.* 1989, Fig. 4). Their calculations using a single-atom tip produced an image similar to the known structure of graphite and virtually free of anomalous features (Gould *et al.* 1989, Fig. 3).

Comparison of our images, the published images of the basal-O layer in sheet silicates, and the double-atom-tip images of graphite calculated by Gould *et al.* (1989) shows that the observed anomalies in images of layer silicates are similar to the calculated anomalies in double-atom-tip images of graphite. This similarity suggests that anomalous features in AFM images of layer silicates, such as the apparent two-fold symmetry and the apparent differences in height between adjacent tetrahedra (Fig. 1), are the result of the interaction of double-atom tips with surface-O atoms.

Possible artifacts in AFM images

The Fourier transforms used for image enhancement are generated from the structural geometry of the image and are applied back to the image to eliminate extraneous signals so that structural detail is enhanced. Criticism of the use of 2DFFT for AFM image-enhancement is based on two arguments: (1) features that are not present in the initial image are introduced, or features that are present are removed, by the use of the 2DFFT; (2) use of 2DFFT averages the atomic positions so that resolution of individual atoms is not obtained. An alternative explanation for (2) is that a harmonic vibration, related to the structural periodicity, is set up in the cantilever as the tip passes over the atoms of the surface, producing an image of an average structure (the

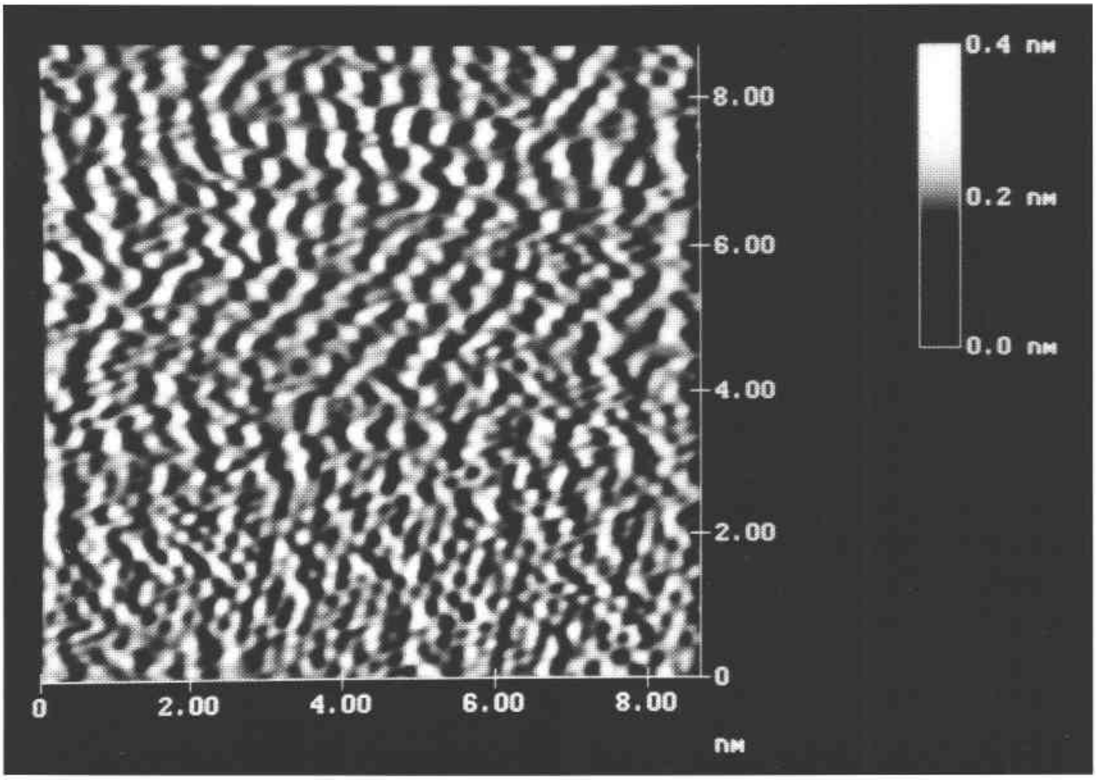

FIG. 4. A 2DFFT-filtered image showing the basal-O atoms of the sheet of tetrahedra in the upper part of the image, and the underlying (O, OH) plane of the sheet of octahedra revealed by the removal of the sheet of tetrahedra in the lower part. The rings of basal-O atoms (upper two thirds) have a 5 Å spacing, and the triangularly arranged OH anions (lower third) have a 3 Å spacing. No averaging (or melding) of the two structural regions has been produced by the 2DFFT filtering (Fig. 5, Wicks *et al.* 1993).

analogue of the vibration of a wheel moving along a brick road is used).

In the first case, it is possible to significantly alter the content of an image by eliminating parts of the Fourier transform (see Fig. 4, Wicks *et al.* 1994); this process can thus significantly change the filtered image. However, this is not intrinsic to the use of 2DFFT, and whether changes are introduced into the image becomes a question of the intent or competence of the individual scientist. Correctly used, the implementation of 2DFFT excludes high-frequency noise that is close to the pixel frequency of the image. The frequency of such noise is some orders of magnitude away from the frequency of the atomic-repeat distances of the mineral surface, and if all the structure information is used in the 2DFFT operation, only spurious noise is removed; atomic details of the structure of the surface remain. Careful comparison of Figures 2 and 3 shows that no structural details are added or subtracted by the application of 2DFFT; only spurious signals are removed.

According to the second line of criticism, an average structure is produced by 2DFFT rather than an image of individual atoms. This is refuted by two images that we have recorded. In Figure 4, two different atomic-repeat units have been recorded on a single image. The image shows both the basal-O surface of the sheet of tetrahedra with a 5 Å repeat in the upper half of the image, and the (O, OH) plane of the underlying sheet of octahedra with a 3 Å repeat in the lower half of the image (where the sheet of tetrahedra has been physically removed by the tip). The use of 2DFFT to enhance this single raw image does not average but maintains two discrete structural elements with 5 Å and 3 Å spacings, respectively (Fig. 4). Thus the criticism that 2DFFT necessarily averages atomic positions is not valid.

Further evidence against the criticism that 2DFFT, or harmonic vibration of the cantilever, must average the atomic position is shown in Figure 3. Distinct spheres are visible, and these spheres coincide with positions of the basal-O atoms in the sheet of tetrahedra of lizardite. What is more important in this image is the displacement of atoms away from their ideal positions, particularly toward the upper right corner. These well-resolved atoms in non-structural positions indicate that 2DFFT or harmonic vibration has not produced an image of the average structure. If averaging had occurred, by either method, these atoms would have been shifted back to their ideal structural positions. In fact, this is atomic-scale resolution of atoms that are in the process of being detached by the tip as a result of the high tracking-force used. It follows that at this high tracking-force the tip also must be disrupted. Why atomic-scale resolution of the basal-O surface is obtained on a disrupted surface with a disrupted tip, and not on a normal surface, is not known, but suggests that resolution can be enhanced during the dynamic process of detachment.

Identification of surface atoms

It is not possible to identify directly the atoms in an AFM image. However, if atoms coincide with known structural positions, their identity can be assumed with some degree of confidence. Lindgreen *et al.* (1991) could not determine if they had recorded images of the basal-O atoms of interlayered illite and smectite or of H_2O molecules adsorbed on the basal-O plane. In our case, the use of high-force scanning strong enough to break Si–O–Mg bonds must have removed any adsorbed H_2O.

Wicks *et al.* (1993) postulated that the empty hexagonal ring is a key factor in the removal of the basal-O and Si atoms during high-force scanning because it allows ambient H_2O groups access to the (O,OH) plane. The dissociation of H_2O to H^+ and OH^- ions during high-force scanning provides the ions necessary to approximately satisfy local bond-valence requirements as Si–O–Mg bonds are broken and basal-O and Si atoms are removed. Thus the (O,OH) plane revealed in Figure 4 will actually consist only of OH groups. One third of these will be the original OH groups and two thirds will be new OH groups produced by hydroxylation of O atoms during high-force scanning. A similar mechanism could operate to fill a defect on a silicate surface where a missing O would be replaced by an ambient H_2O molecule, or an OH group if dissociation occurred. Thus an AFM scan in air or water of such a feature would not record a defect.

CONCLUSIONS

(1) Images of the {001} surface of sheet silicates recorded by AFM in this study represent individual atoms recorded at atomic-scale resolution and not arrays of averaged atoms.

(2) Comparison of the images of the {001} surface of lizardite and kaolinite with calculated images of graphite (Gould *et al.* 1989) suggest that some differences between the surface images and the bulk structures are due to multiple-atom-tip interactions with the surface atoms.

(3) Use of the 2DFFT step of image enhancement does not average the atoms represented in the image of the surface. This conclusion is supported by the occurrence of a high-force scanning image of lizardite that shows two different atomic-repeat units in different parts of the raw and enhanced images.

(4) Harmonic vibration of the cantilever related to the periodicity of the structure being scanned does not occur. This conclusion is supported by the occurrence of a high-force scanning image of lizardite that shows atoms that have been significantly and randomly displaced, and thus not averaged, from their normal structural positions.

(5) Point defects will not be observed by AFM operated in air or water, but can be observed by AFM operated in ultra-high vacuum.

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