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Autor: Nyfeler, Daniel / Berger, Rüdiger / Gerber, Christoph
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Scanning force microscopy on albite cleavage surfaces

by Daniel Nyfeler¹, Rüdiger Berger^{2,3} and Christoph Gerber²

Abstract

Surfaces of low-albite crystals, a member of the natural feldspar group, were studied by scanning force microscopy immediately after cleavage. Data presented exhibit typical steps corresponding to the atomic structure of albite. After exposure of the albite surface to ambient air we observed a time-dependent change in surface morphology emanating from the step edges. To explain such phenomena we propose an atomic model.

Keywords: Scanning force microscopy, feldspar, albite, adsorption, atomic model, hydrous phase.

Introduction

Experimental and theoretical work in mineralogy focuses on the dissolution behaviour of minerals, especially of feldspars (BERNER and HOLDREN, 1977; NESBITT and MUIR, 1988). In particular there is considerable research activity to determine dissolution reactions and rates (see WHITE [1995] for a recent overview). Scientists are interested in estimating the role of surface contamination, surface roughness, specific surface area, or non-stoichiometric behaviour due to crystallographic orientation or nanotextures. Scanning probe microscopy (SPM) techniques offer an ideal method to characterize a wide range of surface properties with unsurpassed resolution in real space. As probe mechanisms, a variety of physical or chemical signal domains can be used. The high spatial resolution of these techniques lead to a better understanding of the morphology of surfaces on an atomic scale (STM: BINNIG et al., 1986; SFM: BINNIG et al., 1982; SNOM: POHL et al., 1984, HEINZELMANN and POHL, 1994; etc.). In particular scanning force microscopy (SFM, see SARID [1994] for an introduction), with a spatial resolution down to atomic level, provides a direct method for real space analysis of surfaces of geological and mineralogical interest (BINDER et al., 1996; CHIARELLO and STURCHIO, 1995; GUT-

MANNSBAUER and HAENNI, 1994; HARTMAN et al., 1990).

SFM was used to image magnetic properties of single-domain particles in bacteria in calcareous sediments (FRITZ et al., 1994). Growth and dissolution on calcite cleavage planes in liquids were also studied (GRATZ et al., 1993; HILLNER et al., 1992). A recent article by STIPP et al. (1996) reported surface changes on terraces and on cleavage steps of natural calcite crystals. They interpreted this as dissolution and reprecipitation within an invisible layer of water that is adsorbed from air following cleavage. Reactivity sites, atomic structure, and morphology of albite surfaces were investigated by SFM in the contact mode (DRAKE and HELLMANN, 1991; HOCELLA et al., 1990).

In this article we present an analysis of albite surfaces by means of SFM directly after cleavage. Moreover, time-resolved data show that these surfaces interact with molecules from ambient air.

SFM Technique

In principle the SFM scans a sharp tip with apex radius smaller than 10 nm over a sample surface. The tip is attached to a cantilever, which acts as a spring. Minute deflections of the spring can be

¹ Laboratory for Chemical and Mineralogical Crystallography, University of Bern, Freiestrasse 3, CH-3012 Bern, Switzerland.

² IBM Research Division, Zürich Research Laboratory, Säumerstrasse 4, CH-8803 Rüschlikon, Switzerland.

³ Also at: University of Basel, Institute of Condensed Matter, CH-4056 Basel, Switzerland.

measured within femtometer accuracy by various methods described elsewhere (SARID, 1994). The corresponding forces in the realm of 10^{-9} N allow a wide range of materials – even soft ones – to be probed nondestructively. A feedback loop keeps the net forces between the tip and the sample constant by controlling their distance with piezoelectric elements. Sample surfaces can be laterally scanned from a few angstroms to a micron level. For a detailed introduction to SFM techniques we refer to the literature (MEYER, 1992; SARID, 1994; WICKRAMASHINGHE, 1989; WIESENDANGER and GUENTHERODT, 1992 a, b, 1993).

Experimental

We chose low-albite samples because of their geological importance and abundance. They were obtained from Amelia, Virginia and provided by the Naturhistorisches Museum Bern. The end-member albites (HELLMANN et al., 1990; HOCHELLA et al., 1990) are tightly intergrown and twinned, which was observed under an optical microscope. Figure 1 shows a structural ball-and-stick model of albite crystals. The structure consists of a framework of (Si/Al) O_4 tetrahedra, with Na occupying the gaps.

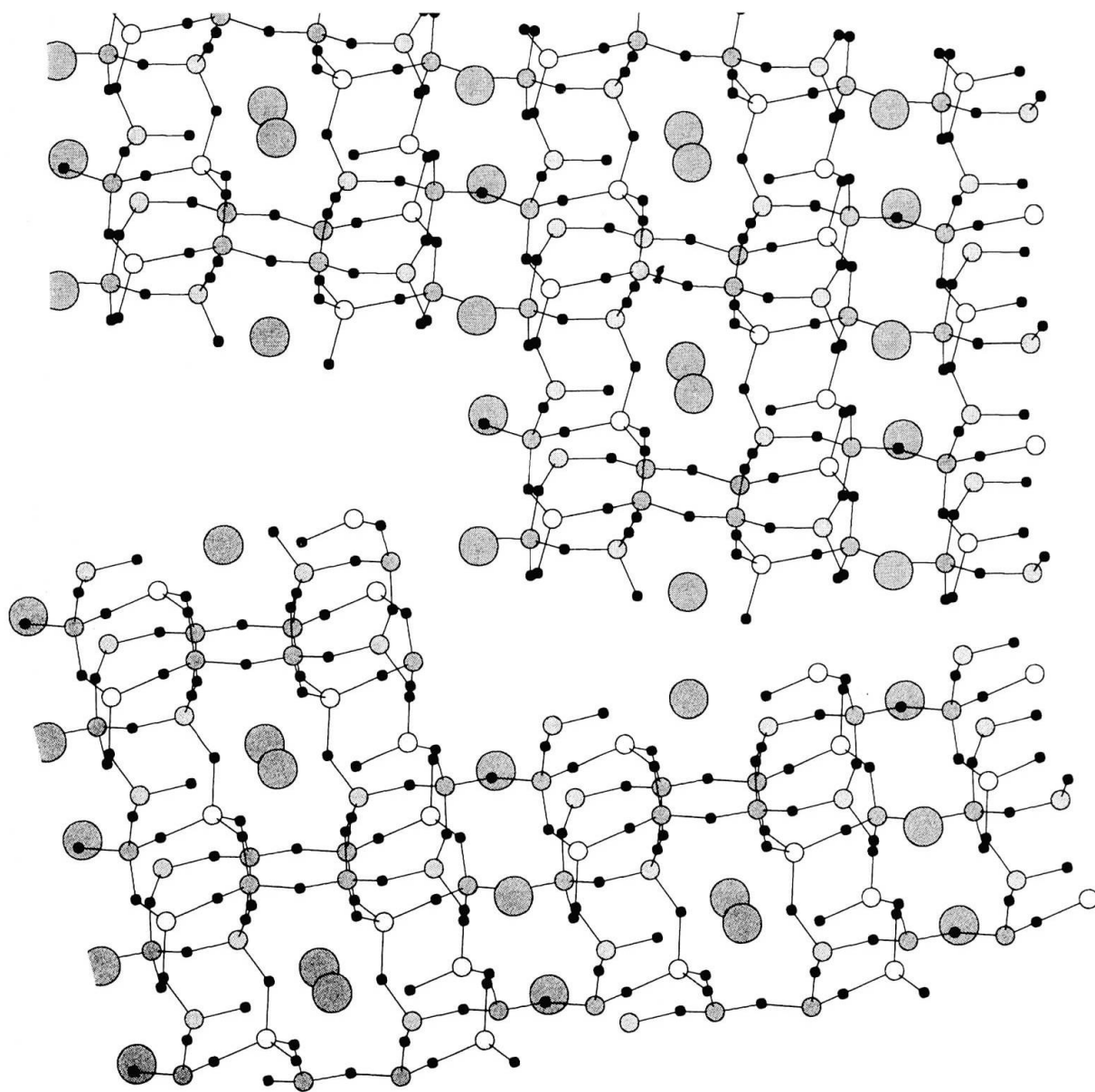


Fig. 1 Ball-and-stick model of the albite structure. Small black spheres represent O, intermediate gray spheres are Si and Al (tetrahedrally coordinated) and the large, isolated spheres represent Na. The Si/Al tetrahedra are corner-linked and form a framework with Na in gaps. Only the bonds between O and Si/Al are shown. Cracking the crystal produces surfaces (including a step dislocation) with different atomic arrangements for the upper and the lower cleavage surface.

In order to identify fresh surfaces of albites, all faces already exposed to air were inked. Afterwards we cleaved the crystals either by hitting them with a hammer or by cracking them with tongs. Then on a chosen crystal a fresh, unstained face was selected, and a pure nitrogen stream was used to remove fine dust particles. SFM was conducted in the tapping mode where the micromechanical silicon cantilevers used had a spring constant of 50 Nm^{-1} (Nanoscope III, Digital Instruments Inc.). Our experiments were performed in air and in an inert argon gas atmosphere at room temperature and pressure. Typically, the first SFM topographs were obtained 5 min after cleaving the feldspar, and data acquisition took place at a rate of 3 min per image.

Results and discussion

The micrograph in figure 2a shows the typical macroscopically flat surface which was obtained for freshly cleaved albites. The entire scale from black to white spreads over 5 nm. The surface offers clearly resolved steps in the SFM topograph. The subparallel steps running $\approx \text{N-S}$ are related to a crystallographic orientation. A corresponding cross-section outlined as a dashed line in figure 2b shows a profile of the topography. The corrugation obtained on a terrace stayed within 0.44 \AA rms. This step height measurement allows the determination of the Miller indices of the probed crystal face, which is especially useful for twinned and/or intergrown crystals. HARLOW and BROWN (1980) determined the structure of low albite from the same locality with three-dimensional neutron and X-ray diffractometry. The structure refinement in space group $\text{C}\bar{1}$ yielded the following cell dimensions: $a = 8.142(2) \text{ \AA}$, $b = 12.785(2) \text{ \AA}$, $c = 7.159(2) \text{ \AA}$, $\alpha = 94.19(2)^\circ$, $\beta = 116.61(2)^\circ$, $\gamma = 87.68(2)^\circ$. The topograph shown in figure 2 yields an average step height of 7.2 \AA , which agrees well with the theoretical value for the c dimension for one unit cell of Amelia albite. We therefore conclude that a (001) face is present for this surface¹. This is an useful method for real-space analysis of crystalline surfaces in addition to other indirect techniques (e.g. XPS, LEED, RHEED). Unlike DRAKE and HELLMANN (1991) and HOCELLA (1990) we did not observe (010) surfaces in our cleavage flakes.

¹ An additional method to confirm crystallographic orientation is Laue backscattering: after SFM experiments, the crystals were mounted on a Laue camera, and a backscattering image was generated and compared with computer-generated (001) and (010) patterns of the albite structure (CHRISTIANSEN, 1985).

However, due to the complexly intergrown and twinned nature of the crystals, each sample requires determination of the surface hkl indices.

In comparison to recent investigations (STIPP et al., 1996) we performed time-resolved measurements of this surface in order to observe changes of a freshly broken cleavage plane due to exposure to ambient air. The micrographs shown in figures 3 a–c represent a selection of typical topographic data obtained within 1 h. Each image was reproduced in both fast scan directions (trace and retrace). As in figure 2, we observe a high density of steps on a macroscopically flat surface. The

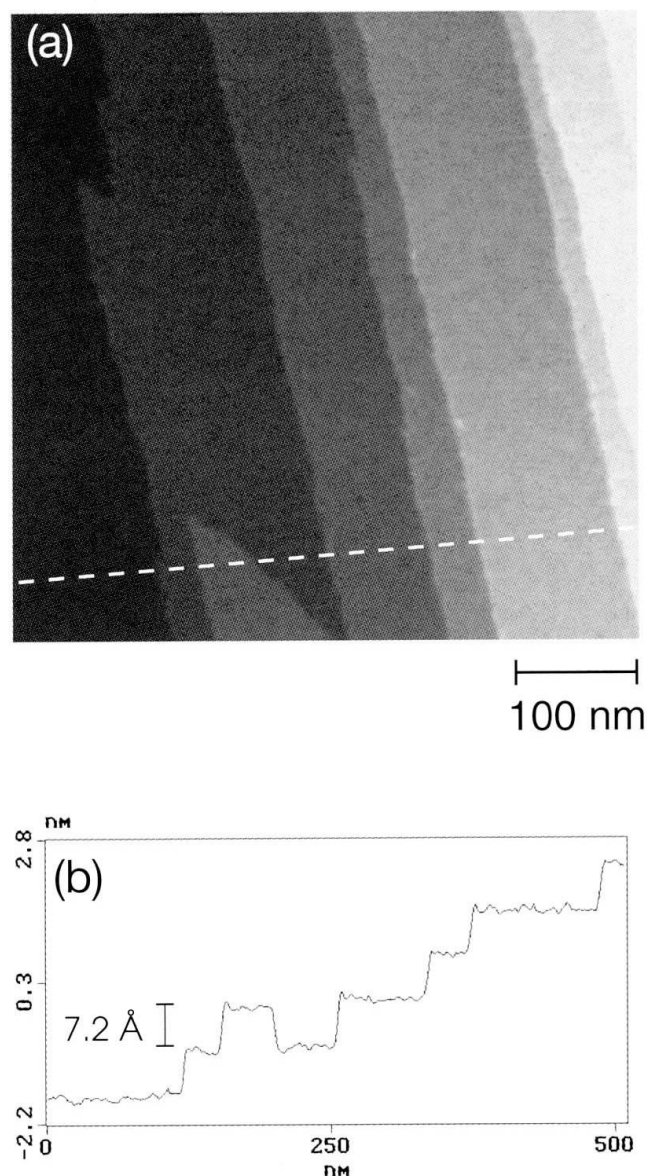


Fig. 2 (a) SFM topograph of a cleaving surface of albite. The image brightness is related to surface height. Subparallel steps run $\approx \text{TN-S}$. A cross section along the dashed line is shown in (b). The measured average step height of 7.2 \AA agrees well with the theoretical value for one unit cell in the (001) direction ($7.159(2) \text{ \AA}$).

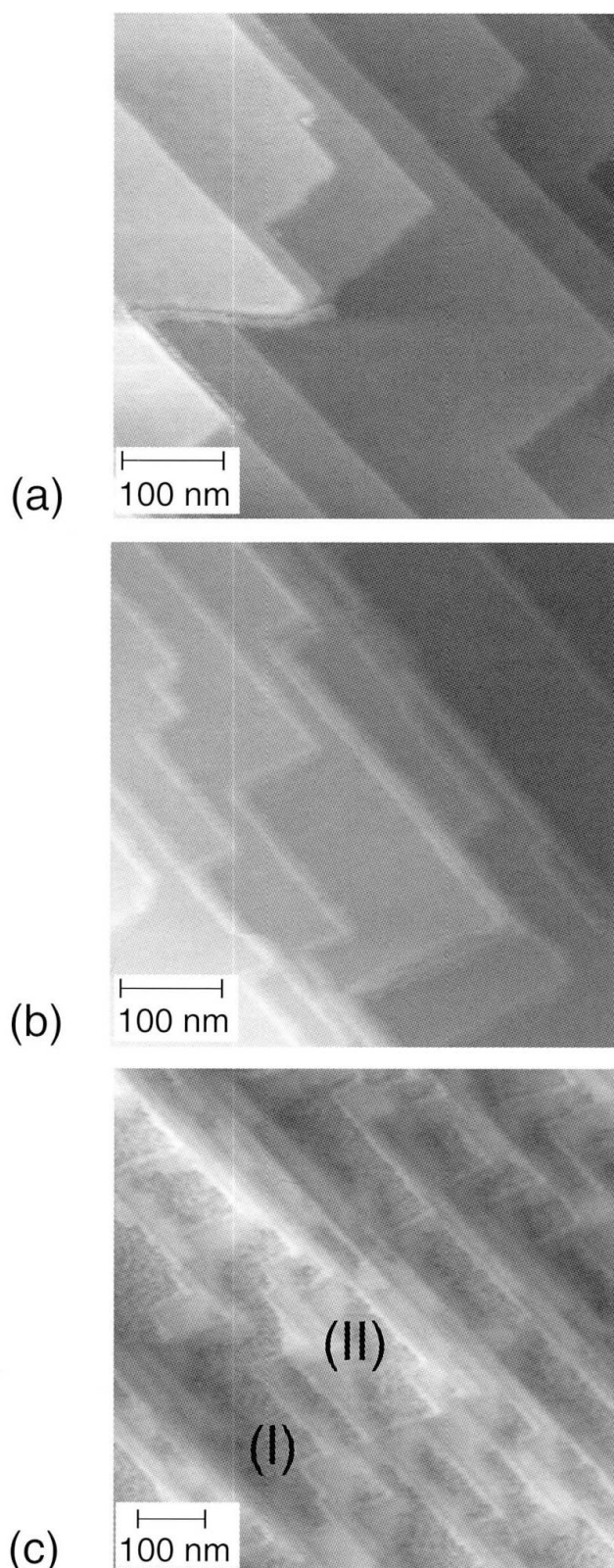


Fig. 3 Time resolved SFM micrographs of a freshly cleaved albite surface exposed to the ambient. The z scale as the gray scale of all three images is 15 nm. (a) Recorded immediately after cleavage. The adsorption of an additional phase started at the step edges. Micrograph (b) presents the same surface area 20 min and (c) 53 min after cleavage. The phase spread laterally over the terraces.

subrectangular terraces in figure 3a are bordered by discrete and well-defined steps. In micrograph 3b, obtained 20 min later, the sharp steps are covered with a diffuse phase. In micrograph 3c, recorded an additional 33 min later, this covering reached an advanced stage. The adsorbing phase starts to cover the terraces, in one case as patches (I), in the other it spreads continuously from step edges (II) as labeled in figure 3c.

In order to gain insight in the growth mechanism of this adsorption we performed a statistical measurement of the average height (henceforth referred to as \bar{h}) and the average width (\bar{w}) of the phase. A closer look at figure 3a reveals that the adsorbate was already present at the steps, where the average dimensions were measured to be $\bar{h}_1 = 0.2$ nm and $\bar{w}_1 = 8$ nm. In the next step (Fig. 3b) the adsorbed phase increased to values of $\bar{h}_2 = 0.5$ nm and $\bar{w}_2 = 12$ nm. In figure 3c, the phase spread out into the flat terraces, therefore \bar{w}_3 was no longer uniform in lateral direction, whereas \bar{h}_3 stayed constant with $= 0.5$ nm. This observed decoration of the surface with progressing time may be interpreted as the formation of a hydrous phase from ambient air. The preferential adsorption is explained by the following model in which the mechanical fracture of a solid involves the breaking of chemical bonds. A cleaved surface is covered with such dangling bonds (outlined in Fig. 1), thereby exposing higher excess surface energies (VAUGHAN and PATTRICK, 1995). Both cleavage surfaces expose such dangling bonds, with a higher density on step edges. These energies decrease as the dangling bonds become saturated, i.e. as they adsorb molecules and / or ions from the environment. As H_2O and hydrocarbons are the most polar and reactive constituents of ambient air, it might be that the phase decorating the surface is a hydrous-hydrocarbon mixture. HU et al. (1995) reported an adsorbed water phase on mica with a height of 0.2 nm, corresponding to \bar{h}_1 of our first step. Ellipsometry measurements carried out on clean gold surfaces confirmed the time-dependent adsorption process in our typical laboratory environment. Furthermore, the measured thickness of 0.5 nm after exposure of the sample to ambient air for 1 h corresponds to exactly $\bar{h}_2 = \bar{h}_3$ obtained by SFM. VAN CAPPELLEN et al. (1993) proposed a model of how adsorption takes place at an atomic scale. The hydrous phase sorbs primarily on edges and corners due to the higher local surface potential, according to our observations. This is confirmed by both experimental and theoretical data for calcium and magnesium oxide (DE LEEUW et al., 1995). They showed that the low-coordinate sites at edges represent the most energetically favorable sites for chemisorbed water.

Conclusions

In this paper we present real-space analysis of natural minerals by means of SFM. Topography and roughness of feldspar cleavage surfaces could be routinely probed. The high spatial resolution and accuracy of this technique allow the determination of Miller indices by measuring step heights. For mineralogists SFM is therefore a useful tool in addition to other indirect methods to obtain local information on mineral surfaces. The technique allows time-resolved measurements of the covering of a freshly cleaved feldspar crystals. The method provides detailed insight into the mechanism of adsorption and the generation of surface phases due to exposure to a specific environment.

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