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Scanning near-field optical microscopy (SNOM) and its application in mineralogy

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Abstract

Scanning near-field optical microscopy (SNOM) is a member of the family of scanning probe microscopes. It combines the high three dimensional resolution of a scanning force microscope with the contrast mechanisms of an optical microscope. An optical resolution beyond the diffraction limit can be achieved. We show the first application of this technique in the field of mineralogy, and we point out its future potential.

Keywords: Scanning near-field optical microscopy, scanning force microscopy, optical microscopy, diffraction limit, resolution, shear force image, spectroscopy.

Introduction

Optical microscopy is still the most popular microscopy technique today. It is fast, inexpensive, provides a wealth of information and allows to investigate samples at ambient conditions. There are many contrast mechanisms, depending on whether polarization, amplitude or phase of the light is detected. Also, its applicapability ranges from imaging of living specimens to spectroscopic analysis of mineralogical samples. To extract more information of a given area, there is a growing need for higher spatial resolution, leading automatically into the nanometric regime. Although optical interferometry gives a vertical resolution down to the nanometer scale, the lateral resolution is limited by diffraction. This was derived by Abbe in his work on diffraction and microscopic imaging (ABBE, 1873). The theoretical diffraction limit of resolution, also known as the Raleigh criterion (1), is essentially given by the wavelength λ of the employed radiation and the numeric aperture $n \sin\Theta$:

$$\Delta \chi \geq 0.61 \frac{\lambda}{n \sin \Theta} \qquad (1)$$

where n is the refractive index of the medium between the object and the microscope, and Θ is the acceptance angle of the detection system. As a consequence, the lateral resolution can only be improved by applying a high numerical aperture ($NA = n \sin\Theta$) or by decreasing the wavelength, such as in electron or X-ray microscopy. Successful efforts have been made in this direction by MANSFIELD and KINO (1990) and by VAN DER OORD et al. (1992). To get even higher resolution one has to go to a regime where wavelength is of no relevance. Studies in this direction resulted in another relatively new and different class of microscopes that have a lateral resolution in the nanometer range: the scanning probe microscopes (SPM), of which the most prominent member is the scanning tunneling microscope (STM) (BINNIG et al., 1986). All of these developments are very interesting but only a minority has proven their usefulness to mineralogical or geological problems. Nevertheless, due to the high lateral resolution of these techniques, new insights into mineralogical questions have been gained (HOCHELLA et al., 1989; HOCHELLA et al., 1990; HARTMAN et al., 1990; WEISENHORN et al., 1990; JOHNSSON et al., 1991; RACHLIN et al., 1992; HILL-

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NER et al., 1992; OHNESORGE and BINNIG, 1993; SCANDELLA et al., 1993; STIPP et al., 1994; HENDERSON et al., 1994; NAGY and BLUM, 1994). It is therefore clear that much could be learned by combining the interaction mechanism of optical microscopy with the resolution of the scanning probe methods. The scanning near-field optical microscope (SNOM) combines the properties of a scanning force microscope (SFM) operated in the non-contact mode and an optical microscope. The result is a simultaneous acquisition of optical and topographic information with almost the resolution of a SFM.

HOW TO OVERCOME THE DIFFRACTION LIMIT?

The basic idea to overcome the diffraction limit in microscopy was already discussed by SYNGE (1928). To understand Synge's proposal we have to introduce two notions that are not applicable to conventional optics but to SNOM: the *far-field* and the *near-field*. With respect to a light source, the observer stays in the *far-field* when the distance between him and the light source is more than the wavelength λ of the emitted light ($d >> \lambda$). The *near-field*, consequently, describes the range within the wavelength of the light ($d << \lambda$). When the observer stays in the near-field the Raleigh criterion(1) is no more applicable and the resolution is only determined by the size of the light source and its separation from the surface. A resolution beyond the diffraction limit is therefore possible. Synge's proposal was based on the following knowledge (see Fig. 1): A tiny aperture is illuminated from the back and acts as a subwavelength sized light source which is scanned in optical *near-field* distance to a sample surface, i.e., at a distance $d << \lambda$. No diffraction limited lens is needed for the image formation at a resolution functionally dependent on only the probe size and the probe-to-sample separation, each of which can be made much smaller than the wavelength of light. But more than 40 years passed until Synge's ideas were taken up and realized by Ash and Nicholls, who achieved a lateral resolution of $\lambda/60$ in the microwave wavelength regime (λ = 3 cm) (ASH and NICHOLLS, 1972).

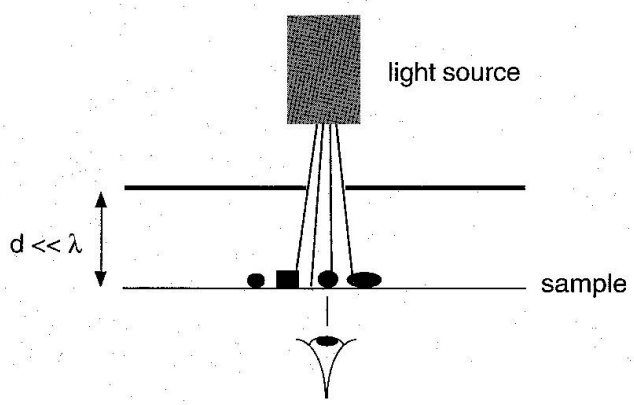


Fig. 1 Generic scanning near-field optical microscope (SNOM) setup: a subwavelength light source is raster-scanned across the sample at small distance.

The realization of the scanning near-field optical microscope (SNOM), sometimes also termed NSOM, was only possible after the invention of the STM, with its ability to actuate probes in a controlled manner at distances of nanometers over surfaces of interest. Indeed, the SNOM borrowed many parts from STM and especially SFM. Shortly after the appearance of the STM, POHL et al. (1984) first demonstrated near-field microscopy in the optical wavelength regime with a spatial resolution of $\lambda/20$ (25 nm).

Despite the attractive possibilities, SNOM was nearly forgotten because of the big rush on the other scanning probe methods such as STM and SFM. But now many scientists realize that certain sample-specific information, especially chemical identification and spectroscopy is nearly impossible to obtain with SFM, or just under very restrictive circumstances, such as friction force microscopy (FFM) (FROMMER and MEYER, 1991; MEYER, 1992). SNOM provides most of the contrast mechanisms known from the conventional microscopy, such as: absorption, polarization, fluorescence, and reflectivity. In some cases the resulting contrast is similar to that observed in the far-field (conventional microscopy), whereas in others, near-field specific eflfects are observed.

Experimental

Figure 2 shows a schematic diagram of a SNOM as it was used in this study (Topometrix Aurora). Laser light (Ar 488 nm) is coupled into a near-field probe formed from an aluminium (Al)-coated, tapered optical fiber (see inset in Fig. 2). The aperture at the apex of the-fiber, usually around 50 nm in diameter, is the only place that is not coated with Al. The tapered end is produced by pulling the fiber while heating with a CO_2 laser. This probe is held in near-field distance of the sample by a force feedback similar to non-contact SFM. The fiber is attached to a piezo tube, that oscillates the fiber end near its resonance frequency. Approaching the probe to the sample surface causes an interaction that results in a damping of its oscillation amplitude and in a

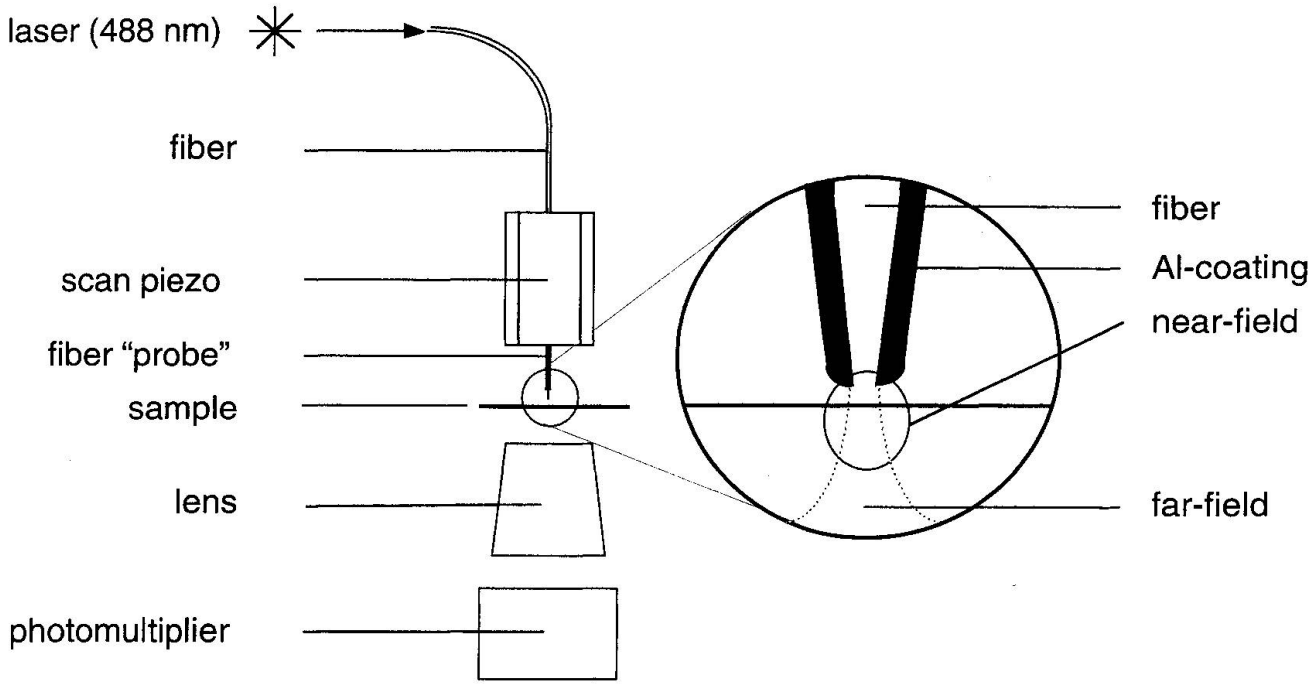


Fig. 2 Schematic of a SNOM in transmission. Inset shows the end of the optical sensor with an aperture of about 50 nm in diameter.

phase shift. This damping is a function of the distance and is used to keep the probe-to-sample distance constant (shear force feedback).

After transmission through the aperture and the sample, the light is collected with an objective lens of the conventional system. It is also possible to collect light that was reflected from the sample surface (BIELEFELDT et al., 1994). This mode of detection can be very useful for opaque samples such as ores, and for local reflection-spectroscopy. In both cases the collected light is detected by a photomultiplier. The sample is scanned under the probe with the aid of piezoelectric transducers, and the SNOM (transmission or reflection) and shear force signals are collected for every point (for a review and an introduction into the other SNOM types see HEINZELMANN and POHL, 1994). As a consequence, non-contact *(shear force)* and optical images are always collected simultaneously. Because the oscillation of the probe is parallel to the surface in SNOM, instead of the perpendicular motion in non-contact SFM, this mode is called *shear force mode.*

Results and discussion

FIRST MINERALOGICAL APPLICATIONS

Figure 3a shows a photomicrograph of a well-polished thin section (25 µm) of a symplectitic intergrowth of amphibole, diopside and plagioclase at the rim of an idiomorphic garnet crystal. The symplectite appears only as a black area (marked with a white arrow) at the border of the relatively idiomorphic garnet. Identification of the phases forming the symplectite was performed by electron probe microanalysis. Figure 3b shows a corresponding shear force image of a symplectite from the same thin section. The shear force image gives information on the topography of the investigated area. It shows mainly scratches, resulting from the polishing process (arrows 1, 2). Figure 3c displays the transmission mode SNOM image which was simultaneously recorded with the shear force image in figure 3b. One can see that this imaging mode yields completely different information. For example, the grain boundary of garnet is clearly visible (arrow 1), and so is the symplectitic intergrowth at the edges of the garnet. This picture reveals, that the symplectite is a mixture of different phases (arrow 2). It appears that the symplectite grows into the garnet, forming pear-like structures or bulges (arrow 3), which are indicative of grainboundary migration. It is not that easy to attribute the clear contrasts to a specific process in this near-field optical image, but it is probably a mixture of absorption and changes of the polarization.

Figure 4 serves to illustrate that SNOM is also suitable to image inclusions in minerals. It emphasizes the power of gaining information that

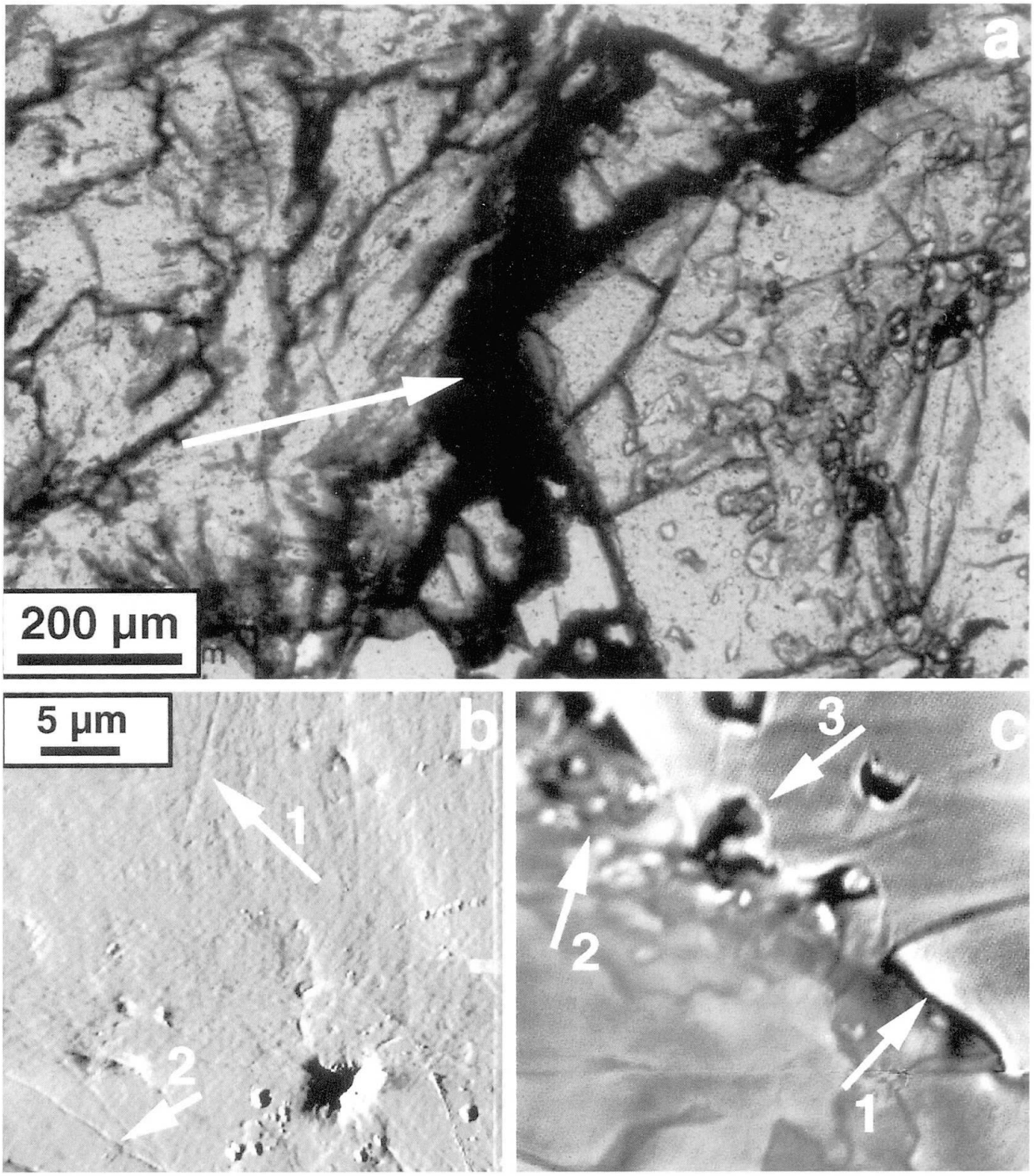
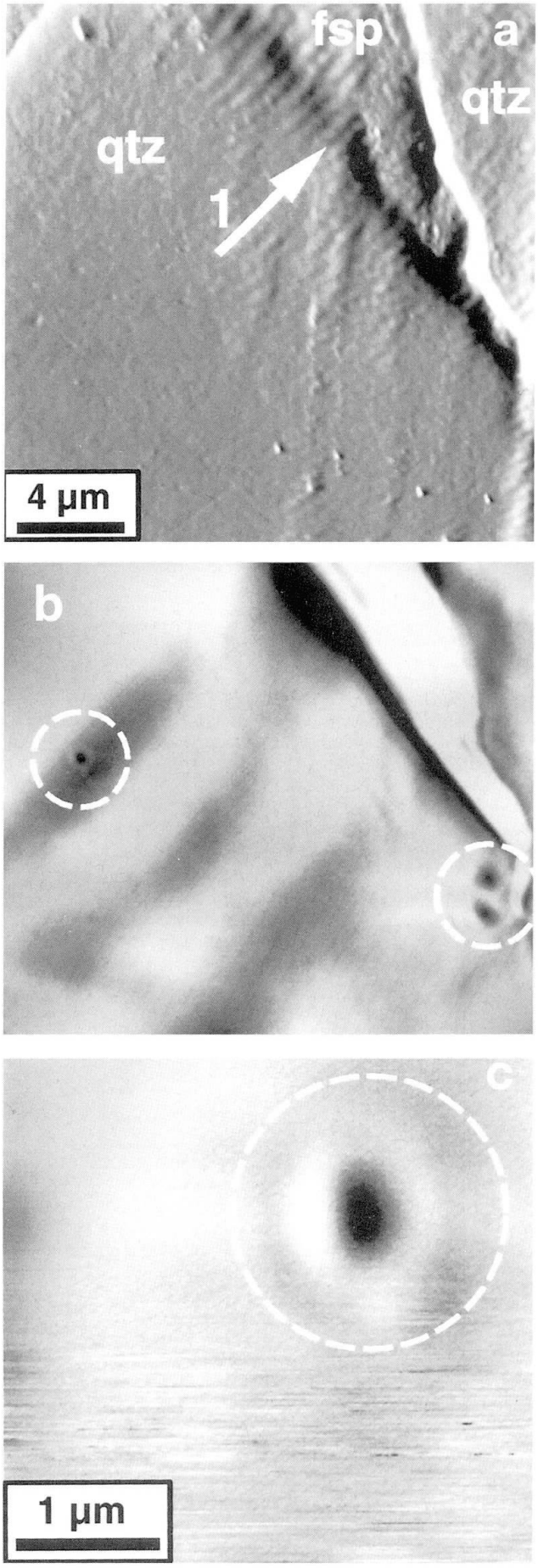


Fig. 3 a) Photomicrograph of a garnet with a symplectite at its border (arrow). b, c) Simultaneously acquired topography (b) and SNOM images (c) of a selected area. Note the different contrast in the scanning near-field optical microscope image. Details are explained in the text.

was not accessible until now. Figure 4a shows the shear force image of a thin section of a rock containing quartz and feldspar. The grainboundaries are clearly visible due to the different relief of quartz and feldspar developed during the polishing process (arrow 1 and bright line). The quartz crystal is harder and consequently higher than the feldspar. These structures can be seen in the shear force (Fig. 4a) as well as in the transmission mode SNOM image (Fig. 4b). But in transmission mode, however, there are some additional features visible: First, one notes that the quartz



◁ *Fig. 4* Topographic (a) and SNOM (b) images of a thin section containing quartz and feldspar. The solid inclusion marked on the left is shown in a separate scan in (c).

crystal is not totally uniform. There are lighter and darker regions arranged in parallel bands. This shows local variations of the sample absorptivity, that could be the result from former tectonic stress (deformation-bands). Second, there are three black spots (circled), that show a different absorptivity and probably represent mineral inclusions. Figure 4c shows a smaller scan of the upper left cycled spot, which has a diameter of about 200 nm.

With conventional optical microscopy, it would be very hard just to see these and other small structures, such as solid and fluid inclusions, symplectites, myrmecites, exsolution-lamellas and polysynthetic twins. An optical identification of these minerals and structures would be impossible. With SNOM, it is possible to additionally perform optical spectroscopy with a spatial resolution in the range of nanometers. The only prerequisite is a tuneable laser, that is a laser that can continuously emit different wavelengths. Other types of spectroscopy that are attainable, and where different groups are working on, are fluorescence, cathodoluminescence, Raman and infrared spectroscopy. If one succeeds to dye the different phases with a dye that is excitable by the wavelength of the laser used, an easy identification with high resolution is possible. The Al-coating of the fiber can be used to very locally emit electrons at the apex of the probe, causing cathodoluminescence effects which can be subsequently detected with SNOM. In very similar ways the fiber can be used to perform Raman and infrared spectroscopy (PAESLER et al., 1990; TSAI et al., 1994).

Conclusions

SNOM is a promising technique for the mineralogical and geological field. It combines the high spatial resolution of a non-contact SFM with the well established contrast mechanisms of optical microscopy. In the near future, it will be useful in order to perform chemical analysis and point spectroscopy in the nanometer range. It is also possible to gain very small scale optical information on rock formation and possible former tectonic activities (mineral deformation, mineral recrystallization at the rims due to tectonic stress) not accessible until today.

Like in conventional microscopy, one is not restricted to transparent samples: opaque minerals also can be investigated with the same apparatus. A major advantage of SNOM is that it does not require new sample preparation techniques. SNOM can be carried out on the same thin sections and single crystals used for microprobe analysis. Attention has to be paid particularly to a very good polish of the surface of the sample so that the tip of the SNOM sensor can follow the surface profile.

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References

ABBE, E. (1873): Beiträge zur Theorie des Mikroskops und der mikroskopischen Wahrnehmung. Archiv für Mikroskopische Anatomie, 9, 413.

ASH, E. and NICHOLLS, G. (1972): Super-resolution aperture scanning microscope. Nature, 237, 510–512.

BIELEFELDT, H., HÖRSCH, I., KRAUSCH, G., LUX-STEINER, M., MLYNEK, J. and MARTI, O. (1994): Reflection-scanning near-field optical microscopy and spectroscopy of opaque samples. Applied Physics A, 59, 103–108.

BINNIG, G., QUATE, C. and GERBER, C. (1986): Atomic force microscope. Phys. Rev. Lett., 56(9), 930–933.

FROMMER, J. and MEYER, E. (1991): Atomic force microscopy: A tool for surface science. Journal of Physics of Condensed Matter, 3, 1–9.

HARTMAN, H., SPOSITO, G., YANG, A., MANNE, S., GOULD, S. and HANSMA, P. (1990): Molecular imaging of clay mineral surfaces with the atomic force microscope. Clays and Clay Minerals, 38(4), 337–342.

HEINZELMANN, H. and POHL, D. (1994): Scanning near-field optical microscopy. Applied Physics A, 59, 89–101.

HENDERSON, G.S., VRDOLJAK, G.A., EBY, R.K., WICKS, F.J. and RACHLIN, A.L. (1994): Atomic force microscopy studies of layer silicate minerals. Colloids and Surfaces, 87, 197–212.

HILLNER, P., GRATZ, A., MANNE, S. and HANSMA, P. (1992): Atomic-scale imaging of calcite growth and dissolution in real time. Geology, 20, 359–362.

HOCHELLA, JR., M.F., EGGLESTON, C.M., ELINGS, V.B., PARKS, G.A., BROWN, JR., G.E., WU, C.M. and KJOLLER, K. (1989): Mineralogy in two dimensions: Scanning tunneling microscopy of semiconducting minerals with implications for geochemical reactivity. Amer. Mineral., 74, 1233–1246.

HOCHELLA, JR., M.F., EGGLESTON, C.M., ELINGS, V.B. and THOMPSON, M.S. (1990): Atomic structure and morphology of the albite 010 surface: An atomic-force microscope and electron diffraction study. Amer. Mineral., 75, 723–730.

JOHNSSON, P.A., EGGLESTON, C.M. and HOCHELLA, JR., M.F. (1991): Imagine molecular-scale structure and microtopography of hematite with the atomic force microscope. American Mineralogist, 76, 1442–1445.

MANSFIELD, S. and KINO, G. (1990): Solid immersion microscope. Appl. Phys. Lett., 57, 2615–2616.

MEYER, E. (1992): Atomic force microscopy. Progress in Surface Science, 41, 3–49.

NAGY, K.L. and BLUM, A.E. (1994): Scanning Probe Microscopy of Clay Minerals, volume 7 of CMS Workshop Lectures. The Clay Minerals Society, P.O. Box 4416, Boulder, CO 80306.

OHNESORGE, F. and BINNIG, G. (1993): True atomic resolution by atomic force microscopy through repulsive and attractive forces. Science, 260, 1451–1456.

PAESLER, M., MOYER, P., JAHNCKE, C., JOHNSON, C., REDDICK, R., WARMACK, R. and FERRELL, T. (1990): Analytical photon scanning tunneling microscopy. Physical Review B, 42(10), 6750–6753.

POHL, D., DENK, W. and LANZ, M. (1984): Optical stethoscopy: Image recording with resolution λ/20. Appl. Phys. Lett., 44, 651–653.

RACHLIN, A.L., HENDERSON, G.S. and GHO, M.C. (1992): An atomic force microscope (AFM) study of the calcite cleavage plane: Image averaging in fourier space. Amer. Mineral., 77, 904–910.

SCANDELLA, L., KRUSE, N. and PRINS, R. (1993): Imagine of zeolite surface structures by atomic force microscopy. Surface Science Letters, 281, 331–334.

STIPP, S., EGGLESTON, C. and NIELSEN, B. (1994): Calcite surface structure observed at micrographic and molecular scales with atomic force microscopy (AFM). Geochim. Cosmochim. Acta, 58(14), 3023–3033.

SYNGE, E. (1928): A suggested model for extending microscopic resolution into the ultra-microscopic region. Philos. Mag., 6, 356–362.

TSAI, D., OTHONOS, A., MOSKOVITS, M. and UTTAMCHANDANI, D. (1994): Raman spectroscopy using a fiber optic probe with subwavelength aperture. Appl. Phys. Lett., 64(14), 1768–1770.

VAN DER OORD, C., GERRITSEN, H., LEVINE, Y., MYRING, W., JONES, G. and MUNRO, I. (1992): Synchrotron radiationas as a light source in confocal microscopy. Rev. Sci. Instr., 63, 632–433.

WEISENHORN, A., DOUGALL, J.M., GOULD, S., COX, S., WISE, W., MASSIE, J., MAIVALD, P., ELINGS, V., STUCKY, G. and HANSMA, P. (1990): Imaging and manipulating molecules on a zeolite surface with an atomic force microscope. Science, 247, 1330–1333.

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