X-ray stereo microscopy for investigation of dynamics in soils

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X-ray stereo microscopy for investigation of dynamics in soils

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Abstract. The here presented combination of stereo imaging and elemental mapping with soft X-ray microscopy reveals the spatial arrangement of naturally aqueous colloidal systems, e. g. iron oxides in soil colloid clusters. Changes in the spatial arrangement can be induced by manipulating the sample in-situ and thus be investigated directly and as a function of time.

1. Introduction

Due to its high spatial and spectral resolution, X-ray microscopy is an important instrument for investigation of colloidal systems, e.g. from environmental sciences [1]. Taking advantage of the natural contrast mechanism, sample preparation is not necessary, it can be imaged in transmission within its original aqueous media up to 10 μm thickness with a resolution in the range of 20-50 nm. In soil science, the element distribution within soil colloid clusters is of great interest [2]. With images taken above and below an absorption edge, distribution maps of the corresponding element are achieved. Thus, the iron distribution in colloidal samples from the environment has been determined. By tilting the object, stereo pairs of images have been taken. The here presented combination of elemental mapping with X-ray microscopy and stereo imaging provides a tool for that and reveals the spatial arrangement of e. g. iron oxides in soil colloid clusters. Changing the chemical conditions of aqueous medium leads to changes in the spatial arrangement, which can be done directly in the X-ray microscope. Since many of the so induced morphological changes are much slower than the time required even for taking stereo pairs, the dynamical behaviour of these changes can be investigated.
Theory and results of such experiments at three different setups will be presented briefly.

2. Parallax equation based stereo reconstruction

Stereo calculations are based on the parallax equation [3]. It relates the parallax ΔY to the vertical distance h and the tilt angle 2θ (θ is the stereo angle). Thus, for a tilt by $\theta_2 - \theta_1 = \theta$

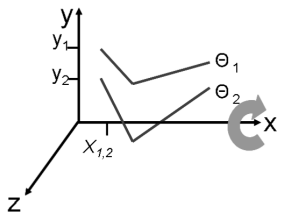

Figure 1. Rotation of a 3-d object around x-axis.

around the x-axis, the z-coordinate of a structure can be calculated by the difference between the y-coordinates (y_1 and y_2) in the respective images projecting the structure onto the x-y-plane (Figure 1). The spatial coordinates become:
$X = x_1 = x_2 \qquad Y = \frac{y_1+y_2}{2\cos\theta} \qquad Z = \frac{y_1-y_2}{2\sin\theta}$
The program xstereo has been written in IDL to mark or select significant structures in a set of tilted x-ray images and get information about the 3-d configuration, distances and lengths. Matching points recognizable in both projections are marked manually. In the 3-d plot, features like curvatures are displayed.

3. Experiments with XM-1, ALS

The soft X-ray microscope XM-1 is a full field microscope at a bending magnet [4]. For the experiments, a 25 nm micro zone plate was used to take high resolution images below and above the Fe L 3 edge at 707 eV. The magnification was about 2800, the spectral resolution E/ΔE≈500. Two kinds of tiltable sample holders for aqueous samples were used: glass capillaries with tips of about 3 μm diameter, and flat holders with two Si_3N_4 foils of 100 nm thickness each. Both holders were sealed, so the samples kept aqueous over several hours.
In Figure 2, images of an aqueous flock of montmorillonite with hematite between two Si_3N_4 foils taken at 704 eV (left) and 707 eV (right) are shown. The hematite particles are clearly appearing at the Fe L 3 absorption edge. Figure 3 presents a detail of the montmorillonite sample shown previously, viewed with an angular difference between both images of 14° around a horizontal tilt axis. Both images are taken at E = 707 eV. Lines, points, and edges are marked for analysis. The results from xstereo are given in Figure 4: view of a 3-d plot of points and edges marked in Figure 3. The unit of the axis is pixel, where 11 pixel relate to 100 nm. Distances had been determined e.g. between structure 6 (center of hematite) and 8 (clay edge) to 180 nm, or between structure 3 (center of hematite) and 7 (clay edge) to 630 nm, revealing results about the interrelation between pH value of the solution and the corresponding edge charge of the clay platelets [5].

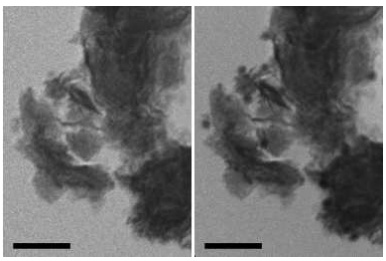

Figure 2. Aqueous sample of montmorillonite particles with hematite colloids. Left: E = 704 eV, right: E = 707 eV. Scale bar: 1 μm.

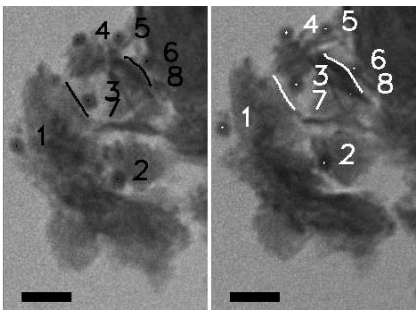

Figure 3. Detail of the sample shown in Figure 2, $\Delta\theta = 14°$, E = 707 eV, features marked. Scale bar: 500 nm.

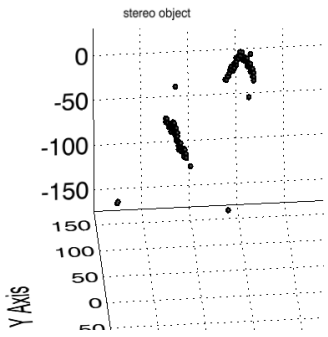

Figure 4. View of 3-d plot of marked features (Figure 3). Unit: pixels. 11 pix. = 100 nm.

4. Experiments with the compact X-ray microscope, KTH

The compact X-ray microscope at the KTH, Stockholm, Sweden, provides a table-top sub-30 nm resolution. It is based on a liquid-jet laser-plasma source, condenser optics and zone plates objectives [6]. The images were taken at $\lambda = 3.37$ nm wavelength with exposure times of 4 to 8 minutes. The image diameter is 20 μm. The vertical tilt given by the stereo mount was 14°, adapting the parallax to human stereo vision. Two pairs of images are shown (Figure 5 and 6).

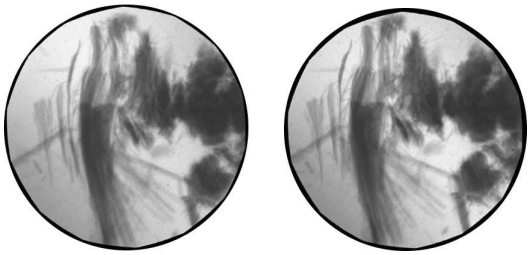

Figure 5. Aqueous nontronite particles, E = 368 eV, $\Delta\theta = 14°, \emptyset = 20\,\mu$m.

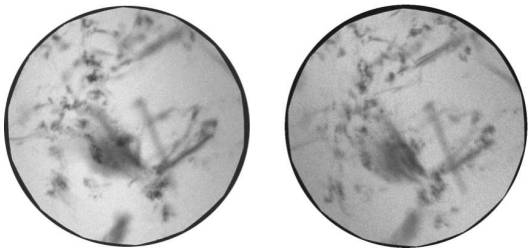

Figure 6. Aqueous nontronite with hematite particles, E = 368 eV, $\Delta\theta = 14°, \emptyset = 20\,\mu$m.

5. Experiments with the STXM, BESSY

The sample in the Scanning Transmission X-ray Microscope at the Undulator U41 at BESSY is scanned with a 50 nm spot, at best resolution a region of up to 40x40 μm^2 [7]. The sample stage has been adapted for tilting capillaries.

Figure 7 shows montmorillonite in a capillary. Two pairs of images were taken of the same sample region with equal image paramaters, one before (Figure 7 up) and one after (Figure 7 below) adding carbon nanotubes. Both image pairs were taken at 400 eV within 30 minutes, respectively, with a horizontal tilt of 15° of a sample region of 8x7 μm^2 onto 160x140 pixel^2 with 16 ms exposure time per pixel. The time between the pairs was 5 hours, resulting in a big change in structure.

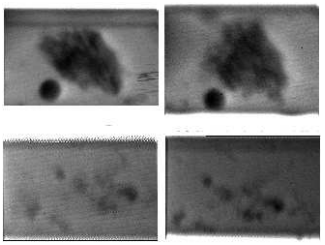

Figure 7. Montmorillonite. Below: after adding C nanotubes.

6. Conclusions

The suitability of a TXM, a STXM, and a TXM with a laboratory source for the investigation of samples from the environment in ambient conditions in combination with stereo imaging and elemental mapping could be shown. Manipulating the sample and tilting the sample was possible even under observation. Therefore, studies of the dynamical behavior of samples will be performed as a next step using both, stereo imaging and elemental mapping, to obtain a complete description of the system under investigation.

Acknowledgments

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