Research area: Mineral Characterisation
"Development of quantitative methods to describe raw materials and mineral products".

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Objective
The project aims at developing automatic SEM based quantitative techniques in order to:

- quantify the mineral content, both major and trace minerals in rocks/ores and in milled products,
- quantify the mineral texture
- quantify the occurrence of minerals in different types of particles in milled materials and mineral products (PTA: Particle Texture Analysis)

Background
All mineral processes are treating particles, and not chemical elements. The particles are containing one or more minerals. To achieve high quality products meeting the different requirements from the customers, and with a minimum waste, a thorough and detailed knowledge concerning the occurrence of the minerals in the raw materials and in the mill products is needed.

In investigations of ore minerals, particularly the base metal sulphides, there has for a long time been used automatic SEM-based image analysers to quantify the occurrence of the minerals. Special emphasis has been laid on determining the liberation properties of the economic minerals to avoid under- or over grinding of the minerals. It is recognized that the behaviour of particles in separation processes is a function of different properties of the particles of which the most important are:

Mineralogical parametres; types of minerals making up the particles, mineral chemistry,..;
Textural properties; size, shape, grain boundaries, type of intergrowths (particle texture)…;
Specific properties of the minerals including specific gravity, surface property, magnetic property…;

In Norway, there has been a strong and positive development within industrial minerals. Due to this the project aims at developing methods which can be used on industrial minerals. In additions the methods also can be used on all kinds of other materials.

Principals of PTA - Particle Texture Analysis

Purpose
Describe mineral liberation, mineral associations and how the minerals are grown together by means of backscatter electron images and X-ray data in the scanning electron microscope (SEM).
Performance

- Polished sections of narrow grain size fractions are analysed by Oxford Inca Feature

- The backscatter electron images are thresholded by grey levels and analysed by means of X-rays aiming to obtain analyses of every single mineral grain.
- The images and the classified mineral analyses are stored.
- Inputs for the PTA software are the Inca database and the uncompressed images from each field.
- Composite particles are found by means of image analysis.
- Results from the image analysis is stored in a database
- Queries are then performed to get desired information
- The results can be presented as distributions of
    - relative amount of minerals in particles
    - mineral associations in particles
    - free grinding distributions
    - statistics

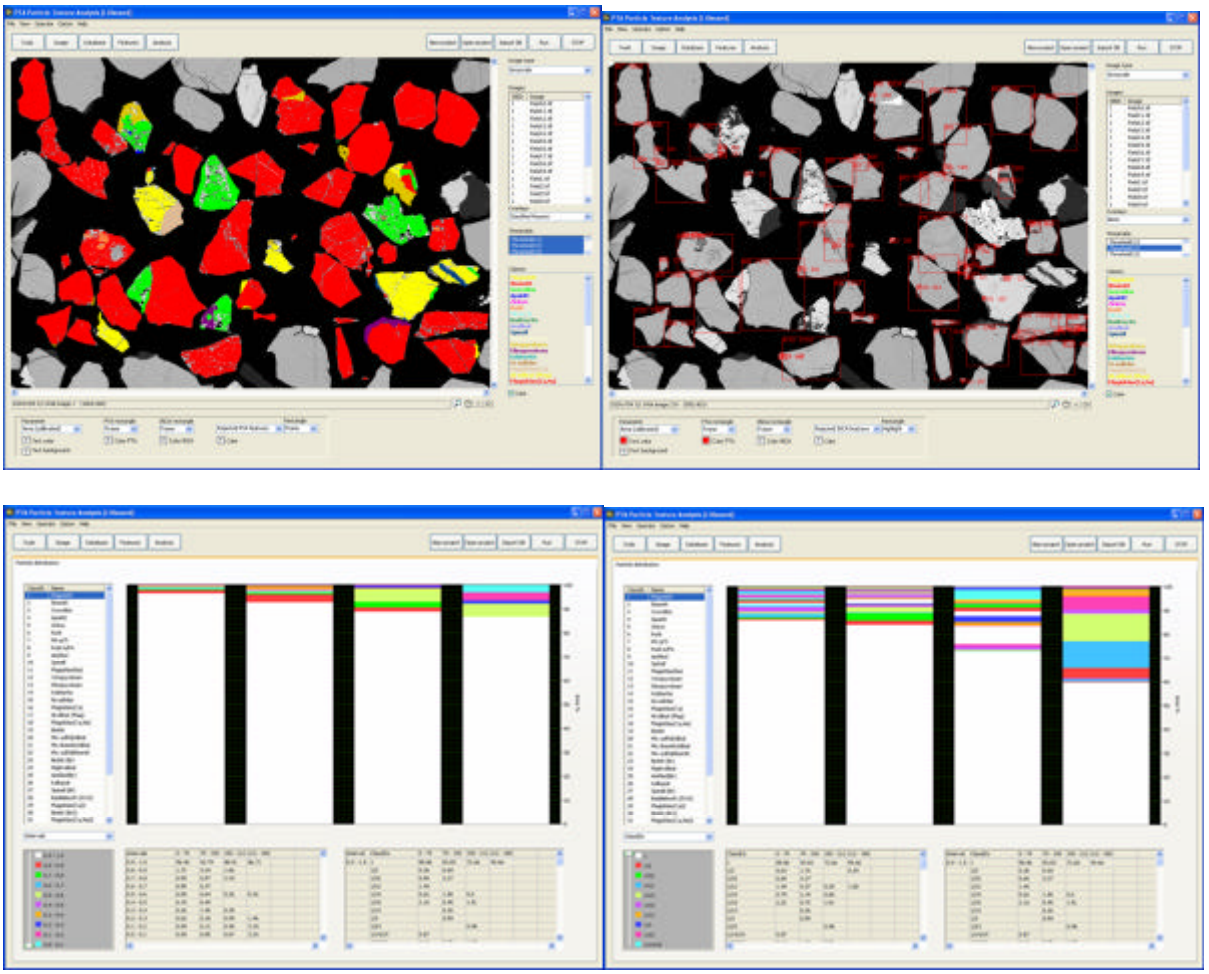


Figure 1 Examples of classified minerals, identified grains, free grinding decisions and mineral associations

Principals of EBSD - Electron BackScatter Diffraction
lattice and grain texture

Electron backscatter diffraction

The EBSD technique is based on the weak diffraction pattern that forms when a focused, stationary, primary electron beam strikes a polished sample, backscatters and diffracts. The diffraction pattern is formed on a fluorescent screen and transferred by a camera to the computer (Hjelen 1990).

Rapid developments in both hardware and software in the past 10 years have made EBSD easy to use and ideal for the rapid analysis of microstructures of crystalline materials.

The diffraction pattern is therefore characteristic for the crystal structure and space orientation of the crystal.

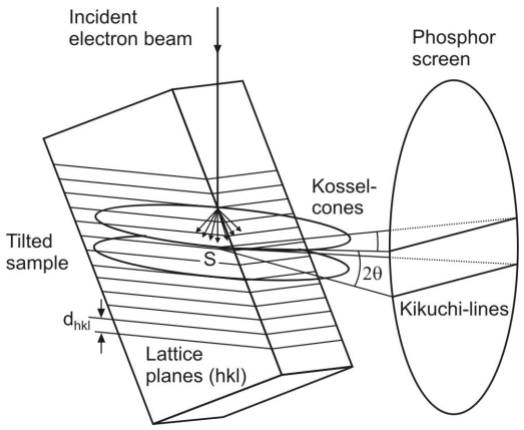


Figure 2 Origin of Kikuchi lines from EBSD (Schwartz et.al. 2000)

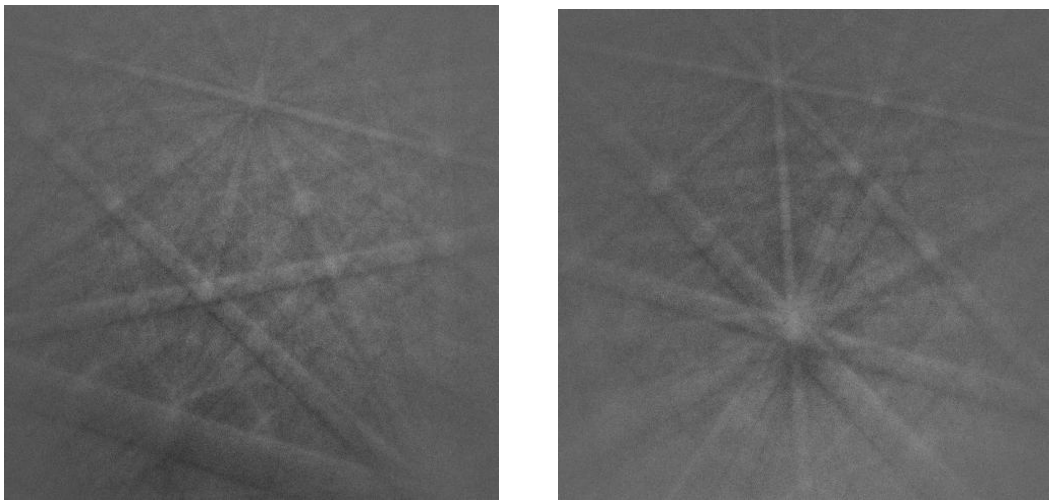
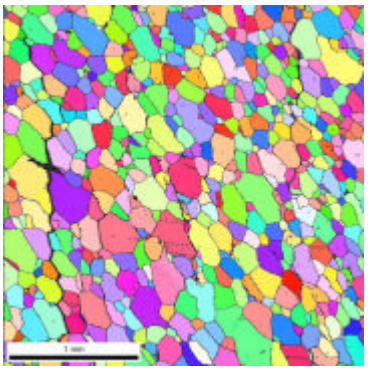
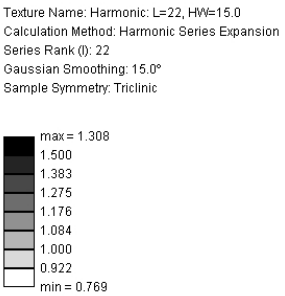
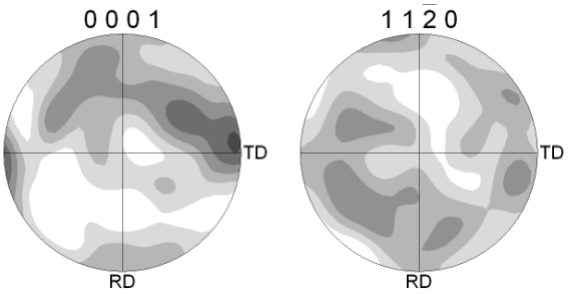


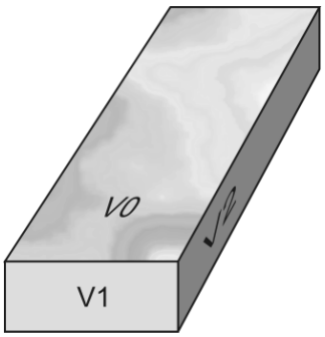
Figure 3 EBSD patterns from 2 orientations of calcite

The EBSD software automatically locates the positions of individual diffraction bands, compares these to theoretical data about the relevant phase and rapidly calculates the 3-D crystallographic orientation. The whole process from start to finish can take from 0.025 sec. to more than one second dependent on the phases analysed.
For each pixel the phase and orientation are stored and can be visualised by maps or plots. Different kinds of plots can show misorientation, grain size, poles etc. The spatial resolution of this technique is superior to x-rays, since elastic backscattered and diffracted electrons have a smaller interaction volume than x-rays. The interaction volume is dependent on acceleration voltage and atomic number of the analysed minerals, but is usually in the sub micron range. (Moen et. al. 2003) (Moen et. al. 2004)









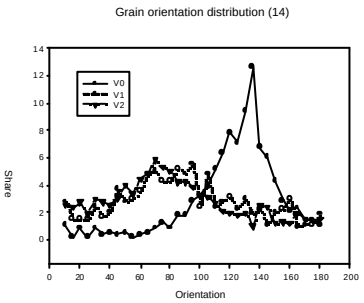


Figure 4 EBSD orientation map, (0001) and (11-20) pole figures and grain shape orientationdistribution for 3 orthogonal directions of a marble block (Moen et. al. 2004).

Kikuchi S. (1928), Diffraction of cathode rays by mica. Jap. J. Phys, 1928. 5:83.

Moen K., Malvik T., Hjelen J., Leinum J.R. (2003), Automatic Material Characterization by means of SEM-techniques, Proceeding, 9th Euroseminar on Microscopy Applied to Building Materials, Ed: M.A:T:M: Broekmans et.al

Schwartz A.J., Kumar M., Adams B.L. (2000), Electron Backscatter Diffraction in Materials Science. NY, Kluwer Acadenic/ Plenum Publishers, ISBN 0-306-46487-X.

Moen K., Malvik, T., Hjelen, J., Leinum J.R., Alnæs L. (2004), EBSD microstructure measurements of marble, Applied Mineralogy 2004, ICAM-BR, Sao Paulo, Brazil, ISBN 85-98656-01-1.

Cooperation with the industry

The importance of being able to quantify the mineralogical parameters is fully recognized by the industry. Several industry partners (TITANIA, Hustadmarmor, Norwegian Crystallite) support the project in order to benefit from the developments being made. In the future, an on line measurement system using the web, and where the industry partner can take an active part in the measurements, is aimed. Combined with flexible solutions and measurement settings this will lead to techniques being able to describe most mineralogical and textural phenomena occurring in rocks/ores and in mineral products.

By use of new and advanced laboratory equipment determine physical and chemical properties of industrial minerals and beneficiated products of industrial mineral with special emphasis on thermal, mechanical and surface properties. The results may be used to define areas for new applications of minerals, establish correlations between material properties and geological processes leading to the different mineral raw materials, and modelling of micro processes occurring during heating and mechanical impact of minerals.

Electron Microscopy Laboratory facilities

The Scanning Electron Microscopy laboratory is the largest SEM-lab. in Scandinavia, with a suite of 7 conventional, low-vacuum and field emission SEMs. In addition a Transmission Electron Microscope (TEM) and an Electron Probe Micro-Analyser (EPMA) belong to the EM-lab. (http://www.material.ntnu.no/lab/material/index.html) These analytical electron microscopes are designed for material characterization using a range of imaging and analytical methods. (Pdf). The instruments are equipped with Energy Dispersive Spectrometers (EDS), Wavelength Dispersive Spectrometers (WDS), Electron BackScatter Diffraction (EBSD) detectors, CL etc.Automatic EBSD analysis is combined with deformation- and heat treatment experiments in a special in-situ substage to study dynamic processes.