

**CRYSTALLISATION AND MORPHOLOGY OF GYPSUM
STUDIED BY SYNCHROTRON X-RAY DIFFRACTION AND SCANNING
ELECTRON MICROSCOPY**

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ABSTRACT

Experiments were performed to investigate the possibility of recording both the crystallisation kinetics and the resulting crystal shape and texture, on the same calcium sulfate specimen by a combination of synchrotron X-ray powder diffraction and scanning electron microscopy techniques.

Calcium sulfate hemihydrate produced from analytical grade calcium sulfate dihydrate was mixed with water and small amounts of setting modifiers, the paste was drawn into a glass capillary and then mounted on the diffractometer. During the hydration X-ray spectra were collected for three minutes. These data were then converted to intensities as a function of time by integrating the area under selected peaks, one for the hemihydrate and one for the dihydrate. After the samples had been dismantled from the X-ray equipment the glass capillaries were crushed in acetone. Specimens for microscopy were prepared from these samples and later studied in a JSM-840A scanning electron microscope.

The relative X-ray intensities were utilised to follow the dissolution of hemihydrate and formation of dihydrate as a function of time. The time for complete reaction increased in the following order depending on the additives: 5% gypsum < 5% gypsum and 0.25% citric acid < 0.5% gypsum < 0.5% potassium sulfate < no additives < 0.25% citric acid. From the microscope pictures it could be seen that when gypsum was added the material was much more homogeneous, due to the increased number of nucleation sites. It could also be seen that when citric acid was added the amount of thin twin crystals increased. Otherwise the gypsum crystals were normally needle shaped.