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Application of synchrotron radiation in cement mineralogy

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Synchrotron based analysis of materials used in the primary industry is mostly limited to basic research, due to the low availability of synchrotron radiation (SR) and poor links to potential industrial users. On the example of cement materials, especially calcium silicate hydrates, recent basic research is summarized. Future possibilities for industrial research and development are outlined. Synchrotron based investigations on the structure of cement gel in literature are mainly done with XAS [1]. At present, work on alkali-rich gels is under way to shed light on structural aspects of the alkali-silica reaction and water glass based binders [2]. SR-based investigations of crystalline calcium silicate hydrates formed under hydrothermal conditions are done with XRD, both in [3] and ex situ [4], XAS [1], XRF and IR [5]. Ex situ studies on single crystals and powders mainly focus on structural investigations whereas special kinetic information can be obtained from in situ measurements. Main applications are steam cured and autoclaved aerated concrete, calcium silicate bricks, oil well cements [3], recycling cements [5] and disposal of conventional or nuclear waste. Promising new methods based on SR are the quantitative phase analysis of cement hydrates including the non crystalline fraction with the Rietveld method and high-resolution microtomography coupled with energy dispersive XRD for real-time and in-situ characterization of the porous microstructures of concrete [6].

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Structural defects and polytypism in moissanite and synthetic SiC crystals

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In recent years, defect characterisation of SiC polytypes has been developed significantly, due to the increasing importance of such polytypes for high temperature, high-power and high-frequency electronic devices [1]. On the other side, moissanite-SiC is a rare mineral that was discovered in 1905 within a meteorite and only recently as a rock-forming mineral (8.4 vol%) in one unique specimen of a terrestrial volcanic rock [2]. Besides it has been found in kimberlitic pipes and can provide some light on the redox conditions of the Earth Mantle. Finally SiC is a particularly interesting species of presolar grain because the polytype distribution can be related to different parameters of the expanding stellar atmospheres of asymptotic giant branch-AGB-stars.

In the frame of a research program in progress on SiC, growth defects of bulky gem-crystals of 6H-SiC were studied by X-ray Diffraction Topography-XRDT and it was found that the main types of defects were dislocations parallel and perpendicular to the growth directions, and micro-channels [3].

This piece of work is devoted to the study of structural defects and degree of order of natural and synthetic moissanite.

Natural and synthetic samples have been investigated by XRD Topography and TEM, focusing on the relationships between defects and growth conditions. XRDT analyses of synthetic 6H-SiC allowed the characterization of dislocations and channels to be made and other polytypes for further investigation by TEM to be localized. All studied sample are characterized by the presence of linear defects, dislocations and micro-channels, uniformly distributed in the crystal. Moreover samples grown by means of Physical Vapour Transport-PVT method, show the same linear defects with different character, strictly related to growth conditions.

TEM images and electron diffractions (EDs) strongly differentiate natural from synthetic samples. ED patterns with [100] incidence of natural crystals are consistent with the 6H polytype and do not show streaks along the [001] stacking direction. This result is confirmed by structure refinement from area detector single crystal X-ray data [4]. Synthetic samples are comparatively much more disordered. Conventional images show high density of (001) faults, not observed in natural samples. Consistently, ED patterns of the [100] zone are streaked along c^* . Atomic resolution imaging shows that synthetic samples mainly consist of (001) stacking sequence described as $(32)_3$ [5]. Locally mixed stacking sequence described by notation $23(3233)_5$, probably referred to a long period polytype, are present.

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