Environmental and In Situ SEM/TEM

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Using hot stage scanning electron microscope for in-situ observations of the crystallization in silicate glass-ceramics

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In-situ observations using a scanning electron microscope (SEM) are very valuable when studying the crystallization process. The formation of the crystal phase can be monitored continuously. This is achieved by a hot stage which can heat up the sample to the desired crystallization temperature.

However only the surface can be studied in the SEM which leads to the requirement of a system where predominantly surface crystallization occurs.

Barium zinc silicate glasses with the mol% composition 30ZnO, 10Na_2O, 10BaO, 50SiO_2 show a pronounced crystallization at the surface. The formed glass-ceramics are promising novel materials for photonic applications. This system enables in-situ experiments within an acceptable time range due to the rapid formation of the crystal phase. Another system under investigation was a fresnoite glass system with the composition 2BaO, TiO_2, 2.75SiO_2. This system is suitable for oriented crystallization and piezo-ceramics. The third system studied in the hot stage is cordierite (2Al_2O_3, 2MgO, 5SiO_2), a long time known and well described system which still shows surprising crystallization behaviour in the in-situ experiment under vacuum condition.

The electron microscope JEOL JSM-7001F was equipped with a GATAN heating stage H1004. The hot stage can be heated up to 1250 °C. The module is cooled by water and a shield-shutter mechanism protects the objective pole-piece and the electron detector. The sample was placed in a crucible which is positioned within a ceramic furnace which is electrically heated by tantalum wires.

The micrographs were recorded every 30 s to 1 min for a duration up to 2 h. The evaluation of the micrograph series enables the determination of crystal growth kinetics. The experiments showed time-dependent crystal growth velocities in the case of barium zinc silicate and fresnoite glass-ceramics.

This leads to the conclusion that the residual glassy phase changes the composition during crystallization which is characteristic for non-isochemical systems.

A secondary effect could be observed during recording the micrographs: The electron beam irradiation directly affects the nucleation and crystal growth. This leads to a dilemma of applying high magnification and the influence on the experiment