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Low Voltage Transmission Electron Microscopy to Study Quantitative Electron-irradiation Damage by *in situ* Investigation of the Phase Transformation from Calcite to Lime

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Calcit ($CaCO_3$) is one of the most common carbonates of the earth crust. The most important use for calcite is for the manufacture of cements and lime for mortars. Limestone is the chief raw material, which when heated about 900° C forms quicklime (CaO) by the reaction: $CaCO_3 \rightarrow CaO + CO_2\uparrow$. Apparently calcite undergoes the same phase transformation to lime (CaO) by electron-irradiation damage. Therefore the new challenge arises to search for conditions and instrumental settings to study in situ the phase transformation on an atomic scale. The irradiation damage is caused predominantely by ionisation, thermal heating or by knock on damage. The influence of knock on damage is studied by reducing the accelerating voltage from 300 kV down to 20 kV by using a CM20 operating at 200 kV, equipped with a LaB6 cathode, a TITAN operating at 300 kV and 80 kV equipped with a field emission gun and an imaging-side Cs-corrector and the newly developed SALVE prototype microscope equipped with a field emission gun, an image-side Cs-corrector, corrected Omega-Filter and a monochromator [1] operating at 20, 40 and 80 kV. At high accelerating voltages the irradiation damage of calcite is starting immediatly with the electron-irradiation and can be divided up in three stages: first the amorphisation of the crystalline structure, second hole production and third recrystalisation in a polycrystalline structure with significant volume and mass loss during the reaction time. We found that the reduction of the accelerating voltages slows down the irradiation damage in calcite and gives the freedom to align the instrument to obtain information on the atomic scale before significant beam damage effects appear. This enables dose rate depend quantitative studies of the volume and mass losses during the phase transformation. [1] U.Kaiser et al Ultramicroscopy 111, 8,(2011), p.1239 [2] This work was supported by the DFG (German Research Foundation) and the Ministry of Science, Research and the Arts (MWK) of Baden-Wuerttemberg in the frame of the SALVE (Sub Angstrom Low-Voltage Electron microscopy and spectroscopy project. We thank the Institut of Mineralogy Münster (P. Schmid-Beurmann) for the samples, S. Groezinger for the sample preparation.