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Acoustic emissions imaging and synchrotron X-ray diffraction analysis of calcite at high pressure and temperature

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We have monitored from *in-situ* X-ray diffraction coupled to Acoustic Emission (AE) imaging, the behavior of a fine grained synthetic calcite aggregate, at 0.66 GPa and for temperatures ranging from ambient to 1200°C.

The powder sample was placed in a boron-epoxy assembly with an 8 mm edge-length and loaded in the MAX80 cubic multi-anvil press installed on the German synchrotron (HASYLAB-DESY, Hamburg). AE were recorded using five piezoceramic transducers (5 MHz eigen frequency) glued on each of the five WC anvils (4 side anvils and upper one). Full waveforms were acquired using an eight channel digital oscilloscope and located using the software Insite (ASC Ltd).

Beyond 600°C, calcite grains started growing as evidenced by huge changes in the relative intensity of the diffraction lines. This is correlated to a sudden burst of AE which all located within the sample volume. These AE may indicate that stress relaxation, going on as intra-crystalline plasticity mechanisms were activated, released enough acoustic energy to be recorded and located. Although the diffraction data showed that grain growth continued beyond 800°C, the acoustic activity progressively decreased to below the sensitivity of our recording device (i.e. the triggering level). However, at temperature higher than 1000°C, a large number of AE were recorded again (\sim 2000 events). AE location revealed that the AE front progressed inwards the sample. The complete loss of diffraction signal and the *post-mortem* recovery of small amounts of CaO suggest that the second AE burst may be related to calcite melting/decarbonation.

Perspectives include thorough microstructural analysis of the samples using electron microscopies (SEM and TEM) as well as a statistical and mechanical analysis of the acoustic data. These experiments will also be repeated (without diffraction) on samples with larger grains using the piston-cylinder apparatus in the same pressure and temperature range.