Acoustic emissions imaging and synchrotron X-ray diffraction analysis at high pressure and high temperature. Part I: calcite

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Geodynamical processes such as phase transitions, dehydration or melting reactions can potentially be the source of a seismic activity. Here we propose to monitor the acoustic emissions produced by a series of reference materials submitted to various pressures and temperatures in a cubic multi-anvil device (MAX-80) in order to evaluate the potential of the experimental approach. Simultaneous collection of XRD patterns of the sample using the synchrotron radiation allows matching the main acoustic events to mineralogical changes in the sample. In order to check whether these acoustic events are really produced within the sample rather than in the surrounding pressure medium, these events are located using the software *Insite* (ASC Ltd).

In a first approach, we have monitored from in-situ X-ray diffraction coupled to Acoustic Emission (AE) imaging, the behaviour 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 then loaded in the MAX80 cubic multi-anvil press (beamline F2.1, HASYLAB). AE were recorded using five piezoceramic transducers (5 MHz eigen frequency) glued on each of the five WC anvils (4 side anvils and the 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 correlates to a sudden burst of AE, all of which located within the sample volume (Figure 1). These AE may indicate that stress relaxation (through the activation of intra-crystalline plasticity mechanisms) released enough acoustic energy to be recorded and located. Although the diffraction data showed that grain growth continued beyond 800°C (Figure 2), 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 (2000 events). AE location revealed that the AE front progressed inwards the sample. The complete loss of diffraction signal (Figure 2) and the post-mortem recovery of small amounts of CaO suggest that the second AE burst may be related to calcite melting/decarbonation.

![Figure 1: Localization of AE, note that most of the acoustic events locate inside the sample (cylinder 4 mm diameter).](image-url)
For the first time, we have succeeded in combining AE continuous monitoring and in-situ X-ray diffraction in time resolved experiments at HP and HT. Acoustic emissions could be located within the sample and signal generated from the assembly could be ruled out. Actually, the boron-epoxy assembly was found to generate very little acoustic emissions. The study of other reference geomaterials such as quartz, kaolinite and gypsum is under progress. In order to favour the production of AE in pressurized sample, two factors will be explored in the near future with this set-up: 1) the effect of deviatoric stress and 2) the role of grain size in sintered samples. We will especially focus on the role of devolatilization reactions in producing AE.