

Report HS-4312

The natural fully ordered sample used was an optically-clear single rhombohedron of dolomite coming from the Eugui complex (Spain) and the disordered sample consists of a fully stoichiometric EU dolomite thermally treated at 1200°C and 3GPa.

The disorder degree of the samples was checked by means of an Oxford Diffraction Xcalibur diffractometer equipped by CCD detector and MoK α radiation ($\lambda = 0.7107 \text{ \AA}$) at the University of Perugia (Italy). Detector distance to the sample was 66 mm with pixel size 60.6 μm . The crystal quality was checked using rotation pictures while 360° phi-scans allowed optimization of the scan width (0.7°) and scan time (50 sec) for the data collection. Collected data were integrated by means of CrysAlis software (Oxford diffraction, 2009) and the absorption correction was applied by the ABSPACK program as implemented in CrysAlis (Oxford Diffraction 2009). Crystal structure refinements were carried out by means of the SHELX-97 program (Sheldrick 1997) in R3 space group, with anisotropic displacement parameters. Disorder parameter s was 0.948(8) and 0.84(2) for EU and EU1200 respectively. Additionally, the latter showed a high twinning percentage of approximately 50% following the {1120} twinning law.

The experiment was performed at the ID09A beamline at the European Synchrotron Radiation Facility (ESRF) (Grenoble, France). The beamline is equipped with angle-dispersive-diffraction apparatus and diamond anvil cells (DAC). Intensities were collected on a Mar555 image-plate detector (430 x 350 mm, 555 mm diagonal active area).

Three single crystal XRD experiments were performed. The first two were carried out on EU sample at HP up to 16 GPa and HP-HT ($T \sim 300^\circ\text{C}$). The third experiment (hereafter EU1200-HP) was performed under HP conditions on EU1200 sample. A membrane-type DAC apparatus with Ne as pressure-transmitting medium was used. Pressure within DAC was measured by ruby fluorescence method (Forman et al., 1972) as regards HP experiments; whereas a Samarium doped SrB₂O₇ (Sm:SrB₂O₇) phase was used for the HP-HT experiment since it has a lower dependence of fluorescence peaks broadening at high temperature, with respect to the one of Rb (Raju et al. 2008). X-ray wavelength was monochromated at 0.415759 \AA . Single crystals were loaded on 250 μm Re-gaskets and the sample-to-detector distance was 309.7706 mm. Operating conditions for HP data collections were 60° ω -scan, 0.5° step size and 1 s step time.

Compressibility of natural dolomite was studied by a second Birch-Murnaghan (BM) fitting of P-V data finding out $K_0 = 94.1(8) \text{ GPa}$ (K' fixed to 4) in agreement with data from Ross and Reeder (1992). Third order BM Equations of State give $K_0 = 93(3)$ and $K' = 4.2(5)$ for the ordered dolomite, $K_0 = 96(5)$ and $K' = 3.3(8)$ for the disordered dolomite and $K_0 = 92(4)$ and $K' = 2.9(5)$ for ordered dolomite at high temperature conditions.