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High-pressure systematic of NaMe3+Si2O6 pyroxenes: volume compression vs Me3+ cation radius

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Recent investigations have been experimentally demonstrated that Na-clinopyroxenes (Na-cpx) can be stable throughout a wide range of temperatures and pressures in the upper mantle and several works have been carried out in order to better constrain their physical properties. In this work the equation of state of a synthetic NaInSi2O6 clinopyroxene characterized by a trivalent non 3d-transition metal at the Me3+ crystallographic site (space group C2/c) was determined up to about 8 GPa by in situ single-crystal X-ray diffraction. Since previous investigations on CaMe2+Si2O6 showed a different effect when 3d- and non 3d-transition elements located at Me site, the aim of this study is to provide a definitive model capable to predict the high-pressure behaviour of (Ca,Na)(Me2+, Me3+)Si2O6 clinopyroxenes.
A single crystal of NaInSi2O6 with size 150*80*50 microns3 was selected for the high-pressure single-crystal X-ray diffraction study. The sample was loaded in an ETH-type diamond anvil cell assembled with a diamond culet of 600 microns in diameter and a gasket preindented to 90 microns with a spark eroded hole of 200 microns in diameter. The cpx was loaded together with a single-crystal of quartz used as an internal pressure standard and some ruby chips used for more approximate determination of the internal pressure. The measurements were performed using a four-circles STOE STADI IV diffractometer on which the software SINGLE08 has been recently installed allowing to perform the eight-position diffracted-beam centering and to fit the diffraction peak profiles. Such centering procedure allows to obtain precise and accurate unit-cell parameters in order to provide values of room pressure bulk modulus affected by a significantly small error.
The NaInSi2O6 cpx was investigated at 12 different pressures up to 7.83 GPa. No evidences of phase transformation were found throughout the pressure range investigated. The sample compresses anisotropically with the b direction being strongly the softest one (as expected for cpx) and a and c axes compressing by similar rates. Using a third-order Birch-Murnaghan equation of state (BM3) to fit the pressure – unit-cell volume data we could refine simultaneously the unit-cell volume V0, the room pressure bulk modulus KT0 and its first pressure derivative K'. Using EoSFIT5.2 software we obtained the following coefficients: V0 = 463.42(3) Å3, KT0 = 109.0(6) GPa, K' = 3.3(2).
In order to obtain a reliable comparison among NaMe3+Si2O6 pyroxenes, we have plotted the relative compression V/V0 calculated to 10 GPa versus the cation radius of Me3+ site for NaVSi2O6, NaAlSi2O6, NaCrSi2O6, NaFe3+Si2O6 end-members (all C2/c space group). For the NaTiSi2O6 end-member we have performed such calculation using the published P-V data up to 4.34 GPa as at greater pressures this cpx transforms to a triclinic symmetry and cannot be considered for a comparison. According to this comparison, a perfect linear relation is showed for those cpx having a 3d-transition element at Me3+ site, while those cpx with Al and In at Me3+ site totally lie out of trend. In good agreement with a previous work on CaMe2+Si2O6 C2/c compounds (Me2+ = Mg, Fe, Ni), the results of this work confirm that the empirical KT0 * V0 = constant relationship is followed in C2/c cpx only if the same valence electron character is shared and provide a definitive model for (Ca,Na)(Me2+,Me3+)Si2O6 clinopyroxene compounds.