CHARACTERISATION OF STRUCTURAL PHASE TRANSITIONS AND MELTING OF SOME GEOMATERIALS UNDER PRESSURE FROM ELECTRICAL IMPEDANCE SPECTROSCOPY

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The phase transformation in calcite I-IV-V and calcite ⇔ aragonite and in SiO_2 have been characterised by electrical impedance measurements at temperatures 500-1200°C and pressures 0.5 – 2.5 GPa in a piston cylinder apparatus. The bulk conductivity σ has been measured from Argand plots in the frequency range 10^5 - 10^{-2}Hz. The cell represents a coaxial cylindrical capacitor with a geometric factor c. 6 cm [1]. As a starting material the synthetic polycrystalline powder of $CaCO_3$, natural crystals of calcite (Spain) and of quartz Mont Rose (France) were used. The transformation temperature was identified from resistivity-temperature curves as a kink point of the activation energy. In a low temperature phase (calcite I), the activation energy E_a of σ x T is c. 1.05±0.05 eV, and in high temperature phase (calcite V) E_a is c. 0.75±0.05 eV. The pressure dependence of T_c for the transformation in calcite V is positive for pressures up to 1 GPa dT_c/dP ~ +20°/GPa and becomes negative for the pressures 1-2 GPa: -20 °C/GPa. The transformation calcite I -IV is less evident from the present observations. The activation energy of electrical conductivity between I and V phases increases gradually to from 0.95 to 1.05-1.15 eV and then decreases to c. 0.75 eV. The gradual increase of E_a may correspond to a kinetic phenomena of retaining R-3c symmetry at temperatures above c. 850°C. However, the pressure dependence of the kink of the activation energy is also + 20°/GPa. The kinetics transformation of calcite in aragonite has been monitored by measuring a variation of the electrical resistance of calcite at 10^3 Hz with time in the stability field of aragonite. Products of the phase transformation have been analysed with a powder diffractometer. Variations of the electrical resistance with time correlate with the degree of phase transformation ξ (t). The degree of transformation calcite to aragonite has been fitted to the expression: $\xi(t) \sim [1- \exp(-t/\tau)]$, where τ is a characteristic time of the phase transformation at a temperature T. For example, τ of the transformation of calcite to aragonite at 2.5 GPa and 760°C is 7.5 h, at 1.5 GPa and 680°C is 8.8 h.
The temperature of α–β-phase transition in quartz has been characterising at 0.5, 1, 1.5 and 2 GPa from the electrical impedance measurements of polycrystalline samples. The difference in activation energies of the *bulk electrical conductivity x T* in α- and β- quartz is rather small and increases with the pressure. For example at 0.5 GPa the activation energies of σ·T for α and β-phase are 1.04 eV and 1.26 eV, at 1 GPa 1.1 eV and 1.3 eV, at 1.5 GPa 1.18 eV and 1.41 eV, at 2 GPa 1.23 eV and 1.53 eV, respectively. The calculated activation volume ΔV for α- and β-phases is about the same 0.18 and 0.25 cm^3/mol. The estimated phase transition temperature T_c from heating and cooling cycles are slightly different. Phase transition temperature obtained in this study from heating and cooling cycles is smaller than has been reported from laser interferometer [2] and close to the T_c data in [3].

References

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