Characterization of the high-temperature modifications of incommensurate tridymite L3-T_o(MX-1) from 25 to 250 °C

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Abstract

Incommensurate tridymite L3- T_0 (MX-1) shows a cascade of five phase transitions at 65, 110, 150, 200, and 380 °C upon heating. The X-ray diffraction patterns were investigated with a Buerger precession camera revealing a sequence of four incommensurate phases in the range from room temperature to 200 °C. The phases formed between 65 and 110 °C and between 110 and 150 °C are new modifications of tridymite. At 65 °C the monoclinic tridymite L3-T₀(MX-1) phase undergoes a first-order transformation to an orthorhombic phase. The incommensurate structural modulation of the room-temperature phase with the wavevector $\mathbf{q}_1 = 0.663 \ \mathbf{a}^* - 0.498 \ \mathbf{c}^*$ flips to $\mathbf{q}_2 = 0.042 \ \mathbf{a}^* - 0.388 \ \mathbf{c}^*$. Simultaneously, a commensurate modulation with tripled b lattice parameter is formed. The wavelengths of both modulations do not depend appreciably on the temperature. The incommensurate modulation discontinuously disappears near 110 °C whereas the commensurate modulation along the **b** axis becomes non-integral with a temperature-dependent wavelength varying between 115 and 100 Å. At 150 \circ C the symmetry is reduced to monoclinic again with γ = 90.4°. Between 150 and 200 °C the monoclinic angle gradually decreases to 90° and the wavelength of the modulation from about 90 to 65 Å. At higher temperatures, the satellite reflections fade into weak streaks and the normal orthorhombic high-temperature modification of tridymite is formed.

The phase transitions are reversible upon cooling except for the first transformation that is partly irreversible for single crystals and reversible but incomplete for pulverized material at room temperature.