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GRADUAL RECRYSTALLISATION OF METAMICT FERGUSONITE: X-RAY DIFFRACTION AND RAMAN SPECTROSCOPY STUDY

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Fergusonite mineral samples, mainly represented as $YNbO_4$, are frequently metamict. Heating experiments induce crystal structure recovery, but also cause phase transition processes, since fergusonite is a polymorphic substance (WOLTEN & CHASE, 1967).

X-ray powder diffraction and Raman spectroscopy were employed to monitor the sequences of fergusonite recrystallisation. The corresponding Raman spectra were also used to reveal possible presence of original structure remnants in the metamict mineral. Two metamict mineral samples previously identified as fergusonite were heated in air at 400, 500, 650, 800, 1000 and 1300°C for 24 hours in each case.

The mineral sample from Bakkane-Steane, Norway, was completely amorphous to X-rays and started to recrystallise at 400 °C with scheelite type structure (space group $I4_1/a$) being stable up to 1000 °C. At 1000 °C monoclinic (space group $I2$) β-fergusonite dominated, with the tetragonal phase still present. At 1300 °C the transformation from tetragonal to monoclinic fergusonite was completed. Raman spectra confirmed the gradual fergusonite recrystallisation by sharpening and intensifying of vibrational bands. The number of bands increased with the lowering of the symmetry. For the metamict mineral the vibrational bands at 779, 685, 697, 310, 208 and 108 cm^{-1} were present indicating the residue of the original crystal structure. At lower heating temperatures the bands were broad, but at higher temperatures reappeared more sharpened, resolved and intensified. The second mineral sample, originating from Ytterby, Sweden, was almost completely metamict, with only a few low-intensity diffraction lines superimposed on the characteristic amorphous diffraction peak. At 400 °C pyrochlore phase started to recrystallise, being continuously present through the whole temperature range. At 1000 °C the monoclinic β-fergusonite appeared with recrystallisation completed at 1300 °C. Raman bands were similar to those of fergusonite from Bakkane-Steanne, although some of them had different intensities and were slightly shifted. Vibration bands were not present in the spectrum of the unheated metamict mineral.

The vibrational spectra of the complex oxide structures are difficult to calculate, but a relation to the analogous synthetic compounds (YASHIMA et al., 1997) could be established. McCONNELL et al. (1976) indicate that LO and TO Nb-O stretching modes of niobium oxides appear in the range 1010-620 cm^{-1} Therefore, the observed lattice vibrations reveal partial preservation of Nb-O polyhedra stacking in metamict fergusonite from Bakkane-Steane.

References

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