CRYSTAL GROWTH AND STRUCTURE CHARACTERIZATION OF RARE EARTH DOUBLE TUNGSTATE

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Monoclinic KEr(WO₄)₂ (KEW) and KSm(WO₄)₂ (KSW) single crystals have been grown by the Top Seeded Solution Growth (TSSG) method. $K_2W_2O_7$ solvent containing up to 25% mol of crystallized compounds was used. This solvent is characterized by a very broad temperature range of crystallization, good transparency and it does not introduce additional ions into the flux. High purity materials: K_2CO_3 , WO_3 and Er_2O_3 or Sm_2O_3 (respectively) ware mixed and loaded in to platinum crucible 50x50mm, with 1 mm thick walls. The mixtures were homogenized by keeping the solution at the temperature of 30° C above saturation temperature for about 32 h. The temperature was stabilized at $\pm 0.3^{\circ}$ C. The radial gradient inside the solution was about 0.2° C/mm and vertical in the range $0.2 - 0.5^{\circ}$ C/mm. The saturation temperature was found at 860° C and 920° C, respectively for KEW and KSW. Rotation rate decreased with increasing diameter of growing crystal from 50rpm at the beginning down to 20rpm. Crystals were pulled up at a rate of 1.5 mm/day. The cooling rate was 0.05° C/h at beginning and then was increased to hold proper diameter.

Structure analysis of obtained crystals was performed by X-Ray powder diffraction using Ni-filtered Cu K α radiation with a Siemens D5000 diffractometer. Data were collected in the angle range $20^{\circ} < 2\theta < 144^{\circ}$ with a step 0.02° and averaging time of 10s/step. The diffraction patterns were analyzed by the Rietveld refinement method. XRD measurements showed the symmetry of monoclinic space group C2/c. The unit cell parameters obtained for KEW crystals are a = 10.61554 Å, b = 10.31332 Å, c = 7.53631 Å, β = 130.750° , V = 625.058 Å 3 and for KSW crystals are a = 10.72871 Å, b = 10.50827 Å, c = 7.62685 Å, β = 130.788° , V = 651.026 Å 3 .

References

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