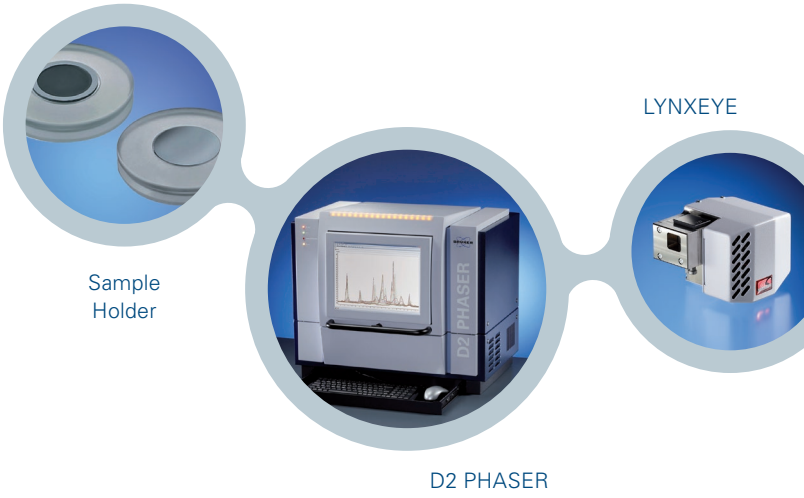

Application Report XRD 9

D2 PHASER Desktop XRD: Crystal Structure of Er-Melilite

The D2 PHASER is a portable desktop XRD instrument for research and quality control. It is easy to operate and independent of external media such as cooling circuits. Thanks to the LYNXEYE detector it is the fastest desktop XRD system on the market.

This report demonstrates its use for investigating crystal structures applying the fundamental parameters approach in the TOPAS software.

Tab. 1: Experimental settings.

D2 PHASER, LYNXEYE detector
Cu radiation (30 kV, 10 mA), Ni filter
Continuous scan from 10 to 100° 2Theta Step width 0.02° Total counting time 2.5 sec per step
Total scan time about 80 min.
2.5° Soller collimators, 0.6 mm divergence slit, anti-scatter screen
LYNXEYE detector opening 5° 2Theta

X-ray powder diffraction helps understanding the properties and the crystal chemistry of new tailor made materials of which no single crystals are available. This information can be accessed from the intensities of the diffraction peaks using the TOPAS software.

Structural variations related to the substitution or Si and Ca against Al and trivalent Lanthanoid ions in the mineral melilite, a layered alumino-silicate with chemical formula $Ln_xCa_{2-x}Al[Al_{1+x}Si_{1-x}O_7]$ $0 \le x \le 1$ and Ln = La, Eu, Er, were recently studied [1]. They form solid solutions and are potential laser materials with interesting optical properties.

About 10 mg Er-melilite of nominal composition x=0.5 were prepared on a low background Si sample holder and measured with the D2 PHASER. Experimental details are given in Tab. 1. The crystal structure, isotropic thermal displacement parameters of the atoms and the unit cell parameters were refined using DIFFRAC TOPAS v4. The fundamental parameters approach (FPA) was used for modeling the resolution function of the D2 PHASER.

Figure 1 presents the TOPAS plot. Refined structural parameters are plotted vs. literature data [1] in the inset. The high

data quality and the excellent refinement become obvious from the only minor deviations from linearity. From the cell parameters c/a ratio a composition of x=0.52 is calculated, in excellent agreement with the nominal value 0.5. The little residuum between calculated and measured data suggests that FPA perfectly models the measured peak shape of D2 PHASER data.

To conclude, our cost-effective desktop XRD system D2 PHASER allows for accurate, FPA based Rietveld analysis applying the TOPAS software.

[1] Peters, L., Knorr, K. & Depmeier, W.: Z. Anorg. Allg. Chem. 2006, 632, 301-6

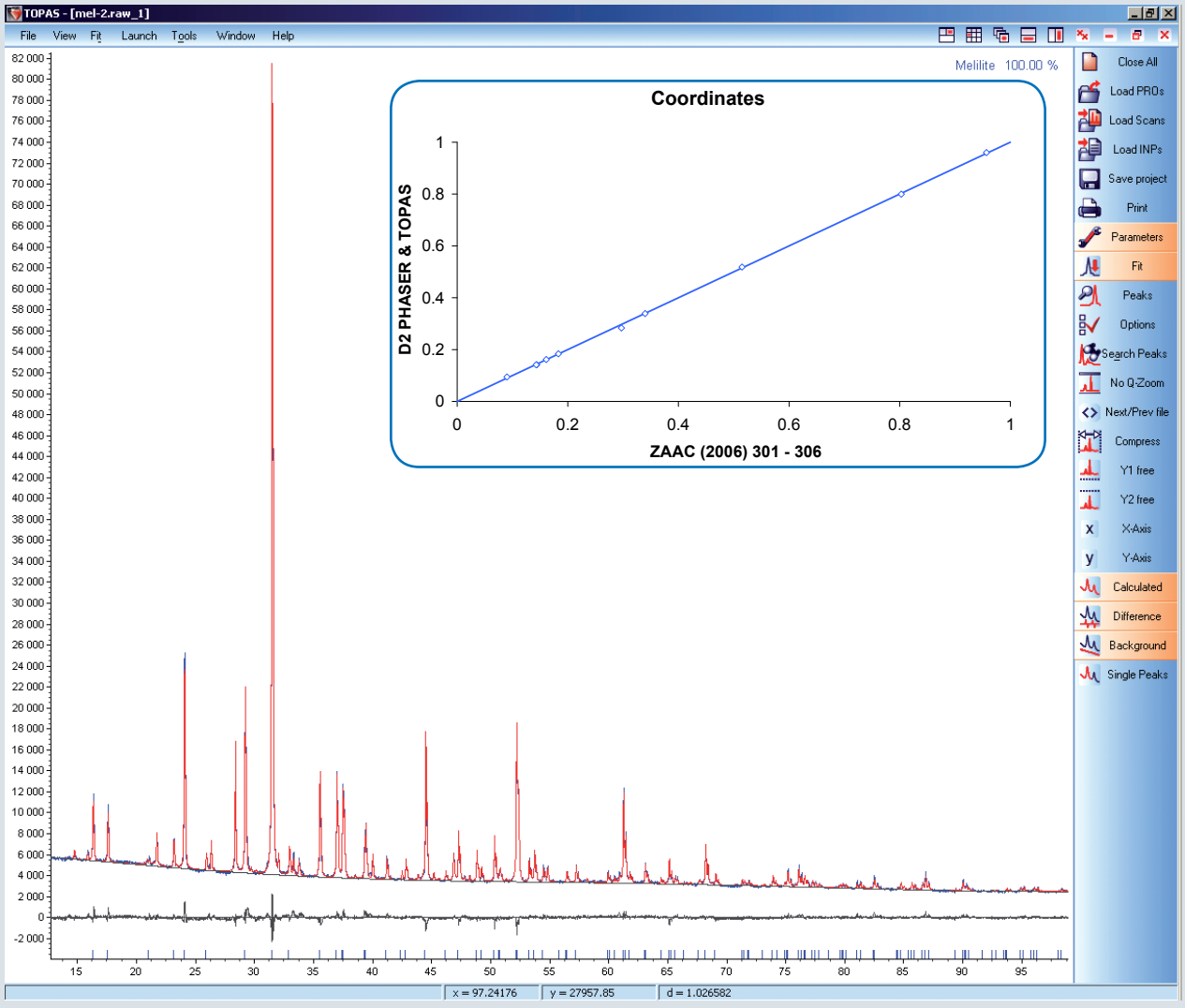

Fig. 1: TOPAS plot of x=0.5 Er-melilite. Lattice parameters of the tetragonal unit cell are a=7.6931(9) Å and c=5.0550(6) Å. The refined fractional coordinates are plotted in the inset vs. literature data [1]. Agreement parameters of the TOPAS refinement are Rwp=3.14, Rp=2.38, and GoF=2.02.

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