Supplementary materials for:

Lithium diffusion in lithium nitride by Pulsed-Field Gradient NMR

The composition of our sample has been checked before and after the heat treatment and high temperature NMR experiment by XRD, after removal from the BN crucible, using a Bruker D8 Advance X-ray diffractometer (Cu Kα) with an airtight sample holder. We show here the XRD spectrum of our starting material:

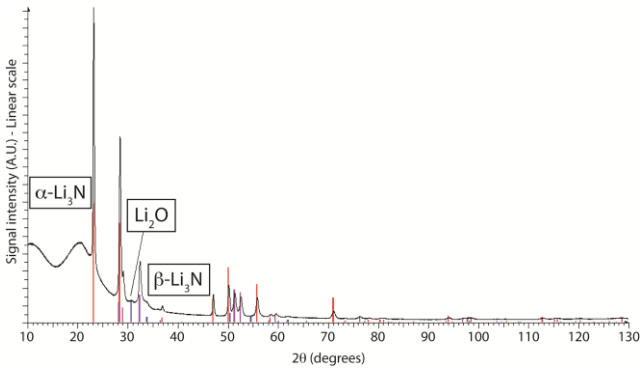

Fig. SI 1 XRD spectrum of the commercial Li_3N starting material, featuring α-Li_3N and β-Li_3N with a very small amount of Li_2O. The use of a protective dome during the XRD experiments induces the distortion in the baseline.

The cell parameters and the quantitative analysis of impurities in the sintered material were obtained by XRD Rietveld refinement using Topas. Cell parameters, sample displacement, scale factor, and profile parameters were refined along with up to five background parameters with α-Li_3N (space group P6/mmm), Li_2O (space group Fm-3m), BN (space group R3mH) and three structures models, where BN comes from the BN crucible for the diffusion experiment. For the sample after the high temperature experiment, the R-factors of the Rietveld refinement are Rp = 6.42%, Rwp = 9.38% and RB = 2.77%, 0.52%, 2.39% and 1.16% for the three structure models, respectively. It shows 87.56 w% of Li_3N, 11.45 w% of Li_2O and 0.99 w% of BN, corresponding to 85.5, 13 and 1.5 mol% respectively. For lithium, this corresponds to a fraction of 90.8 mol% in Li_3N and 9.2 mol% in Li_2O. The pulse sequence we used combined stimulated echo bipolar, gradient pulses and longitudinal eddy-current delay (STE-BP-LED) and is shown below:

Fig. SI 2 STE-BP-LED pulse sequence, indicating the trapezoidal gradient pulses (yellow squares) of maximum strength *g* (from 50 to 1100 G/cm) and duration δ, beginning with a ramp from 0 to *g* and ending with a ramp from *g* to 0 in both 100 µs, included in δ (1 to 1.2 ms duration). The RF pulse power was set to11 kHz, Δ corresponds to the diffusion delay (10 to 12 ms) and τ is the echo time (420 µs). To reduce eddy-current distortions, a longitudinal eddy-current delay (led) of 3 ms was used.

The fits and the standard errors have been obtained with the following Maple 16© worksheet:
Supplementary_Materials_MAPLE_Diffusion_Li3N.mw