

## Analysis of LIB cathodes by using MATPASS with ultra fast MAS

Product used : Nuclear Magnetic Resonance (NMR)

NMR is extremely useful for characterizing local structure of materials for lithium ion battery (LIB) because it is able to observe lithium ions directly. However, a solid state NMR analysis of cathode materials using  $^7\text{Li}$  by conventional MAS frequency ( $\sim 20$  kHz) has been difficult due to a series of spinning side band (SSB) originated from paramagnetic shift anisotropy.

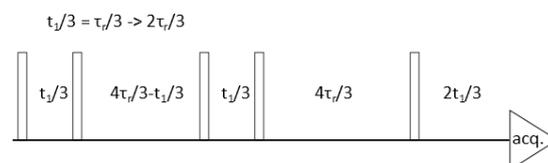
The ultra-fast MAS probes (1mm and 0.75mm) give high resolution and less SSB  $^7\text{Li}$  spectra as shown in an application note NM-140020. However, for many of LIB cathode materials the ultra fast MAS alone may not be sufficient to obtain spectra without effect of SSBs. An excellent solution to avoid the problem is MATPASS (Magic Angle Turning – Phase Adjusted Spinning Sideband) technique developed by Hung et al. <sup>1</sup> The experiment allows SSB free  $^7\text{Li}$  spectra of paramagnetic cathode materials. This technique is available for moderate speed MAS probes, but the ultra fast MAS probe still has great advantage because it allows wide range excitation leading to quantitative spectrum and experiment with small sample amount.

### Ultra fast MAS + MATPASS

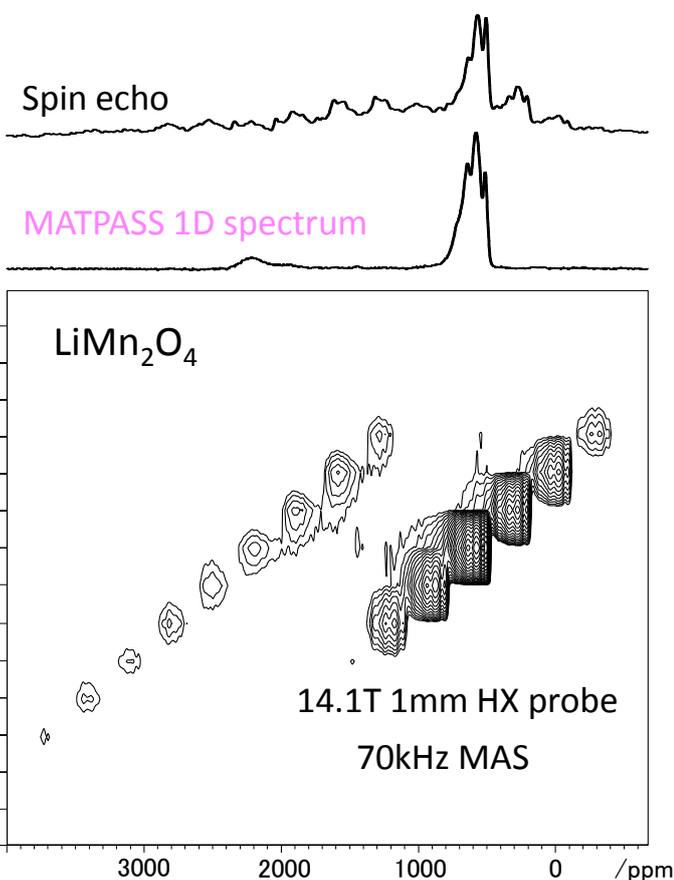
- ✓ SSB free  $^7\text{Li}$  spectrum
- ✓ Wide range excitation
- ✓ Small sample amount



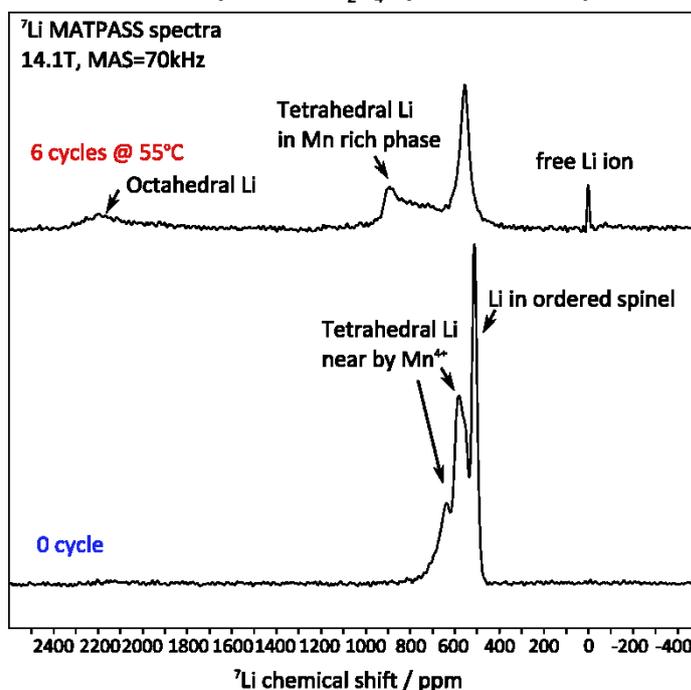
1mm sample tube



1. I. Hung et al., *J. Am. Chem.Soc.* 2012, **134**, 1898–1901



### Deterioration analysis of $\text{LiMn}_2\text{O}_4$ by $^7\text{Li}$ MATPASS spectra



Samples before and after cycles were kindly provided by Prof. Y.Oka (Univ.Hyogo)

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