Probing the Oxygen Proximities of γ-Al_2O_3 of Under Ultra-High Magnetic Field (35T)

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Introduction

γ-Al_2O_3 is of great importance in heterogeneous catalysis, which is widely applied either as catalysts or supports in a wide range of important industrial reactions. The knowledge on the surface oxygen species is critical for the understanding of the structure and property of γ-Al_2O_3, which however, remains as a challenging task. Our previous work characterized the surface oxygen species by DNP-surface-enhanced NMR spectroscopy (DNP-SENS).[1] In this report, we show that the proximities between different oxygen species of γ-Al_2O_3 can be unambiguously investigated by the first well-resolved 2D ^{17}O double quantum (DQ)- single quantum (SQ) homonuclear correlation spectra obtained under ultra-high magnetic field (35T).

Experimental

^{17}O enriched (ca. 10% labeled) of γ-Al_2O_3 samples were prepared according to previously reported method on oxides.[2] ^{17}O MAS NMR experiments were performed using the 35T (1.5G MHz) spectrometer equipped with a 3.2 mm MAS probe at the NHMFL.

Results and Discussion

Although increasing the strength of the magnetic field will exacerbate the offset effect on the low-power recoupling experiment, the remarkable improvement of NMR sensitivity at 35T (1.5G) permits recording of a 2D ^{17}O DQ-SQ homonuclear correlation spectrum (Fig. 1) of γ-Al_2O_3 in 1.2 hrs. by using the $BR2^1_2$ sequence with WURST central-transition enhancement.[2,3] Besides the auto-correlations from tetrahedral oxygen or tri-coordinated oxygen species separately, the cross-correlations ambiguously provide direct evidence for the spatial proximities between tetrahedral oxygen species and tri-coordinated ones. Combined with our previous DNP results, it allows us to readily identify topology structure of oxygen species in γ-Al_2O_3

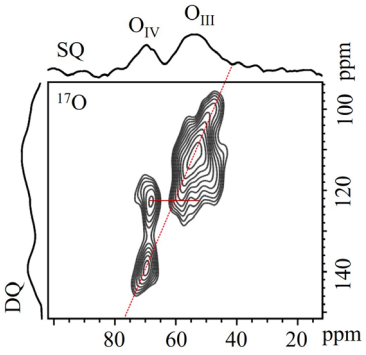

Fig.1 ^{17}O DQ-SQ homonuclear correlation spectrum of γ-Al_2O_3 at 35T.

Conclusions

Benefitting from the dramatic improvement of the NMR sensitivity, the through-space correlation information of complex oxygen species in γ-Al_2O_3 were explored by the first 2D ^{17}O DQ-SQ homonuclear MAS NMR experiment. It should provide a feasible and valuable avenue for the structural characterization of oxygen containing materials.

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References

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