NMR evidence for gapless quantum spin liquid state in the ideal triangular-lattice compound Yb(BaBO$_3$)$_3$

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(Dated: December 10, 2019)

In this paper, we employ nuclear magnetic resonance as a local probe to present the first experimental identification of the gapless quantum spin liquid ground state in the two dimensional triangular lattice antiferromagnet Yb(BaBO$_3$)$_3$. One evidence is the absence of any spin ordering or freezing with temperatures down to $T = 0.26$ K, far below its Curie-Weiss temperature $\theta_w \sim 2.3$ K. From the spin dynamics, precise temperature-independent spin-lattice relaxation rates is observed at low temperatures under a weak magnetic field, indicating the gapless spin excitations and the strongly correlated quantum disordered phase. This fact supplies the other more important evidence for the QSL in our samples. Under higher magnetic fields, a spin excitation gap is induced, whose gap size is directly proportional to the field intensity. The origin for this field induced spin gap is thoroughly discussed.

Quantum spin liquid (QSL) refers to the highly non-trivial state of matter, where spins are highly entangled and strongly fluctuate but never order or freeze even at zero temperature$^{[1]}$. Various novel quantum properties such as fractional spin excitations, topological order, et al. can be expected in the QSLs$^{[2]}$. Besides, the research on the QSLs is believed to be of significant importance for solving the puzzle of high-$T_c$ superconductivity$^{[3, 4]}$ as well as the quantum communication$^{[5]}$. Antiferromagnetically coupled small spins on the geometrically frustrated triangular or kagome lattice have supply people with promising route to realizing the QSL state.

Several geometrically frustrated antiferromagnets are argued to be the promising QSL candidates, including the hot materials of ZnCu$_3$(OH)$_6$Cl$_2$[6, 8] and Cu$_3$Zn(OH)$_6$FBr[9] with kagome structure, and $k$-BEDT-TTF)$_2$Cu$_2$(CN)$_3$[10, 12], EtMe$_3$Sb[Pd(dmit)$_2$]$_2$[13, 14] and the newly discovered YbMgGaO$_4$ with the triangular lattice. To experimentally identify the QSL ground state, these two aspects should be confirmed: one is the absence of magnetic order or spin freezing at sufficient low temperatures as compared with the antiferromagnetic coupling strength; the other key feature is the fractional spin excitations related with the long-range spin entanglement. However, the anti-site disorder effect in a real sample often makes the situation be very complex.

The ground state of YbMgGaO$_4$ is still highly controversial. The Curie-Weiss temperature $\theta_w \sim -4$ K is indicated from $dc$ susceptibility measurement, while no long-range magnetic order is observed down to ultra-low temperatures[16]. The magnetic excitation contributed specific heat shows a power-law behavior $C_m \propto T^\alpha$ with $\alpha \approx 2/3$. Additionally, a broad continuum in the spin excitation spectrum is observed from inelastic neutron scattering$^{[17]}$. Consequently, the gapless U(1) spin liquid with spinon fermi surface is proposed for the ground state of YbMgGaO$_4$[18]. However, no significant magnetic excitation contribution is observed in the thermal conductivity of YbMgGaO$_4$[19]. In its isostructural counterpart YbZnGaO$_4$ with very similar properties, the magnetic exchange coupling is found to be as weak as $~0.1$ meV$^{[20]}$, while the disorder effect due to the random mixing of Mg$^{2+}$/Zn$^{2+}$ and Ga$^{2+}$ can strongly influence the ground state through its significant impact on the local environment of Yb$^{3+}$. The $ac$ susceptibility measurements on both compounds show frequency-dependent peaks around $0.1$ K, indicative of the spin-glass ground state$^{[20]}$. A recent theoretical study shows that the disorder-free YbMgGaO$_4$ should exhibit a robust collinear magnetic order. It is the mixed Mg$^{2+}$/Zn$^{2+}$ and Ga$^{2+}$ site disorder effect that results in the spin disorder and give birth to the spin-liquid like behavior$^{[21]}$.

To clarify the controversial results about the ground state in YbMgGaO$_4$, studying new materials containing Yb$^{2+}$-triangular lattice without inherent structural disorder is an alternative way with significant importance. In this letter, we employ nuclear magnetic resonance (NMR) as a local probe to perform detailed study on the static magnetism and spin dynamics in the structural-disorder-free triangular lattice antiferromagnet Yb(BaBO$_3$)$_3$. The Curie-Weiss fit to the low temperature $dc$ susceptibility gives a value of $\theta_w \sim -2.3$ K$^{[22]}$, comparable to that in YbZnGaO$_4$. While neither spin ordering nor freezing is observed with the temperature down to 0.26 K evident by spectral analysis and spin-lattice relaxation rate measurements, yielding the frustration factor $|\theta_w|/T_N > 8.8$. For the spin dynamics, the spin-lattice relaxation rates show a temperature-independent behavior for 0.26 K$< T < 25$ K under a weak magnetic field.
This is a strong indication for the gapless spin excitations and a constant sum of dynamic spin susceptibility in the momentum space, consistent with the strongly correlated quantum disordered phase. With strengthening applied magnetic field, a spin excitation gap is induced, whose gap size is found to be proportional to the field strength. This spin gap should originate from the spin-wave excitations, as the spinon description of the excitation fails in the strong field regime. These facts supply strong evidence for the gapless QSL ground state in the newly discovered Yb(BaBO$_3$)$_3$ without inherent structural imperfection.

Both polycrystal and single crystal of Yb(BaBO$_3$)$_3$ are used in this work. The polycrystal are synthesized by the solid reaction method and the single crystals are obtained by the conventional flux method. For the NMR study, about ten milligram of polycrystal and single crystals with typical dimensions of 4 $\times$ 4 $\times$ 0.1 mm$^3$ are selected. The single crystal is placed on a piezoelectric nano-rotation stage for precise alignment of the field direction. Our NMR measurements are conducted on the $^{11}$B nuclei($\gamma_B = 13.655$ MHz/T, $I = 3/2$) with a phase-coherent NMR spectrometer. The spectrum is obtained by summing up or integrating the spin-echo intensities at different frequencies or magnetic fields. The spin-lattice relaxation rate is measured by the standard inversion-recovery method.

The Yb(BaBO$_3$)$_3$ crystalizes in the hexagonal structure with space group $P6_3cm$ [22] (See Fig.1(a)) and the supplemental material]. We stress that the inherent structural disorder, such as the anti-site mixing of the nonmagnetic Mg$^{2+}$/Zn$^{2+}$ and Ga$^{3+}$ observed in YbMgGaO$_4$ and YbZnGaO$_4$ [22,24], is completely absent here, as the ionic radius is very different. For the magnetic layer, the corner-sharing YbO$_6$ octahedra and BO$_3$ form a very stable framework, strong disorders in the magnetic layer as that occurs in ZnCu$_3$(OH)$_6$Cl$_2$ [23, 26] is also not likely present here.

Typical single crystal $^{11}$B NMR spectra are presented in Fig.1(c) for both field directions. With an in-plane magnetic field, the spectrum is composed by two groups of peaks denoted by the down arrows and shadows. The $^{11}$B nucleus has a nuclear spin of 3/2, thus we can expect three NMR transitions for the nucleus in a non-zero local electric field gradient (EFG). In Yb(BaBO$_3$)$_3$, there exist three inequivalent $^{11}$B sites, with the interlayer and in-plane B sites respectively denoted by B2 and B1/B3 in Fig.1(a). The nuclei contributing to the sharp $\alpha$ peaks have a weaker hyperfine coupling with the magnetic layer, as evidenced by the nearly temperature independent Knight shift (the relative line shift with respect to the Larmor frequency, shown below), and a much slower spin-lattice relaxation rate as compared with the $\beta$ peaks. Hence, the $\alpha$ peaks can be assigned to the B2 site, and $\beta$ peaks are from the in-plane B1/B3 site. Under a field along the crystalline c-axis, the $\beta$ peaks from B1/B3 site smear out and become undetectable as a result of too fast spin-spin relaxations, again confirm their strong coupling to the magnetic site. The nuclear quadruple resonance frequency $\nu_Q$ is calculated to be $\sim$ 1.34 MHz for B2 and $\sim$ 1.31 MHz for B1/B3 at $T = 1.7$ K.

We present the full spectrum of both the single crystal and polycrystal respectively in Fig.2(a) and (b). The full width at half maximum (FWHM) of the central transition for the B2 and B1/B3 peaks are respectively 10 kHz and 19 kHz at $T = 50$ K. In the recently discovered QSL candidate material NaYbS$_2$ [27], the FWHM of $^{23}$Na central transition in the single crystal is calculated to be $\sim$ 50 kHz at $T = 120$ K. The narrow line width in our sample demonstrate the high homogeneity of our single crystal samples. For the polycrystal, the well-defined powder pattern is observed due to the random distribution of the crystalline axis with respect to the field direction.

Neither spin ordering nor freezing behavior is observed with the temperatures down to $T = 0.26$ K, despite the strong antiferromagnetic correlations between Yb$^{3+}$ moments. With the sample cooling down to $T = 0.26$ K, both the central transitions of the single crystal shift to a higher frequency (Fig.2a)), and the peaks originating from the B1/B3 sites in the polycrystal moves to the low field side. These behaviors indicate the enhancing spin susceptibility at low temperatures, consistent with $dc$ susceptibility measurements [22]. The slight line broadening at low temperatures results from the combined effect of the increasing Knight shift, different hyperfine coupling strength between B1 and B3 sites, as well as some minor disorder effect inevitable in the sample synthesis. During the cooling process, neither noticeable spectral weight loss nor any peak-splitting due to spontaneous
The field-swept $^{11}$B NMR spectra of the single crystal under a 2 Tesla in-plane field. (b) The field-swept $^{11}$B NMR spectra of the polycrystal under a 2 Tesla field. (c) The Knight shift with the magnetic field perpendicular to the crystalline c-axis as a function of temperature. The saturated Knight shift as a function of field intensity is shown in the inset. The hollow circles for $\mu_0 H = 12$ T and 15 T is deduced from the level-off behavior of the Knight shift below $T = 4$ K. The red star denotes the expected Knight shift at zero field limit (See the text).

Symmetry breaking is observed in the single crystal for the entire temperature range studied in this work. For the polycrystal, the rectangular line shape typical for the magnetic order in the powder sample is also not observed with the temperature down to 0.27 K. These facts supply strong evidence for the absence of spin ordering or freezing, supporting the existence of the QSL state.

To study the intrinsic spin susceptibility, we further plot the Knight shift as a function of temperature in Fig.2(c) under different field intensities. With the sample cooling down, all the Knight shifts share a similar temperature dependence, first increase mildly and begin to level off at low temperatures. Under high magnetic fields, the temperature for the level-off behavior appearing further rises and the saturated Knight shift is suppressed (see Fig.2(c) inset). We further extrapolate the saturated Knight shift to zero field limit (shown by the * symbol), and its temperature dependence is proposed by the dashed line in Fig.2(c). Actually, the low-temperature level-off behavior of the spin susceptibility and its field dependence are analogous to what observed in dc susceptibility measurements in the recent Yb-based QSL candidate materials YbMgGaO$_4$ and NaYbS$_2$ with triangular lattice hosting $j_{\text{eff}} = 1/2$ spins$^{27,28}$. The level-off behavior and its field dependence is related with the gapless spin excitations in the highly anisotropic spin system and their suppression under applied field.

Another important typical characteristic for the QSL ground state is the fractional quantum spin excitation (spinon), besides the absence of magnetic ordering at ultra-low temperature. The spin lattice relaxation rate $(T_1)^{-1}$ formulated as $(T_1)^{-1} \propto T \sum_q |A(q)|^2 [\chi''(q,\omega_L)]$, is good probe for the low-energy spin fluctuations in solids, where $A(q)$ and $\chi''(q,\omega_L)$ respectively denote the hyperfine coupling tensor as a function of the wave vector $q$ and the imaginary part of the dynamic susceptibility at the nuclear Larmor frequency $\omega_L$. In Fig.3 we present the temperature dependence of $(T_1)^{-1}$ under a wide field intensity range to gain insights into the spin excitations in Yb(BaBO$_3$)$_3$. With the sample cooling from room temperature to $T \sim 70$ K, the $(T_1)^{-1}$ strongly increases, and show a field-independent characteristic. Below $T \sim 70$ K, the $(T_1)^{-1}$ begins to flatten out, and show a precise temperature-independent behavior below $T = 10$ K down to 0.26 K under a in-plane 0.51 Tesla weak magnetic field. With strengthening the magnetic field intensity, the $(T_1)$ drops gradually at low temperatures, and show a fan-like shape. For both our single crystals and polycrystal, very similar behavior is observed (See Fig.3(a) and (b)), confirming these phenomena to be completely intrinsic. The slight difference between the numerical values of $(T_1)^{-1}$ results from the
mixing of signals of B1/B3 sites with that from intercalated B2 sites, whose hyperfine coupling is much weaker.

The \((11T_1)^{-1}(T)\) behavior for the high temperature region \((70 \text{ K} < T < 300 \text{ K})\) results from the slowing down of spin fluctuations contributed by the thermally excited Yb\(^{3+}\) multiplet with strong correlations. This is consistent with the previous reported large \(|\theta_w|\) obtained from the high temperature dc susceptibility \(^{22}\). More intriguing is the quantum spin excitations reflected in the low temperature \((11T_1)^{-1}(T)\) behavior. The temperature independent \((11T_1)^{-1}\) directly indicate the maintaining spin excitations down to the temperature far below \(|\theta_w| \sim 2.3 \text{ K}\). Neither \(\lambda\)-like critical slowing down behavior typical for spin ordering nor "hump"-like slow spin dynamics resulting from spin freezing \(^{29}\) is observed in our sample, again demonstrating the strong magnetic frustration effect. Actually, this behavior is reported previously in YbMgGaO\(_4\) \(^{18}\), NaYbSe\(_2\) \(^{20}\), NaYbO\(_2\) \(^{31}\) as well as other geometrically frustrated magnets \(^{32}\) \(^{33}\). The temperature independent spin-lattice relaxation rate demonstrate a constant sum of dynamic spin susceptibility in the momentum space, indicating the gapless quantum spin excitations in our sample. As the Knight shift mainly measures the spin susceptibility at \(T = 0\), both the constant Knight shift and \((11T_1)^{-1}(T)\) at low temperature limit imply that the spin fluctuation should have a weak \(T^2\) dependence. These two fact are consistent with the U(1) QSL ground state with spinon fermion surface \(^{36}\) \(^{38}\).

For field up to \(\mu_0H = 15.8 \text{ T}\), typical behaviors for the spin ordering or freezing, such as line splitting or mild broadening in the spectrum and the slowing down of spin fluctuations from the spin-lattice relaxation measurement, are completely absent in our samples (data not shown). This indicates that the spin system stays far away from the magnetic instable point, in sharp contrast with that in the recently discovered \(J_{\text{eff}} = 1/2\) triangular lattice NaYbO\(_2\) \(^{41}\) \(^{42}\), NaYbSe\(_2\) \(^{43}\), CsYbSe\(_2\) and the Kitaev QSL candidate material \(\alpha\)-RuCl\(_3\) \(^{44}\) \(^{45}\), where the magnetically ordered state can respectively be induced or fully suppressed by a moderate applied magnetic field. For the field higher than 5 Tesla, double \(T_1\) components are needed to fit the nuclear magnetization recovery curve below \(T \sim 1.7 \text{ K}\), and both the \((11T_1)^{-1}\) values are also shown in Fig.\(3\) (a). We think the component with faster relaxation rates is contributed from the tiny disorder effect, corresponding to the in-gap state observed in neutron scattering \(^{10}\). However, no such in-gap state is observed in our polycrystal samples, suggesting a better homogeneity.

We step further to explore the properties of novel quantum excitation by tracing its evolution under different magnetic field. In Fig.\(4\) (a) and (b), we plot the \(\ln((11T_1)^{-1})\) versus \(T^{-1}\), and fit the data with a linear function to demonstrate the magnetic field induced spin excitation gap. The gap size obtained from the fittings is shown in Fig.\(4\) (c) as a function of the field intensity. Surprisingly, the spin excitation gap size is simply proportional to the applied field intensity. One scenario for the physical mechanism is the Zeeman splitting of the spinon fermi surface in the magnetic field. Recent theoretical study suggests that the spinon bands splits in magnetic field due to the Zeeman effect, and the energy gap is directly proportional to the field intensity \(^{46}\). However, this theory is based on the fact that the spinon remains a good description of the spin excitation in the weak field regime \(^{39}\). The well-defined dispersive spin-wave excitation is already observed by the inelastic neutron scattering for fields above \(\mu_0H = 7.8 \text{ T}\) in YbMgGaO\(_4\) \(^{40}\). For the present sample, the saturated magnetic field is comparable to that in YbMgGaO\(_4\) as seen from the susceptibility data \(^{22}\). The QSL state should not be preserved for such a high magnetic field, and this field dependence of the gap size may have no obvious connections with the spinon excitations. Thus, the most possible origin of the evolution of the field induced spin gap should be related with the spin-wave excitations at the strong magnetic field regime \(^{47}\). Further studies on the \(T^2\)-dependence of the spin excitation should be very helpful.

To conclude, we performed the first NMR study on the newly discovered \(J_{\text{eff}} = 1/2\) triangular lattice com-
pound Yb(BaBO$_3$)$_3$ without inherent disorder. Absence of any spin ordering or freezing is evidenced by both the spectral analysis and the spin-lattice relaxation rate measurements, with the temperature down to 0.26 K. At low magnetic fields, the temperature independent $T_1^{-1}$ at low temperatures indicates the maintaining of strong quantum spin excitations and a constant sum of dynamic spin susceptibility in the momentum space. These facts supply strong evidence for the QSL ground state with gapless spin excitations in Yb(BaBO$_3$)$_3$. Under high magnetic fields, the field-induced spin excitation gap is observed, with the gap size proportional to the field intensity. We think this gap should be the gap of the dispersive spin-wave excitations at the strong magnetic field regime of the QSL state.

We thank Y. S. Li, Y. P. Cai, J. H. Zhou and Gang Chen for helpful discussions. This research was supported by the National Key Research and Development Program of China (Grant No. 2016YFA0401802), the National Natural Science Foundation of China (Grants No. 11504377, 11874057, 11574288, 11874158 and U1732273) and the Users with Excellence Program of Hefei Science center CAS (Grant No. 2019HSC-UE008). A portion of this work was supported by the High Magnetic Field Laboratory of Anhui Province.

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