Evidence for s-wave superconductivity in the new $\beta$-pyrochlore oxide RbOs$_2$O$_6$

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We report the results of $^{87}$Rb NMR measurements on RbOs$_2$O$_6$, a new member of the family of the superconducting pyrochlore-type oxides with a critical temperature $T_c = 6.4$ K. In the normal state, the nuclear spin-lattice relaxation time $T_1$ obeys the Korringa-type relation $T_1 T = $ constant and the Knight shift is independent of temperature, indicating the absence of strong magnetic correlations. In the superconducting state, $T_1^{-1}(T)$ exhibits a tiny coherence enhancement just below $T_c$, and decreases exponentially with further decreasing temperatures. The value of the corresponding energy gap is close to that predicted by the conventional weak-coupling BCS theory. Our results indicate that RbOs$_2$O$_6$ is a conventional s-wave-type superconductor.

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Geometrical frustration of spin systems has recently attracted much attention because, instead of long-range magnetic order, novel ground states, including unconventional superconductivity, may be adopted[1, 2]. Pyrochlore-type oxides with tetrahedral networks of magnetic ions, the so-called pyrochlore lattice, are well known physical realizations of geometrically frustrated magnetic systems. This class of materials has recently received enhanced attention because superconductivity was found in systems. This class of materials has recently received enhanced attention because, instead of long-range magnetic order, novel ground states, including unconventional superconductivity, may be adopted[1, 2]. Pyrochlore-type oxides with tetrahedral networks of magnetic ions, the so-called pyrochlore lattice, are well known physical realizations of geometrically frustrated magnetic systems. This class of materials has recently received enhanced attention because superconductivity was found in systems. This class of materials has recently received enhanced attention because superconductivity was found in systems. This class of materials has recently received enhanced attention because superconductivity was found in systems.

Very recently, a new ternary compound RbOs$_2$O$_6$ with the $\beta$-pyrochlore structure, the same as KOs$_2$O$_6$, has been discovered[13]. This compound exhibits superconductivity below $T_c = 6.4$ K[13, 14]. The results of recent specific-heat[14] and magnetic-field penetration depth measurements[13] were claimed to indicate a conventional BCS-type behavior of this superconducting state. The zero-temperature upper critical magnetic field $H_{c2}$ ($\sim 6$ T), extracted from specific-heat measurements on RbOs$_2$O$_6$, is lower than the Pauli-limiting field[14], in contrast to the above cited claims for KOs$_2$O$_6$. From the results of electrical resistivity measurements, however, the value of $H_{c2}$ for RbOs$_2$O$_6$ at zero temperature was claimed to be larger than the Pauli limit of 12 T[13]. This estimated value for the Pauli limit may, however, be substantially modified towards higher values by spin-orbit interactions, as suggested by the results of band-structure calculations for the related pyrochlore-type oxides Cd$_2$Re$_2$O$_7$ and Cd$_2$Os$_2$O$_7$[15].

In this report, we present the results of magnetic susceptibility and $^{87}$Rb NMR measurements on samples of polycrystalline RbOs$_2$O$_6$ in both the normal and the superconducting state. Our results support the view of conventional superconductivity in this material.

The samples were prepared from polycrystalline material of RbOs$_2$O$_6$, synthesized from the starting materials OsO$_2$ and Rb$_2$O. The experimental details of the synthesis and the purification of RbOs$_2$O$_6$ are described elsewhere[17]. The material was confirmed to have the correct structure by X-ray diffraction. Most of the reflections could be indexed on the basis of a pyrochlore unit cell with a lattice parameter $a = 10.1137(1)$ Å. A small amount of OsO$_2$ (less than 5%) was detected as an impurity. Our NMR experiments probe the local environment of the Rb ions. The Rb cations occupy the 8b site in the pyrochlore lattice, which provides a local environment with cubic symmetry.

The magnetic susceptibility $\chi(T) = \frac{M(T)}{H}$, where $M(T)$ represents the temperature dependent magnetization, was measured upon cooling the sample at temperatures between 300 K and 2 K in external magnetic fields $\mu_0 H = 50$ G and 2.94 T, using a SQUID magnetometer. The NMR measurements were performed at temperatures between 0.4 K and 35 K in an external magnetic field of 2.9427 T using a standard phase-coherent-type pulsed spectrometer. The $^{87}$Rb NMR spectra were obtained by Fast-Fourier-Transformation (FFT) of the spin-echo signals, following a $\pi/2$, $\pi$, $rf$ pulse sequence. The nuclear spin-lattice relaxation time $T_1$ was measured by the saturation recovery method, where the spin-echo signals were measured after the application of a comb of $rf$ pulses.
FIG. 1: Temperature dependences of the magnetic susceptibility $\chi(T)$ measured on a powdered sample of RbOs$_2$O$_6$ in external magnetic fields of (a) 50 G and (b) 2.94 T. Note the very different vertical scales. The sample was cooled in the field. Inset: Temperature dependence of $\chi(T)$ above $T_c$ in an external magnetic field of 2.94 T.

FIG. 2: Evolution of the FFT spectra of $^{87}$Rb NMR in an external magnetic field of 2.9427 T between 1.2 K and 5.0 K. Inset: Temperature dependence of the full-width at half-maximum (FWHM) of the signal.

$\chi(T)$ of a powdered sample of RbOs$_2$O$_6$. The superconducting transition is reflected in the onset of a large diamagnetic signal due to the Meissner effect (Fig. 1(a)). In case of $\mu_0 H = 50$ G, $\chi(T)$ reflects the onset of diamagnetism at 6.4 K. As usual, increasing external magnetic fields shift the transition to lower temperatures. In an external magnetic field $\mu_0 H = 2.94$ T, the same that we used in our NMR measurements, the $\chi(T)$ data reveal the onset of superconductivity at $T_c = 3.8$ K (Fig. 1(b)). This value is consistent with the results of previous specific-heat measurements. The inset of Fig. 1(b) shows the temperature dependence of $\chi(T)$ of RbOs$_2$O$_6$ in the normal state for $\mu_0 H = 2.94$ T. At temperatures exceeding 100 K, $\chi(T)$ is, to a good approximation, temperature-independent. Upon cooling to below 50 K, the susceptibility increases gradually, such that $\chi(T) = \chi_0 + \frac{C}{T}$, where $C$ is the Curie constant and $\chi_0 = 4.8 \times 10^{-7}$ emu/g is the temperature-independent susceptibility. For common metals, $\chi_0 = \chi_{\text{Pauli}} \pm \chi_{\text{Landau}} + \chi_{\text{shell}}$, where for free electrons $\chi_{\text{Landau}} = -\frac{1}{3} \chi_{\text{Pauli}}$. Assuming that the core-electron diamagnetism term $\chi_{\text{shell}}$ is negligibly small, $\chi_{\text{Pauli}} = \frac{1}{2} \chi_0$ and we can calculate the electronic density of states at the Fermi surface from $\chi_{\text{Pauli}} = \mu_B^2 \frac{D(E_F)}{3}$. Using this relation, we obtain $D(E_F) = 1.38$ states/eV-atom. This value of $D(E_F)$ implies that the electronic specific-heat coefficient $\gamma = \frac{2 k_B^2 D(E_F)}{3} = 31$ mJ/mol$^{-1}$K$^{-2}$, in very good agreement with the value estimated from the result of specific-heat measurements. The Curie-type upturn of $\chi(T)$ at low temperatures is attributed to the presence of a small concentration of impurity moments. The effective paramagnetic moment deduced from the Curie constant is small, of the order of 0.1 $\mu_B$/Os. Thus, the intrinsic behavior of $\chi(T)$ of RbOs$_2$O$_6$ in the normal state is that of a simple metal, consistent with the results of measurements of the Knight shift and the nuclear spin-lattice relaxation rate, to be discussed below.

Figure 2 shows the evolution of the FFT spectra of $^{87}$Rb NMR at low temperatures. The spectrum contains a single resonance line. As described above, the Rb nuclei occupy highly symmetrical sites and thus, the influence of the quadrupole interaction is quenched ($I = \frac{5}{2}$ for the $^{87}$Rb nucleus). The inset of Fig. 2 shows the temperature dependence of the full-width at half-maximum (FWHM) of the $^{87}$Rb NMR spectrum. In the normal state, this width is of the order of 12 kHz and independent of temperature. In the superconducting state, the NMR spectrum broadens appreciably due to a distribution of local
fields produced by the vortex lattice. The inhomogeneous broadening of the NMR line of type-II superconductors can approximately be calculated as \( \Gamma \sim \frac{\phi_0}{\lambda(16\pi^2T)^{1/2}} \), the square root of the second moment of the expected field distribution due to the vortices. With \( \lambda = 4100 \text{ Å} \) as the zero-temperature London penetration depth, and \( \phi_0 = \frac{hc}{2e} \) as the flux quantum, we calculate \( \Gamma \sim 5.5 \text{ Oe} \) \( \sim 8 \text{ kHz} \) at \( T = 0 \text{ K} \). This value is close to the observed total enhancement of the line width of approximately 12 kHz.

Next, we consider the nuclear spin-lattice relaxation. Figure 3 shows the temperature dependence of \((T_cT)^{-1}\). The \( T_1 \) measurements were made at the peak positions of the resonance signals, but the employed rf pulses were short enough to irradiate the entire NMR line. The observed magnetization recovery (data not shown) followed a single-exponential curve.

In the normal state, \((T_1T)^{-1}(T)\) obeys the Korringa relation \((T_1T)^{-1}_n = 0.117 \text{ (sK)}^{-1}\), as expected for simple metals and indicating the absence of significant magnetic interactions in RbOs\(_2\)O\(_6\). Recently, it was reported that \( T_1^{-1} \) of \(^{39}\text{K}\) nuclear spins in KOs\(_2\)O\(_6\) exhibits an unusual temperature dependence in the normal state. This was interpreted as evidence for considerable antiferromagnetic correlations in the itinerant electron system. Assuming that both data sets are reliable, it must be concluded that the magnetic features of RbOs\(_2\)O\(_6\) are quite different from those of KOs\(_2\)O\(_6\).

In the superconducting state, \((T_1T)^{-1}\) reveals no clear coherence peak just below \( T_c \) (\( \sim 3.8 \text{ K} \) for \( \mu_0H = 2.94 \text{ T} \)) but drops sharply only below 3 K, i.e., at a temperature significantly lower than \( T_c \). The maximum value of \((T_1T)^{-1}\) below \( T_c \) is only 4 % larger than the value in the normal state. A similar result has recently been obtained by other workers. This behavior is distinctly different from that of well identified unconventional superconductors where \((T_1T)^{-1}\) drops sharply just below \( T_c \). We argue that the superconducting state of RbOs\(_2\)O\(_6\) is of conventional type and that the data between 3 K and \( T_c(H) \) reflects a strongly reduced coherence peak in \((T_1T)^{-1}(T)\). The coherence peaks for s-wave type-II superconductors are often reduced for various reasons. In the case of \( V_3\text{Sn} \), it was argued that the application of an external magnetic field causes the observed reduction. The coherence peak may also be suppressed by finite life-time effects on the quasiparticles due to, for instance, electron-phonon interactions. Fibich showed that an effective broadening of the electronic energy levels is brought about by absorption processes of thermal phonons. More accurately, the gap function has a (negative) imaginary part, which has the effect of removing the singularity in the electrical density of states at non-zero temperature. Although these life-time effects are expected to be particularly important for strong-coupling superconductors, the mechanism was originally invoked in order to explain the only modest enhancement of \((T_1T)^{-1}\) below \( T_c \) in the case of Al. Fibich has derived the ratio \((T_1n/T_1s)\) of the relaxation rates in the superconducting state to those in the normal state as

\[
\frac{T_{1n}}{T_{1s}} = 2f(\Delta_1)[1 + \frac{\Delta_1(T)}{k_BT}(1 - f(\Delta_1)) \ln\frac{2\Delta_1(T)}{|\Delta_2(T)|}] \tag{1}
\]

with

\[
\frac{\Delta_2(T)}{\Delta_0} = C\left(\frac{\Delta_0}{\Delta_1(T)}\right)^{1/3}(\frac{T}{T_c})^{8/3} \tag{2}
\]

where \(\Delta_1(T)\) and \(\Delta_2(T)\) are the real and the imaginary part of the gap function, \(f\) is the Fermi distribution function, and \(C\) is a fitting parameter. Our simulations (data not shown) using Eq. 1 do not yield satisfactory results for the case of RbOs\(_2\)O\(_6\), however, and we conclude that this type of reasoning is not adequate for explaining the only weakly developed coherence peak in \((T_1T)^{-1}(T)\) in RbOs\(_2\)O\(_6\).

In an attempt to elucidate the reduction of the coherence peak in \((T_1T)^{-1}\) below \( T_c \), we tried to consider the effect of the applied magnetic field using the approach suggested by Goldberg and Weger. Here, the basic assumption is that the total nuclear spin-lattice relaxation rate is the sum of two terms, where the first describes the relaxation in the normal-state vortex cores and the other captures \((T_1T)^{-1}\) in the remaining superconducting volume. Near \( T_c \), this leads to

\[
(T_1T)^{-1} = (T_1T)_n^{-1}(H)^2 + (T_1T)^{-1}_{BCS}(1 - \frac{H(\xi^2)}{\Phi}) \tag{3}
\]

where \((T_1T)_n^{-1} = 0.117 \text{ (sK)}^{-1}\). The coherence length is given by \(\xi(T) = \frac{\tau c}{(\tau c/\sigma_{\text{rf}})^{1/2}}\) with \(\xi(0) = 74 \text{ Å}\), and \((T_1T)^{-1}_{BCS}\) represents the relaxation in the superconducting volume. Inserting the parameters for RbOs\(_2\)O\(_6\), the first term on the r.h.s. of Eq. 3 turns out to be...
negligibly small and hence for the field strength used in our experiments, the influence of the applied magnetic field in the manner described above plays no role in our problem. This may not be the case for higher applied magnetic fields, however.

At very low temperatures and high magnetic fields, yet another process, namely spin diffusion, will play a dominant role. In this case, the observed relaxation rate $T^{-1}$ will be dominated by fast processes in the normal core of the vortices. Note that due to the small value of $\xi$ at low temperatures, finite-size effects may have to be taken into account in the vortex cores and the corresponding relaxation rate may not simply be given by $(T_1)^{-1}$ of the bulk in the normal state.

Below 3 K, $T^{-1}$ decreases exponentially with temperature upon cooling. Below 1 K, where $T^{-1}$ is less than $7 \times 10^{-3} \text{s}^{-1}$, the relaxation tends towards a temperature-independent value with decreasing temperature. This deviation may be caused by paramagnetic impurities and/or by relaxation via spin diffusion to the normal vortex cores, which are regions of fast relaxation. The onset of anomalous relaxation follows a more than an order of magnitude reduction of the energy gap can be accounted for by incorporating spin-orbit and a spin part. In the normal state of the conduction electrons, seen at a particular nuclear site. This finding, however, should be considered with some caution because, as we shall see below, a more detailed inspection of the data suggest a considerable anisotropy in the gap parameter. Nevertheless, the observed thermally activated temperature dependence of $T^{-1}$ provides clear evidence for a nodeless gap configuration and the remnants of the coherence peak just below $T_c$ indicate a conventional s-wave-type pairing of the quasiparticles. This is consistent with previous interpretations of results of specific-heat[14] and magnetic-field penetration depth measurements[15].

In order to reproduce $T^{-1}(T)$ at intermediate temperatures below $T_c$, we fitted the data to the BCS model and assumed a distribution of energy gap amplitudes in the range between $\Delta - \delta$ and $\Delta + \delta$ across the Fermi surface[20]. The solid line below $T_c$ in the mainframe of Fig. 3 represents the result of a calculation using $\Delta(0) = 3.5$ and $\Delta'(T_c) = 0.5$. In this way, we obtain good agreement with the experimental results at temperatures between 1 K and 3 K. The calculation cannot reproduce the behavior just below $T_c$ quantitatively. The distribution of the energy gap can be accounted for by incorporating a $k$-space anisotropy of the conventional $s$-wave gap[20],

$$\Delta(H, \Omega) = <\Delta(H) > [1 + a(\Omega)]$$

where $\Omega$ is the solid angle in $k$-space, $< \Delta(H) >$ the mean gap value over all orientations in $k$-space, and $a(\Omega)$ is the anisotropy function satisfying the condition $a(\Omega) >= 0$. Our value for $\frac{\Delta}{\Delta(0)}$ implies $< a^2(\Omega) > = 0.25$. This anisotropy can substantially affect various thermodynamic quantities. For instance, it has been shown[21, 25] that it renormalizes the ratio $\frac{\Delta}{\Delta(0)}$, as $\frac{\Delta}{\Delta(0)} = \frac{\Delta}{\Delta(0)}(1 - \frac{\delta}{\alpha} < a^2 >)$, where $\frac{\alpha}{\Delta(0)}$ is the ratio in the absence of anisotropy. In our case, since $\frac{\Delta}{\Delta(0)} = 3.2$ and $< a^2(\Omega) > = 0.25$, it follows that $\frac{\Delta}{\Delta(0)} = 5.1$, a substantially enhanced value with respect to the expectations for the BCS theory in the weak-coupling limit, and raising some questions about the validity of the approximation. An refined quantitative discussion of the NMR data obviously requires additional efforts in numerical calculations.

Figure 4 shows the temperature dependence of the Knight shift of the $^{87}$Rb NMR signal. The Knight shift is a measure of the uniform magnetic susceptibility of the conduction electrons, seen at a particular nuclear site. In general, the Knight shift consists of a $T$-independent orbital part and a spin part. In the normal state of RbOs$_2$O$_6$, the Knight shift is very small, about 0.045 $\%$, and practically independent of temperature, reflecting the influence of the temperature-independent contribution of the electronic spin susceptibility. In the superconducting state, the diamagnetic shift $H_{dia}$ is estimated as $-\frac{H_{dia} \ln(\frac{\xi}{\xi})}{\ln(\frac{\lambda}{\lambda})}$. With $\xi = 74 \AA[14]$, $\lambda = 4100 \AA[15]$, $\beta = 0.381$ for the triangular vortex lattice, and $d = 285 \AA$ as the nearest-neighbor vortex-lattice spacing in 2.9427
T, we calculate $K_{\text{dia}} = -0.004\%$. The observed Knight shift variation below $T_c$ is much larger than $K_{\text{dia}}$, thus reflecting the spin-singlet-pairing of the quasiparticles. The residual line shift at very low temperatures is of the order of 0.020\%.

The inset of Fig. 4 shows the temperature dependence of the Knight shift $K_s$ normalized by its value at $T_c$. Here, the residual line shift attributed to orbital effects has been subtracted from the raw data. The dotted line represents the result of a calculation using the conventional BCS model with the same parameters and the same gap distribution as those in the previously discussed analysis of $T_1^{-1}(T)$ below $T_c$. Because the calculation does not agree with the experimental data, the deviation of the temperature dependence of the Knight shift below $T_c$ from the BCS expectation needs further examination.

The ratio $K_o = \frac{\gamma_{\text{orb}}}{\gamma_{\text{el}}}$ in the normal state provides a useful measure for the importance of electron-electron magnetic correlations. The parameter $S = \frac{\gamma_{\text{orb}}}{\gamma_{\text{el}}}$ and $\gamma_{\text{orb}}$ and $\gamma_{\text{el}}$ are the electronic and nuclear gyromagnetic ratios, respectively. Depending on the value of $K_o$ being much smaller or larger than unity, substantial ferro- or antiferro-magnetic correlations in the itinerant electron systems are significant. If we assume that the residual line shift $K(T = 0)$ is due to the orbital contributions, $K_o$ is estimated to be 4.6, providing some evidence for the existence of antiferromagnetic electron-electron correlations.

We present and discuss the results of $^{87}$Rb NMR measurements on the new superconducting pyrochlore-type oxide RbOs$_2$O$_6$. In the normal state, the nuclear spin lattice relaxation rate $T_1^{-1}$ obeys the Korringa-type relation $(T_1T)^{-1} = 0.117$ (sK)$^{-1}$ and the line shift is independent of temperature. In the superconducting state, $T_1^{-1}$ reveals a very much reduced coherence peak just below $T_c$, and eventually decreases with a thermally activated behavior upon further cooling. The $T_1^{-1}(T)$ data can qualitatively be explained by the BCS model considering some anisotropy of the gap function. In spite of some remaining numerical inconsistencies, we claim that our NMR results imply that the superconducting state of RbOs$_2$O$_6$ is characterized by singlet-pairing of the electrons and that the gap function exhibits a conventional $s$-wave-type symmetry with some $k$-dependent variation of the amplitude, however.

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