NMR investigations of U_2Ru_2Sn: A possible Kondo insulator

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NMR measurements on various samples of U_2Ru_2Sn, a tetragonal Kondo insulating system, are presented. We observed anisotropic Knight shifts in all the samples. The anisotropy increases at low temperatures. The Knight shifts as well as the spin-lattice relaxation rates show signatures of gap opening at low temperatures. The hyperfine coupling exhibits an anomalous temperature dependence near the Kondo temperature. The results are discussed in the light of a low-temperature gap with residual density of states in this system.

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I. INTRODUCTION

A subset of Kondo lattice systems, in which the lattice of magnetic ions (f and d elements) interact with conduction electrons giving rise to exotic low-temperature properties, falls into the category of Kondo insulators (or heavy fermion semiconductors[1]). In addition, the magnetic elements are valence fluctuating.[2] This set of compounds has the common property of a high-temperature paramagnetic phase and a low-temperature nonmagnetic (or at least partially moment compensated) phase. However, unlike the better studied heavy fermion metals, the low-temperature phase is insulating or at least semimetallic, indicating a presence of an energy gap in the excitation spectrum of electrons at low temperatures.

After the first reported compound SmB_6,[3] a series of Ce based, U based, and Yb based compounds have been discovered[1] to belong to this class of materials in which, at high temperatures, the magnetic susceptibility follows Curie-Weiss (CW) behavior with the effective moment of the free ion, goes through a deviation from CW behavior and a maximum in the susceptibility in the intermediate temperatures, and decreases to a value that corresponds to only a fraction of the moment expected from the CW behavior. The resistivity at high temperature has a $-\log T$ behavior; however, at low temperature the exponential increase with decreasing temperature, associated with the presence of gap in the spectrum, is often masked by a saturation indicative of in-gap states or impurity states which contribute to conductivity. An extensive compilation of properties of Kondo insulators has been made by Riseborough[1]. The high-temperature Curie-Weiss susceptibility and logarithmic temperature dependence of resistivity are consistent with scattering of conduction electrons by independent magnetic moments. At low temperatures, the electrically insulating state (and an energy gap) is a result of f-electron correlations.

Two different characteristic temperatures have been identified with the Kondo insulators. One is the magnitude of the gap at low temperature and the other is the temperature below which the gap opens. The temperature dependence of the gap has been studied in FeSi (Ref. 4) and in $Ce_3Bi_4Pt_3$ (Ref. 5). Moreover, the gap value depends on the physical property from which it is extracted. A compilation of gap opening temperatures for selected Ce based Kondo insulators is given by Takabatake *et al.*[6] Recently, Adroja *et al.*[7] have made a compilation of data showing the universal relation between the susceptibility maximum (T_{max}) to the spin gap value Δ, which is $T_{max}=3T_K$. However, the temperature dependence of the gap has not been studied so extensively as to have a clear understanding.

Nuclear magnetic resonance (NMR) is a very useful tool for determining the gap at the Fermi surface. The spin-lattice relaxation rate $\frac{1}{T_1}$ is sensitive to excitations of quasiparticles across the gap structure. The study of temperature dependence of relaxation rate has given a wealth of information and has been a method of extracting the gap value for various Kondo insulators.[8–10] Unlike the magnetic susceptibility, which contains contributions from extrinsic impurities, Knight shift that arises due to the hyperfine interaction of nuclear spin with the electronic spin susceptibility is less influenced by impurities and not influenced at all by other phases and hence is an ideal tool for studying the spin compensation and opening of spin energy gap.

U_2Ru_2Sn was initially identified as a weak paramagnet[11] in the class of U_2T_2Sn, and later classified as the first tetragonal Kondo insulating system.[12] The magnetic susceptibility and thermal and electrical transport properties were extensively investigated[13] in both polycrystallites[14] and single crystals.[15] The magnetic susceptibility is anisotropic and shows a maximum around 180 K and decreases at low temperature. At temperatures above 300 K, the susceptibility follows CW behavior with nearly free ion moment. The resistivity at high temperatures shows $-\log T$ behavior, which is attributed to incoherent Kondo scattering. At intermediate temperatures the resistivity decreases, signaling a coherence between the $5f$ moments of the U atoms. Below 25 K the resistivity increases, indicating a semimetallic state.[16]

Like in the other Kondo insulators, the gap values obtained from various physical properties such as specific heat, resistivity, and NMR are different. A gap value of 150 K was estimated from specific heat and NMR relaxation rate of a polycrystalline sample.[17] The relaxation rate measurements confirm the presence of residual density of states and also provided evidence for the scaling of relaxation rate measurement with CeNiSn.

In this paper, we report our NMR studies on single-crystal, oriented microcrystalline, and polycrystal samples of U_2Ru_2Sn. We compare our results with that of CeNiSn and $Ce_3Bi_4Pt_3$ to point out the similarities in the behavior of the

Knight shift and relaxation rates in these systems.

II. EXPERIMENT

The NMR measurements have been carried out using a field sweep NMR instrument developed in our laboratory, using the automation software written in LABVIEW, where it is possible to achieve the field stability of 0.1 G between different scans.

Polycrystalline U_2Ru_2Sn was obtained by arc melting the individual components. The detailed method of preparation is published elsewhere.[13] The single crystal was obtained using a traveling floating-zone technique. Four different types of samples were used for this measurement. They are randomly oriented polycrystalline powder (RP), polycrystalline powder oriented in 7 T field and sealed in epoxy (OP), pieces of single crystals (OX), and oriented (in 7 T) microcrystalline samples obtained by powdering single crystals and sealed in epoxy (OM).

The oriented samples were obtained by mixing the powder (or microcrystals) in epoxy in a cylindrical shape and applying a magnetic field perpendicular to the axis of the cylinder. A near complete orientation is obtained within 9 h while the epoxy was setting. In this process, the sample orients with the *c* axis parallel to the magnetic field. The orientation was verified using the NMR spectrum obtained in the single crystals at 4.2 K (see Fig. 1).

The magnetic field for orienting the sample was applied in the direction perpendicular to the axis of the cylinder so that the same sample can be used for both orientations with the *c* axis parallel to the field (parallel direction) and the *c* axis perpendicular to the field (perpendicular direction) in the NMR coil by rotating the sample by 90° about the cylindrical axis. The samples were well characterized using x-ray diffraction and χ vs T measurements. The single-crystal sample was found to have traces of URuSn (less than 400 ppm). The polycrystalline sample was found to contain traces of pure Sn (detected by its superconducting transition). However, no NMR signal could be observed from pure Sn. The signals from OX samples were obtained below 75 K; however, it was impossible to obtain signal above 75 K due to the poor penetration depth of rf in the sample. However, the signal could be observed, though very weak, once again above 200 K. In order to observe the spectra in the entire temperature range, we powdered a piece of single crystal and oriented the microcrystallites in a magnetic field of ~7 T while it is being set in an epoxy. The extent of the orientation could be verified from the Knight shift values as to how well the *ab* axis value coincides with the shift value of the perpendicular direction in the powder pattern.

The preparation method for the nonmagnetic reference sample Th_2Ru_2Sn is described elsewhere.[18] The reference compound has a Knight shift of about 0.1% relative to the listed Larmor frequency and is temperature independent between 4.2 and 200 K. The Knight shift values of U_2Ru_2Sn are obtained with respect to this reference.

III. RESULTS

Figure 1 shows the compilation of field sweep ^{119}Sn-NMR spectra at various temperatures. The spectra in the middle panel are obtained from RP samples. The spectra below ~100 K show powder pattern due to axially symmetric Knight shift. The development of the increased anisotropy below 100 K is evident. Above 100 K, the line fits to a Gaussian line shape and the anisotropy is not resolved at any higher temperature. In order to investigate the anisotropic Knight shift directly, we carried out field sweep NMR measurements on pieces of single crystals oriented in parallel and perpendicular directions. The top and bottom panels show the spectra obtained in oriented single crystal at 10 and 200 K, respectively. From this figure, one can see that the $K_{\parallel}$ and $K_{\perp}$ directions correspond to parallel and perpendicular directions, respectively, indicating that the axis of the anisotropy coincides with the *c* axis.

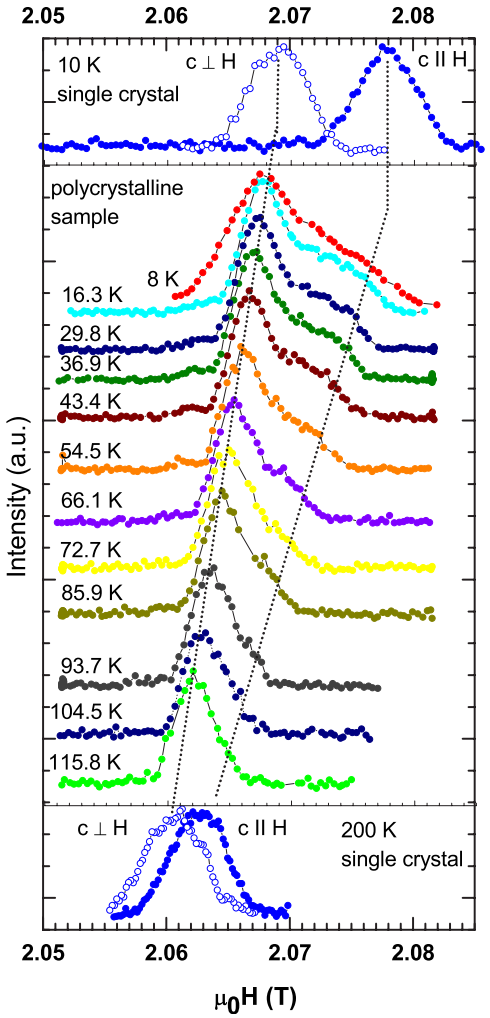

FIG. 1. (Color online) NMR spectra obtained by sweeping field at fixed frequency of 33 MHz in U_2Ru_2Sn. The top and bottom panels are the lines obtained in OX samples and the middle panel shows the polycrystalline unoriented powder sample. The dotted line is a guide for the eyes.

A. Knight shift

The Knight shift in the reference compound Th_2Ru_2Sn is isotropic and temperature independent (~0.1%). The tem-

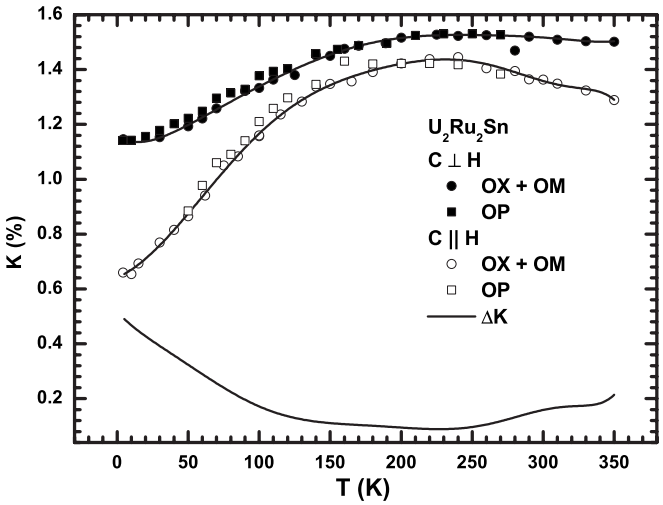

FIG. 2. Knight shift as a function of temperature in the parallel and perpendicular directions in U_2Ru_2Sn.

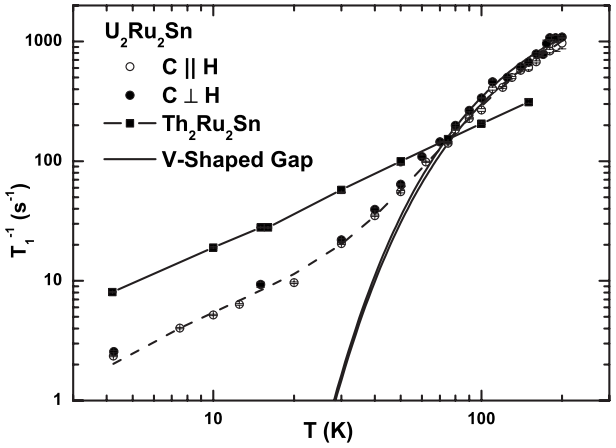

FIG. 3. The relaxation rate of U_2Ru_2Sn in parallel (open) and perpendicular (filled) directions. The relaxation rate of Th_2Ru_2Sn follows the Korringa law (filled square with solid line). The solid curves show the straight energy gap of 230 K. The dashed line is the best fit for V-shaped gap fitting well with the low-temperature residual relaxation rate as well.

perature dependence of the Knight shift values for both the directions in U_2Ru_2Sn from 350 to 4.2 K is given in Fig. 2. The Knight shift is positive and anisotropic in the entire temperature range of measurement. However, below ~150 K, the Knight shift in the parallel direction exhibits a rapid decrease as indicated by the ΔK plot.

The temperature dependence of the Knight shift in the parallel direction is qualitatively similar to the magnetic susceptibility, where a drop in the susceptibility is observed below 150 K. The drop in the susceptibility following a broad maximum at 150 K is a characteristic feature of Kondo system and is related to the Kondo temperature (T_K). The Knight shift in the perpendicular direction, however, does not show such remarkable change near 150 K. This is a clear indication of an anisotropic energy gap arising due to the anisotropic interaction of the $5f$ electrons with the conduction band. The anisotropic residual Knight shifts at the lowest temperature could also be associated with various other reasons. For instance, the partial compensation of U moments, which is evident from the low-temperature susceptibility, could result in an anisotropic residual $5f$ moments. It could also be because of the anisotropy in the residual density of states in the gap.

It is important at this point to compare our results with those of other Kondo insulators in order to show that the results are not entirely anomalous. A similar behavior of different temperature dependence in different crystallographic directions is observed in orthorhombic Kondo insulator CeNiSn.[9] Though $Ce_3Bi_4Pt_3$ is cubic, the ^{209}Bi Knight shift is anisotropic.[8] This shows that our observations in U_2Ru_2Sn are typical of Kondo insulating system and the observed anomaly at 150 K is signature of the gap opening.

B. Relaxation rate

^{119}Sn NMR relaxation rate in the reference sample Th_2Ru_2Sn exhibits linear temperature dependence, following the Korringa relation (Fig. 3) indicating that the relaxation is dominated by free conduction electron. In U_2Ru_2Sn, near 200 K, the relaxation rates are much higher than those in Th_2Ru_2Sn. However, the relaxation rate drops rapidly below this temperature, signaling opening of the energy gap, and decreases to a value less than that in Th_2Ru_2Sn. Below 80 K, the relaxation rate is linear. In U_2Ru_2Sn, one can consider that there are two contributions to the relaxation process which can be written as

$$\frac{1}{T_1} = \frac{1}{T_{1-CE}} + \frac{1}{T_{1-5f}}. \quad (1)$$

The first term is the contribution to the relaxation from the conduction electrons due to contact interaction and the second is the transferred hyperfine term from the f electrons of U. One can consider that the conduction-electron contribution in U_2Ru_2Sn will be similar to that in Th_2Ru_2Sn. It is clear that at high temperatures, both the contributions are present and hence the $\frac{1}{T_1}$ in U_2Ru_2Sn is higher than that of Th_2Ru_2Sn. However, below 80 K, the $\frac{1}{T_1}$ decreases below that of Th_2Ru_2Sn, indicating that *both* contributions are decreasing. Below 80 K, the relaxation rate exhibits a Korringa behavior indicating, the presence of residual density of states. We do not observe an anisotropy as clearly in the relaxation rate as in the Knight shift.

The relaxation rate in the temperature range of 80–200 K can be fitted to the form $\frac{1}{T_1} \sim e^{-\Delta/k_B T}$ with $\Delta = 230$ K. This value is higher than what was obtained in the polycrystalline randomly oriented powder,[17] indicating a sample dependence.

The features of $\frac{1}{T_1}$ observed in U_2Ru_2Sn are common in many of the Kondo insulating systems especially CeNiSn.[19] There are different models for the shape of the density of states near the Fermi surface in order to explain the observed temperature dependence of relaxation rate. Considering the linear temperature dependence at low temperatures, a V-shaped energy gap was proposed by Nakamura *et al.*,[20] which was found to fit the temperature dependence of relax-

ation rate of CeNiSn. Nakamura *et al.* approximated the general expression[21] for the temperature dependence of relaxation rate by assuming a $\vec{q}$ independent spin fluctuation and $\vec{q}$ averaged hyperfine interaction to get

$$\frac{1}{T_1} \propto A_{HF}^2 T \int N_{eff}^2(E)\left[-\frac{\partial f(E)}{\partial E}\right]dE. \tag{2}$$

The expression given below for the V-shaped gap is similar to that used by Nakamura *et al.* in their calculations:

$$N(E) = \frac{C}{E^2 + \left(\frac{D}{2}\right)^2} \quad \text{when } |E| > \frac{\Delta}{2}$$

$$= \frac{|E - E_f|}{\Delta} \frac{C}{(E-\Delta)^2 + \left(\frac{D}{2}\right)^2}$$

$$\text{when } |E| < \frac{\Delta}{2} \text{ and } N(E) > \alpha,$$

$$N(E) = \alpha \quad \text{when } |E| < \frac{\Delta}{2} \text{ and } N(E) < \alpha.$$

The first two expressions represent a V-shaped gap (Δ) in a finite band with a width D. The third expression ensures that the minimum value of $N(E)$, inside the V-shaped gap, is the residual density of states (α). The finite bandwidth helps explain the saturation of the relaxation rate at high temperatures. $N(E)$ as well as the integral in the Eq. (2) are evaluated numerically. The solid line through the data points is the best fit of the model, which agrees well with the data with the values of $\frac{\Delta}{K_B}=230\pm5$ K and $\frac{D}{K_B}=2300\pm50$ K. The value of α corresponds to 0.3% of the high-temperature $\left(T \gg \frac{\Delta}{K_B}\right)$ density of states. The value of gap is much smaller than that predicted by the universal relation connecting the T_{max} to T_K described by the work of Adroja *et al.*[7] Further work is needed to find out the reason for such a deviation. In order to see the coupling of the electron to probe nuclei (^{119}Sn), we need to look at the hyperfine interaction.

C. Hyperfine interaction

The relation between spin susceptibility and Knight shift can be written as

$$K_s(T) = \frac{A_{HF}}{N_A \mu_B} \chi_s(T), \tag{3}$$

where A_{HF} is the hyperfine coupling constant, and N_A and μ_B are Avogadro's number and the Bohr magneton, respectively. The plot of $K(T)$ against the corresponding value of magnetic susceptibility $\chi(T)$ is given in Fig. 4 and the slope is the value of A_{HF}. In the parallel direction, the low-T and the high-T regimes are independent straight lines connected through a turnover regime which corresponds to the maximum of the susceptibility. In the perpendicular direction, the low-T data could be fitted to a straight line. The individual slope values are given in Fig. 4. The intercept values are

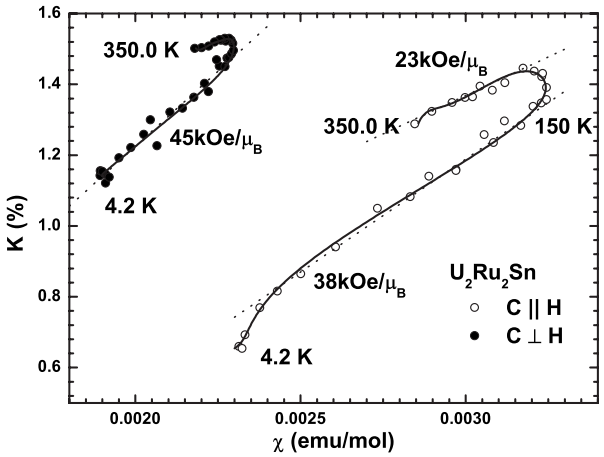

FIG. 4. The $K(T)$ vs $\chi(T)$ shows marked difference in the two directions. The solid lines produced by fitting the data with a seventh-order polynomial serve as a guide for the eyes. The dotted straight lines are linear fits to the data in that range and the corresponding values of A_{HF} are indicated.

−0.006, 0.0002, and −0.001 in the low T, high T of parallel direction, and low T of perpendicular direction, respectively. In the perpendicular direction, the high-temperature regime has a temperature independent Knight shift though the magnetic susceptibility shows a weak temperature dependence.

Such a turnover in the K vs χ plot is observed in $Ce_3Bi_4Pt_3$ and CeNiSn. In both the cases, the axial Knight shift exhibits the turnover. In the case of $Ce_3Bi_4Pt_3$ the isotropic part is nearly linear, whereas in CeNiSn the K vs χ behavior is much more complicated in the other crystallographic directions. The turnover temperature has been attributed to the Kondo temperature in these materials. Kim and Cox[22] have worked out the K vs χ relation for single parameter Kondo system and predict a turnover at the characteristic temperature T_0. Moreover, Ohama *et al.*[19] identify a deviation from linearity of K vs χ near 200 K. However, such a temperature could not be identified within the range of our measurements on U_2Ru_2Sn.

The striking feature is that the magnetic susceptibility in the perpendicular direction is less than that in the parallel direction, whereas K exhibits a trend that is reverse. Though this feature is more prominent at low temperatures, it is so in the entire temperature range, indicating that the hyperfine coupling is much stronger in the ab plane than in the c direction. Moreover, in the low-temperature region, after the gap opening and the moment compensation take place, both contributing to the decrease of hyperfine coupling constant, one observes an enhanced hyperfine coupling constant in the parallel direction and probably in the perpendicular direction as well. In the case of $Ce_3Bi_4Pt_3$, K_{ax} shows very similar behavior.[8]

In order to understand the results, one must consider various contributions to the Knight shift from the susceptibility by estimating a possible value for each one of them. In systems such as U_2Ru_2Sn, which has both conduction electrons and localized spins, the Knight shift can be written as $K(T) = K_0 + K_f(T)$. The temperature independent part K_0 contains the direct Fermi contact term and the dipolar contribution

coming from the non-*s*-nature of the conduction electron and can be written as $K_0 = K_{CE} + K_{orb}$. The temperature dependent part, $K_f(T)$, arises from the interaction of *f* electrons and the probe nuclei through the conduction-electron polarization otherwise called the transferred hyperfine coupling of the *f* electrons. In uniform conduction-electron polarization model,[23] the temperature dependence of the total susceptibility can be written as

$$K(T) = K_{CE}\left[1 + \frac{\langle\Gamma\rangle\chi_f\langle\mathbf{J}\cdot\mathbf{S}\rangle}{g_f g_s \mu_B N J(J+1)}\right].$$

Here, the χ_f is the *f*-electron susceptibility, *N* is Avogadro's number, and $\langle\Gamma\rangle$ is the spatial average of the exchange coupling between the *f* electron and the conduction electron. $\langle\mathbf{J}\cdot\mathbf{S}\rangle$ is the average projected spin. According to this equation for uranium in $5f^2$ configuration corresponding to a $J = L - S = 4$, where $\mathbf{S}\cdot\mathbf{J}$ is negative, a positive Knight shift (in the entire temperature range) and a positive hyperfine coupling, as in the present case, indicate an antiferromagnetic exchange[23] $\langle\Gamma\rangle$ between the conduction-electron spins and the U $5f$ spins, which is according to the expectations in a Kondo system. This shows that the exchange coupling is stronger in the low-temperature regime where the gap is open. It is also possible that the exchange could be very anisotropic and the simple picture of uniform polarization may not be valid at all. It is clear from the anisotropy and temperature dependence of A_{hf} that the effects we observe are due to the correlations set up by opening of the energy gap.

Kim and Cox[22] had discussed in detail the Knight shift in a Kondo system describing the hyperfine interaction in heavy fermion systems such as $CeSn_3$. They describe two different contributions to the Knight shift coming from two different types of polarization of the conduction electrons surrounding the f atom. One is the oscillatory Ruderman-Kittel-Kasuya-Yourda (RKKY)-type interaction and the other is the Kondo interaction (which results in the singlet) which are present at the same time. Such a picture of two components resulted in a turnover in the K-χ plot at a temperature related to the Kondo temperature (T_0). This picture is valid for a heavy fermion system where the ground state is metallic. In the case of U_2Ru_2Sn, the ground state is insulating. Nevertheless, one can consider that the turnover in K-χ graph is due to the interplay of RKKY and Kondo interactions.

IV. CONCLUSIONS

We have observed Knight shift and spin-lattice relaxation in U_2Ru_2Sn between 4.2 and 325 K. The Knight shift is anisotropic over the entire temperature range and below 150 K the anisotropy increases, indicating the opening of a gap. The temperature dependence of relaxation rate also indicates the opening of a gap. At high temperatures, the contribution to relaxation rate is predominantly from the $5f$ electrons of U, and at low temperature the same is from the residual density of states. A model of a V-shaped density of states fits the relaxation data well. From the Knight shift and relaxation data, one could conclude that U_2Ru_2Sn can be classified as a Kondo insulator along with $CePt_3Bi_4$, CeNiSn, etc.

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