Nodule superconductivity and time-reversal symmetry breaking in the noncentrosymmetric superconductor \( \text{Re}_{24}\text{Ti}_5 \)

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Superconductors with an inversion center can host either even-parity singlet-or odd-parity spin-triplet states. These strict symmetry-imposed requirements, however, are relaxed in noncentrosymmetric superconductors (NCSCs), where parity-mixed superconducting states are also allowed. In these materials the lack of an inversion symmetry often induces an antisymmetric spin-orbit coupling (ASOC), which can lift the degeneracy of conduction band electrons. Since the extent of parity-mixing is determined by the strength of SOC, formally similar compounds, but with different spin-orbit couplings, can exhibit different degrees of parity mixing.

The recent interest in NCSCs is related to the complex nature of their superconducting properties.¹,² Because of the mixed pairing, noncentrosymmetric superconductors can display significantly different properties compared to their conventional counterparts. Some NCSCs, such as CePt₃Si³,⁴ CeRhⅢSi₂⁴, Li₂Pt₂B,⁵,⁶ and Mo₃Al₁C₅⁷ exhibit line nodes, while others, as LaNiC₂⁸, and (La,₁Y₂)₂C₃⁹ show multiple superconducting gaps. Furthermore, because of the spin-triplet pairing, the upper critical field often exceeds the Pauli limit, as has been found, e.g., in CePt₃Si¹⁰ and Ce(Rh,Ir)Si₄¹¹,¹² Finally, some NCSCs, as e.g., LaNiC₂¹³, Re₃(Zr,Hi)¹⁴,¹⁵ and La₃Ir₃,¹⁶ are known to break the time-reversal symmetry (TRS).

The binary alloy Re₂₄Ti₅ is a NCSC with superconducting temperature \( T_c = 6\text{ K} \), as reported already in the 1960s.¹⁷ Its physical properties were studied in detail only recently,¹⁸ yet to date the microscopic nature of its SC remains largely unexplored. Similarly to Re₂₃Zr₅ and Re₂₄Nb₃, also Re₂₄Ti₅ adopts an \( \alpha-\text{Mn} \) type crystal structure with space group \( I-4\text{m}m \). However, while the former compounds have been widely studied by means of macroscopic techniques,¹⁹,²⁰ much less is known about Re₂₃Ti₅. A simple analogy, based on structural similarity, can lead to wrong conclusions, since a SOC-dependent parity mixing can bring about rather different superconducting properties. Since its sister compounds, Re₃(Zr,Hi), are known to break the TRS in the superconducting state,¹⁴,¹⁵ Re₂₄Ti₅ represents an ideal opportunity to search for TRS breaking and unconventional SC in a material with a modified SOC value. Moreover, the study of an additional NCSCs can bring new insights into the nature of unconventional superconductivity in general.

Considering the key role played by muon-spin relaxation and rotation (μSR) techniques in unraveling the presence of TRS breaking in unconventional superconductors, twenty in this paper, we report on the systematic magnetization, transport, thermodynamic, tunnel-diode oscillator (TDO) and μSR studies of Re₂₄Ti₅ with particular focus on the latter. We find that below \( T_c \), spontaneous magnetic fields appear, implying a superconducting state which breaks TRS and has an unconventional nature. The low-temperature penetration depth, superfluid density and electronic specific heat all suggest a nodeless s-wave pairing mechanism.

Polycrystalline Re₂₄Ti₅ samples were prepared by arc melting Re and Ti metals under argon atmosphere and then annealed at 900°C for two weeks. The x-ray powder diffraction, measured on a Bruker D8 diffractometer, confirmed the \( \alpha-\text{Mn} \) structure of Re₂₄Ti₅. Magnetic susceptibility, electrical resistivity, and specific heat measurements in different applied magnetic fields were performed on a Quantum Design magnetic property measurement system (MPMS-7 T) and a physical property measurement system (PPMS-14 T). The μSR measurements were carried out using the general-purpose (GPS) instrument located at the \( \pi \mu \text{M} \) beamline of the Swiss Muon Source (SμS) of Paul Scherrer Institut (PSI) in Villigen, Switzerland. The temperature-dependent shift of magnetic-penetration depth was measured by using a tunnel-diode oscillator (TDO) technique in a \( \text{He}_2 \) cryostat, at an operating frequency of 7 MHz.

The magnetic susceptibility, measured at 1 mT using field-cooling (FC) and zero-field cooling (ZFC) procedures, is shown in Fig. 1(a). The splitting of the two curves is typical of type-II superconductors, and the ZFC-susceptibility indicates bulk superconductivity with \( T_c = 6\text{ K} \). The electrical resistivity drops at the onset of superconductivity at 6.8 K, becoming zero at 6 K [see Fig. 2(a)]. The bulk nature of SC is further confirmed by specific-heat data [see Fig. 2(b)].

In transverse field (TF) μSR measurements of superconductors, the applied magnetic field should exceed the lower \( \mu_0H_c^1 \) critical value, so that the additional field-distribution...
The dashed-line in Fig. 1(a) Temperature dependence of magnetic susceptibility \( \chi (T) \) for Re\(_{24}\)Ti\(_5\). (b) Magnetization versus applied magnetic field recorded at different temperatures up to \( T_c \). For each temperature, \( \mu_0 H_{c1} \) was determined as the value where \( M(H) \) deviates from linearity. (c) \( \mu_0 H_{c1} \) vs. temperature: the solid line, a fit to \( \mu_0 H_{c1}(T) = \mu_0 H_{c1}(0)[1- (T/T_c)^2] \), determines a \( \mu_0 H_{c1}(0) = 8.3(1) \text{ mT} \).

broadening due to the flux-line lattice (FLL) can be quantified from the muon decay rate. To determine \( \mu_0 H_{c1} \), the field-dependent magnetization was preliminarily measured at various temperatures below \( T_c \), as shown in Fig. 1(b). The derived \( \mu_0 H_{c1} \) values are plotted in Fig. 1(c) as a function of temperature. The solid line is a fit to \( \mu_0 H_{c1}(T) = \mu_0 H_{c1}(0)[1- (T/T_c)^2] \), which provides a lower critical field \( \mu_0 H_{c1}(0) = 8.3(1) \text{ mT}, consistent with the 8.4-mT value calculated from magnetic penetration depth \( \lambda(0) \). In the Ginzburg-Landau theory of superconductivity, the magnetic penetration depth \( \lambda \) is related to the coherence length \( \xi \) and the lower critical field \( \mu_0 H_{c1} \) via \( \mu_0 H_{c1} = (\Phi_0/4\pi\lambda^2)[\ln(\kappa)+\alpha(\kappa)] \), where \( \Phi_0 = 2.07 \times 10^{-3} \text{ Tm}^2 \) is the quantum of magnetic flux, \( \kappa = \lambda/\xi \) is the Ginzburg-Landau parameter, and \( \alpha(\kappa) \) is a parameter which converges to 0.497 for \( \kappa \gg 1 \). By using \( \mu_0 H_{c1} = 8.3 \text{ mT} \) and \( \xi = 5.41 \text{ nm} \) (calculated from \( \mu_0 H_{c1} \)), the resulting \( \lambda(0) = 286 \text{ nm} \) is consistent with the experimental value from \( \mu\text{SR} \) [see Fig. 3(c)]. With a Ginzburg-Landau parameter \( \kappa \approx 53 \gg 1 \), Re\(_{24}\)Ti\(_5\) is clearly a type-II superconductor. The temperature dependence of penetration depth \( \lambda(T) \) can be estimated also from \( \mu_0 H_{c1}(T) \) and \( \xi(T) \), where \( \xi(T) \) is related to the upper critical field, \( \mu_0 H_{c2}(T) = \Phi_0/2\pi\xi^2(T) \).

To investigate the behavior of the upper critical field \( \mu_0 H_{c2} \), we measured the electrical resistivity \( \rho(T) \) and specific heat \( C(T)/T \) at various magnetic fields. As shown in Figs. 2(a) and (b), the superconducting transition in both cases shifts towards lower temperature upon increasing the magnetic field. Note that, for \( \mu_0 H = 11 \text{ T} \), the large upturn of specific heat at low-\( T \) is due to a Schottky anomaly from nuclear moments, which hinders the superconducting transition. Similar features were also observed in other Re\(_{24}\)Ti\(_5\). The superconducting transition temperatures vs. the normalized temperature \( T/T_c \) as derived from both \( \rho(T) \) and \( C(T)/T \) are summarized in Fig. 2(c). Data taken from Ref. 18 are also plotted. The temperature dependence of the upper critical field \( \mu_0 H_{c2}(T) \) was analyzed following the Werthamer-Helfand-Hohenberg (WHH) model. The dashed-line in Fig. 2(c) shows two representative TF-\( \mu\text{SR} \) spectra collected above and below \( T_c \). Below \( T_c \), the fast decay of muon-spin polarization reflects the inhomogeneous field distribution due to the FFL in the mixed superconducting state. The time-domain spectra were fitted by means of the following model with a Gaussian decay:

\[
A_{\text{TF}} = A_0 \cos(\gamma_\mu B_t t + \phi) e^{-\sigma t^2/2} + A_{bg} \cos(\gamma_{bg} B_t t + \phi). \tag{1}
\]

Here \( A_0 \) and \( A_{bg} \) are the initial muon-spin asymmetries for

![FIG. 2. Temperature-dependent electrical resistivity (a) and specific heat (b) at different applied magnetic fields up to 12 T. From the suppression of \( T_c \) with increasing field (c) we determine an upper critical field \( \mu_0 H_{c2}(0) = 11.2(1) \text{ T} \). The dashed line represents a fit to the WHH model without spin-orbit scattering, whereas the dash-dotted line is a fit to the Ginzburg-Landau model (see text).](image-url)
was evaluated. The solid lines in (c) and (d) both represent fits with the latter not undergoing any depolarization.

Given the nonmagnetic nature of the sample holder, \( B \) practically coincides with the applied magnetic field and its temperature, as determined from \( \phi \) vs. \( T \). The insert shows the shift of penetration depth shown in the inset of Fig. 3(c). Since \( \lambda_{ab} \) is well described by an s-wave model with a single SC gap of about 1.08 meV and a \( \lambda(0) \) of 298 nm. Such superconducting gap is similar to that of other Re-based intermetallic superconductors, e.g., Re\(_2\)Zr (1.21 meV).\(^{14,28}\) Re\(_2\)Hf (0.94 meV).\(^{22,29}\) Re\(_2\)Nb\(_5\) (0.89 meV).\(^{19}\) Also the 2\(\Delta\)/\(k_B T\), values of these compounds [e.g., 4.2(1) for Re\(_2\)Ti\(_5\)] are higher than 3.53, the value expected for a weakly-coupled BCS superconductor, thus indicating moderately strong electron-phonon couplings in these materials. Moreover, the low temperature penetration depth shown in the inset of Fig. 3(c), exhibits an exponential temperature dependence, providing further evidence of fully-gapped superconductivity in Re\(_2\)Ti\(_5\).

Since the specific heat in the superconducting state also offers insights into the superconducting gap and its symmetry, the zero-field specific heat data were further analyzed. The electronic specific heat \( C/E \) is obtained by subtracting the phonon contribution from the experimental data. As shown in the inset of Fig. 3(d) by a dashed-line, the normal-state specific heat is fitted with \( C/E = \gamma_e + \beta T^2 + \delta T^4 \), from which the phonon contribution was evaluated. The solid lines in (c) and (d) both represent fits using a fully-gapped s-wave model.
low (1.5 K) \( T_s \) show clear differences, especially at long times. To exclude the possibility of stray magnetic fields (which in any case would affect uniformly all datasets), the magnets were quenched before the measurements and we made use of an active field-nulling facility. Without an external field, the relaxation is determined mostly by the nuclear magnetic moments, normally described by a Gaussian Kubo-Toyabe relaxation function.

\[
\sigma(t) = A_s \left[ \frac{1}{3} + \frac{2}{3} (1 - \sigma^2 t^2 - s t) e^{-\frac{t^2}{\tau^2}} \right] + A_{bg}.
\]

(3)

Here \( A_s \) is the initial sample-related muon-spin asymmetry, whereas \( A_{bg} \) represents a time- and temperature-independent background. As already shown in the TF-\( \mu \)SR case (see Fig. 3), both the background and the nuclear contributions to the decay are independent of temperature. This is clearly the case also with ZF-\( \mu \)SR [see Fig. 4(b)], where \( \sigma(T) \) remains constant (within the experimental error) in the studied temperature range. On the other hand, the exponential component, related to the presence of spontaneous magnetic fields, shows a small yet distinct increase as the temperature is lowered below \( T_s \) [see Fig. 4(c)].

Such an increase in \( \Lambda(T) \), similar to that found also in Re\(_6\)(ZrH\(_x\)),\(^{14,15}\) represents the signature of spontaneously occurring magnetic fields and of TRS breaking in the Re\(_6\)Ti\(_5\) noncentrosymmetric superconductor. Given the small size of the considered effect, to rule out the possibility of an impurity-induced relaxation (typically relevant at low temperatures), we performed auxiliary longitudinal-field (LF)-\( \mu \)SR measurements at 1.5 K. As shown in Fig. 4(a), a field of 30 mT only is sufficient to lock the muon spins and to completely decouple them from the weak spontaneous magnetic fields, thus removing any relaxation traces related to them.

Up to now, several NCSCs, including LaNiC\(_2\),\(^{13}\) Re\(_6\)(ZrH\(_x\)),\(^{14,15}\) and La\(_2\)Ir\(_3\)\(^{16}\) have been found to exhibit a TRS breaking in the superconducting state. Yet, in many others, as e.g., Mo\(_3\)Al\(_2\)C,\(^7\) Mg\(_{10}\)Ir\(_9\)B\(_{16}\),\(^{23}\) Re\(_3\)W\(_{34}\) and PbTaSe\(_2\),\(^{35}\) the TRS is preserved. The Re\(_{32}\)Ti\(_5\) considered here, a sister compound to Re\(_6\)(ZrH\(_x\)), is a new member of the TRS-breaking NCSCs, despite a relatively reduced ASOC. This strongly suggests that, while the presence of an ASOC seems essential to induce a TRS breaking in NCSCs, its strength is not a crucial condition. Indeed, although LaNiC\(_2\)\(^{13}\) has a much weaker ASOC compared to La\(_2\)Ir\(_3\),\(^{16}\) the respective changes in zero-field muon relaxation rates are comparable (\( \Delta \Lambda \sim 0.01 \mu s^{-1} \)). In our case, too, the replacement of the 5d HF with the 3d Ti, reduces remarkably the ASOC, yet the effects on TRS breaking remain comparable. Hence, we believe that TRS breaking in NCSCs is mostly related to the crystal-structure symmetry and, to test such hypothesis, La\(_2\)Ti\(_3\) compounds (\( T = \) transition metal, e.g., Ni, Pd, Rh, Pt) represent good candidates, since all of them exhibit a Th\(_1\)Fe\(_5\)-type crystal structure, with the 3d to 5d transition metals covering a wide ASOC range.

The spin-triplet states can give rise to spontaneous fields in the superconducting state, which break the TRS. Most of these TRS-broken phases exhibit nodes in the superconducting gap, as e.g., Sr\(_2\)RuO\(_4\).\(^{21}\) However, in highly-symmetric systems, the TRS breaking can also occur in fully-gapped states.\(^{36}\) Thus, the cubic Re\(_6\)(ZrH\(_x\))\(^{14,15}\) and Re\(_{32}\)Ti\(_5\) or the hexagonal La\(_2\)Ir\(_3\)\(^{16}\) all exhibit fully gapped superconducting states, but with TRS breaking. A point-group analysis of Re\(_6\)Zr\(_{14}\) reveals that a mixed singlet and triplet state is allowed to break the TRS. The continuous search for other low-symmetry NCSCs provides a good opportunity to find non-s-wave superconductors with TRS breaking, hence, furthering our understanding of the NCSC physics.

In summary, we investigated the noncentrosymmetric superconductor Re\(_{32}\)Ti\(_5\) by means of \( \mu \)SR and TDO techniques. Bulk superconductivity with \( T_c = 6 \) K was characterized by magnetization, transport, and specific heat measurements. Both the low-temperature penetration depth, superfluid density and the zero-field specific-heat data reveal a nodeless superconductivity in Re\(_{32}\)Ti\(_5\), well described by an isotropic s-wave model with a single gap. The spontaneous fields, which appear below \( T_s \) and increase with decreasing temperature, provide strong evidence that the superconducting state of noncentrosymmetric Re\(_{32}\)Ti\(_5\) breaks TRS and has an unconventional nature.

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