Unconventional superconductivity in the nickel-chalcogenide superconductor, TlNi$_2$Se$_2$

E. Jellyman$^1$, P. Jefferies$^1$, S. Pollard$^1$, E. M. Forgan$^1$, E. Blackburn$^1$, A. T. Holmes$^2$, R. Cubitt$^1$, J. Gavilano$^3$, Hangdong Wang$^5,6$, Jianhua Du$^5$, and Minghu Fang$^5,7$

$^1$School of Physics and Astronomy, University of Birmingham, Birmingham B15 2TT, United Kingdom
$^2$European Spallation Source, Lund, Sweden
$^3$Institut Laue Langevin, 71 Avenue des Martyrs, 38000 Grenoble, France
$^4$Paul Scherrer Institute, 5232 Villigen, Switzerland
$^5$Department of Physics, Zhejiang University, Hangzhou 310027, China
$^6$Department of Physics, Hangzhou Normal University, Hangzhou 310036, China and
$^7$Collaborative Innovation Centre of Advanced Microstructure, Nanjing 210093, China

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We present the results of a study of the vortex lattice of the nickel chalcogenide superconductor TlNi$_2$Se$_2$, using small angle neutron scattering. This is a heavy-fermion superconductor with the same crystal symmetry as the iron-arsenides. Previous work points to it being a two-gap superconductor, with an unknown pairing mechanism. No structural transitions in the vortex lattice are seen in the phase diagram, and only small variations in the anisotropy. Modeling of the form factor and penetration depth indicates that this material is an unconventional superconductor, with nodes in the gap structure.

INTRODUCTION

Nickel-chalcogenides are a new class of superconductor [1, 2, 5–8], with TlNi$_2$Se$_2$ synthesised in single crystal form and characterised in 2013 by Wang et al. [1]. TlNi$_2$Se$_2$ becomes superconducting below 3.7 K, and appears to be a heavy fermion material with an effective mass of $m^* = (14 - 20)m_e$. There is conflicting evidence as to the nature of the pairing mechanisms in this material [1, 2]. Thermal conductivity data and deviations from the Wiedemann-Franz law [9, 10] do not support a $d$-wave interpretation. However, the heat capacity shows a power-law dependence of the Sommerfeld coefficient: $\gamma_N = 58.33H^{0.5}$ [1]. This is typically associated with $d$-wave superconductors [11–13]. In the normal state, TlNi$_2$Se$_2$ shows Pauli paramagnetism [1]. Additional evidence from the heat capacity and thermal conductivity [2] infers a two-gap model with a lower gap suppressed at $H^* = 0.36H_{c2} = 0.29$ T. The two gaps are estimated to be $\Delta_1 = 0.84k_BT_c$ and $\Delta_2 = 2.01k_BT_c$ [1, 2]. To investigate this further, we have undertaken a survey of the vortex lattice (VL) using small-angle neutron scattering (SANS).

TlNi$_2$Se$_2$ has a tetragonal structure (Figure 1), with lattice parameters $a = (3.870 \pm 0.001)\AA$ and $c = (13.435 \pm 0.001)\AA$. It belongs to the $I4/mmm$ space group, like the iron-arsenides and CeCu$_2$Si$_2$ [14] (the first heavy-fermion superconductor discovered). The resistivity has an anisotropy ratio of $\rho_c/\rho_ab = 1.57$ [1]. The estimated ratio of the electron mean free path to the coherence length is $l_e/\xi_0 = 33.3 \gg 1$ (where $\xi_0 = 20.3$ nm [1]), which places our samples in the clean limit [29].

EXPERIMENTAL DETAILS

The work presented here was done on the D33 instrument at the Institut Laue-Langevin (ILL) [3]. Preliminary studies were carried out at SANS-I at the Paul Scherrer Institute (PSI).

The neutron wavelength used was $7\AA$ for $B \geq 0.2$ T and $12\AA$ for $B < 0.2$ T with a bandwidth of $\Delta\lambda/\lambda = 0.1$. The collimation was set to 12.8 m with the 2D multidetector 12 m from the sample. An aperture area of $1.08 \times 10^{-4}$ m$^2$ was used, mounted at the end of the waveguide of the beam between the sample and beam collimation. The samples were mounted in a 17...
in situ could be altered
insert [4]. The sample orientation about the vertical axis
T horizontal-field cryomagnet equipped with a dilution
with \( \phi \) rocks a rotation about the \( x \)-axis and \( \omega \) rocks a rotation about the
y-axis.

A mosaic of seven single crystals was prepared (Figure 1). The \( c \) axes are parallel to the field, \( B \) (approximately parallel to the neutron beam). The \( a \) and \( b \) axes were aligned at 45° to the vertical axis, so that when the sample was rotated relative to \( B \) about the vertical axis, the crystal symmetry is broken. This permits a single VL domain to be selected where two domains are visible.

To prepare the vortex lattice at a given temperature and field, the sample was cooled in an oscillating field through \( T_c \) to the target temperature. The size of the oscillation was always ±5 mT. This improves the quality and order of the VL [15], particularly at low fields. For temperature scans, data were collected by raising the temperature, rather than warming and cooling through \( T_c \) for each point.

At set values of field (\( B \)) and temperature (\( T \)), the diffraction pattern was collected by rocking through \( \omega \) and \( \phi \). Background scans were taken in the normal state at \( T > T_c \), in low- and high-field set-ups then subtracted from the VL foreground measurements. The resulting diffraction patterns were analysed using the software package GRASP [19]. Figure 3 are examples of such diffraction patterns.

The VL was measured with \( c \) at angle \( \Omega = 0^\circ \), 10° and 30° to \( B \) so as to select one VL domain. Field dependent measurements were taken over the range 0.05 T to 0.5 T.

Figure 2: Image of the mosaic of the seven single crystal samples. These samples are approximately 0.13mm thick and have a total volume of \( 4.68 \times 10^{-9} \text{m}^3 \). The solid lines indicate the \( ab \)-plane alignment of the crystals, rotated by 45° relative to the \( xy \)-axes. The dashed lines indicate the rocking axes relative to the crystal axes, with \( \phi \) rocks a rotation about the \( x \)-axis and \( \omega \) rocks a rotation about the \( y \)-axis.

\[ \Gamma_{VL} = \left( \frac{Q_0}{Q_{VL}} \right)^2 \] (1)

where \( Q_{VL} \) is the measured \( Q \) value in reciprocal space of the VL spots, and \( Q_0 \) is the expected position of the VL spots for an isotropic hexagonal lattice. \( Q_0 \) depends on \( B \), such that \( Q_0 = 2\pi \sqrt{2B/\sqrt{3} \Phi_0} \). A result of \( \Gamma > 1 \) suggests \( Q_{VL} \) is smaller than expected, while \( \Gamma < 1 \) suggests a larger \( Q_{VL} \) than expected. This can be summarised as a contraction or expansion, respectively, of the VL along the \( x \)-axis in reciprocal space.

A lack of change in the VL is unusual for unconventional pairing mechanisms, multi-gap systems and heavy fermion behaviour. These systems are usually accompanied by some form of VL structural change such as anisotropy or a transition from hexagon to square/rhombus [21-24] observed directly in the diffraction patterns with respect to angle and field modulation.

We might naively expect the VL structure to reflect the anisotropy seen in the structure, both crystalline (\( c/a = 3.47 \)), and electronic (\( \rho_c/\rho_{ab} = 1.57 \)) [1]. Instead Figure 4 would suggest that there is a general trend of a decrease in the magnitude of \( \Gamma_{VL} \) as the field is increased. Along the \( x \)-axis, the VL is slightly contracted at low fields and slightly stretched at higher fields. This effect is small, with some results for the higher fields still sitting within error of \( \Gamma_{VL} = 1 \); the effect is clearest at \( \Omega = 0^\circ \).

The most significant argument against an anisotropic interpretation of the VL is the lack of a relationship between \( \Omega \) and \( \Gamma_{VL} \). Typically we would expect anisotropic
Figure 4: Anisotropy of the VL calculated using equation 1. The reference line at $\Gamma_{VL} = 1$ indicates the area where there is no anisotropy in the VL. All three values of $\Omega$ indicate a similar linear relationship of the anisotropy with respect to $B$ with a negative gradient, passing through isotropy at $\approx 0.3$ T.

Figure 5: Anisotropy of the VL with respect to $\Omega$, at 0.1 T, 0.2 T and 0.3 T. The data is fitted to equation 2 with the values for $\Gamma_{ac}$ taken from the measured values of $\Gamma_{VL}(\Omega = 0)$ in Figure 4. Behaviour that conforms to the following relationship:

$$\Gamma_{VL} = \frac{\Gamma_{ac}}{\sqrt{\cos^2 \Omega + (\Gamma_{ac} \sin \Omega)^2}}$$

where $\Gamma_{ac}$ is the ratio between the major and minor axes of the VL in reciprocal space. This can be taken from the zero angle results given $\Gamma_{VL}(\Omega = 0) = \Gamma_{ac}$.

From Figure 5, we can see that the fit for 0.3 T is very poor, while the fits for 0.1 T and 0.2 T are slightly better. Overall we can see that there is some small presence of anisotropy in this system, but the fits do not entirely conform to observed behaviour in general. This suggests that the orientation of the VL spots and the shape of the VL changes by only a small amount with $\Omega$ and more sensitive to $B$ variation. These results illustrate the need for more intermediate angles and higher angles to improve the resolution and the range over which an anisotropy analysis can take place. More angles would likely improve the fitting procedure for analysing $\Gamma_{VL}(\Omega)$.

The anisotropy is very low in comparison to structural equivalents like KFe$_2$As$_2$ [23], where $\Gamma_{ac} \to 10$. An alternative approach is seeing if the spots rotate around the $z$ axis (|| to the beam) in $\chi$ rather than shift along the $x/y$-axes in $Q$. The $\chi$ angle is measured from the $y$-axis to a point in the diffraction pattern (such as a VL spot). Figure 6 illustrates the average angular separation between the pairs of top and bottom spots (Figure 3a). The opening angle is $\eta = 60^\circ$ for an isotropic hexagonal VL. Our results show that there is a weak linear relationship with $B$ giving a compressed $\eta$ below 0.3 T and expanded $\eta$ above. This is consistent with what we see in $\eta$ in Figure 6.

The observed behaviour of the top/bottom spots suggests that these spots are drifting outwards along the $x$-axis more rapidly than they should with $B$. ARPES results in [20] indicate nodes in the gap structure that are concurrent with the fourfold symmetry of the crystal structure. Given the orientation of the crystal (see Figure 2), this suggests that these spots are moving closer to alignment with the fourfold symmetry axis of the nodes, parallel to the $a - b$ axes, with respect to $B$.

Overall the available evidence suggests a weak anisotropy for $\Omega \leq 30^\circ$, with a maximum anisotropy of $\Gamma_{ac} \approx 1.2$ with respect to $B$ and $\Omega$. We can posit that the anisotropy present in Tln$_2$Se$_2$ is weak but linked to the underlying gap and crystal structure, as previously...
discussed. We may also conclude that the $B$ dependent anisotropy shows little evidence for the suppression of a smaller gap at the predicted field of $H^* = 0.29$ T. If a second gap were suppressed it would likely present as a discontinuity in the signal.

Despite Refs. [1] and [2] identifying the material as a two-gap heavy fermion superconductor with potentially unconventional pairing mechanisms, there appears to be no significant structural transitions of the VL and no significant discontinuities in the VL signal for $\Omega$ and $B$ variation. Unconventional superconductors, particularly $d$-wave systems, tend to be accompanied by some form of VL structural change such as anisotropy or a transition to square/rhombus [21–24]. This does not however, rule out the unconventional case for this material.

**Integrated intensity and form factor**

By rotating in $\Omega$ we select a single domain of the VL for analysis. The 2nd domain signal for $\Omega = 0^\circ$ is comparable to the 1st domain signal. The 2nd domain rapidly diminishes with increased $\Omega$. In Figure 7 we see both domains summed together. The larger errors for $\Omega = 10^\circ$, $30^\circ$ are due to the weak 2nd domain signal here. The intensity of the VL spots with respect to the rocking angle ($\omega, \phi$) was fitted with a Gaussian function. We obtain the signal strength of the VL by integrating over the area between the foreground Gaussian curve and the background at for each diffraction spot. The integrated area is the integrated intensity, $I(q)$. We observe an increase in $I(q)$ of the VL with increased angle, $\Omega$, in Fig. 7.

According to [1][2] We would expect to see a suppression of the smaller gap at $H^* = 0.29$ T. In the case of the integrated intensity this would manifest as a sudden drop in the the VL signal, $I(q)$. A suppressed gap would decrease the number of Cooper pairs supporting the superconducting state above $H^*$. We do not observe a discontinuous or smooth transition in the vicinity of $H^*$ to indicate such a gap suppression.

The form factor, $F(q)$ gives a measure of the modulation of the VL field on top of the applied field. This is calculated using the Christen formula for the integrated intensity [25]

$$I(q) = 2\pi V \lambda_n^2 \phi_n \left( \frac{\gamma_n}{4} \right)^2 \frac{(|F(q)|)^2}{\Phi_0 \cos(\zeta) q} \tag{3}$$

where $V = 4.68 \times 10^{-9}$ m$^3$ is the total volume of the sample mosaic, $\lambda_n = 7$ Å, $12$ Å is the neutron wavelength, $\phi_n = 7.71 \times 10^9$ cm$^{-2} s^{-1}$ is the neutron flux (extracted via a direct beam measurement with known aperture size of 1.08 $\times$ 10$^{-4}$ m$^2$), $\gamma_n = 1.92$ is the gyromagnetic ratio for a neutron, $F(|q|)$ is the form factor, $\Phi_0$ is the flux quantum, $q$ is the q-value associated with the applied field for the diffraction pattern and $\zeta$ is the Lorentz angle (the angle between the spot being analysed and the normal of the rocking angle axis). In this investigation all of the spots are averaged to get $\langle I(q) \rangle$ to analyse how the whole VL is behaving. Equation 3 when rearranged, gives us the FF results seen in Fig. 8 and Fig. 11.

Additionally, a single set of results was taken at $\Omega = 30^\circ$ rotation of the sample and 0.15 T with temperature variation from 100 mK up to 3.5 K. The previous, field dependent, data in Fig. 7 indicates that the strongest VL signal is at a $30^\circ$ rotation from the field and 0.15 T at $\lambda_n = 7$ Å and so would give the largest intensity for the VL at $T \simeq T_c$ for $\lambda_n = 7$ Å (and ensures the 2nd domain has the smallest possible contribution). The $T$
The modified London model is used \cite{29} to account the finite core size of the vortices in the VL, a fermion so a BCS fit will likely be insufficient. This material has already been established as heavy; we need to have a good representation of the behaviour of the flux cores. By substituting equation 4 into equation 3, \( \lambda(0) \) can be extracted from \( I(q) \).

For a complete analysis, we must compare ideal models of \( s \)-wave and nodal (unconventional) superconductivity to the original \( I(q) \) data, such that the representation of the data is not affected by assumptions for the variation of the \( \xi \) (of which little is known for this material). To do this the models are created from equation 3 and equation 4. In order to calculate the models we need to have a good representation of the behaviour of the \( \lambda(T) \). This is the step where we involve variation in the pairing symmetry. We use previous attempts by Prozorov et al. \cite{26,28} using a modified BCS theory that can accommodate unconventional pairing mechanisms and nodes. The Prozorov framework is a simplification of the work by Izawa et al. \cite{33,34} on nodal pairing for point and line nodes. We start at the Lewis two-fluid model for \( \lambda(T) \),

\[
\lambda(T) = \frac{\lambda(0)}{\sqrt{1 - t^2}};
\]

where \( t = T/T_c \). Although this can give a reasonable estimate for \( \lambda(T) \) it only represents an ideal for a clean, local BCS superconductor. Instead we must use the procedure derived by Prozorov et al. \cite{26} as follows

\[
\lambda(T) = \frac{\lambda(0)}{\sqrt{1 - p^2}}
\]

where \( p \) is estimated to be \( p = 2 \) for \( s \)-wave. Empirical fits have shown \( p = 4 \) is not universal \cite{28,29}, with \( p = 2 \) being a better representation of \( s \)-wave behaviour. \( p = 4/3 \) has similarly been shown to fit for nodal gap structure \cite{28}. We can generate comparative models for \( F(q, T) \) and \( I(q, T) \) and see how the empirical results compare. This approach helps classify the pairing symmetry of the gap function and potentially highlights any suppression of specific pairing mechanisms based on changes in \( p \). The models created with this method are given in Figures 9, 11 and 12 for \( I(q, T) \), \( F(q, T) \) and \( \lambda(T) \), respectfully.

We see from Figure 9 a strong correlation of the integrated intensity to the nodal model. The integrated intensity is calculated by averaging the integrated intensity of all six VL spots in the first domain with respect to temperature, giving an impression of the overall behaviour of the VL signal. This result is surprising given the lack of obvious VL structural reordering with field, temperature or angle.

Linear behaviour in a logarithmic plot of \(|F(q)|\) with field variation is usually indicative of conventional behaviour with respect to field as it indicates little change.
in the coherence length with field, whereas non-linear behaviour could be indicative of unconventional superconductivity. In the case of Figure 10 we see areas of linear behaviour in the logarithmic plot, but not across the entire range of fields accessed; specifically the lowest and highest temperatures of the range.

Figure 11 very much indicates the same as Figure 9; the signal conforms most closely to a nodal interpretation. This is very surprising considering the lack of structural changes but there is room for error at the extremities of the temperature range to suggest an evolution of the pairing mechanism. Overall we see a strong fit in Figures 9 and 11 to an unconventional picture dominating the pairing symmetry in the superconducting state.

Figures 9 and 11 represent the most visually useful analysis of the available temperature dependent data for trying to identify the pairing mechanism. A direct comparison with ideal models lends more weight to the argument for a nodal interpretation contrary to conclusions by Hong et al. [2]. In order to compliment the data fits and $|F(q)|$ calculations we also present $\lambda(T)$ calculations using equation 4 for the data and equation 6 for the comparative models. The penetration depth analysis is presented in Figure 12.

Although $\lambda(T)$ is presented in comparison to models, a fit of the data was also performed using equation 6. The fitting procedure yields the following values: $\lambda_\omega = 156.52 \pm 1.75$ nm, $p = 1.37 \pm 0.09$, $T_c = 3.60 \pm 0.05$ K, $\lambda_\phi = 149.60 \pm 4.58$ nm, $p = 1.20 \pm 0.24$ and $T_c = 3.90 \pm 0.25$ K. These separate fits indicate an interesting divergence in the penetration depth on approaching $T_c$, but with values of $p$ within $<1\sigma$ of each other. This could be suggestive of multiple gaps with their presence only becoming apparent as $T \to T_c$ and the smaller gap experiences greater degrees of suppression as a result of a larger penetration depth.

**DISCUSSION**

We can likely rule out multiple gaps in the gap structure of TlNi$_2$Se$_2$. If we were seeing multiple gaps, two gaps as proposed previously [1], we would see evidence of
In previous work TiNi$_2$Se$_2$ showed some evidence of potentially being a $d$-wave superconductor \cite{1}. Generally speaking $d$-wave superconductivity can be identified in SANS studies by a rearrangement of the VL with respect to field or angle \cite{24}. In this investigation we have seen no such rearrangement. This does not preclude the existence of $d$-wave pairing entirely, but it is far less likely. Anisotropy is small in the VL with field and angle variation but is consistent with field and reflects a possible shift of flux lines attempting to align with the fourfold crystal and nodal structure.

The most likely candidate for the gap structure is a nodal gap due to the enhancement of $\lambda(T)$, the linear behaviour of $F(q,T)$ and the agreement between the data and the nodal gap behaviour outlined by the work of Prozorov et al. \cite{25,26}. The conspicuous lack of structural changes in the VL is unusual for an unconventional superconductor and certainly indicates that the material has nodes, but does not likely have a fully $d$-wave pairing system \cite{24}. This is comparable to the interpretation of the analogous material KFe$_2$As$_2$, which is an unconventional, nodal $s$-wave superconductor, sometimes denoted as $s\pm$-wave \cite{21,23}. Given that a lack of structural reordering in the VL or anisotropy is an indicator of a lack of any $d$-wave pairing present \cite{24} in the Fermi surface, this raises the question of whether the VL anisotropy is in fact being inhibited in some way which could be made clear by probing at much higher angles of $\Omega$. What seems more probable is that the apparent $d$-wave preference is an artifact of nodes in the superconducting gap, supported by the consistent adherence to the nodal models used in this work.

**CONCLUSION**

We can conclude that TiNi$_2$Se$_2$ is a nodal superconductor given the linear behaviour of the form factor. However, given the weak anisotropy and lack of rearrangement of the VL we cannot necessarily attribute this nodal behaviour to $d$-wave pairing. Given the lack of features in the vicinity of the predicted suppression field, $H^* = 0.29$ T in field dependent results, we must also conclude that there is only a single nodal gap controlling the superconductivity in this material.

Continued investigation of this material will clarify some of the unusual results given in this work. It would be prudent to continue SANS studies of the VL up to much larger angles of rotation with respect to the field in order to probe for any structural changes in the VL and to see how the form factor signal continues to evolve with angle.

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*EXJ001@bham.ac.uk

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