Raising $T_c$ in charge density wave superconductor $\text{ZrTe}_3$ by Ni intercalation

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received 17 March 2011; accepted in final form 18 May 2011
published online 23 June 2011

PACS 74.70.Ad - Metals; alloys and binary compounds (including A15, MgB$_2$, etc.)
PACS 74.62.Dh - Effects of crystal defects, doping and substitution
PACS 74.25.-q - Properties of superconductors

Abstract - We report on the discovery of bulk superconductivity in $\text{Ni}_{0.05}\text{ZrTe}_3$ at $T_c = 3.1\,\text{K}$, obtained through Ni intercalation. Superconductivity coexists with charge density wave (CDW) state with $T_{\text{CDW}} = 41\,\text{K}$. When compared to the parent material $\text{ZrTe}_3$, the filamentary superconducting transition is substantially increased whereas $T_{\text{CDW}}$ is suppressed. The analysis of superconducting state indicates that $\text{Ni}_{0.05}\text{ZrTe}_3$ is an intermediately coupled superconductor.

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Introduction. – Charge density wave (CDW) and superconductivity are distinctive quantum orders that emerge due to Fermi surface (FS) instabilities [1]. Competition and coexistence between CDW and superconductivity in low-dimensional materials is one of the most fundamental problems in condensed matter physics. Among CDW superconductors, layered MX$_2$ compounds and chain-type MX$_3$ compounds ($M = \text{transition metal}$, and $X = \text{S, Se or Te}$) are well known [2–4]. It was recently pointed out that chalcogenide superconductors represent a weak-coupling limit of the melting of the stripe (or smectic) order in cuprates [5]. Furthermore, the dome-like $T_c(x)$ [4] in Cu$_x$TiSe$_2$ and pairing mechanism was proposed to arise from the quantum criticality related to fluctuations in CDW order [6,7]. Therefore the discovery of new superconductors in this materials class is of significant interest.

$\text{ZrTe}_3$ belongs to a family of trichalcogenides MX$_3$ ($M = \text{Ti, Zr, Hf, U, Th, and } X = \text{S, Se, Te}$). The structure consists of infinite X-X chains formed by stacking MX$_3$ prisms along the crystallographic b-axis. The polyhedra are arranged in double sheets and stacked along monoclinic c-axis by van der Waals forces (fig. 1(a)) [8]. $\text{ZrTe}_3$ exhibits CDW transition at $T_{\text{CDW}} = 63\,\text{K}$. The CDW nesting vector $\vec{\nu}_{\text{CDW}} = (1/14, 0, 1/3)$ has no component in the chain-axis direction [9]. This is different from another well-known quasi-one-dimensional CDW compound NbSe$_3$ with X-X chains which exhibits a resistivity anomaly along the chain axis [10]. The $\rho(T)$ of $\text{ZrTe}_3$ is quasi-two-dimensional, metallic below 300 K with anomalies due to CDW transition [11], and superconductivity below $T_c = 2\,\text{K}$. The superconductivity is not bulk but filamentary [12]. Theoretical calculations [13] and photoemission study [14] indicate that the CDW transition is driven by the nesting of parallel planar sections of the FS which are related to the Te $5p_x$ band along the Te-Te chains. Other parts of the FS remain unaffected and are

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Fig. 1: (Colour on-line) (a) The structure of $\text{ZrTe}_3$ with green (orange) symbols for Zr (Te) atoms. (b) Powder XRD pattern of $\text{Ni}_{0.05}\text{ZrTe}_3$. Inset: photo of typical single crystals of $\text{Ni}_{0.05}\text{ZrTe}_3$. (c) XRD result of $\text{Ni}_{0.05}\text{ZrTe}_3$ single crystal.
responsible for superconductivity. Therefore, CDW and filamentary superconductivity coexist in ZrTe₃ due to the multiband nature of the Fermi surface.

On the other hand, ZrTe₃ (fig. 1(a)) exhibits an uncommon pressure dependence of CDW and superconductivity [15]. With increasing pressure $T_{CDW}$ initially increases, then decreases up to 2 GPa and abruptly vanishes near 5 GPa. $T_c$ falls to below 1.2 K at 0.5 GPa and cannot be observed up to 5 GPa. At higher pressures superconducting transition emerges again and increases continuously from 2.5 K to 4.7 K at 11 GPa. $T_{CDW}(P)$ and $T_c(P)$ above 5 GPa can be explained by pressure-induced FS modifications. High pressures favor three-dimensionality, therefore the area of the planar portions of FS will decrease and the CDW transition will be suppressed. Consequently to superconducting $T_c$, a competing FS instability will be favored. However, the pressure dependence above 2 GPa is still unclear. $T_c(P)$ and $T_{CDW}(P)$ indicate that the changes in FS are rather sensitive to crystal structure modification. Besides hydrostatic pressure, doping and intercalation can be used to tune the crystal structure, lattice properties, shape and characteristics of the FS. For example, intercalating external atoms into the interlayer weak van der Waals gap in ZrTe₃ single crystals. Ni intercalation induces bulk superconductivity with enhanced $T_c$ while the $T_{CDW}$ shifts to lower temperatures, indicating coexistence of CDW and superconductivity.

**Experimental.** Single-crystal Ni₃ZrTe₃ was grown via the chemical vapor transport (CVT) method. The source and growth zone were set at 700°C for 2 days and then kept at 720°C and 645°C, respectively, for 10 days. Golden plate-like single crystals with a typical size of $2 \times 2 \times 0.2$ mm³ were obtained. The crystal structure and phase purity were examined by powder and single-crystal X-ray diffraction pattern (XRD) with Cu $K_{\alpha}$ radiation ($\lambda = 1.5418$ Å) using a Rigaku MiniFlex X-ray machine. The structure parameters are extracted by fitting the XRD spectra using the Rietica software [18]. The composition of the Ni₃ZrTe₃ single crystal was determined by examination of multiple points on the crystals using energy dispersive X-ray spectroscopy (EDS) in a JEOL JSM-6500 scanning electron microscope. The measured compositions are Ni₀.₀₅ZrTe₃ as $Ni_{0.05}ZrTe_3$, abbreviated as $Ni_{0.05}ZrTe_3$. Physical property measurements were performed in Quantum Design PPMS-9 and MPMS XL 5 instruments.

**Results and discussion.** Powder X-ray diffraction (XRD) result (fig. 1(b)) of ground samples indicates that all peaks of Ni₀.₀₅ZrTe₃ can be indexed using the structure of ZrTe₃, i.e. the intercalation does not change the structure of the mother compound. The fitted lattice parameters are $a = 0.5895(1)$ nm, $b = 0.3923(1)$ nm, $c = 1.0160(2)$ nm, and $\beta = 97.76(1)^{\circ}$. Our refinement of pure ZrTe₃ crystals gave lattice parameters $a = 0.5863(3)$ nm, $b = 0.3927(7)$ nm, $c = 1.0095(5)$ nm and $\beta = 97.75(1)^{\circ}$. Hence, the $a$- and $c$-axis lattice parameters of Ni₀.₀₅ZrTe₃ are larger than those of ZrTe₃. The substitution on the Zr site is unlikely due to the smaller size of the Ni ion ($r_{Ni^{2+}} = 69$ pm) than the Zr ion ($r_{Zr^{4+}} = 72$ pm). This indicates that Ni is most likely intercalated in the van-der-Waals–bonded crystallographic layers in ZrTe₃. The XRD pattern of single crystals (fig. 1(b) inset) reveals that the crystal surface is normal to the $c$-axis with the plate-shaped surface parallel to the $ab$-plane (fig. 1(c)).

Figure 2(a) shows the temperature dependence of resistivity at zero field from 1.9 K to 300 K. Both $\rho_a(T)$, and $\rho_b(T)$ undergo a relatively sharp superconducting transition at $T_{c,0\text{set}} = 3.74(3)$ K and 3.59(2) K, respectively (upper inset). It should be noted that $\rho_c(T)$ is metallic in the normal state with no anomalies due to CDW and with a residual resistivity ratio (RRR) of 6. $\rho_c(T)$ exhibits a transition located at 41 K (lower inset fig. 2(a)). This can be ascribed to CDW since it is similar to
resistivity anomalies in ZrTe$_3$ [11]. However, the transition has been depressed from around 65 K to 41 K. Therefore, there is a coexistence of superconductivity and CDW in Ni$_{0.05}$ZrTe$_3$ similar to ZrTe$_3$ but with nearly double bulk $T_c$ [12]. The filamentary superconductivity and the CDW originate from different sections of FS in ZrTe$_3$. From the difference in $T_c$, $T_{CDW}$ in Ni$_{0.05}$ZrTe$_3$ and ZrTe$_3$ we can conclude that Ni intercalation dramatically changes the FS related to the superconductivity but has a minor effect on the FS parts associated with the CDW transition. Inter- calation has a distinctively different effect when compared to pressure. The reason could be that intercalation only modifies one part of the FS related to superconductivity whereas pressure increases the three-dimensionality of all parts.

Figure 2(b) shows the temperature dependence of the ac susceptibility of Ni$_{0.05}$ZrTe$_3$ single crystal with dimension of $2 \times 1.5 \times 0.24$ mm$^3$ for $H \parallel ab$ and $H \parallel c$. The single sharp peak of $4\pi\chi''$ accompanied by a very steep transition in $4\pi\chi'$, indicates that the sample is rather homogeneous. The onset temperature for both field directions is 3.1 K. This is slightly lower than that obtained from resistivity measurement. The transition width $\Delta T_c$ is 0.3 K. For $H \parallel ab$, the value of $-4\pi\chi'$ at 1.8 K is 110% without any demagnetization factor correction, indicating that the superconductivity is bulk. This is different from the mother compound ZrTe$_3$ where superconductivity is undetectable in magnetization due to its filamentary nature below $T_c = 2$K. The inset in fig. 2(b) shows the dc magnetic susceptibility for field along $\hat{a}$, $\hat{c}$ axes with zero-field cooling (ZFC) and field cooling (FC). For ZFC curves, $T_{c, \text{onset}}$ and $\Delta T_c$ are consistent with the results of ac susceptibility and the values of $-4\pi\chi$ at 1.8 K are nearly identical. On the other hand, the volume fraction estimated from the FC curve is about 2% at 1.8 K for $H \parallel c$, similar to other intercalated compounds such as (Pyridine)$_{1/2}$TaS$_2$ [19], YbC$_6$ [20], Ni$_{0.05}$TaS$_2$ [21]. The small magnetization values for FC is likely due to the complicated magnetic flux pinning effects in the intercalated compounds [19].

The results of the dc magnetization vs. field $M(H)$ at various temperatures for both directions are shown in fig. 3(a). The shape of the $M(H)$ curves points that Ni$_{0.05}$ZrTe$_3$ is a typical type-II superconductor. Figures 3(b) and (c) show the initial $M(H)$ curves at the low-field region. All curves clearly fall on the same line and deviate from linearity for different temperatures. Linear fits describe the Meissner shielding effects (“Meissner line”) (figs. 3(b) and (c)). The obtained slope of the linear fit up to 30 Oe at the lowest temperature of our measurement $T = 1.8$ K is $-0.995(5) \approx -1$. This corresponds to $-4\pi M = H$ for $H \parallel ab$, where the demagnetization factor is negligible.

The value of $H_{c1}^*$, at which the field starts to penetrate into the sample can be determined by examining the point of deviation from the Meissner line on the initial slope of the magnetization curve. $H_{c1}^*$ is not the same as the real lower critical field, $H_{c1}$, due to the geometric effect. $H_{c1}$ can be deduced from the first penetration field $H_{c1}^*$, assuming that the magnetization $M = -H_{c1}$ when the first vortex enters into the sample. Thus $H$ has been rescaled to $H = H_o - NM$ and $H_{c1} = H_{c1}^*/(1 - N)$, where $N$ is the demagnetization factor and $H_o$ is the external field [22]. We estimate demagnetization factors 0.075 and 0.781 for $H \parallel ab$ and $H \parallel c$ by using $H_{c1} = H_{c1}^*/\tanh(\sqrt{0.366b/a})$, where $a$ and $b$ are the width and thickness of a plate-like superconductor [23]. This is consistent with previous results, i.e. the demagnetization factor for $H \parallel ab$ is negligible. However, the demagnetization factor must be considered for $H \parallel c$. The $M(H)$ curve for $H \parallel c$ with demagnetization correction is shown in fig. 3(c) and the slope of the fitted line in the linear region is $-0.963(1) \approx -1$. From the above and constrained with the resolution limit of our magnetization measurement $\Delta m = 3 \times 10^{-5}$ emu we extract the temperature dependence of $H_{c1}$ for both field directions (fig. 3(d)). The $H_{c1}(T)$ can be well explained by $H_{c1}(T) = H_{c1}(0)(1 - (T/T_o)^2)$. The obtained $H_{c1,ab}(0)$ and $H_{c1,c}(0)$ are 50(1) and 321(8) Oe, respectively. On the other hand, the anisotropy of $H_{c1}$, $\gamma_{H_{c1}} = H_{c1,ab}/H_{c1,c}$, increases with decreasing temperature and is estimated about 6.4 at $T = 1.8$ K. This is much larger than in MX$_2$ compounds [21].

The main panel of fig. 4(a) shows the temperature dependence of the specific heat of Ni$_{0.05}$ZrTe$_3$ below 5 K at $H = 0$, 10, and 50 kOe. A jump at 3 K can be clearly seen at $H = 0$, indicating bulk superconductivity of Ni$_{0.05}$ZrTe$_3$ sample. The $T_{c, \text{onset}}$ of 3.1 K determined
from the specific-heat jump is consistent with that obtained from the susceptibility and transport measurements. At $H = 1$ kOe, the superconducting anomaly is shifted to lower temperatures. The normal state is recovered at $H = 50$ kOe. In order to obtain the electronic specific-heat coefficient $\gamma$ and Debye temperatures $\Theta_D$, the low-temperature specific heat at $H = 50$ kOe is fitted using $C_p/T = \gamma + \beta T^2$. The obtained parameters are $\gamma = 2.7(1)$ mJ/molK$^2$ and $\Theta_D = 192.4(1)$ K using $\Theta_D = (12\pi^4NR/5\beta)^{1/3}$, where $N = 4$ is the number of atoms per formula unit and $R$ is the gas constant. The high-temperature specific heat approaches the value of $3NR$, in accordance with the Dulong-Petit law (fig. 4(a), inset). According to the McMillan formula for electron-phonon–mediated superconductivity [24], the electron-phonon coupling constant $\lambda$ can be determined by

$$T_c = \frac{\Theta_D}{1.45} \exp \left[ -\frac{1.04(1+\lambda)}{\lambda - \mu^*(1+0.62\lambda)} \right],$$

where $\mu^* \approx 0.13$ is the common value for Coulomb pseudopotential. By using $T_c = 3.1$ K and $\Theta_D = 192.4$ K, we obtain $\lambda = 0.63$, indicating an intermediate coupling superconductor. The electronic specific-heat part $C_{es}$ obtained by subtracting the phonon part from the total specific heat is shown in fig. 4(b). From fits to $C_{es}/T - T$ below $T_c$ using the BCS formula for the electronic contribution to the specific heat [25], the ratio of the gap and the critical temperature is about, $2\Delta/k_BT_c = 3.00(2)$. This is smaller than the typical BCS value (3.53) in the weak-coupling limit [25]. The solid red line in fig. 4(b) shows the data simulation for an isotropic $s$-wave BCS gap. The specific-heat jump at $T_c$, $\Delta C_{es}/\gamma T_c = 1.20(3)$, is somewhat smaller than the weak-coupling value 1.43 [24]. The calculated coupling strength from the heat capacity anomaly could be influenced by the rather large fitting inaccuracy near $T_c$ and somewhat broad superconducting transition width in $C/T(T)$.

![Fig. 4](image_url) (Colour on-line) (a) Low-temperature specific-heat behavior of Ni$_{0.05}$ZrTe$_3$ plotted as $C_p/T$ vs. $T$ at $H = 0$, 1 and 50 kOe. The solid line is a fit described in the text. Inset: temperature dependence of $C_p(T)$ from 1.95 K to 300 K at $H = 0$ kOe. (b) Temperature dependence of the electronic specific heat plotted as $C_{es}/T$ vs. $T$ at $H = 0$ kOe. The solid line shows fitted result of $C_{es}/T$ assuming an isotropic $s$-wave BCS gap.

The temperature-dependent resistivity $\rho_0(T)$ of Ni$_{0.05}$ZrTe$_3$ below 5 kOe in magnetic fields for $H \parallel a$ and $H \parallel c$ is shown in figs. 5(a) and (b). With increasing magnetic fields, the resistivity transition width becomes broader and the onset of superconductivity gradually shifts to lower temperatures. At $H = 5$ kOe, for $H \parallel c$, the superconducting transition cannot be observed above 1.9 K, whereas for $H \parallel a$, the superconductivity above 1.9 K is nearly suppressed at $H = 10$ kOe.

![Fig. 5](image_url) (Colour on-line) Temperature dependence of the resistivity $\rho_0(T)$ of Ni$_{0.05}$ZrTe$_3$ for (a) $H \parallel a$ and (b) $H \parallel c$ at the various magnetic fields. (c) Temperature dependence of the upper critical field $H_{c2}$ for $H \parallel a$ and $H \parallel c$. Solid lines show the fitted result using WHH formula described in the text.

The temperature-dependent resistivity $\rho_0(T)$ of Ni$_{0.05}$ZrTe$_3$ below 5 kOe in magnetic fields for $H \parallel a$ and $H \parallel c$ is shown in figs. 5(a) and (b). With increasing magnetic fields, the resistivity transition width becomes broader and the onset of superconductivity gradually shifts to lower temperatures. At $H = 5$ kOe, for $H \parallel c$, the superconducting transition cannot be observed above 1.9 K, whereas for $H \parallel a$, the superconductivity above 1.9 K is nearly suppressed at $H = 10$ kOe.

Figure 5(c) shows the upper critical field $H_{c2}(T)$ of Ni$_{0.05}$ZrTe$_3$ corresponding to the temperatures where the resistivity drops to 90% of the normal-state resistivity $\rho_{n,b}(T,H)(T_{c,\text{onset}})$ at 5 K. Since the Pauli limiting field $H_{P}(0) = 1.84T_c > 55$ kOe, the orbital effect should be the dominant pair-breaking mechanism. According to the conventional one-band Werthamer-Helfand-Hohenberg (WHH) theory, which describes the orbital limited upper critical field of dirty type-II superconductors [26], $H_{c2}$ can be described by

$$\ln \frac{1}{T} = \psi \left( \frac{1}{2} + \frac{\bar{h}}{2t} \right) - \psi \left( \frac{1}{2} \right),$$

where $t = T/T_c$, $\psi$ is a digamma function and

$$\bar{h} = \pi^2 T_c \left( \frac{4H_{c2}}{\pi^2 T_c (-dH_{c2}/dT)_{T=T_c}} \right).$$
Table 1: Superconducting parameters of Ni$_{0.05}$ZrTe$_3$.

| Ni$_{0.05}$ZrTe$_3$ | H$_c$(0) (Oe) | H$_c$(0) (kOe) | H$_c$(0) (Oe) | ξ(0) (nm) | λ(0) (nm) | κ(0) |
|-------------------|---------------|---------------|---------------|-----------|-----------|------|
| H || ab           | 50(1)         | 12.28(860)    | 449(13)      | 26(1)     | 78(11)   | 19.3(8) |
| H || c            | 321(8)        | 4.862(340)    | 10(1)        | 1242(100) | 3.0(3)    |      |

The $H_c(T)$ fits are shown by solid and dotted lines in fig. 5(c). The obtained $-dH_c/dT|_{T=T_c} = 5.049(285)$ kOe, $T_c = 3.51(4)$ K for $H || a$ and $-dH_c/dT|_{T=T_c} = 2.016(122)$ kOe, $T_c = 3.48(4)$ K for $H || c$ are consistent with the $T_{c,\text{onset}}$ ($\rho_b = 90\%\rho_{ab}(T, H = 0) = 3.59(2)$ K). The estimated upper critical fields are $H_{c2}(0) = 12.28(860)$ kOe and 4.862(340) kOe for $H || a$ and $H || c$. From the $H_{c2}(0)$ zero-temperature coherence length $\xi(0)$ can be estimated with Ginzburg-Landau formula $H_{c2,c}(0) = \Phi_0/[2\pi\xi_0^2(0)]$, and $H_{c2,ab}(0) = \Phi_0/[2\pi\xi_{ab}(0)\xi_c(0)]$, where $\Phi_0 = 2.07 \times 10^{-15}$ Wb. Based on the values of $H_{c1}(0)$ and $H_{c2}(0)$, the Ginzburg-Landau (GL) parameter $\kappa$ is obtained from $H_{c2}(0)/H_{c1}(0) = 2\kappa^2/(\ln\kappa + 0.08)$. And the thermodynamic critical field $H_{c1}(0)$ can be obtained from $H_{c1}(0) = H_{c1,ab}(0)/[\sqrt{2}\kappa_{ab}(0)]$. The GL penetration length $\lambda_{ab}(0)$ and $\lambda_c(0)$ can be evaluated using $\kappa_{c}(0) = \lambda_{ab}(0)/\xi_{ab}(0)$, and $\kappa_{ab}(0) = [\lambda_{ab}(0)\lambda_c(0)/\xi_{ab}(0)\xi_c(0)]^{1/2}$ [27]. All of the obtained parameters are listed in table 1.

Conclusion. – In summary, we have discovered the bulk superconductivity in Ni-intercalated ZrTe$_3$ and presented detailed characterization of the superconducting state. The bulk superconducting $T_c$ was nearly doubled when compared to filamentary $T_c$ in the parent material ZrTe$_3$. The normal-state electrical resistivity indicates that the CDW transition is somewhat suppressed with Ni intercalation, coexisting with bulk superconductivity. The Ni intercalation substantially changes the FS related to the superconductivity but has a minor effects on the FS parts associated with CDW. This is rather different from the pressure effects. Our results suggest intermediate coupled superconductivity and possible rich vortex physics.

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We thank J. WARREN for experimental support at Brookhaven National Laboratory (BNL). This work was carried out at BNL, which is operated for the U.S. Department of Energy by Brookhaven Science Associates DE-Ac02-98CH10886.

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