Superconductivity at 5.2 K in ZrTe$_3$ polycrystals and the effect of Cu and Ag intercalation

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Abstract
We report the occurrence of superconductivity in polycrystalline samples of ZrTe$_3$ at temperature 5.2 K at ambient pressure. The superconducting state coexists with the charge density wave (CDW) phase, which sets in at 63 K. The intercalation of Cu or Ag does not have any bearing on the superconducting transition temperature but suppresses the CDW state. The feature of a CDW anomaly in these compounds is clearly seen in the DC magnetization data. Resistivity data are analyzed in order to estimate the relative loss of carriers and reduction in the nested Fermi surface area upon CDW formation in ZrTe$_3$ and the intercalated compounds.

1. Introduction

The interplay of charge density wave and superconducting (SC) states continues to be a subject of significant interest [1]. Low dimensional chalcogenides of transition metals are among the most well studied systems that show the coexistence of these competing phenomena. 2H–NbSe$_2$ shows a CDW at 32 K and SC at 7 K, whereas Cu intercalation of 1T–TiSe$_2$ suppresses the CDW and introduces SC at low temperature [2, 3]. In recent reports, copper and nickel intercalated compounds of ZrTe$_3$ have been shown to exhibit bulk superconducting properties at 3.8 K and 3.1 K respectively [4, 5]. The parent compound ZrTe$_3$ shows an SC transition below 2 K but the volume of the SC fraction is very small (<5%) [6]. It has unique crystal structure among the IV trichalcogenides and has triangular prismatic chains. There are two identical chains connected by inversion symmetry in the monoclinic unit cell, in such a way that neighboring chains (parallel to the $b$ direction), make alternate chain pairs [7–9]. The electrical resistivity of ZrTe$_3$ single crystals shows metallic behavior with an anisotropy ratio of 1:1:10 for $\rho_a$, $\rho_b$, $\rho_c$ respectively [7]. The metallic properties in ZrTe$_3$ emerge as a result of enhanced overlap between bands of the different chains (the interchain Zr–Te distance is shorter than the intrachain distance). The lesser difference in the electronegativities of Zr and Te compared to other chalcogen atoms makes more electrons available for conduction [6, 10].

The electrical resistivity of ZrTe$_3$ measured along the different directions shows the CDW anomaly at $T_{CDW}$ = 63 K in $a$ and $c$ directions but not in the $b$ direction [6, 7]. Photoemission studies and band structure calculations attribute this behavior as due to the nesting in a small electron pocket of the highly directional Te–Te chains, while other sheets of the Fermi surface remain unaffected [11]. The opening of a gap in the electronic dispersion of the band follows the BCS model of a Peierls transition with a mean field transition temperature about four times higher than $T_{CDW}$ [12]. The dependence of the CDW transition on the pressure was very unusual. $T_{CDW}$ initially increased to 114 K at 1 GPa and then decreased monotonically before abruptly vanishing at 5 GPa [13, 14]. The increase in the pressure enhances the three dimensionality of the structure and thus diminishes the area of the planar portion of the Fermi surface (FS), resulting in the reduction of the nested FS. The effect of pressure on the CDW is akin to that of the doping of the foreign atoms in the lattice, where redistribution of charge lead to a reduction in the nested FS [13, 14].

Previous studies have reported the presence of filamentary SC in single-crystalline ZrTe$_3$ with $T_c$ < 2 K, with weakly coupled SC filaments aligned parallel to the $a$ axis with the
spacing of 4 nm [6, 7]. These SC filaments run parallel to the a axis, the perpendicular direction of the triangular prismatic chain of ZrTe₃. There is a strong pressure dependence on Tc as in the case of TCDW. Tc initially falls below 1.2 K at 0.5 GPa, then increases again to 4.7 K at 11 GPa [13]. Since the coupling constant for SC pairing is proportional to the electronic constant divided by the mean square phonon energy, the softening of the phonon modes enhances the coupling constant and increases Tc [13, 15].

In the literature, most of the work is focused on single-crystalline samples and there is no report on polycrystalline ZrTe₃ highlighting the superconducting or charge density wave properties. A recent report has shown bulk superconductivity in the Cu and Ni intercalated ZrTe₃ crystals with Tc values of 3.8 K and 3.1 K respectively. In the present work we have undertaken a study of polycrystalline ZrTe₃, and intercalation with a small amount of Cu and Ag. The polycrystal samples have the one advantage of homogeneous distribution of intercalants compared to the crystals grown by the vapor transport method. This becomes even more important when the intercalant percentage is very low, as in our case (5%). The main finding of the present work is the occurrence of superconductivity at the enhanced temperature of 5.2 K in polycrystalline ZrTe₃ at ambient pressure. However, the superconductivity is still filamentary, just as for the single crystals. Unlike for the single crystals, Cu or Ag intercalation cannot be used to enhance Tc for polycrystal samples. We have also studied the effect of intercalant on the CDW state of the compound. The CDW anomaly is clearly visible in the high field magnetization data for these weakly magnetic materials. The charge transfer by the intercalant Cu and Ag is found to enhance the electrical conductivity of the ZrTe₃ and suppress the CDW anomaly.

2. Sample preparation and experiments

Polycrystalline samples of ZrTe₃, Cu₀.₀₅ZrTe₃, and Ag₀.₀₅ZrTe₃ were prepared using the high purity elements Zr (99.9%), Te (99.9%), Cu (99.999%), and Ag (99.9%) taken in the stoichiometric ratio, inside evacuated (10⁻⁶ mbar pressure) quartz tubes at 975 °C for 48 h. The reacted material contained micron size crystallites. For resistivity and heat capacity measurements, the compounds were pelletized at 10 ton pressure. The pellets obtained were of density ≈6 g cm⁻³. The electrical contacts were made using the high quality silver paint and the contact resistances were found to be 1–2 Ω. The x-ray diffraction patterns of the compounds were taken using a Philips X’Pert PRO diffractometer in Bragg–Brentano geometry. A Zeiss Ultra field emission scanning electron microscope (FESEM) was used for compositional and microscopic analysis. A Quantum Design SQUID magnetometer was used for magnetic measurement and a QD PPMS (Physical Properties Measurement System) was used for resistivity and heat capacity measurements.

3. Results and discussion

The x-ray diffraction patterns of the ZrTe₃, Cu and Ag intercalated ZrTe₃ compounds were analyzed using the Rietveld (GSAS) program and were found to fit with a monoclinic (space group P2₁/m) structure (figure 1). The lattice parameters as obtained from the Rietveld fit are a = 5.863 Å, b = 3.923 Å, c = 10.089 Å, and β = 97.74° for ZrTe₃. It is difficult to detect any appreciable change in the lattice parameters for the 5% intercalation of Cu/Ag in ZrTe₃ using our x-ray diffraction data. It is unlikely that the intercalant atoms are substituting for the Zr atoms because of the big mismatch in atomic size. However the change in the relative peak intensities is one signature of intercalation in the compound. A good quality neutron diffraction pattern is required to see the effect of small amounts of intercalants on the lattice parameters. SEM (scanning electron microscope) images for the ZrTe₃, Cu₀.₀₅ZrTe₃, and Ag₀.₀₅ZrTe₃ samples (figure 2) show the presence of small crystallities of average size a few microns in these polycrystalline samples. The intercalated compounds seem to have slightly bigger crystallites compared to the parent compound. The samples were characterized using EDX (energy dispersive x-ray) spectroscopy, to ascertain the composition of the compounds, which were found to be the same as the desired stoichiometry, within the instrument accuracy limit (±2%). Figure 2 also shows the EDX graphs for Cu₀.₀₅ZrTe₃ and Ag₀.₀₅ZrTe₃, and ascertains the presence of Cu and Ag intercalants in the compounds.

The electrical resistivities of the ZrTe₃, Cu₀.₀₅ZrTe₃, and Ag₀.₀₅ZrTe₃ (figure 3), show the onset of the SC transition (Tc) at 5.2 K. The higher value of Tc for polycrystalline ZrTe₃, in comparison to <2 K for single crystals of previous studies, is highly remarkable. However the transition widths (ΔTc) of these polycrystalline samples are large, with ΔTc ≈ 2 K, showing the large effect of grain boundary interaction on the transition temperature. The normal state resistivity of these compounds initially shows semiconducting behavior, changing to metallic below 200 K. Similar behavior has
been observed in recently discovered iron chalcogenide superconductors FeTe$_1-x$Se$_x$, where the weak localization of the charge carriers leads to a rise in the resistivity upon cooling [16–18]. The grain boundary effects in the polycrystalline samples become more important and affect the electrical properties. The room temperature resistivity of the compounds decreases upon intercalation of Ag and Cu. The room temperature resistivity values are 13 m$\Omega$ cm for ZrTe$_3$, 7 m$\Omega$ cm for Ag$_{0.05}$ZrTe$_3$, and 4.2 m$\Omega$ cm for Cu$_{0.05}$ZrTe$_3$. The ZrTe$_3$ is highly anisotropic in nature and has high resistivity along the $c$ direction. The room temperature resistivity of the polycrystalline ZrTe$_3$ sample is 10 times higher than for the single-crystalline form of ZrTe$_3$ along the $c$ axis [6, 10]. The polycrystallinity of samples does not have any bearing on the CDW phase of the compound, and $T_{\text{CDW}}$ for ZrTe$_3$ is found to be 63 K, the same as for the single crystals. Further analysis of the CDW state is presented in a later section.

The magnetic field dependence of the $T_c$ is shown in the inset of the respective figures. All the compounds show strong field dependence on the SC transition and have very similar upper critical fields ($H_{c2}$). The $HT$ phase diagram for ZrTe$_3$ and Ag$_{0.05}$ZrTe$_3$ for the field values taken from the point where resistivity drops to 10%, 50% and 90% of the onset $T_c$ is shown in the figure 2(d). The $HT$ phase diagram for the polycrystalline Cu$_{0.05}$ZrTe$_3$ sample is similar to that of ZrTe$_3$. The Werthamer–Helfand–Hohenberg (WHH) relation $\mu_0H_{c2}(0) = -0.693T_c \left(\frac{dH_{c2}}{dT}\right)_{T_c}$ gave the values of $H_{c2}(0) = 1.3$ T for ZrTe$_3$, Cu$_{0.05}$ZrTe$_3$, and Ag$_{0.05}$ZrTe$_3$ ($T_c$ is taken at the midpoint of transition), which is slightly higher than the 1.2 T for Ni$_{0.05}$ZrTe$_3$ in the $H \parallel a$ direction [5]. Though the $HT$ diagram follows the linear behavior rather than the empirical relation $H_{c2}(T) = H(0)(1 - T/T_c)^2$, the value of $\mu_0H_{c2}/k_B T_c = 0.23$ T K$^{-1}$, is under the Pauli weak coupling limit of $\mu_0H_{c2}/k_B T_c = 1.84$ T K$^{-1}$ for the singlet pairing. The coherence length ($\xi$) obtained using the Ginzburg–Landau (GL) formula $\xi = (\Phi_0/2\pi \mu_0H_{c2})^{1/2}$, where $\Phi_0 = 2.07 \times 10^{-7}$ Oe cm$^2$, is calculated as 14.8 nm, which is comparable to those observed for the Cu and Ni intercalated ZrTe$_3$ single crystals [4, 5].

The low field ($H = 20$ Oe) DC magnetization (ZFC and FC) data for the ZrTe$_3$, Cu$_{0.05}$ZrTe$_3$, and Ag$_{0.05}$ZrTe$_3$ are shown in figure 4. The SC transition is broad and the onset $T_c$ is lower ($T_c = 3.8$ K) than that observed in the electrical resistivity data. The estimated superconducting volume fraction for these compounds is as low as 6%, 5%, and 8% for ZrTe$_3$, Cu$_{0.05}$ZrTe$_3$, and Ag$_{0.05}$ZrTe$_3$ respectively. The smaller SC volume fraction and broad transition temperature are suggestive of a filamentary nature of the superconductivity and a distribution of $T_c$ within the material. We think that like the CDW state, which is observed in the $a$ and $c$ directions only, superconductivity may also set in only along these directions in the remanent part of the Fermi surface, and therefore is not a bulk feature. The $MH$ hysteresis loop (not shown here) at 1.8 K is like the typical butterfly loop of type II superconductors. The $MH$ loop at 1.8 K for all the systems showed a large jump in $M$ upon field reversal, which is indicative of a higher value of the critical current.
Figure 3. Temperature dependence of the electrical resistivity of ZrTe$_3$ (a), Cu$_{0.05}$ZrTe$_3$ (b), and Ag$_{0.05}$ZrTe$_3$ (c), showing the SC $T_c$. The field dependence of $T_c$ is shown in the insets of the respective figures for $H = 0$, 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, and 0.50 T. The HT phase diagram for ZrTe$_3$ and Ag$_{0.05}$ZrTe$_3$ is shown in (d). The dotted line is the extrapolation of the linear $H$–$T$ behavior to $T = 0$ K. The $H_C(0)$ values obtained are 1.6 T (ZrTe$_3$) or 1.7 T (Ag$_{0.05}$ZrTe$_3$), 1.3 T, and 1.0 T for the field values taken from 90%, 50%, and 10% of the $\rho(T_{onset})$.

Figure 4. Temperature dependence of the DC magnetization ($H = 20$ Oe) of ZrTe$_3$, Cu$_{0.05}$ZrTe$_3$, and Ag$_{0.05}$ZrTe$_3$ (upper panel). The magnetic field isotherms at temperatures $T = 1.8$, 2.0, 2.2, 2.5, 3.0, 3.3, and 4.0 K are shown in the lower panel for these compounds.
density $J_c$ (according to Bean’s model $J_c \propto \Delta M$) [19]. This is quite expected, as various types of disorder act as extra pinning centers and thus lead to higher $J_c$. Figure 4 shows low field $MH$ isotherms for all three compounds. The $H_{c1}$ values for ZrTe$_3$, Cu$_{0.05}$ZrTe$_3$, and Ag$_{0.05}$ZrTe$_3$, calculated from the point of deviation from the linear state in the $MH$ behavior, are 6 Oe, 7.5 Oe and 8.5 Oe respectively. These values are similar to that for the Cu$_{0.05}$ZrTe$_3$ single crystals for $H \parallel ab$ [4, 5]. However, the field values taken from the point where the magnetization deviates from linearity do not represent the actual $H_{c1}$, but the field where the magnetic field starts penetrating into the sample. However, for polycrystalline samples in powder form we can take this value as approximately $H_{c1}$. Like the upper critical field $H_{c2}$, the lower critical field ($H_{c1}$) shows a linear dependence on the temperature.

The specific heats ($C$) of ZrTe$_3$, and Ag$_{0.05}$ZrTe$_3$ are shown in the figure 5. The $C$ versus $T$ plot does not show any sharp jump. However, similar to the HC reports for the single-crystalline ZrTe$_3$, a weak jump in heat capacity is seen in the $C/T$ versus $T^2$ plot. We have shown a $C/T$ versus $T^2$ plot for Ag$_{0.05}$ZrTe$_3$ in the upper inset of the figure 5. The values of $\gamma$ and $\beta$ obtained from the low temperature intercept and slope of the $C/T$ versus $T^2$ curves in the normal state are respectively $\gamma = 0.6$ mJ mol$^{-1}$ K$^{-2}$, $\beta = 2.95$ mJ mol$^{-1}$ K$^{-4}$ for ZrTe$_3$, and $\gamma = 3.35$ mJ mol$^{-1}$ K$^{-2}$, $\beta = 0.97$ mJ mol$^{-1}$ K$^{-4}$ for Ag$_{0.05}$ZrTe$_3$. These values are slightly different from the reported values of $\gamma = 3.20$ mJ mol$^{-1}$ K$^{-2}$ and $\beta = 2.31$ mJ mol$^{-1}$ K$^{-4}$ for ZrTe$_3$, and $\gamma = 2.64$ mJ mol$^{-1}$ K$^{-2}$ and $\beta = 1.21$ mJ mol$^{-1}$ K$^{-4}$ for Cu$_{0.05}$ZrTe$_3$ single crystals [4, 20, 21]. The evolution of the phonon density of states, $(C-\gamma T)/T^3$, with the temperature shows a clear drop around the SC transition (lower inset of figure 5).

The DC magnetization at $H = 0.5$ T shows the paramagnetic behavior for ZrTe$_3$ and Ag$_{0.05}$ZrTe$_3$ (figure 6). The $MT$ data for Cu$_{0.05}$ZrTe$_3$ (not shown here) are also similar to those for ZrTe$_3$ and Ag$_{0.05}$ZrTe$_3$, with the reduced anomaly at the CDW transition temperature. The magnetic susceptibility of the metal depends on the electronic structure in the vicinity of the Fermi energy. For a metal with a high density of states $D(E_F)$ at $E_F$, $\chi$ is dominated by the paramagnetic Pauli component $\chi_P$ in the absence of exchange enhancement and spin–orbit coupling. $\chi_P(T)$ is proportional to the average density of states within $k_BT$ of the Fermi level. We observe a drop in susceptibility at $T_{CDW}$ in ZrTe$_3$, Cu$_{0.05}$ZrTe$_3$, and Ag$_{0.05}$ZrTe$_3$, confirming the change in the electronic structure near the Fermi surface. The onset of the CDW leads to the opening of a gap at the Fermi surface and a reduction in charge carriers, results in a drop of susceptibility. However the susceptibility anomaly is weaker for Ag$_{0.05}$ZrTe$_3$ and Cu$_{0.05}$ZrTe$_3$ compared to ZrTe$_3$.

The electrical resistivity ($\rho$) and its temperature derivative ($d\rho/dT$) for the compounds are shown in figure 7, on an enlarged scale, to elucidate the CDW onset temperature. As seen from the figure, the onset of the CDW at $\sim 80$ K (determined from $d\rho/dT$) remains the same for all compositions and the full CDW sets in near 63 K. The $T_{CDW}$ for polycrystalline ZrTe$_3$ is the same as that reported for
the single crystal ZrTe$_3$. Though the intercalation of Cu or Ag does not affect the CDW state in our polycrystalline samples, the amplitude of the CDW anomaly changes with intercalation.

In figure 8, we have plotted the normalized electrical resistivity at room temperature for the ZrTe$_3$, Ag$_{0.05}$ZrTe$_3$, and Cu$_{0.05}$ZrTe$_3$ compounds to compare their normal state behaviors. The resistivity increases down to 200 K upon cooling before decreasing again. The relative change in the slope of the resistivity is consistent with the room temperature resistivity values of these compounds. The CDW transition is clearly visible, and the resistivity rises again at lower temperature below the CDW state, before collapsing at the SC transition. We observed a bifurcation of the resistivity data upon cooling and warming, above the CDW transition, the reason for which is not clear to us, and needs further study. Doping with 5% Ag or Cu affects the CDW state slightly. Though there is no change in the $T_{\text{CDW}}$, the magnitude of the CDW anomaly decreases upon Ag/Cu intercalation, which can be estimated from the size of the resistivity anomaly ($\Delta \rho_{\text{CDW}}$) at $T_{\text{CDW}}$ [13, 22]. The change in resistivity for the CDW ($\Delta \rho_{\text{CDW}}$) is related to the reduction of the density of states at the Fermi level ($\text{DOS}(E_F)$). The relative change in the resistivity due to the CDW is given by $\alpha = (R_1 - R_2)/R_1 = (\sigma_1 - \sigma_2)/\sigma_1$, where $R_1$ and $R_2$ are the resistivities in the CDW state and the expected value in the absence of the CDW [13, 22]. The resistivities $R_1$ and $R_2$ are shown schematically in the inset of figure 8. The conductivity of the metal is defined as $\sigma = N_0 e^2 v^2 f \tau$, where $N_0$, $v_f$ and $\tau$ are the DOS at $E_F$, the Fermi velocity and the relaxation time of the conduction electrons, respectively. The formation of the CDW state reduces $N_0$ but does not change $v_f$. Therefore, under the assumption that $\tau$ is not affected by the CDW formation, $\alpha$ can be rewritten as $\alpha = (\sigma_1 - \sigma_2)/\sigma_1 = (N_0 - (N_0 - \Delta N))/N_0 = \Delta N/N_0$, where $N_0$ is the total density of states at $E_F$ in the normal state and $\Delta N$ denotes the reduction of $N_0$ due to the CDW formation. $\Delta N$ is proportional to the reduction of the Fermi surface due to the CDW formation; therefore $\alpha$ can give information on the size of the nested Fermi surface. The values of $\alpha$ for ZrTe$_3$, Ag$_{0.05}$ZrTe$_3$, and Cu$_{0.05}$ZrTe$_3$ are found to be 0.04, 0.027, and 0.025. It has been observed for single crystals of ZrTe$_3$ that the pressure dependence of the superconducting transition temperature follows the pressure dependence of $\alpha$ [13]. $\alpha$ for polycrystalline ZrTe$_3$ is the same as for the crystals at 5 GPa pressure [13]. It is possible that the pressure due to strains in the polycrystalline compound is the reason for the enhancement of $T_c$. In this context, it should be mentioned that in NbSe$_3$ single crystals, superconductivity was never observed down to 50 mK and it is argued that a very high pressure is required for inducing superconductivity, the conditions for which exist at the grain boundaries of polycrystalline samples [14, 23, 24].

4. Conclusions

In conclusion, polycrystalline samples of ZrTe$_3$ are found to superconduct at the enhanced temperature of 5.2 K. The volume of the superconducting fraction is less and we speculate that, like the CDW state, which is observed in the $a$ and $c$ directions only, superconductivity also sets in only along these directions in the remanent part of the Fermi surface, and therefore is not a bulk feature. Though the nature is filamentary, this is akin to the effect of external pressure on $T_c$ in ZrTe$_3$ single crystal. The strains between the agglomerated small single crystallites in polycrystalline samples could be a possible reason for the higher superconducting transition.
temperature $T_c$. Ironically we did not observe any effect of 5% intercalation of Cu and Ag on the SC state. This is in contrast to findings of the recent study in Cu intercalated ZrTe$_3$, where only the intercalated phase is observed to show bulk superconductivity, whereas our polycrystalline samples of Cu$_{0.05}$ZrTe$_3$ and Ag$_{0.05}$ZrTe$_3$ do not show any improvement in the superconducting volume fraction. We have shown that Ag and Cu intercalation increases the electrical conductivity and affects the CDW anomaly, but has no bearing on the transition temperature of either the CDW or the SC phase.

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