Pressure induced superconductivity in the compound ScZrCo

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Abstract
We report on the synthesis and physical properties of a new compound, ScZrCo, which crystallizes in the Ti2Ni structure with the space group of Fd3mS without a spatial inversion center. The resistivity at ambient pressure shows a semiconducting-like behavior. Furthermore, specific heat and magnetic susceptibility measurements yield a rather large value of Wilson ratio $\approx 4.47$. Both suggest a ground state with correlation effect. By applying pressure, the up-going behavior of resistivity in the low temperature region is suppressed and superconductivity emerges. Up to 36.1 GPa, a superconducting transition at about 3.6 K with a quite high upper critical field is observed. Our discovery here provides a new platform for investigating the relationship between the correlation effect and superconductivity.

1. Introduction
The discoveries of superconductivity in cuprate [1, 2] and iron-pnictides/chalcogenides [3–6] have revived the enthusiasm of exploring new high temperature superconductors in correlated systems. In many compounds containing 3d transition metal elements, the strong interaction of electrons cannot be neglected, leading to a correlation effect and subtle balance between localization and itinerancy of d-orbital electrons [7]. And this correlation effect induces tremendous and rich physics, including unconventional superconductivity, Mott insulating state, bad metal, spin density wave, etc [8–11]. And superconductivity has been found in many 3d transition metal compounds, such as Na$_x$CoO$_2$·1.3H$_2$O, MnP, CrAs, Ba$_1$−$_x$Na$_x$Ti$_x$Sb$_2$O$_6$, etc [12–16].

As cobalt is a typical 3d transition metal element and lies between iron and copper, the superconductors containing cobalt are generally interesting and unconventional. Superconductors containing Co include compounds such as Na$_x$CoO$_2$·1.3H$_2$O and Zr$_2$Co [12, 17]. The superconductor Na$_x$CoO$_2$·1.3H$_2$O with $T_c \approx 4$ K is fascinating for its strong electron correlation effect, and the superconductivity may have a d-wave gap symmetry reflecting unconventional pairing mechanism [18]. In the cobaltite Na$_{0.7}$CoO$_2$, a Curie–Weiss metallic phase was reported, which was argued to be induced by correlation effect [19, 20]. The cobalt alloy Zr$_2$Co, which crystalizes in the CuAl$_2$ structure (space group: I4/mcm) was also reported as a superconductor with $T_c = 5$ K [17]. And the NMR investigation revealed that it could be an itinerant antiferromagnet in the normal state [21].

Moreover, non-centrosymmetric structure may result in the spin-triplet superconducting pairing. Due to the non-trivial antisymmetric spin–orbit coupling effect induced by heavy atoms and the lacking of inversion symmetry, it was argued that the superconductivity CePt$_3$Si may have such an odd parity [22]. Here, we report the synthesis and physical properties of the compound ScZrCo with a non-centrosymmetric structure. This compound was slightly mentioned before in the way that by doping Sc into ZrCo alloy, however the structure and physical properties have never been determined [23]. We find that ScZrCo does not crystallize in the tetragonal CuAl$_2$ structure like Zr$_2$Co, but in the cubic Ti$_2$Ni structure with the space group Fd3mS [24]. In this paper, an anomalous ground state of ScZrCo is found and superconductivity is induced by applying high pressure.
2. Experimental

The polycrystalline ScZrCo samples were fabricated by the arc-melting method. The raw materials scandium (Alfa Aesar, powder, purity 99.8%), zirconium (Aladdin, powder, purity 99.9%) and cobalt (Alfa Aesar, powder, purity 99.99%) were weighed in the mole ratio 1:1:0.9, mixed well, and pressed into a pellet in a glove box filled with argon. The samples were fabricated on the copper base of the arc furnace filled with argon atmosphere. To ensure the homogeneity of the samples, we re-melted the pellets by turning up and down at least 5 times. Finally, we got the silver-shining polycrystalline samples. The mole ratio 1:1:1 has also been tried, but the products always have the impurity ScZrCo$_2$.

The Bruker D8 Advance diffractometer (Cu-K$_\alpha$ radiation) was used to measure the x-ray diffraction pattern. The energy dispersive x-ray microanalysis spectrum and the SEM photograph of the polycrystalline samples were obtained by Phenom ProX (Phenom). The measurements were performed at an accelerating voltage of 15 kV. The DC magnetization measurements were performed with a SQUID-VSM-7T (Quantum Design). The specific heat was measured by the thermal-relaxation method with the PPMS-16T (Quantum Design) which allows us to realize high vacuum for measuring the specific heat down to 2 K. To ensure the good thermal conduction for the specific heat measurements, we polish one side of the ScZrCo sample well and paste with the thermal conduction grease. The measurements of resistivity under high pressure were accomplished by using the diamond based system cryoDAC-PPMS (Almax easyLab). The culet size of diamonds is 400 $\mu$m. The holes i.e. for the high pressure measurements is 200 $\mu$m. The gasket is made by T301 stainless steel which was insulated by the cubic-BN-epoxy mixture, and a small piece of ruby was used as a pressure manometer. The relation of pressure and ruby $R(1)$ fluorescent peak is calculated by the equation $P \text{ (GPa)} = 380.81(\Delta \lambda / \lambda_0 + 1)^3 - 1$ [25]. Here $\lambda_0$ is the position of ruby R(1) fluorescent peak at ambient pressure. And $\Delta \lambda = \lambda - \lambda_0$ ($\lambda$ is the position of ruby R(1) fluorescent peak at the pressure $P$). The pressure transmitting medium was the ScZrCo polycrystalline powder itself, this is a way used quite often for the high pressure measurements. To obtain a relatively better homogeneity of pressure, we grind the polycrystalline powder in the glove box filled with argon atmosphere. The four-probe van der Pauw method was used to detect the resistivity under high pressure [26]. The high pressure resistivity measurements were done with an option adapted to the physical property measurement system (PPMS-9T, Quantum Design).

3. Results and discussion

3.1. Identification of the structure

Figure 1 (a) shows the x-ray diffraction pattern for the sample ScZrCo and the Rietveld fitting curve (the red line) to the data [27]. Our XRD can be well fitted by the structure model of Ti$_2$Ni, which means that the two compounds should have the same structure. The Rietveld refinement gives a quite good agreement between the measured data and the calculated profile with the agreement factor R-weighted pattern $R_{wp} = 1.79\%$, R pattern $R_p = 1.39\%$, and goodness of fit GOF = 1.57. No obvious impurity peaks are found except for two tiny ones at around 34.3 and 39.7 degrees reflecting trace impurity of scandium. The lattice parameters for the cubic unit cell are determined to be $a = b = c = 12.227 \text{Å}$. Figure 1(b) shows the schematic structure of ScZrCo with the space group Fd3mS. The locations in periodic table of elements Sc and Zr are adjacent to Ti, while Co is located...
Thus, it is reasonable that Sc and Zr locate at the same site of Ti in Ti$_2$Ni (48f and 16c), Co locates at the site of Ni. However, there might be the mix-occupancy of Zr and Co or some other possibilities. In order to clarify that, we fitted the data with the possibility that the Zr occupies the 32e site (the assigned Co position).

However, we find that the occupancy ratio of Zr at the 32e site and the related $U_{iso}$ yielded from the fitting by the TOPAS software are negative, which is unreasonable. This means that there is no or very tiny amount of Zr at the Co position (32e site). We also did the similar fittings, such as, Sc occupying the 32e site or Co occupying the Sc/Zr positions (16c/48f). They all result in negative or zero occupancy ratio. Therefore, in this sample, we incline to conclude that Co does not occupy the mixed sites of Zr and Sc, and vice versa. As shown in table 1, there are two sites for Sc and Zr, which are occupied by Sc and Zr atoms in approximately the ratio of 1:1. And the occupancies of each site for Sc and Zr are also shown in table 1. Apparently, all Co atoms occupy the same site. As shown in figure 1(b), every four nearest Co atoms connected by Sc/Zr (at the site of 48f) form a tetrahedron. This tetrahedron is surrounded by Sc/Zr atoms at the 16c site. Every four nearest tetrahedron assembles a larger tetrahedron. As we know, this kind of tetrahedron has a typical non-centrosymmetric geometric structure. Compounds with non-centrosymmetric structure usually behave in a unique way, especially about their superconducting order parameter. Recently, unconventional superconducting properties were observed in the non-centrosymmetric compounds CePt$_3$Si [22] and Li$_2$Pt$_3$B [28] for the possible existence of spin–triplet pairing symmetry.

In figures 2(a)–(c) we show the spatial distribution of the composition ratio for Sc, Zr and Co, respectively. Actually we did the measurement of two-dimensional composition mapping. Since the mapping does not precisely give the numeric content of each element, here we choose to show the spatial composition distribution through a line scan of EDS measurement. The EDS scanning line is shown on the SEM image in figure 2(e). As one can see in figures 2(a)–(c), Co is quite uniformly distributed on the line. While, Sc and Zr have a fluctuation to some extent. Interestingly, when the composition of Sc shows a suppression, that of Zr exhibits an enhancement at the same position, namely, they are anti-correlated each other. This indicates that Sc and Zr are complementary in doping at the same position in space. As shown in figure 2(d), the sum content ratio of Sc and Zr are quite homogeneous and exhibits a stable ratio of about 2.3 over the averaged Co composition, which is consistent with the results of XRD that the Co is deficient.

| Atom | Site | Occupancy | $x$ | $y$ | $z$ | $U_{iso}$ |
|------|------|-----------|----|----|----|----------|
| Co   | 32e  | 0.904     | 0.9115 | 0.9115 | 0.9115 | 0.013(6)  |
| Sc1  | 48f  | 0.467     | 0.3099 | 0.3099 | 0.3099 | 0.015(2)  |
| Zr1  | 48f  | 0.533     | 0.3099 | 0.3099 | 0.3099 | 0.002(8)  |
| Sc2  | 16c  | 0.445     | 0.125  | 0.125  | 0.125  | 0.018(8)  |
| Zr2  | 16c  | 0.557     | 0.125  | 0.125  | 0.125  | 0.009(4)  |

Note. Space group Fd3mS; lattice parameter $a = 12.227 \text{Å}$; $R_{wp} = 1.79\%$, $R_p = 1.39\%$, GOF = 1.57.
The temperature dependent magnetic susceptibility is shown in 3.2. Resistive, magnetic and thermodynamic properties

The temperature dependent magnetic susceptibility is shown in Figure 3(a) with an applied magnetic field of 1 T (the background signal of SQUID has been subtracted). Using the Curie–Weiss law, we fit the data by the equation

\[ \chi(T) = \frac{c}{T + T_0} + \chi(0) \left[ 1 - \left( \frac{T}{T_E} \right)^2 \right]. \]  

(1)

The first term of the equation arises from the Curie–Weiss susceptibility which is given by local magnetic moments of paramagnetic ions, and the second one is the Pauli paramagnetic susceptibility term which is related to the quasiparticle density of states at Fermi level [29, 30]. The fitting yields \( c = 0.0013 \) emu K mol\(^{-1}\) Oe\(^{-1}\), \( T_0 = 0.8 \) K, \( \chi(0) = 0.0006 \) emu mol\(^{-1}\) Oe\(^{-1}\), \( T_E = 1295.7 \) K. Considering the quite large value of \( T_E \), the Pauli paramagnetic susceptibility is weakly temperature dependent in the range of 2–300 K. Taking \( c = \mu_0 \mu_{eff}^2 / 3k_B \), the effective magnetic moment per Co can be estimated to be around 0.10 \( \mu_B \).

In Figure 3(b), we present the specific heat coefficient \( C/T \) versus \( T^2 \). As shown in the inset, in the low temperature region (\( T < 10 \) K), we fit the experimental data with the Debye model \( C/T = \gamma_c + \beta T^2 + \eta T^4 \) containing a fourth order term is used to fit the data from 2 to 8.5 K. Using the value of Avogadro constant \( N_A = 6.02 \times 10^{23} \) mol\(^{-1}\), Boltzmann constant \( k_B = 1.38 \times 10^{-23} \) J K\(^{-1}\), \( Z = 3 \) (the number of atoms in a primitive cell), the Debye temperature \( \Theta_D \) is estimated to be 248.7 K and \( \gamma_n = 9.75 \) mJ mol\(^{-1}\) K\(^{-2}\). In the inset of Figure 3(b), an enlarged view is shown and the upturn is clearly seen, which may be induced by the Schottky anomaly or the correlation effect. However its smooth temperature dependent evolution together with the large Wilson ratio discussed later may exclude the former case. The Curie–Weiss type of magnetic susceptibility is very similar to that of Na\(_{0.7}\)CoO\(_2\), which was also argued to be induced by correlation effect [18, 19]. Usually the Wilson ratio can be used to determine whether there is correlation effect in the system. By definition the Wilson ratio is expressed in the form [31]

\[ R = \frac{4\pi^2 k_B^2}{3(\mu_B)^2} \frac{\chi(0)}{\gamma_n}. \]

(2)

Here \( g \) is Lande factor which is 2 for a bare electron, and \( \mu_B \) is the Bohr magneton. If \( R \approx 1 \), the material is considered to be the non-interacting electron gas. If \( R \) is less than 2 but larger than 1, it corresponds to an interacting Fermi liquid. If the Wilson ratio \( R > 2 \), a strong electron correlation effect would be expected. For the present compound ScZrCo, \( R \) is determined to be 4.47, which suggests a moderate correlation effect in the compound. In correlated systems, the localization and itinerancy of electrons is subtly balanced and the ground state can vary from a Mott insulator to an itinerant metal [9, 10].

Usually, high pressure can tune the ground state properties of the correlated systems, which may trigger unconventional superconductivity. The temperature dependence of resistivity for ScZrCo at various pressures is presented in Figure 4(a). At ambient pressure, the resistivity curve exhibits a negative temperature dependent
resistivity in the whole temperature region from 1.9 K to 300 K, which reveals a semiconducting-like behavior and cannot be fitted with the model of a band insulator \( \rho = \rho_0 \exp(E_F/k_B T) \). It is similar to some alloy materials such as TiCoAl, which is characterized by strong scattering of charge transport. Naively this negative slope of temperature dependent resistivity may be also induced by strong scattering since we have probably mutual occupation of the Sc and Zr atoms at the sites of 48f and 16c. However, this picture can be removed since under pressure this semiconducting-like behavior is strongly suppressed and a metallic feature finally appears. This would not happen if the up-turning of resistivity in the low temperature region at ambient pressure were induced by scattering of impurities or disorders.

By applying a pressure, the semiconducting-like feature of resistivity is gradually suppressed and the resistivity curve turns to a metallic one. At the pressure of about 2 GPa, the resistivity keeps almost as a constant in whole range of temperature. A systematic evolution is illustrated in figure 4. As the pressure reaches 19.5 GPa (shown in the inset of figure 4(a)), the superconductivity shows up in the low temperature region with a small but sharp up-turning, which may result from the inhomogeneity of the sample at high pressure under the van der Pauw methods. This feature is quite common in the high pressure resistance measurement. This upturn can also be suppressed with increasing magnetic field. For these reasons we regard the point where this upturn starts as the onset transition temperature of superconductivity. As we increase the pressure further, the superconducting transition temperature gets increased. At 36.1 GPa, the highest pressure we can reach in this run of experiment, superconductivity with an onset transition temperature \( T_c = 3.6 \) K has been observed, and meanwhile the zero resistivity is also observed at a lower temperature. Figure 4(b) shows the normalized resistivity at different pressures. As we can see, the value of residual-resistivity-ratio \( (\rho_{300K}/\rho_{4K}) \) increases with pressure, and changes gradually from below 1 to about 1.26 at about 24.5 GPa. Then, it keeps almost unchanged with further increasing pressure up to 36.1 GPa. The inset of figure 4(b) shows the enlarged view of the low temperature resistivity at 16.3 and 36.1 GPa.

3.3. Pressure dependent phase diagram
To determine the superconducting transition temperature, we have adopted different criterions: the onset superconducting transition temperature \( T_{c, onset} \) determined from the very beginning of the deviating point of the normal state resistivity curve; the critical temperatures with 50\% \( \rho_{4K} \) and 90\% \( \rho_{4K} \), where \( \rho_{4K} \) is the resistivity at 4 K. In figure 5, we present a phase diagram which reveals the pressure dependence of the superconducting transition temperature \( T_c \) and the ratio \( \rho_{4K}/\rho_{300K} \). As shown in the phase diagram, the value of \( \rho_{4K}/\rho_{300K} \) is suppressed gradually when the pressure is increased up to 19.5 GPa. At the pressure of 19.5 GPa, we observe a deviation of resistivity from the normal state background, which as explained above, is due to the presence of superconductivity. The superconducting transition temperature increases gradually by applying higher pressures and \( T_c \) goes up to 3.6 K under a pressure of 36.1 GPa. The transition temperature is expected to rise further under higher pressures. From figure 4(b), we find that just after the semiconducting-like behavior is...
suppressed, superconductivity starts to emerge. This can easily get a reflection from the phase diagram shown in figure 5. This set of data manifest that superconductivity shows as a subtle balance between the localization and itinerancy of electrons [8].

3.4. Upper critical field

In figure 6, we present the low temperature resistivity under different magnetic fields up to 4 T at 36.1 GPa. The systematic evolution of resistivity curve under a magnetic field tells that the drop of resistivity at around 3 K is due to superconductivity. We thus determine the upper critical field \( H_{c2}(T) \) versus temperature and display the data in the inset of figure 6. Here, we use the criterions of 90%\( \rho_n \) (filled olive squares) and 50%\( \rho_n \) (filled blue squares), and the fitting curves (dashed lines).

According to Ginzburg–Landau theory, the terms \( 1 - (T/T_c)^2 \) and \( 1 + (T/T_c)^2 \) are the two basic components in constructing the functions of the coherence length and upper critical field in a type-II superconductor. The fitting curve is shown by the dashed line. For the criterion of 90%\( \rho_n \), the parameters of \( a \) and \( b \) used in the fitting are 0.74 and \(-0.68\), respectively. The estimated \( H_{c2}(0) \) is about 5.14 T (see the filled olive squares in the inset of figure 6).
Considering $T_c(90\%\rho_c) = 2.7$ K, we obtain the ratio of $H_c(0)/T_c = 1.9$ T K$^{-1}$. It is curious to know whether the superconductivity has an odd parity provided the system has a non-centrosymmetric structure. Measurements of $\mu SR$, NMR or specific heat under pressure would be highly desired to resolve this issue.

One may argue that, only the resistivity measurements under high pressure could not prove the bulk superconductivity, and it may arise from the impurity phases, such as Sc or Zr. However, we argue that this cannot be the case. Although there are tiny peaks of Sc in the XRD patterns of the sample, usually such tiny amount of impurity could not lead to a zero resistivity. Moreover, the features of superconductivity of our sample are completely different from that of Sc under pressure. For the different phases of Sc under pressure with $T_c$ of 8.31 K (111 GPa), 10.6 K (81 GPa) and 12.8 K (87 GPa) [33], according to the values of $dT_c/dH$ in previous studies, the upper critical fields estimated through the linear extrapolation with the slope of $H_c2/dT$ near $T_c$ are less than 1.1 T, 1.7 T and 2.3 T, respectively. They are much smaller than the value of 5.14 T ($T_c = 2.7$ K, 36.1 GPa) determined from our sample. In addition, as we know, the $T_c$ of Zr under a pressure of 30 GPa is about 11 K [34]. It is much higher than the $T_c$ value of our sample. Furthermore, we also synthesized another material ScZrCo$_2$ by arc-melting method as that for ScZrCo and studied the resistivity under high pressure. No any trace of superconductivity was found in the temperature dependence of resistivity in ScZrCo$_2$ under high pressure up to 32.1 GPa. Except for the different stoichiometric ratios, all preparation procedures of ScZrCo and ScZrCo$_2$ are the same, and they should have very similar impurities. If the superconductivity would arise from the impurity phases in ScZrCo, it should also appear in ScZrCo$_2$. This may rule out the possibility that the superconductivity arises from the impurity phases in our present sample ScZrCo.

4. Conclusion

In conclusion, we have successfully synthesized a new compound ScZrCo and determined the structure. It has a cubic structure with the space group Fd3mS without a spatial inversion center. The sample at ambient pressure shows a weak semiconducting-like behavior which cannot be expressed by the model of band insulator. The up-turning of low temperature specific heat coefficient and large Wilson ratio suggest a ground state with correlation effect. By applying pressure the system gradually becomes metallic. When the pressure is beyond 19.5 GPa, superconductivity emerges. A superconducting transition temperature at about 3.6 K can be observed under 36.1 GPa. This material may provide a typical platform for achieving superconductivity by finely tuning the subtle balance between localization and itinerancy of d-orbital electrons.

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