Interfacial superconductivity above Pauli limit in homogeneously disordered tungsten carbide - Ga composite

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A directed narrow jet of an organo-metallic gas containing a heavy metal can be decomposed by an accelerated beam of gallium ions, leaving behind a track made up of a complex residue of gallium, heavy metal and carbon. The process is highly controllable and in certain cases, the residue has remarkable superconducting properties, like an upper critical field ($H_{c2} \sim 10$ T) that is higher than the paramagnetic limit. Werthamer-Helfand-Hohenberg (WHH) analysis shows the presence of moderate spin-orbit (SO) scattering and a Maki parameter compatible with unconventional (e.g., FFLO-like) superconducting states. Using a spatially resolved mass spectrometric technique (Atomic probe tomography), we show that the possible origin of the SO effects lies in the formation of nanocrystalline tungsten carbide (WC) with a possible non-centrosymmetric crystal structure. We also show that when Ga is distributed on the surface of nano-crystallite WC, the sp-orbitals of Ga give rise to bands with a significant density of states near the Fermi energy. The superconductor is in the dirty limit where the mean free path ($l$) is much smaller than the zero temperature coherence length, i.e., $l \ll \xi_0 \approx 5$ nm. Low-temperature magnetotransport with in-situ rotation of the sample in a magnetic field shows clear anisotropic effects that weaken as the width of the tracks are increased from ~ 100 nm to ~ 1 µm [Phys. Rev. B 103, L020504, 2021]. The combination of the transition temperature ($T_c \approx 5$ K), the critical field $H_{c2} \geq 10$ T and nanometer-scale patternability of these tracks make them an attractive component for engineered mesoscopic structures.

INTRODUCTION

A superconducting state that survives at high magnetic field can open up interesting possibilities - e.g. potential coexistence of quantum Hall edge states and Cooper-pairs [1, 2]. The superconducting state is suppressed by a magnetic field in two ways. The kinetic energy of the shielding currents builds up with increasing external field and acts against the condensation energy. Secondly the magnetic field tries to align the spins and hence makes the spin-singlet state unfavourable. Compared to the free electron Fermi sea, the paramagnetically polarised state is lower in energy by an amount $E_{Mag} = \frac{\mu_B^2 D(E_F) (\mu_0 H)^2}{2}$, where $\mu_B$ is the Bohr magneton and $D(E_F)$ is the density of states at the Fermi level. The BCS ground state has a condensation energy $E_{BCS} = \frac{D(E_F) \Delta(0)^2}{2}$, where $D(E_F)$ is the density of states at the Fermi level and $\Delta(0) = 1.76 k_B T_c$ is the BCS gap. $T_c$ is the critical temperature. For $\mu_0 H > 1.8 T_c$, the paramagnetic state would be lower in energy. We shall refer to this field as $H_P$. $D(E_F)$ cancels from both sides of the expression leading to a certain degree of material independence. This very well known limit is referred to as the Pauli or the Clogston-Chandrasekhar limit in literature [3, 4]. Upper critical field of elemental superconductors are well below this limit. There has been a number of efforts to find exotic superconductors (e.g. FFLO, spin-triplet, topological) in recent past which might compatible with relatively high magnetic field. Several methods including metal intercalation between layers of a material [5, 6], applying high pressure, hard and soft tip contact [7, 8] and interface between two materials [9, 10] has been tried for achieving this. In this paper, we point out an emerging class of materials, composed of Ga and a heavy metal carbide, fabricated using FIB which are undoubtedly granular and highly disordered, but with $H_{c2}$ significantly above the Pauli limit. It also appears that these materials are characterised by anisotropic in plane $H_{c2}$ and supersonic vortex velocities at low magnetic fields.

A metalorganic gas, constituting of a heavy metal (W, Pt, Nb) bonded with organic functional groups is injected close to the area of interest on a sample. Either an accurately directed electron beam or an ion beam of...
TABLE I. Tungsten sample id and different relevant parameters. Resistivity was calculated using the formula $\rho_n = \frac{L_y \times L_z}{L_x} \times R_N L_x$; where, $L_x$, $L_y$, $L_z$ are length, width and thickness of those samples respectively. The justification of treating this as 3D rectangular blocks comes from the fact that the mean free path and coherence lengths are both much smaller than the shortest of the three dimensions in all the samples. Orbital limiting field is calculated using the relation: $H_{orb}^{c_2}(0) = 0.7 \left| \frac{T dH_{c_2}}{dT} \right|_T$.

| ID (Width) | Deposition parameter (kV, pA) | $T_c$ (K) | $\rho_n$ ($\mu\Omega$ cm) | $H_{orb}^{c_2}(0)$ (T) |
|------------|-------------------------------|-----------|--------------------------|------------------------|
| A (100 nm) | 30, 52 | 5.20 | 65 | 18.0 |
| B (500 nm) | 30, 52 | 5.02 | 110 | 12.4 |
| C (1000 nm) | 30, 52 | 5.10 | 115 | 13.0 |
| D01 (440 nm) | 30, 20 | 4.50 | 125 | 16.8 |
| D02 (280 nm) | 30, 20 | 5.02 | 110 | 14.0 |

Gallium is used to decompose the gas, leaving behind a residue of Ga, C and the heavy metal. The process leads to a product whose stoichiometry is not precise but its superconducting properties are robust [12–21]. In our samples, tungsten was carried by a gas containing hexacarbonyl tungsten (W(CO)$_6$) and then selectively decomposed by a gallium beam (30 keV, 52 pA or 20 pA Ga$^+$ ion current) leaving an amorphous Ga-W-C residue at programmed locations forming the tracks on Si/SiO$_2$ and sapphire substrates (Table I). These composite films are superconducting with a critical temperature $T_c \approx 4.8$ K. The highest magnetic field at which dissipationless transmission can be observed is clearly higher than 10 T. This was already reached at $T \approx 1.6$ K and had not saturated. The $T = 0$ value is clearly going to be higher than the Clogston–Chandrasekhar limit.

The observed upper critical field arises as a combined effect of the orbital and spin paramagnetism related pair breaking mechanisms. The manner in which these two parts combine is generally described by the Werthamer-Hohenberg-Helfand (WHH) theory [22–24]. The $\mu_0 H_{c_2}(T)$ curves depend on two important dimensionless parameters, the Maki parameter $\alpha = \sqrt{2} H_{c_2}^{orb}/H_P$ and $\lambda_{so}$ the spin orbit scattering strength. The limiting orbital field, $H_{orb}^{c_2}$ may be calculated from experimental data as $H_{orb}^{c_2}(T = 0) \approx 0.7 \left| \frac{T dH_{c_2}}{dT} \right|_T$. The applicability of WHH theory to amorphous superconductors is well established [25–27]. Later on we shall show quantitatively, that the application of the WHH theory is well justified for this system. However it is also important to point out that there are known examples where the WHH framework doesn’t work [28, 29].

Figure [1] shows the behaviour of five superconducting samples listed in table [I] along with the fit-parameters.

These fits are described by

$$\ln \left( \frac{1}{T} \right) = \sum_{\nu=-\infty}^{\infty} \left( \frac{1}{|2\nu+1|} - \left[ \frac{1}{|2\nu+1|} + \frac{b_c}{t} + \left( \frac{(\alpha b_c/t)^2}{|2\nu+1|+(b_c+\lambda_{so})/t} \right)^{-1} \right] \right)$$
where \( t = T/T_c \), \( \alpha = \frac{\sqrt{2}H_{orb}^2}{H_P} \) is the Maki parameter, the dimensionless magnetic field \( b_c = \mu_0 h_c = \frac{e\hbar}{2m\alpha \kappa_B T_c} \) and \( \lambda_{so} \) is the spin-orbit scattering constant [22]. Figure 1(b) shows the values of the spin-orbit scattering and Maki parameter obtained from these fittings. Critical field enhancement of 1.15 to 1.27 times \( H_P \) has been observed in our samples. We find an overall trend of \( \alpha \) decreasing with increasing thickness and \( \lambda_{so} \) increasing with increasing thickness. The value of \( \alpha (= 2 - 3) \) is more than the 1.8 in all the films, suggesting that this system can potentially have an FFLO phase. Non-zero values of \( \lambda_{so} \) points to the presence of moderate spin-orbit scattering in this system that can enhance the \( H_{c2} \) significantly by suppressing the spin paramagnetic effect. This has been observed in dirty layered superconductors [31], consisting of transition metal dichalcogenide intercalated with organic molecules, with strong SOC (Klemm–Luther–Beasley (KLB) theory) [32]. Enhancement of critical field in the presence of atoms with high atomic number \( (Z) \) is also observed before [33].

However the mere presence of heavy atoms in their elemental state would not imply SOC effects. The lattice and its chemical environment are obviously important. To understand the composition of these wires we have carried out Atomic Probe Tomography (APT) analysis of the samples. In APT, the sample is prepared in the form of a needle, and atom by atom “evaporation” from the sample is done utilizing a strong electric field [34]. Ions are evaporated from the apex and projected into a position sensitive single ion detector [35]. From the positions and time-of-flight data, 3D reconstruction of the analyzed volume is done. It is possible to individually identify the chemical makeup of samples from the mass spectrum \((e/m \text{ ratio})\). All the APT measurements were done using CAMECA local electrode atom probe (LEAP 5000 XR). It has a lateral resolution of 0.3 – 0.5 nm and a depth resolution of 0.1 – 0.3 nm. Tip evaporation was carried out in laser mode with pulse energy 30 pJ and frequency and 200 kHz at 40 K.

There are certain clear conclusions that may be drawn from the APT results:

1. The key question of the state in which the heavy metal is present, is clearly answered. All the W is in the form of its carbide \((\text{WC})\). The two peaks (marked in figure 2(c)) correspond to singly and doubly ionised species with mass 195 amu. There is no W \((\sim 184 \text{ amu})\) in free from at all. While it is not possible to determine crystal structure of WC, there are indeed forms of WC that are non-centrosymmetric and hence capable of supporting spin-orbit interaction in the bulk [36]. Presence of WC is further verified by observation of two Raman peaks at 667 and 817 cm\(^{-1}\) can be associated with the W-C stretching modes (figure 2(d)) [37].

2. The fractional abundance of W, C and Ga if calculated from the APT data agrees very well with separately carried out energy dispersive X-ray spectroscopy (EDS) data from various locations of the samples. Atomic composition of these samples turns out to be C \((52\%)\), W \((36\%)\) and Ga \((12\%)\) which is almost same for all the samples.

3. It is also interesting to note (figure 3(b)) that there is a noticeable precipitation of the carbon towards the bottom of the nanowires. This may be of some significance as thin polarizable underlayers of materials like Ge (for example) have been shown to increase the \( T_c \) of strongly disordered quench condensed films of metals. Such polarizable dielectrics are thought to be able to modify the electron-electron interactions in the granular thin films [35].
The distributions of the four major components WC, C, and Ga. Cross sectional distribution of every component is shown in the bottom panel of each image.

Transport measurements with in-situ rotation of the sample in a magnetic field was carried out in fields of up to 10 T. The magnetic field was aligned parallel (with ±1° error) to the current direction. Decrement of resistance 

\[ D = v_F l / 3 \] 

and 

\[ l = \frac{6\pi^2 h}{e^2 S \rho_n} \] 

where \( S \) is the free electron fermi surface area in the normal state. We obtain \( v_F \approx 3.4 \times 10^6 \) m/sec (for D01), consequently \( l \) turns out to be extremely small. The calculated value of \( l \) is in fact smaller than the typical lattice constant justifying the assumption \( l \ll \xi_0 \). The APT results suggest that these films are essentially extremely small WC granules embedded in a matrix of carbon and gallium. This feature influences the vortex dynamics in these samples that we will show later.

Even though all the physical dimensions of the sample are \( \gg \xi_0 \) we observe a pronounced anisotropy in \( H_{c2}(\phi) \), that is very prominent \(( \sim 30\% \) at \( T = 0.8T_c \) for a sample with width of about \( 25 \xi_0 \). It weakens but does not disappear even when the lateral dimensions are of the order of \( \sim 200 \xi_0 \) [43], as shown in figure 4.

We now validate the key assumptions related to the applicability of WHH theory. Three key requirements are: an isotropic Fermi surface, weak electron-phonon coupling \( (\lambda_{ep}) \) and the rate of spin-flip scattering \( (1/\tau_{so}) \) being lower than the non-spin-flip \( (1/\tau_{tr}) \) scattering. In homogeneously disordered samples like these fermi surface is almost certainly isotropic. For all our samples, the transport scattering time \( \tau_{tr} \approx l/v_F < 10^{-16} \) sec. \( \tau_{so} \) is obtained from the WHH fit using

\[ \frac{\hbar}{4\pi k_B T c \lambda_{so}} \approx 10^{-13} \] 

It is clear that \( 1/\tau_{tr} \gg 1/\tau_{so} \). The electron-phonon coupling strength \( \lambda_{ep} \) can be estimated using the McMillan equation [46]

\[ \lambda_{ep} = \frac{1.04 + \mu^* \ln(\frac{\Theta_D}{T})}{(1 - 0.62\mu^*) \ln(\frac{\Theta_D}{1.467}) - 1.04} \] 

Here, \( \mu^* \) is Coulomb pseudopotential, \( \Theta_D \) is the Debye temperature. For metal films, a typical value of \( \mu^* \) around 0.1 to 0.15 is often assumed to get a fair estimate of \( \lambda_{ep} \). Debye temperature of crystalline tungsten and its alloys are not very different, \( \Theta_D \approx 400 K \) [17]. For device D01 we obtained \( \lambda_{ep} \approx 0.5 \) consistent with the weak coupling assumptions [48] [49].

To gain insight about the possible role of Ga in the composite, we have done ab initio calculations for Ga
adsorbed WC system. Electronic structure simulations were carried out using density functional theory (DFT) implemented within the Vienna ab initio simulation package (VASP) \[50, 51\]. Plane wave basis set using the projector augmented wave (PAW) \[52\] method was used with an energy cutoff of 550 eV. To describe the exchange and correlation, we have employed generalized-gradient approximation by Perdew, Burke, and Ernzerhof \[51\]. Total energy(force) convergence criterion was set up to $10^{-5}$ eV (0.01 eV/Å). WC crystallizes in the space group P6m2. We have adopted lattice constants \(a = b = 2.198 \text{ Å}, \) and \(c = 2.846 \text{ Å}\) for WC \[53\]. A (001) surface slab with 16 unit cell was constructed with tungsten on the top and carbon on the bottom surface respectively. A vacuum of around 15 Å was added to nullify the interaction between the top and bottom surfaces. Further, we have added a single gallium adsorbant on the top surface of the slab. Geometry of the surface slabs for WC and with gallium adsorbant are shown in figure 5 (see caption). We have also checked another possibility where top surface termination is carbon instead of tungsten and we put a Ga adsorbant on top of the carbon terminated layer. However, it’s turns out that Ga adsorption on top of tungsten layer is energetically most favorable than the other case. The Brillouin zone (BZ) integration of the slab was performed using a $11 \times 1 \times 1$ Γ-centered k-mesh. We have also carried out the ionic relaxation to optimize the slab geometry.

Figure 5(c, e) shows the band structure for surface slab in figure 5(a) and 5(b) respectively. Both the surface slabs show metallic behavior with a large band density near the Fermi level. In figure 5(e), there are additional bands that arise from the Ga adsorbant on the surface of the slab in figure 5(b). Red-colored dots show the orbital contributions of the gallium atom. The density of states (in states/atom) of bare WC and Ga adsorbed WC are also shown in Fig. 5(d) and 5(f) respectively. Figure 5(f) clearly shows significant contribution of Ga density of states around the Fermi level. Relatively sharp peak of the Ga density of states justify some flatness of the Ga dominated bands near Fermi level in Fig. 5(e). This might be responsible for the observed $T_c$ enhancement \[20\]. From nearly 2 K to 5 K, in Ga beam prepared composite compared to that of electron beam prepared one. A recent band structure calculation supported by ARPES data also identified non-trivial topological aspects of surface states of WC \[53\]. The interface of single-crystal WC and metal have shown superconductivity till considerably high magnetic fields \[7, 10\]. The large interfacial area of the nanocrystals with the Ga-C composite may be fulfilling similar conditions in these samples. It has been predicted recently \[51\] that materials having triple-band crossing, like in WC, can uniquely stabilize spin-triplet superconductivity.

We further find that at low magnetic fields, when the density of vortices is low, there is very little pinning and the system behaves like a very clean system. This happens because on the scale of the size of a vortex ($\xi_0$), the system is essentially homogeneous. The radial distribution of the individual constituents obtained from the APT date as well as the very small mean free path indicate that the inhomogeneity occurs at an extremely small scale. It is for this reason the vortex sees the system as smooth and clean without any significant pinning centers. Two devices (Device A and C), of two extreme thicknesses, were measured at 4 K (0.8 $T_c$) with a different in-plane tilt angle of the magnetic field. Data from the device A is shown in figure 6(a). At low current the vortex motion is expected to be dominated by the weak pinning forces present in the system. Consider a case when the magnetic field is along y direction and the current flow ($j_x$) is along x. \(\rho_n\) denotes the normal state resistivity of the wire (see table 1). The Lorentz force \((F_L = L_y j_x \Phi_0)\) acting on a single vortex, will be balanced by the viscous drag \(F_d = L_y \eta v_y\), where the “viscosity” is given by \(\eta = \frac{\Phi_0^2}{2\pi L_y^2 \rho_n}\) \[59\]. The number of vortices \((N)\) can be estimated as \(N \Phi = \mu_0 H L_x L_z\). The total energy dissipation due to all vortices can be written as \[57, 58\] as a product of the force and velocity summed over all the vortices and equated to a flux-flow resistivity \(\rho_{ff}\).
FIG. 6. (a) Angular dependence resistivity of device-A with in plane $H$ at 4.2 K; (c) to (f) Angular variation of resistivity at different magnetic field values marked with dotted vertical line in (a). Measurement current of 100 nA was used in this experiment, which is three order of magnitude less compared to these devices’ critical current ($50 - 100 \mu$A). The sample was mechanically rotated to a certain angular position and the magnetic field was then swept. All the solid lines in figure (c) to (f) are $\sin^2(\phi)$ fitting discussed in text.

Equation 3 can be used to get an independent estimate of $\xi_0$, that does not use the slope of $H_{c2}$ near $T_c$. By using typical values of $\rho_{ff}$ at $\phi = \pi/2$ at 2.5 T we obtain $\xi_0 \approx 6.4$ nm. Given the approximate nature of the vortex flow model, we consider the agreement to be very good. When the field is applied at an angle $\phi$ as shown in figure (b), the dissipation should clearly vary as the square of the component of the force along $y$, and hence have a $\sin^2(\phi)$ dependence at low fields. This simple proportionality is expected to hold when the vortex density is low. This behaviour, indicative of smooth flow of the vortices at low fields is clearly seen till 2.5 T. We have also estimated the velocity of the vortices and find that they reach supersonic speeds [3].

In conclusion, we have shown that a highly disordered granular system like a FIB deposited track exhibits $H_{c2}$ above the paramagnetic limit. Systems like this have widespread use in mesoscopic devices and superconducting electronics. We showed by APT-based mass spectrometry that the heavy metal is present in the form of its carbide. The crystal structure and band structure of WC potentially has several features that can give rise to spin-orbit effects as well as robust surface states when in contact with other metal ions.

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