Hydrogen-Intercalated Graphene on SiC as Platform for Hybrid Superconductor Devices

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Nanodevices based on hybrid graphene–superconductor structures have recently attracted much attention owing to both fundamental and application aspects. However, atomic-level investigations of proximity-induced superconductivity in graphene, especially on technologically relevant substrates remain rare. Here, the atomic-scale study of electronic properties and the superconducting proximity effect in hydrogen-intercalated single-layer graphene on SiC decorated with epitaxial lead (Pb) islands is reported. The graphene layer is thoroughly characterized by means of Landau level spectroscopy which confirms its quasi-free-standing nature. Scanning tunneling spectroscopy performed at 1.8 K on the graphene layer in the vicinity of Pb islands shows a reduced superconducting gap of $\Delta_{gr} = 0.20(1)$ meV, which points to a graphene/superconductor junction of moderate transparency. The variations of the proximity-induced superconducting gap on graphene are measured as function of spatial position as well as of magnetic field strength. Spatially resolved measurements yield a coherence length of about 175 nm in the graphene monolayer. The study provides a foundation for realization of graphene–superconductor heterostructures on large-scale SiC(0001) wafers suitable for future technological applications.

Hybrid graphene–superconductor structures show a large variety of unique features,[1–5] which are related to the fascinating electronic properties of graphene like the massless Dirac fermion spectrum or gate-tunable charge carrier density. Up to now superconductivity in single layer graphene has been achieved either by alkali metal doping[6] or by proximity to a superconducting material.[7–9] Relying on the latter mechanism a number of possible applications has been proposed, such as spin current filters,[10] valley sensors,[11] or current switches.[12,13] Both the fundamental aspects and the application realization perspectives motivated a large number of transport experiments in high-quality graphene–superconductor junctions.[14–20] In spite of the enormous progress in device fabrication and transport studies, atomic scale investigations of proximity superconductivity in graphene still remain a rare issue. In the last years, local characterization has been reported employing scanning tunneling microscopy (STM) and scanning tunneling spectroscopy (STS) of proximity-induced superconductivity in monolayer graphene on Re(0001),[21] on the high-temperature superconductor Pr$_2$Ce,CuO$_4$[8] as well as in graphene decorated by large Al islands.[22] Recently, Pb/graphene has been investigated as a particularly promising system for the proximity effect in graphene.[23–25] However, the actual local-scale studies on proximity induced superconductivity in this system are still missing.

In this work, we perform a scanning tunneling microscopy investigation of the superconducting proximity effect induced by compact lead (Pb) islands in a graphene layer. We use a quasi-free-standing monolayer graphene (QFMLG) obtained by large scale hydrogen intercalation underneath graphene on 6H-SiC(0001).[26] The electronic properties of the graphene layer and particularly its decoupling from the underlying substrate are investigated by Landau level spectroscopy. A thorough characterization of superconductivity in the Pb nanoislands on top of graphene is performed and serves as a basis for the study of the proximity effect in graphene. Further on, we carefully investigate the proximity-induced superconducting gap on graphene both as function of spatial position and magnetic field strength.

Figure 1a shows a typical STM image of the surface after room-temperature evaporation of Pb on QFMLG on SiC prepared by hydrogen intercalation.[26,27] Large Pb islands of triangular-truncated shape are visible with the edges preferably oriented along the main crystallographic directions of the graphene lattice. The islands are measured to be 5-25 monolayers (ML) in height, indicated individually in the figure (for details of evaluation see Figure S1, Supporting Information). We express the island lateral size by introducing an effective diameter $d_{eff} = 2\sqrt{A/\pi}$ with
A being the measured area in the STM topography. In our experiments we observe islands with \( d_{\text{eff}} = 10 \sim 60 \) nm. Atomic resolution imaging on top of Pb islands (Figure S1a, Supporting Information) shows a hexagonal close-packed lattice with the periodicity of 3.4(1) Å suggesting the (111) bulk truncation of Pb. Upon imaging at temperatures down to 1.7 K, Pb islands can still be moved by the STM tip pointing toward a rather weak interaction between Pb and the underlying graphene, being in coincidence with previous reports.\(^{[24,28-30]}\) We further performed a detailed investigation of the electronic properties of the islands by \( dI/dU \) spectroscopy and quasiparticle interference (QPI) mappings (Figure S1d–f, Supporting Information). The observed electronic behavior is dominated by quantum well states showing a very good agreement with the previous reports on the Pb/graphene system.\(^{[24]}\)

Figure 1b shows an atomically resolved STM image of graphene between the Pb islands. The honeycomb lattice of graphene is clearly discernible without any notable Moiré pattern. Dark depressions are most possibly inhomogeneities at the interface as the graphene lattice remains uninterrupted. Also a small amount of point defects (see inset in Figure 1b) is observed giving rise to strong local density of states (LDOS) modulations due to quasiparticle scattering.\(^{[11-13]}\) Figure 1c shows a \( dI/dU \) spectrum measured on clean graphene at zero magnetic field. The observed step-like features, symmetrically positioned around zero bias, are generally attributed to phonon-mediated inelastic tunneling.\(^{[34-36]}\) As evident from the second derivative, the features occur at energies of \( \pm 57(2) \) meV and \( \pm 78(2) \) meV corresponding to the excitation of out-of-plane acoustic phonons at \( \overline{M} \) as well as out-of-plane optical and transverse acoustic phonons at \( \overline{M} \), respectively.\(^{[37-41]}\)

Upon application of a magnetic field perpendicular to the sample surface well-pronounced Landau-level (LL) peaks develop (see Figure 1d and Figure S2a,b, Supporting Information). The high-field \( dI/dU \) spectra are comparable to those previously reported for a decoupled graphene monolayer.\(^{[41-44]}\) The energies of the LLs depend linearly on the square-root of magnetic field \( B \) and level index \( n \). By fitting the field-dependent positions of the single LLs, we obtain the Fermi velocity \( v_F = 1.011(4) \times 10^6 \) m s\(^{-1} \) and the position of the Dirac point with respect to \( E_D \). \( E_B = \pm 252(1) \) meV. Both values are additionally confirmed by the analysis of QPI mappings at different bias voltages (Figure S2d–f, Supporting Information). The position of the Dirac point and the Fermi velocity are in excellent agreement with the angle-resolved photoemission data obtained on comparable samples.\(^{[26,27,45]}\) The peak width analysis of the LL sequence at 6 T yields a quasiparticle lifetime of \( \tau_0 = 49(19) \) fs at \( E_F \) (Figure S2c, Supporting Information). The value for the quasiparticle lifetime can be converted into a characteristic mean free path giving \( l = \tau_0 v_F = 50(9) \) nm. We observe no pronounced quasiparticle scattering in close proximity to Pb islands as well as only few point scatterers on graphene and thus suggest the characteristic mean free path being possibly limited by scattering at defects at the graphene/SiC interface. The observation of LLs together with the observed quasiparticle scattering at point defects and pronounced phonon signatures in \( dI/dU \) spectra deliver a clear evidence that graphene is very well decoupled showing only small hole doping and thus can be indeed described as quasi-free-standing monolayer graphene as proposed before.\(^{[26,27,46]}\)

In the following we focus on the investigation of the superconducting properties of Pb islands. Figure 2a shows the superconducting gap of a 15 ML thick Pb island, measured at 0 T and 0.5 T. In zero field, we observe pronounced quasiparticle Bardeen-Cooper-Schrieffer (BCS) peaks around \( \pm 2.5 \) meV, which is almost twice the bulk superconducting gap of Pb of \( \Delta_{\text{bulk}} \approx 1.35 \) meV\(^{[47]}\) thus indicating tunneling between two superconducting electrodes (indexed by E1 and E2 in the following). This is supported by a small zero-bias conductance peak which stems from the finite temperature. At 0.5 T the gap is decreased by roughly a half, indicating a breakdown of the superconductivity in E1. The high mobility of the islands suggests only weak interaction with the graphene layer and thus a high probability for such clusters to be picked up by the STM tip. As a consequence in most of the measurements presented here, a Pb cluster is attached to the apex of the STM tip. In the following, we therefore describe the procedure to reliably characterize the tip before and after each measurement in order to ensure the comparability of the results. The \( dI/dU \) spectrum at 0.5 T can be fitted assuming a normal conductor (NC) to superconductor (SC) tunneling process with the BCS density of states (DOS) in the SC state and a constant DOS for Pb in the NC state (see Supporting Information for fit procedure details). The fit yields a BCS gap of \( \Delta = 0.95(1) \) meV in E2. The deviation of critical field and BCS gap size compared to bulk values stems from confinement\(^{[48,49]}\) and the reduction of the superconducting order parameter in.
an external magnetic field.\(^{[50]}\) The latter effect can be described within the frame of the Ginzburg–Landau (GL) theory and behaves like \(\Delta_{\text{GL}}(B) \propto \sqrt{T - \frac{\Delta_0^2}{B}}\), where \(\Delta_0\) denotes the critical field at which superconductivity vanishes.

We determine \(B_{\text{c}1}\) by following the evolution of (1) the fitted superconducting gap \(\Delta\) as well as (2) the zero-bias conductance (ZBC)\(^{[51,52]}\) upon sweeping the magnetic field from 0.5 to 1.5 T. We fit each \(dI/dU\) spectrum assuming a NC-SC tunneling process and plot the resulting gap widths \(\Delta_c\) and the ZBC values as shown in Figure 2b. Both field dependencies can be excellently fitted using \(\Delta_{\text{GL}}(B) + c\), with \(c = 0\) for the ZBC values and \(c \neq 0\) for the gap widths. We note that the exact evolution of \(\Delta(B)\) strongly depends on the electronic mean free path and shape of the island.\(^{[51-56]}\) The values for the critical field are well comparable, that is, \(B_{\text{ZBC}} = 1.39(2)\) T and a slightly smaller \(B_{\text{GL}} = 1.36(1)\) T (see Figure S3a, Supporting Information, for details). We obtain a zero-field gap value \(\Delta_c(0\,\text{T}) = 1.07(1)\) meV. Extrapolating the fit function \(B_{\text{GL}}(B)\) it is then possible to fit the \(dI/dU\) spectra recorded below 0.5 T, that is, for the SC-SC tunneling process, and to obtain the values \(\Delta_c(B)\).

The best fit for the magnetic field dependence of the data for the low field region can be obtained using a function \(\Delta_{\text{GL}}(B) \propto \sqrt{\cos((\pi/3)(B/B_0)^2)}\), adapted from the BCS theory.\(^{[57]}\) We obtain \(\Delta_c(0\,\text{T}) = 1.36(2)\) meV, which is in good agreement with the bulk value. The corresponding critical field is calculated to \(B_{\text{c}1} = 0.45(1)\) T. Evaluation of the experimental data for several islands \((N = 21)\) consequently measured with the same tip reveals that the value of \(B_{\text{c}1}\) stays constant, whereas \(B_{\text{c}2}\) varies from point to point. We thus conclude that \(\Delta_1\) and \(B_{\text{c}1}\) represent the superconducting parameters of the Pb cluster at the STM tip. The abrupt collapse of the gap \(\Delta_c\) with increasing field indicates a first order phase transition, pointing to a sharp STP tip apex.\(^{[58]}\) In each experiment, we determined and used parameters \(\Delta_1 \equiv \Delta_{\text{tip}}\) and \(B_{\text{c}1} \equiv B_{\text{c}1,\text{tip}}\) in order to subsequently deduce superconducting gap \(\Delta_1 \equiv \Delta_{\text{island}}\) and critical fields \(B_{\text{c}1,\text{island}}\) of Pb islands. We further observe that the obtained value for \(\Delta_{\text{island}}\) exhibits a temperature dependence following the BCS theory (see Figure S3b, Supporting Information). The value for \(\Delta_{\text{island}}\) obtained from the BCS fit indicates that the superconducting gap closes upon decreasing the island size. In Figure 2c, we plot \(\Delta_{\text{island}}\) for 33 islands as a function of inverse island thickness. A linear fit to the experimental data yields \(\Delta(1/\text{island}) = 1.29(8)\) meV, which is in good agreement with the bulk value of \(\Delta_{\text{bulk}}(1.8\,\text{K}) = 1.34\) meV. Although the smallest island height taken into account here is 8 ML, the linear fit suggests a breakdown of superconductivity between 1 and 2 ML thickness, when the gap size reaches 0 meV at \(d = 1.8(2)\) ML. This behavior is in agreement with previous studies on thin Pb islands.\(^{[49,51,59]}\)

In contrast to the thickness, the influence of the island’s area and effective diameter on the superconducting gap size is negligible. However, the lateral confinement of thin islands shows a pronounced impact on their critical field, when a magnetic field perpendicular to the surface plane is applied.\(^{[60]}\) In Figure 2d we plot the measured critical fields \(B_{\text{c}1,\text{island}}\) as a function of the effective diameter \(d_{\text{eff}}\). Despite a certain scatter of the values which we relate to different island shapes, we observe a clear decrease of critical field with increasing the island diameter. This behavior generally can be fitted by a \(1/d\) function as derived from the Ginzburg–Landau theory\(^{[50]}\) and yields \(B_{\text{c}1}(d_{\text{eff}}) = c_1/d_{\text{eff}}\) with \(c_1 = 64(1)\) nm T. A similar value has been reported for Pb islands on Si(111) for a range of smaller diameters, although the authors modify the function to \(c_1/(d + d_0)\) to account for an effective superconducting area slightly different to the topographic one.\(^{[61]}\) In the present study, the diameter independent offset is fitted to \(d_0 = -8(2)\) nm with \(c_1 = 49(3)\) nm T. However, the deviation remains small compared to the observed spread of critical fields. We do not observe vortex formation in our measurements up to effective diameters of \(\approx 70\) nm (see Figure S3c–f, Supporting Information, for more details about \(B_{\text{c}1}\) as function of island size).

In the following we focus on proximity-induced superconductivity in QFMLG. Figure 3a,b shows the \(dI/dU\) spectra measured on top of a 15 ML thick Pb island as well as on the bare graphene surface approximately 20 nm away from the island edge. Both measurements are performed at an applied magnetic field of 0.5 T, which is high enough to suppress superconductivity in the STM tip. On graphene a reduced but finite superconducting gap is observed that can be fitted assuming a BCS SC-SC tunneling process. We obtain a gap of \(\Delta_g = 0.15(2)\) meV and relate it to proximity-induced superconductivity.\(^{[22,62]}\) The quality of the BCS fit and the absence of pronounced subgap features or zero-bias conductance peaks point to a conventional
superconductivity in 2D electron systems. The size of the superconducting gap measured beyond the border to graphene however exhibits a far more interesting behavior. First, an abrupt change of $\Delta$ is observed at the edge of the Pb island within a distance of 1–2 nm. A comparable behavior has been reported earlier for Al/graphene. Second, a gradual decrease of the gap size is observed upon moving away from the edge of the Pb island due to the finite superconducting coherence length $\xi$ in the graphene layer. To obtain the value of $\xi$ we perform a fit to an exponential decay $\Delta(x) = \Delta_0 e^{-x/\xi}$, assuming zero gap size at a distance far away from the superconducting island. The fit yields an initial gap size $\Delta_0 = 0.20(1)$ meV and a coherence length of $\xi = 175(8)$ nm on graphene. Third, we observe significant short-range variations of $\Delta(x)$ exceeding a certain spread of fitted values that stem from low energy resolution. These modulations are due to proximity of further Pb islands within the measurement region (Figure 3a). For the same reason the gap size flattens out for the last third of the measurement line, where the tip comes closer to another island within the area. We note that the calculation of $\xi$ with a single exponential decay just serves as a first-order approximation and could overestimate the real value. A comparable study of Pb islands on SiC revealed a diverse range of spatial evolutions of the proximity-induced gap from linear to constant behavior, depending on the island arrangement in the vicinity of the measurement point. Apart from these geometric effects, oscillations in the gap behavior have been discussed in terms of local disorder in form of step edges, cavity effects or strain in the graphene lattice. In our case, no step edges or any other defects were observed within the measurement area.

We would like to compare our result with the previous experiment on few-layer graphene on SiC capped with superconducting aluminum, where the authors obtain a coherence length of 429(9) nm. This is roughly twice as large as observed in our measurement and correlates with the graphene elastic mean free paths, that is, 101(4) nm versus 50(9) nm in our system. Furthermore, the remaining superconducting gap in graphene adds up to roughly 70% of the Al gap energy, in contrast to a drop to 20% in the case of Pb/graphene. Both findings could be caused by the transition from graphene as buffer layer to the hydrogen termination of the SiC surface, with a slightly higher graphene-substrate interaction in the latter case. Experimentally, this is to some extent supported by the enhanced doping level of 250 meV compared to roughly 50 meV on multilayer graphene, combined with a reduced quasiparticle lifetime in our system as determined from Landau level line widths. The differences in the measured size of the proximity gaps could stem from a different interface coupling to the graphene when changing from Pb to Al. The expected induced gap size under the Pb island $\Delta_{\text{ind}}$ can be estimated following a procedure reported recently, which mainly relies on theoretical results for the proximity-induced superconductivity in 2D electron systems.
induced gap is determined by the tunneling rate $\Gamma^\prime$ at the NC/SC interface, which can be written as \(^{24,65}\):

$$\Gamma^\prime = \frac{h v_F^2}{8 |E_F - E_D| e^2 A R}$$

(1)

with $A$, the contact area between superconductor and graphene, $R$, the interface resistance, $v_F$, Fermi velocity in graphene, and $E_D$, the energy position of the Dirac point. The product $AR = 3.25 \times 10^{-12} \Omega \text{m}^2$ can be estimated from dynamical coulomb blockade measurements of Pb islands on HOPG.\(^{166}\) With the values of $v_F$ and $E_D$ determined from our Landau level spectroscopy measurements, the tunneling rate (in units of energy) amounts to $\Gamma^\prime = 0.28(1) \text{meV}$. It has been shown that for $\Gamma^\prime \ll \Delta$ the tunneling rate corresponds to the induced gap size, that is, $\Delta_{\text{ind}} = \Gamma^\prime$ \(^{64,65}\) where $\Delta = 1.16 \text{meV}$ denotes the energy gap of the Pb island. A more accurate calculation of $\Delta_{\text{ind}}$ can be performed using the equation $\Delta_{\text{ind}}^2 = (1 + 2\Gamma^\prime/\sqrt{\Delta^2 - \Delta_{\text{ind}}^2}) - (\Gamma^\prime)^2 = 0$, yielding $\Delta_{\text{ind}} = 0.23(1) \text{meV}$. This value is in very good agreement with the proximity-induced energy gap $\Delta_{\text{prox}} = 0.20(1) \text{meV}$ observed next to the Pb island. We emphasize that the estimated values strongly depend on the assumption concerning the Pb/graphene interface, that is, the value of $AR$. With the induced gap size observed for the Al/graphene system, the model estimates a tunneling rate of $0.30(1) \text{meV}$, indicating comparable interface resistances of Pb and Al. This finding would point to the different graphene–substrate interaction of both systems as origin for the observed variations of induced gap size.

In order to investigate the impact of an external magnetic field on the proximity-induced superconductivity in graphene we record a series of $dI/dU$ spectra upon tuning the magnetic field from $0$ T to $1.25$ T (Figure 3d). These measurements are carried out with a Pb cluster at the tip showing a slightly different critical field. The measurements are performed on clean graphene at a point which is 15 nm away from a different Pb island with a thickness of 15 ML and a critical field of $1.34(5)$ T. On graphene, we observe a persistent superconducting gap above $B_{\text{c},\text{tip}}$. The gap vanishes around 1 T as can be seen from Figure 3d. The temperature limit of the energy resolution does not allow a precise analysis of gap size based on BCS fitting. We therefore consider the ZBC to be a reasonable indicator of the superconducting state in graphene, which is plotted in Figure 3e.\(^{31,54}\) The ZBC indeed shows a step-like behavior following the breakdown of superconductivity first in the tip and then in the sample. Except for a small intermediate region right above $B_{\text{c},\text{tip}} = 0.49(1)$ T the evolution of the superconducting gap in graphene can be well described with the same function used before on the Pb islands. Note that we do not observe a constant pseudogap after breakdown of superconductivity like in most measurements over the Pb islands, originating from the interplay between different quantum-size related effects\(^{67}\) (for more details see Supporting Information S4). We determine the critical field to $B_{\text{c},\text{gr}} = 1.24(5)$ T that is only slightly smaller than the observed value on the Pb island in the proximity to the measurement point. Thus we conclude that the proximity-induced superconducting gap in graphene scales linearly with the energy gap of Pb islands.

In conclusion, we performed a detailed characterization of structural and electronic properties of hydrogen-intercalated graphene on SiC decorated with epitaxial Pb islands. The observation of Landau levels confirms the quasi-free-standing behavior of the monolayer graphene indicating a slight p-doping. Pb islands on top of graphene show the superconducting gap $\Delta$ and critical field $B_c$, which vary with thickness, diameter, and volume. The observed superconducting properties of Pb islands coincide mostly with previous studies on other substrates and provide us a basis to study the superconducting proximity effect that arises at the Pb/graphene interface. We observe an induced gap of $\Delta_{\text{gr}} = 0.20(1) \text{meV}$ on graphene close to the Pb island edge, which coincides well with an estimation for a metal/superconductor junction of moderate transparency. Spatially resolved measurements yield a coherence length of $\xi = 175(8) \text{nm}$ in the graphene monolayer. We show that the induced superconducting gap scales linearly with the energy gap of the corresponding Pb island upon applying a magnetic field. Our findings pave the way for the large-scale fabrication of graphene–superconductor heterostructures, which can be implemented for quantum information processing.

**Experimental Section**

The SiC samples were cut from a commercial wafer purchased from SiCrystal, chemically cleaned, and hydrogen etched for removing polishing scratches. Graphene growth was performed by RF heating in Ar atmosphere.\(^{68}\) A single carbon layer, the so-called buffer or zero layer graphene (ZLG) was obtained using a growth temperature of $1400^\circ\text{C}\(^{27,69}\). The ZLG was decoupled from the SiC substrate by heating in H$_2$ atmosphere at 800 °C resulting in QFMLG.\(^{26}\) After being transferred in air, the samples were introduced into the ultrahigh vacuum chamber (base pressure: $\approx 5 \times 10^{-11}$ mbar) and annealed for several hours to remove surface contaminations. Pb was deposited from an effusion cell, while the substrate was kept at room temperature. A nominal thickness of 15 Å was estimated relying on the evaporation rate calibration with a quartz microbalance. Directly after the growth, the samples were transferred into the cryogenic STM (Omicron Nanotechnology GmbH) without breaking the vacuum conditions. All STM and STS measurements were carried out at temperatures of 1.7–6 K. Differential conductance ($dI/dU$) spectra were recorded using a standard lock-in technique with a modulation voltage $U_{\text{mod}} = 500 \mu \text{V}$ (amplitude) at a frequency $f_{\text{mod}} = 537.8$ Hz added to the sample bias. The magnetic field is applied perpendicular to the sample surface. Grinded and polished PtIr tips (Nanoscore GmbH) were used. The sign of the bias voltage corresponds to the potential at the sample.

**Supporting Information**

Supporting Information is available from the Wiley Online Library or from the author.

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**Conflict of Interest**

The authors declare no conflict of interest.

**Author contributions**

M.F. conceived the experiment. F.P., T.B., and M.F. performed the measurements. S.F. and U.S. grew and characterized the graphene/SiC
samples. F.P. and M.F. analyzed the data and wrote the manuscript draft. All authors discussed the results and contributed to writing the paper.

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epitaxial graphene, hybrid superconductor devices, proximity effect, scanning tunneling microscopy, superconductivity

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